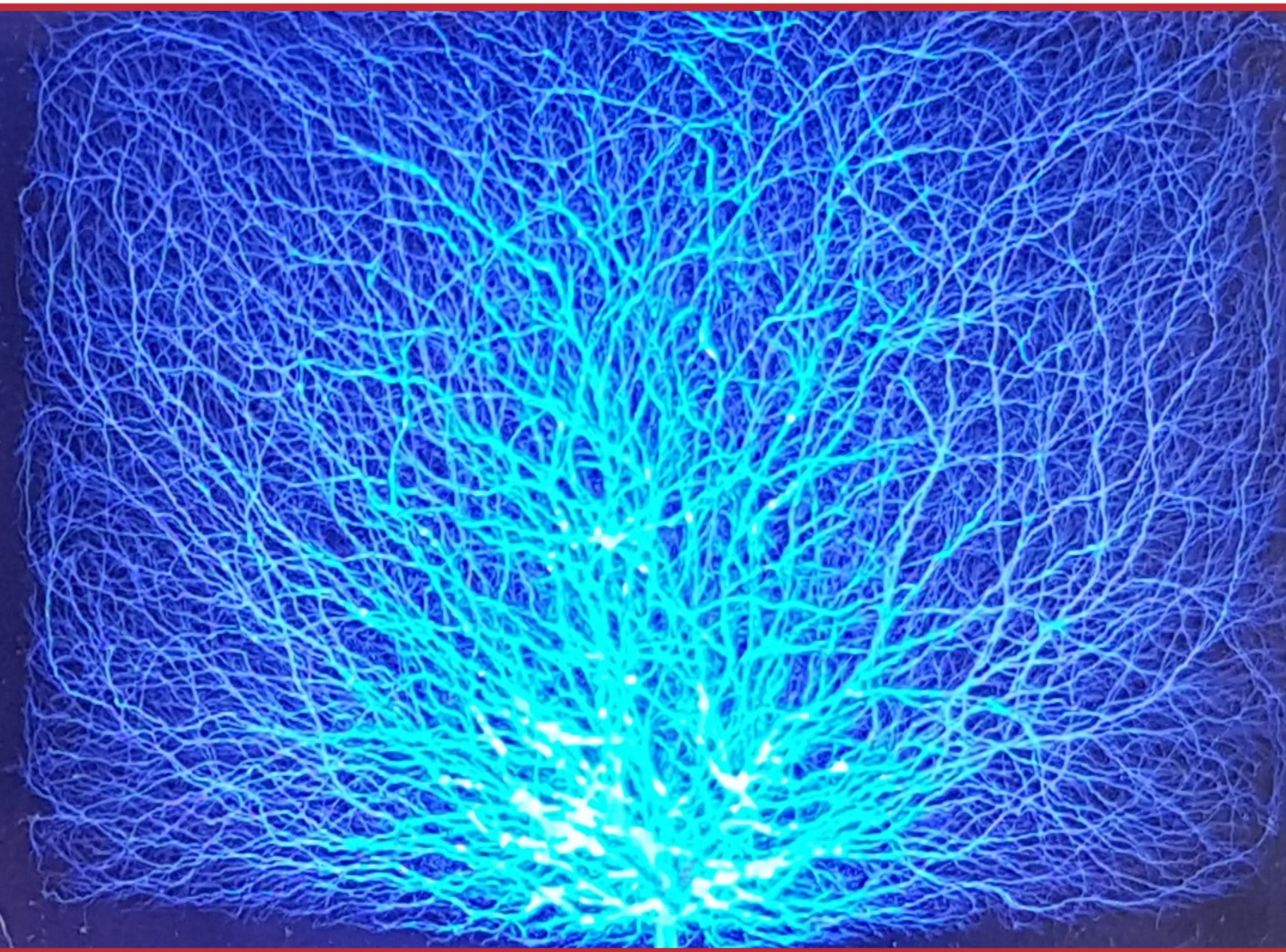
LSC 2017



An International conference on
advances in Liquid Scintillation Spectrometry

1-5 May 2017
Copenhagen, Denmark

ABSTRACT BOOK

LSC2017 -Advances in Liquid Scintillation Spectrometry

Organisers:
DTU - Technical University of Denmark, www.dtu.dk
SSM - Swedish Radiation Safety Authority, www.ssm.se IAEA - International Atomic Energy Agency, www.iaea.org

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Abstract Book

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Welcome to LSC2017

Welcome to LSC2017, an international conference on advances in liquid scintillation spectrometry. LSC2017 conference follows the LSC series conferences initiated in 1957 and the last one in 2013 in Barcelona, becomes the 23rd edition of this conference. This is the first time this conference come to Nordic countries, the LSC2017 conference is organized by Technical University of Denmark (DTU) in cooperated with IAEA (International Atomic Energy Organization) and Swedish Radiation Safety Authority (SSM). We, the Center for Nuclear Technologies in DTU, are happy to host this conference in Copenhagen.

Liquid scintillation spectrometry has being an important radiometric technique for measurement of radionuclides and widely used not only in the nuclear field, but also many other fields. Compared to the newly developed techniques, such as accelerator mass spectrometry (AMS) and ICP- MS, liquid scintillation spectrometry is considered as a traditional and mature technique. However, the LSC technique continues to develop in the recent years, and remarkable progress on the LSC methodology has been achieved. In particular, the TDCR (triple-to-double coincidence ratio) based LSC method and instruments have obtained a great success. The tedious quench correction and counting efficiency calibration in the conventional LSC measurement can be avoid, and even absolute measurement of radionuclide activity using LSC become realistic. The application of LSC techniques shows constantly increasing, indicated by the increasing LSC instruments installation and research publications. Meanwhile, there are still some challenges in the LSC methodology and its application in some research fields. LSC2017 conference provides a platform to all participants to present your recent achievements, exchange the experience, knowledge and idea, and discuss the challenges we faced. The topics of the LSC2017 conference aims to cover all aspects related to LSC technique, from new development on LSC methodology to its application in different fields.

We are happy to have about 200 participants and exhibitor representatives from 36 countries registered in the conference. More than 180 peer-reviewed papers are accepted for presentation. The high quality program including 14 invited lectures, 75 oral presentations and more than 85 poster presentations, give an excellent overview in all aspects in the LSC methodology and applications. The LSC2017 proceedings will be published in a special issue in JRNC (J. Radioanal. Nucl. Chem.), the submission system have opened from 1st April and will close on 1st June. Full papers of all presentations both oral and poster are invited to submit to the LSC2017 conference proceedings, the selected papers through JRNC ordinary peer-review process will be published.

I would like to thank all participants and exhibitors for your efforts and interest to the LSC2017 conference, special thanks to the organizing institutions DTU Nutech and Swedish Radiation Safety Authority for their finically support. I want to thank the scientific committee members for their great work on reviewing the abstracts and organizing the sessions. Grateful thanks are also dedicate to my colleagues in DTU Nutech and the organizing committee members for their hard and productive work in the preparation of the conference.

Hope all of you enjoy the conference and your stay in our wonderful Copenhagen!

Xiaolin Hou
Chairman of LSC2017 conference
Professor of Technical University of Denmark

Preliminary program of LSC 2017 conference

Sunday	Monday	Tuesday	Wednesday	Thursday	Friday
	8:00 Registration/Breakfast	8:00 Breakfast	8:00 Breakfast	8:00 Breakfast	8:00 Breakfast
	Opening Session	Nuclear chemistry	Natural radionuclides and other topics	Plastic scintillator	Tritium studies
	9:00 Welcome speech	8:30 Invited talk	8:30 Invited talk	8:30 Invited talk	8:30 Invited talk
		8:55 Oral	8:55 Oral	8:55 Oral	8:55 Oral
		9:10 Oral	9:10 Oral	9:10 Oral	9:10 Oral
	9:40 Opening lecture	9:25 Oral	9:25 Oral	9:25 Oral	9:25 Oral
		9:40 Oral	9:40 Oral	9:40 Oral	9:40 Oral
		9:55 Oral	9:55 Oral	9:55 Oral	9:55 Oral
		10:10 Oral	10:10 Oral	10:10 Oral	10:10 Oral
	10:25 Coffee break	10:25 Coffee break	10:25 Coffee break	10:25 Coffee break	10:25 Coffee break
	New development and spectrum analysis-1	Metrology and quality assurance-1	Metrology and quality assurance-2	Environmental radioactivity-2	Environmental radioactivity-3
	10:45 Invited talk	10:45 Invited talk	10:45 Invited talk	10:45 Invited talk	10:45 Invited talk
	11:10 Oral	11:10 Oral	11:10 Oral	11:10 Oral	11:10 Oral
	11:25 Oral	11:25 Oral	11:25 Oral	11:25 Oral	11:25 Oral
	11:40 Oral	11:40 Oral	11:40 Oral	11:40 Oral	11:40 Oral
	11:55 Oral	11:55 Oral	11:55 Oral	11:55 Oral	11:55 Oral
	12:10 Oral	12:10 Oral	12:10 Oral	12:10 Oral	12:10 Close ceremony
	12:25 Lunch	12:25 Lunch	12:25 Lunch	12:25 Lunch	12:40 Lunch
Registration/Reception	Neutrino and neutron detection	New development and spectrum analysis-2	Social activity	Medicine and other topics	
	13:40 Invited talk	13:40 Invited talk	13:40-17:30 Tour to Hamlet castle	13:40 Oral	
	14:05 Oral	14:05 Oral		14:00 Oral	
	14:20 Oral	14:20 Oral		14:20 Oral	
	14:35 Oral	14:35 Oral		14:35 Oral	
15:00 -18:00 Registration	14:50 Oral	14:50 Oral		14:50 Oral	
	15:05 Oral	15:05 Oral		15:05 Oral	
	15:20 Oral	15:20 Oral		15:20 Oral	
	15:35 Oral	15:35 Oral		15:35 Oral	
	15:50 Coffee break	15:50 Oral		15:50 Oral	
	Environmental radioactivity-1	16:05 Coffee break		16:05 Coffee break	
16:00-18:00 Reception	16:10 Invited				
	16:35 Oral				
	16:50 Oral	16:20-17:40 Poster session-1		16:20-17:40 Poster session-2	
	17:05 Oral				
16:00-17:00 Concert by Zapolski Strings	17:20 Oral				
	17:35 Coffee break				
		17:40 Coffee break	18:00-22:00 Conference dinner (Tivoli)		
	18:00-20:00 Workshop-1	18:00-20:00 Workshop-2			

Monday

8:00-9:00	Registration					
Opening Session (Chaired by Prof. Xiaolin Hou)						
9.00	Welcom speeches		HOU Xiaolin	Technical University of Denmark	Denmark	
			LYNOV Jens-Peter	Technical University of Denmark	Denmark	
9.20			NIELSEN Sven	Technical University of Denmark	Denmark	Six decades of environmental-radioactivity measurements
09.40	Opening lecture		Dr. KOSSERT Karsten	Physikalisch-Technische Bundesanstalt	Germany	Radionuclide metrology using LSC: Current status and limitations
	Coffee break					
Session: New development on LSC instrumentation methodology, scintillator and spectrum analysis-1 (Chaired by: Dr. Philippe Cassette, France and Prof. Chunli Liu, China)						
Time	ID	Title	Author	Affiliation	Country	Presentation Title
10.45	166	Dr.	JANDA Jiri (invited)	University of Defence, NBC Defence Inst.	Czech Republic	The comparison of scintillation properties of YAP:Ce, YAG:Ce and ZnO:Ga powders as a potential substitution of liquid scintillation cocktail
11.10	130	Prof.	OMTVEDT Jon Petter	University of Oslo, Dep. of Chemistry, P.O. Box 1033 - Blindern, NO-0315 Oslo	Norway	On-line LS Spectroscopy of Super-heavy Elements
11.25	113	Dr.	FENG Xiaogui	Institute of Nuclear and New Energy Technology, Collaborative Innovation Center of Advanced Nuclear	China	A performance comparison of two liquid scintillation counters from PerkinElmer, Inc.
11.40	126	Dr.	MITEV Krasimir	Sofia University, St. Kliment Ohridski, Faculty of Physics	Bulgaria	Design and performance of a miniature TDCR counting system
11.55	170	Prof.	DEVOL Timothy A	Dep. of Environmental Engineering and Earth Science, Clemson University	USA	New pyrazoline fluorophores for more efficient organic scintillators
12.10	147	Mr.	WANG Yadong	China Inst. for Radiation Protection	China	Effect of sodium salicylate on the determination of Pb-210/Bi-210 and Ra-228/Ac-228 by Cerenkov counting
12:25	Lunch					

Monday ... continued

Session: LSC in neutrino and neutron detection (Chaired by: Dr. Xiongxin Dai, Canada and Dr. Sven Nielsen, Denmark)						
13:40	226	Dr.	YEH Minfang (invited)	Brookhaven National Laboratory	USA	Water-based and Metal-doped Liquid Scintillator for Neutrino Detection
14:05	175	Dr.	DAI Xiongxin	China Inst. for Radiation Protection	China	Boron-10 and Lithium-6 Loaded Scintillator for Neutron Detection
14:20	122	Dr.	BERGERON Denis	Physical Measurement Lab., Nat. Inst. of Standards and Technology	USA	Phase stability and lithium loading capacity in a liquid scintillation cocktail
14:35	173	Prof.	FAN Tieshuan	Inst. for Heavy Ion Physics, School of Physics, Peking Uni.	China	The Deuterium-Deuterium neutron time-of-flight spectrometer at EAST tokamak
14:50	291	Prof.	LIU Hongjie	Chinese Academy of Engineering Physics	China	Liquid Scintillator Neutron Detection System for Fast-ignition
15:05	218	Dr.	TIAN Lianpeng	Queens University	Canada	Synthesis and Characterization of Organic Tellurium Compounds for SNO+ Liquid Scintillator
15:20	161	Mr.	BEAUMONT Jonathan S.	International Atomic Energy Agency	Austria	A Fast-neutron Coincidence Collar Using Liquid Scintillators for Fresh Fuel Verification
15:35	228	Prof.	ZHANG Xiaodong	School of Nuclear Science and Technology, Lanzhou Uni.	China	Next-Generation, Micron-order Resolution Neutron Imaging with Gel Scintillator Filled Glass Capillary Arrays
15:50	Coffee break					
Session: Application in environmental radioactivity-1 (Chaired by Prof. Gabriele Wallner, Austria and Dr. Jost Eikenberg, Switzerland)						
16:10	123	Dr.	PITOIS Aurelien (invited)	International Atomic Energy Agency, IAEA Environment Lab.	Austria	IAEA's ALMERA Effort Towards Harmonization of Radioanalytical Procedures: Development and Validation of a Rapid Procedure for Simultaneous Determination of ^{89}Sr and ^{90}Sr in Soil Samples Using Cerenkov and Liquid Scintillation Counting
16:35	142	Dr.	KIM Hyuncheol	Environmental Radioactivity Assessment Team, Korea Atomic Energy Research Inst.	Korea	Determination of radiostrontium in seawater using automated separation system at routine and emergency situation
16:50	135	Dr.	LANDSTETTER Claudia	Austrian Agency for Health and Food Safety Ltd.	Austria	Tritium and Gross Alpha and Beta Measurements performed for the Official Radioactivity Monitoring in Austria
17:05	141	Dr.	BRAEKERS Damien	IRE Elit	Belgium	Rapid analysis of ^{90}Sr and ^{99}Tc in low active effluent using extraction disks: the advantages of the use of liquid scintillation counting
17:20	35	Mr.	MASHABA M.	Northwest University, CARST	South Africa	Gross Alpha – Beta Measurements of Water Samples from Wonderfonteinspruit Catchment Area in the Gauteng Province South Africa using LSC
17:35	Coffee break					
Hidex Workshop: Hidex – Serving LSC Community, How to Get the Most out of Your TDCR triple Coincidence Detector						
18:00-20:00		Dr.	EIKENBERG Jost	Paul Sherrer Institute	Switzerland	Practical approach to TDCR - theory, how to use TDCR on different applications, advantages and limitations (Invited speaker, open)
	296	Mr.	JUVONEN Risto	Hidex	Finland	Triple Coincidence Detector facilitating Luminescence Free Counting of H-3 (Risto Juvonen, product manager, Hidex)
	297	Mr.	JUVONEN Risto	Hidex	Finland	Optimization of alpha/beta separation conditions using 2D Graphical tool (Risto Juvonen, product manager, Hidex)
There will be a Hidex 300 SL counter in working condition at the workshop so the participants can have a chance to test TDCR and other instrument features. Hidex will provide a bag with a small gift and product material to participants. Drinks and light foods are provided.						

Tuesday

Session: LSC in nuclear chemistry (decommissioning , waste processing and nuclear materials)
(Chaired by: Prof. Nora Vajda, Hungary and Prof. Phil E. Warwick, UK)

Time	ID	Title	Author	Affiliation	Country	Presentation Title
8:30	235	Prof.	WARWICK Phillip (invited)	University of Southampton	UK	Rapid on-site screening of aqueous waste streams using dip stick technology & liquid scintillation counting
8:55	82	Prof.	VAJDA Nóra	RADANAL Ltd.	Hungary	Validation of radiochemical methods for the determination of difficult-to-measure nuclides using LSC
9:10	184	Mr.	MAXWELL Sherrod	Savannah River Nuclear Solutions, LLC	USA	Rapid Method for the Determination of Sr-90 in Steel and Concrete Samples
9:25	248	Dr.	MARSH Richard	Raddec Intl. Ltd /GAU-Radioanalytical	UK	A new bomb-combustion system for tritium extraction
9:40	286	Dr.	BRENNETOT René	Den - Service d'Etudes Analytiques et de Réactivité des Surfaces (SEARS), CEA, Université Paris-Sacla	France	Application of LSC for ^{3}H, ^{14}C, ^{36}Cl, ^{41}Ca and ^{63}Ni determination in various matrix from nuclear waste
9:55	149	Dr.	SHI Keliang	Radiochemistry Lab, School of Nuclear Science and Technology, Lanzhou Uni.	China	Determination of technetium-99 in UO$_2$ samples using liquid extraction separation and LSC measurement
10:10	106	Dr.	BAGÁN Héctor	Universitat de Barcelona	Spain	PSresins for the characterization of nuclear wastes: application to ^{126}Sn and ^{63}Ni
10:25	Coffee break					
Session: Radionuclides metrology using LSC, standardization and quality assurance-1 (Chaired by Dr. Brian E. Zimmerman USA and Prof. Leifeng Cao, China)						
10:45	105	Dr.	THIAM Cheick (invited)	CEA, LIST, Lab. National Henri Becquerel (LNE-LNHB)	France	Adaptation of PTB's analytical modelling for TDCR-Cherenkov activity measurements at LNE-LNHB
11:10	104	Dr.	KOSSERT Karsten	Physikalisch-Technische Bundesanstalt (PTB)	Germany	Primary activity standardization of ^{134}Cs
11:25	270	Dr.	MARGANIEC-GALAZKA Ju	Physikalisch-Technische Bundesanstalt (PTB)	Germany	Activity determination of 88Y by means of 4Pi(LS)Beta-Gamma coincidence counting
11:40	211	Mr.	LIU Haoran	National Institute of Metrology	China	Standardization of Na-22 by the CIEMAT/NIST method
11:55	269	Mr.	KELLETT Mark A.	CEA, LIST, Lab. National Henri Becquerel (LNE-LNHB)	France	The use of recommended decay data from the DDEP for activity determinations using LSC
12:10	134	Dr.	VAN ROOY Milton	NMISA	South Africa	First absolute standardization of ^{18}F at NMISA via 4π (LS)β-γ counting for SIRTI comparison
12:25	Lunch					

Tuesday ... continued

Session: New development on LSC instrumentation methodology, scintillator and spectrum analysis-2 (Chaired by Dr. Jean Aupiais, France and Prof. Jon Petter Omtvedt, Norway)						
13:40	136	Mr.	NICHOLS Nathaniel J. (invited)	Environmental Eng. and Earth Sciences, Clemson University	USA	Computational modeling of organic fluor molecules
14:05	181	Prof.	KIM Hong Joo	Dep. of Physics, Kyungpook National University	Korea	Development of multifunctional digital pulse processing module for particle identification in liquid scintillator
14:20	205	Dr.	Ryszard Broda	NCBJ RC POLATOM	Poland	An influence of the TDCR system settings on the radionuclides standardization
14:35	243	Dr.	BORRAS Antoni	University of the Balearic Islands	Spain	Design and development of a miniaturized detector for radionuclide determination in automated flow systems
14:50	194	Dr.	BÉLIER Gilbert	CEA, DAM, DIF, DPTA Centre du Grand rue	France	Use of active scintillating targets in nuclear physics experiments - measurement of spontaneous fission
15:05	305	Prof.	LIU Shenye	Chinese Academy of Engineering Physics	China	Fabrication and characterization of low afterglow liquid scintillator
15:20	168	Mr.	FONS-CASTELLS Jordi	Lab. of Environmental Radioactivity of the Uni. of Barcelona	Spain	Combination methods for rapid determination of mixtures of alpha and beta emitters in water samples
15:35	237	Mr.	LEE Ukjae	Ulsan National Inst. of Science and Technology	Korea	Multiple Beta Spectrum Analysis Method Based on Spectrum Fitting
15:50	139	Prof.	WU Wangsuo	Radiochemistry Lab., Lanzhou University	China	A method for ^{237}Np determination with liquid scintillation counting in the experiment of neptunium sorption onto bentonite
16:05	Coffee break					
16:20-17:40	**Poster session-1**					
	Sessions: New development on LSC instruments, scintillators and spectrum analysis; Radionuclides metrology and quality assurance; LSC for Neutrino and neutron detection; LSC in Nuclear chemistry; Plastic scintillator and application (Chaired by Dr. Jixin Qiao, Denmark)					
17:40	Coffee break					
PerkinElmer Workshop: Advances in Ultra low level detection and alpha beta separation						
18:00-20:00	Presentation-1			PerkinElmer		GCT technology combined with BGO guard background reduction to accurately measure down to near-background levels
	Presentation-2			PerkinElmer		Dual PSA Discriminators to categorize marginal events for optimal Alpha Beta separation and improved quality metric

Wednesday

Session: Application in natural radionuclides measurements and other topics
(Chaired by Dr. Siegurd Möbius, Germany and Dr. Dennis Ringkjøbing Elema, Denmark)

Time	ID	Title	Author	Affiliation	Country	Presentation Title
8:30	275	Prof.	Chałupnik Stanisław (Invited)	Central Mining Institute (GIG)	Poland	LSC for the measurements of radon and its decay products
8:55	252	Prof.	MATHUTHU Manny	North-West University (Mafikeng)	South Africa	Determination of Lead isotope ratios for Nuclear Forensic signatures from uranium mine products in South Africa
9:10	114	Dr.	BANU Ozden	Institute of Nuclear Sciences, Ege Uni.	Turkey	Evaluation of Methods for the Determination of ^{210}Pb and ^{210}Po in Ca-rich Ash Samples
9:25	262	Prof.	JULL A J Timothy	University of Arizona	USA	Studies of anthropogenic Iodine-129 using accelerator mass spectrometry in corals and ocean water
9:40	36	Dr.	QIAO Jixin	DTU Nutech, Center for Nuclear Technologies	Denmark	Development of radiochemical methods at DTU Nutech, Denmark
9:55	145	Prof.	CAO Leifeng	Laser Fusion Research Center, CAEP	China	Simulation of neutron encode imaging with liquid scintillator filled capillary array
10:10	278	Ms.	CHOUDHURY Dibyasree	Saha Institute of Nuclear Physics	India	Estimation of α emitting polonium radionuclides in proton irradiated lead bismuth targets by LSC-TD
10:25	Coffee break					
Session: Radionuclides metrology using LSC, standardization and quality assurance-2 (Chaired by Dr. Karsten Kossert, Germany and Prof. Timothy Jull, USA)						
10:45	283	Dr.	ZIMMERMAN Brian (invited)	Physical Measurement Lab., National Institute of Standards and Technology	USA	Uncertainty Assessment in Liquid Scintillation Counting
11:10	203	Dr.	CASSETTE Philippe	CEA, LIST Lab. National Henri Becquerel (LNE-LNHB)	France	With a little bit of effort, manufacturers could make better LS counters, even suitable for radionuclide metrology
11:25	129	Dr.	PUJOL Luis	Centro de Estudios y Exp. de Obras Públicas (CEDEX)	Spain	Implementation of ISO/IEC 17025 in a low level liquid scintillation tritium laboratory
11:40	284	Dr.	CAPOGNI Marco	ENEA National Institute of Ionizing Radiation Metrology	Italy	Bilateral comparison of C-14 activity measurements at the NCBJ RC POLATOM and the ENEA-INMRI
11:55	223	Dr.	VERREZEN Freddy	Belgian Nuclear Research Center (SCK-CEN)	Belgium	Long-term performance of liquid scintillation laboratory of LRM, SCK•CEN
12:10	236	Dr.	GACA Pawel	University of Southampton	UK	LSC calibration stability over long timescales
12:25	Lunch					

13:40-17:30	Social activity - Tour to Hamlet castle
18:00-22:00	Conference dinner (Tivoli)

Thursday

Session: Plastic scintillator and application
(Chaired by Prof. Jose F. Garcia and Dr. Alex Tarancon, Spain)

Time	ID	Title	Author	Affiliation	Country	Presentation Title
8:30	165	Dr.	GARCIA Jose F. (invited)	Dept. Chemical Engineering and Analytical Chemistry. Uni. of Barcelona	Spain	Plastic scintillators and related analytical proposals for radionuclide analysis
8:55	124	Dr.	ETSUKO Furuta	Ochanomizu University	Japan	Measurement of tritium with plastic scintillators in large vials of a low background LSC -an organic waste-less method
9:10	154	Dr.	MITEV Krasimir	Sofia University St. Kliment Ohridski	Bulgaria	Synthesis and characterisation of scintillating microspheres made of polystyrene/ polycarbonate for ^{222}Rn measurements
9:25	169	Prof.	DEVOL Timothy A	Environmental Engineering and Earth Sciences Dep., Clemson Uni.	USA	Development of stable extractive scintillating materials for quantification of radiostrontium in aqueous solutions
9:40	190	Mr.	BAE Jun Woo	Ulsan National Inst. of Science and Technology	Korea	Development of continuous inflow tritium in water measurement technology by using electrolysis and plastic scintillator
9:55	207	Ms.	SAEZ-MUÑOZ Marina	Lab. de Radiactividad Ambiental. Uni. Politècnica de Valènc	Spain	^{89}Sr/^{90}Sr determination in milk in emergency situations by using PSresins
10:10	225	Dr.	DEAKIN Tom Deakin	The University of Sheffield	United Kingdom	Development of low cost per unit area plastic scintillator materials for radiation detection and monitoring applications
10:25	Coffee break					
Session: Application in environmental radioactivity-2 (Chaired by Mr. Aurelien Pitois, IAEA and Prof. Mikael Jensen, Denmark)						
10:45	128	Dr.	PUJOL Luis (invited)	Centro de Estudios y Exp. de Obras Públicas (CEDEX)	Spain	Comparison of several methods for measuring ^{222}Rn in drinking water
11:10	267	Prof.	LIU Chunli	College of Chemistry & Molecular Engineering, Peking Uni.	China	Application of LSC Techniques in Environmental Radiochemistry Research in China
11:25	150	Dr.	ASLAN Nazife	Turkish Atomic Energy Auth., Sarayköy Nuclear Research and Training Center	Turkey	Adsorptive Removal of Lead-210 Using Hydroxyapatite Nanopowders Prepared From Phosphogypsum Waste
11:40	167	Dr.	KRAJCAR BRONIC Ines	Ruđer Bošković Institute	Croatia	Experience after 10 years of monitoring ^{14}C in the vicinity of the Nuclear Power Plant Krško, Slovenia
11:55	178	Ms.	VOS VAN AVEZATHE A.	RIKILT Wageningen University & Research	The Netherlands	Comparison study on low-level Sr-90 measurements for the Dutch food monitoring program
12:10	185	Dr.	RIDONE Sandro	ENEA - Radiation Protection Inst. - Integrated Lab. of Radioactivity Measurement and Monit	ITALY	Determination of ^{90}Sr and ^{89}Sr in water and urine samples by chemical separation and Cherenkov count
12:25	Lunch					

Thursday ... continued

Session: Application in medicine and other topics
(Chaired by Dr. Mats Eriksson, Sweden and Dr. Steffen Happel, France)

13:40	306	Prof.	JENSEN Mikael	DTU Nutech, Center for Nuclear Technologies	Denmark	Liquid Scintillation Counting in Quality Control of PET radiopharmaceuticals
14:00	25	Prof.	HOU Xiaolin	DTU Nutech, Center for Nuclear Technologies	Denmark	LSC for quality control of ^{99m}Tc eluate from ^{99}Mo-^{99m}Tc generator
14:20	285	Prof.	GREIFE Uwe	Department of Physics, Colorado School of Mines	USA	Functionalization of Polymers with Fluorescent and Neutron Sensitive Groups for Efficient Neutron and Gamma Detection
14:35	255	Ms.	KHUMALO Ntokozo	North-West University (Mafikeng)	South Africa	A comparison of LSC and Isotopic Techniques in Resolving Nuclear Forensic Signatures in Uranium Mini
14:50	296	Mr.	JUVONEN Risto	HIDEX Oy	Finland	Luminescence free counting of H-3 facilitated by Hidex 300 SL/600 SL TDCR triple coincidence counter
15:05	297	Mr.	HAASLAHTI Ville	HIDEX Oy	Finland	Optimization of a/b separation using 2D graphical tool
15:20	300	Mr.	SERRALUNGA Michel	ZINSSER ANALYTIC GMBH	Germany	Alternative technical solutions for L.S.Counting of low activities environmental samples
15:35	132	Dr.	EDLER Ronald	PerkinElmer	Germany	Measurement of Tritium, gross alpha/beta and ^{222}Rn with the new Quantulus GCT 6220 according to council directive 2013/51 Euratom
16:05	Coffee break					
16:20-17:40	Poster session 2					
	Sessions: Application environmental radioactivity; Application in medicine; Application in tritium studies; LSC for natural radionuclides measurement ; Applications based on alpha/beta spectrometry and other topics (Chaired by Dr. Kasper Andersson)					

Friday

Session: Application in tritium studies
(Chaired by Dr. Nicolas Baglan, France and Dr. Per Roos, Denmark)

Time	ID	Title	Author	Affiliation	Country	Presentation Title
8:30	271	Dr.	BAGLAN Nicolas (invited)	CEA/DAM/DIF	FRANCE	Tritium analysis strategy regarding activity concentration levels in monitoring situations
8:55	117	Dr.	ETSUKO Furuta	Ochanomizu University	Japan	Apparatus for measurement of tritium in expiration with plastic scintillator
9:10	213	Dr.	VARLAM Carmen	Tritium Lab., Inst. for Cryogenics and Isotopic Technologies ICSI	Romania	Homogeneity Assessment for Grass Items from Organically Bound Tritium Proficiency Test
9:25	176	Ms.	GOMES Ana RIta	Instituto Superior Técnico, Uni. de Lisboa	Portugal	Determination of tritium in water using isotopic enrichment. Methodology improvements
9:40	182	Ms.	XU Qinghua	Inst. of Materials, China Academy of Engineering Physics	China	A new efficiency measuring way for the liquid scintillation counting method
9:55	231	Mr.	CHEN Qianyuan	Radiation Monitoring Technical Center, Ministry of Environmental Protection	China	Quench correction in the analysis of Organic Bonded Tritium (OBT) in biota samples
10:10	180	Dr.	KOŽAR LOGAR Jasmina	University of Yaoundé I, Faculty of Science	Cameroon	Tritium content in tissue free water (TFWT) in African coconuts
10:25	Coffee break					

Session: Application in environmental radioactivity-3
(Chaired by Prof. Stanisław Chałupnik, Poland and Dr. Mun Ja Kang, Korea

Time	ID	Title	Author	Affiliation	Country	Presentation Title
10:45	281	Dr.	CHUNG Kun Ho (invited)	Environmental Radioactivity Assessment Team, Korea Atomic Energy Research Inst.	Korea	Automation of the radiochmical procedures for the sequential separation of radionuclides
11:10	272	Mr.	JEROME Simon Mark	National Physical Laboratory	United Kingdom	Determination of ^{242m}Am by Extraction Chromatography and Liquid Scintillation Counting
11:25	277	Dr.	ROOS Per	DTU Nutech, Center for Nuclear Technologies	Denmark	A comparison of methodologies in analysing 210Pb-210Bi-210Po
11:40	201	Ms.	MULLINS Sarah	Department of Health	Australia	The comparison of two liquid scintillation instruments for analysis of highly quenched samples
11:55	245	Dr.	NODILO Marijana	Ruđer Bošković Institute	Croatia	Subsequent determination of ^{90}Sr and ^{210}Pb in goat bones samples from Island of Mljet, Croatia
12:10	Close ceremony					
12:40	Lunch					

Poster session 1

Id	Title	Author	Affiliation	Country	Abstract title	Session
131	Dr.	BUZYNNYI Mykhailo	Inst. of Environmental Geochemistry NAS of Ukraine	Ukraine	Teflon vials for precise C-14 in benzene measurements by LSC techniques	New development on LSC instruments, scintillators and spectrum analysis
153	Dr.	AUPIAIS Jean	CEA, DAM, DIF, F-91297 Arpajon France	France	Evaluation of several liquid & solid scintillators in terms of pulse shape discrimination for fission studies	New development on LSC instruments, scintillators and spectrum analysis
163	Mr.	THOMSON James	Meridian Biotechnologies Ltd.	UK	LSC Counting - Dos and Donts	New development on LSC instruments, scintillators and spectrum analysis
164	Dr.	KRAJCAR BRONIC Ines	Ruđer Bošković Institute	Croatia	Determination of biogenic component in liquid fuels by the ^{14}C method and direct LSC measurement	New development on LSC instruments, scintillators and spectrum analysis
189	Dr.	DEAKIN Tom	LabLogic Systems Ltd.	UK	Development of the 'WILMA' LSC platform for the online monitoring of radionuclides in air and water	New development on LSC instruments, scintillators and spectrum analysis
202	Dr.	SABOT Benoit	CEA/LNE-LNHB	France	A new compact TDCR-Cerenkov counter for the online measurement of ^{56}Mn activity	New development on LSC instruments, scintillators and spectrum analysis
212	Mr.	HAASLAHTI Ville	Hidex	Finland	Improved counting efficiency determination by core function modelling in triple to double coincidence ration counter	New development on LSC instruments, scintillators and spectrum analysis
215	Mr.	JUVONEN Risto	Hidex Oy	Finland	Luminescence free liquid scintillation counting method	New development on LSC instruments, scintillators and spectrum analysis
232	Mrs.	KATO Yuka	Measuring Systems Engineering Dept., Hitachi, Ltd.	JAPAN	Development of a method to measure filter sample using plastic scintillator	New development on LSC instruments, scintillators and spectrum analysis
292	Ms.	KABAI Eva	Federal Office for Radiation Protection	Germany	Approach to the uncertainty estimation of the ^{89}Sr/^{90}Sr determination by LSC	New development on LSC instruments, scintillators and spectrum analysis
148	Dr.	ÖZÇAYAN Gülten	Turkish Atomic Energy Auth., Sarayköy Nuclear Research and Training Center	TURKEY	Assignment of Reference Value for ^{90}Sr in Black Tea Powder used as TAEK Proficiency Test Sample	Radionuclides metrology and quality assurance
138	Mr.	YELTEPE Emin	Turkish Atomic Energy Auth. - Sarayköy Nuclear Research and Training Center	Turkey	Standardization of ^{170}Tm with CIEMAT/NIST efficiency tracing, TDCR and gamma spectrometric methods	Radionuclides metrology and quality assurance
143	Dr.	ZIMMERMAN Brian	Physical Measurement Lab., National Inst. of Standards and Technology	USA	Correlation in the Application of the Triple-to-Double Coincidence Method with Unequal Photomultiplier Tube Efficiencies	Radionuclides metrology and quality assurance
151	Dr.	JIANG Jun	AWE plc	UK	The Measurement of Tb-160 and Tb-161 by Liquid Scintillation Counting	Radionuclides metrology and quality assurance
192	Ms.	GAIGALAITÉ Lina	Center for Physical Sciences and Technology (FTMC)	Lithuania	Standardization of tritium with the TDCR instrument	Radionuclides metrology and quality assurance
177	Mrs.	KATO Yuka	Measuring Systems Engineering Dept., Hitachi, Ltd.	Japan	New calibration method for liquid scintillation counter using plastic scintillator	Radionuclides metrology and quality assurance
251	Mr.	CHAE Jung-Seok	Korea Inst. of Nuclear Safety	Korea	Determination of ^{210}Pb by liquid scintillation counting of ^{210}Pb and its progenies	Radionuclides metrology and quality assurance
276	Prof.	CHALUPNIK Stanislaw	Silesian Centre for Environmental Radioactivity, Central Mining Inst.	Poland	Application of LSC for measurements of concentrations of radon and thoron decay products in air	Radionuclides metrology and quality assurance
279	Ms.	ERIKSSON Sofia	Swedish Radiation Safety Auth., Dept. of Radiation Protection	Sweden	DON'T TRUST YOUR MEMORY IN THE LAB – the implementation of a quality control program for LSC	Radionuclides metrology and quality assurance
298	Mr.	TEMPLE Simon	Meridian Biotechnologies Ltd	UK	External Standard Channels Ratio on Hitachi Aloka AccuFLEX LSC-8000	Radionuclides metrology and quality assurance
303	Ms.	TAN Hiong Jun Angela	DSO National Laboratories	Singapore	Simultaneous Analysis of Strontium and Cesium in a Single Process	Radionuclides metrology and quality assurance
304	Mr.	LOO Howe Kiat	DSO National Laboratories	Singapore	Application of DGA Resin for Yttrium Extraction during Strontium Analysis	Radionuclides metrology and quality assurance
127	Prof.	ZHANG Zhiyong	Inst. of High Energy Physics, The Chinese Academy of Sciences	China	Preparation of Gadolinium Loaded Liquid Scintillator for Daya Bay Neutrino Experiment	LSC for Neutrino and neutron detection
137	Mr.	LI Jinghuai	China Institute of Atomic Energy	China	Fast Neutron Multiplicity Detectors Based on Liquid Scintillators for uranium Mass determination	LSC for Neutrino and neutron detection
145	Prof.	CAO Leifeng	Laser Fusion Research Center, CAEP	China	Simulation of neutron encode imaging with liquid scintillator filled capillary array	LSC for Neutrino and neutron detection

Poster session 1

Id	Title	Author	Affiliation	Country	Abstract title	Session
198	Prof.	KOSHIMIZU Masanori	Tohoku University	Japan	Synthesis of ZrO_2 nanoparticles for liquid scintillators for neutrinoless double beta decay detection	LSC for Neutrino and neutron detection
81	Mr.	RAJEC Pavol	Comenius University, Dep. of Nuclear Chemistry	Slovakia	Radionuclides determination by LSC during routine operation of nuclear power plants and its decommissioning	LSC in Nuclear chemistry
90	Mr.	GOUDEAU Vincent	CEA, Direction of Fundamental Research, Unities for Protection Health and Security, Environment	France	In situ measurement of ^{90}Sr activity using direct liquid scintillation counting (ISMAD LSC): application to decommossioning projects	LSC in Nuclear chemistry
125	Ms.	GAUTIER C.	Den - Service d'Etudes Analytiques et de Réactivité des Surfaces (SEARS), CEA, Uni. Paris	France	Is it easy to improve radiochemical methods in respect of REACH regulation?	LSC in Nuclear chemistry
256	Mr.	YUN Yong-Gi	Korea Inst. of Nuclear Safety	Korea	Pre-concentration method using oxalate co-precipitation with MnO_2 for determination of ^{90}Sr	LSC in Nuclear chemistry
260	Dr.	KIM Daeji	NA	South Korea	Evaluation of ^{14}C analysis using Pyrolyser system for decommissioned materials	LSC in Nuclear chemistry
261	Dr.	DIRKS Carina	TrisKem International	France	On the development of an extraction chromatographic resion for the separation and determination of selenium	LSC in Nuclear chemistry
295	Ms.	WOLF Sarah	Helmholtz-Zentrum Berlin für Materialien und Energie GmbH	Germany	Quantitative determination of Ni-63 wipe tests by liquid scintillation analysis (LSA)	LSC in Nuclear chemistry
301	Dr.	LIM Jong Myoung	Korea Atomic Energy Research Institute	Korea	The Radiological Characterization of Concrete and Soil from Decommissioning Process of Research Reactor	LSC in nuclear Chemistry
302	Ms.	JEONG Kahee	KHNP Central Research Institute	Korea	Laboratory infrastructure and utilization plan for radiochemical analysis	LSC in nuclear Chemistry
119	Dr.	ETSUKO Furuta	Ochanomizu University	Japan	A plastic scintillation counter for tritiated water measurement as vapor	Plastic scintillator and application
120	Dr.	GARCIA Jose.F.	Department of Analytical Chemistry. Uni. of Barcelona	Spain	^{90}Sr and ^{99}Tc determination in Radioactive Ion Exchange Resins coming from Nuclear Wastes	Plastic scintillator and application
204	Mr.	HIYAMA Fumiyuki	Dep. of Applied Chemistry, Graduate School of Engineering, Tohoku Uni.	Japan	X-ray detection capabilities of plastic scintillators incorporated with hafnium oxide nanoparticles	Plastic scintillator and application
224	Dr.	TARANCON Alex	University of Barcelona	Spain	Plastic scintillation foils for the measurement of the alpha and beta emitting radionuclides	Plastic scintillator and application
238	Prof.	KIM Sunghwan	Cheongju University	Korea	Fabrication and Characterization of 3D Printed Plastic Scintillator	Plastic scintillator and application
239	Dr.	MINOWA Haruka	The Jikei University School of Medicine	Japan	Rapid separation and easy measurement using plastic scintillator for radiostrontium in seawater	Plastic scintillator and application
250	Dr.	MITEV Krasimir	Sofia University St. Kliment Ohridski	Bulgaria	A Laboratory Study of Scintillation Counting of Polycarbonates	Plastic scintillator and application

Poster session 2

Id	Title	Author	Affiliation	Country	Abstract title	Session
80	Dr.	SAUEIA Ca¡tia	IPEN - CNEN/SP	Brazil	Assessment of ^{90}Sr by liquid scintillation counting (LSC) in marine fish species consumed in the city of São Paulo	Application environmental radioactivity
93	Mrs.	BAO Li	China Inst. for Radiation Protection	China	Carbon-14 concentration in aqueous samples from southeast of China	Application environmental radioactivity
109	Dr.	STOJKOVIC Ivana	University of Novi Sad, Faculty of Technical Sciences	Serbia	Optimization of rapid ^{90}Sr/^{90}Y screening method in waters via Cherenkov radiation detection	Application environmental radioactivity
110	Dr.	STOJKOVIC Ivana	University of Novi Sad, Faculty of Technical Sciences	Serbia	Evaluation of different LSC methods for ^{222}Rn determination in waters	Application environmental radioactivity
115	Dr.	IDOETA Raquel	Dpto. de Ingeniería Nuclear y Mecánica de Fluidos, ETSI de Bilbao, UPV	Spain	Use of the IAEA rapid simultaneous determination of ^{89}Sr and ^{90}Sr in milk as a routine method for environmental purposes	Application environmental radioactivity
116	Dr.	IDOETA Raquel	Dpto. de Ingeniería Nuclear y Mecánica de Fluidos, ETSI de Bilbao, UPV	Spain	Ra-226 determination by LSC and alpha-spectrometry in by-products from oil industry	Application environmental radioactivity
155	Mr.	WIATR K.	National Veterinary Research Institute	Poland	Pilot Studies on ^{90}Sr in Meat, Milk and Dairy Products from the Polish Market	Application environmental radioactivity
156	Dr.	KRIŠTOF Romana	Jožef Stefan Institute	Slovenia	Biolubricant measurements by direct LSC method	Application environmental radioactivity
172	Ms.	COHA Ivana	Ruđer Bošković Institute	Croatia	Optimisation of Tri-Carb Liquid Scintillation Counter for Cherenkov Counting of ^{90}Sr/^{90}Y	Application environmental radioactivity
174	Dr.	HERMANSPAHN Nikolaus	Inst. of Environmental Science and Research Ltd	New Zealand	Sample pre-treatment for HTO measurements in biota and seawater	Application environmental radioactivity
187	Dr.	FUNDA Barlas Şimşe	Turkish Atomic Energy Aut., Çekmece Nuclear Research and Training Center	Turkey	Determination of Sr-90 in soil and sediment samples with double energetic window Method by LSC	Application environmental radioactivity
200	Dr.	YAN Zeyi	Lanzhou University	China	Determination of Pu in environmental water samples using TEVA microextraction chromatography separation and liquid scintillation counter measurement	Application environmental radioactivity
208	Dr.	JOBBAGY Viktor	European Commission, Joint Research Centre (EC-JRC)	Belgium	Optimization of liquid scintillation counting technique for a European interlaboratory comparison in gross alpha/beta activity concentrations in water	Application environmental radioactivity
209	Mrs.	WALLNER Gabriele	Institut für Anorganische Chemie, Uni. Wien	Austria	Radionuclide extraction from aqueous solutions by Ionic Liquids	Application environmental radioactivity
210	Dr.	VASILE Mirela	SCK-CEN	Belgium	Applicability of the 3M EmporeTM RAD disks for rapid determination of ^{90}Sr and ^{99}Tc by liquid scintillation counting	Application environmental radioactivity
222	Mr.	DOUMONT Cédric	IRE Elit	Belgium	The Belgian St-Adele fountain as a natural sample source for an interlaboratory comparison exercise	Application environmental radioactivity
233	Dr.	VAGNER Irina	National R&D Inst. for Cryogenics and Isotopic Technologies - ICSI	Romania	Comparison of two methods for C-14 analysis from essential oils using LSC	Application environmental radioactivity
257	Ms.	GAIGALAITÉ Lina	Center for Physical Sciences and Technology (F TMC)	Lithuania	Measurement of radiocarbon and tritium in a single sample: simultaneous and radiochemistry approaches	Application environmental radioactivity
282	Dr.	OSVATH Szabolcs	National Public Health Center, National Research Directorate for Radiobiology and Radiohygiene	Hungary	Determination of Ra-226 in natural water samples by liquid scintillation counting	Application environmental radioactivity
290	Dr.	CHUNG Sungwook	Korea Basic Science Institute	Republic of Korea	Radon-222 Radioactivity in Groundwater	Application environmental radioactivity
288	Dr.	SALMINEN-PAATERO Susanna	University of Helsinki, Department of Chemistry	Finland	^{90}Sr in surface air of Rovaniemi (Finnish Lapland) in 1965-2011	Application environmental radioactivity
274	Prof.	CHALUPNIK Stanislaw	Central Mining Inst., Silesian Centre for Environmental Radioactivity	Poland	The role of LSC methods in implementation of the Euratom Directive 51/2013 for the monitoring of radioactivity of water supplies in Poland	Application environmental radioactivity
244	Dr.	BORRAS Antoni	University of the Balearic Islands	Spain	Automated dispersive liquid-liquid microextraction of ^{99}Tc from biological samples and hospital residues previous liquid scintillation counting	Application in medicine and other topics
259	Mr.	KO Raymond	Radiation Protection Bureau, Health Canada	Canada	Radiobioassay method for the determination of isotopes from fuel particles	Application in medicine and other topics
213	Dr.	VARLAM Carmen	Tritium Lab., Inst. for Cryogenics and Isotopic Technologies ICSI	Romania	Homogeneity Assessment for Grass Items from Organically Bound Tritium Proficiency Test	Application in tritium studies

Poster session 2

Id	Title	Author	Affiliation	Country	Abstract title	Session
157	Dr.	KRIŠTOF Romana	Jožef Stefan Institute	Slovenia	Tritium in environment around NPP Krško	Application in tritium studies
180	Dr.	KOŽAR LOGAR Jasmina	University of Yaoundé I, Faculty of Science	Cameroon	Tritium content in tissue free water (TFWT) in African coconuts	Application in tritium studies
247	Ms.	PÁNTYA Annamária	Hungarian Acad. of Sciences, Centre for Energy Research	Hungary	Tritium internal dose estimation with liquid scintillators	Application in tritium studies
146	Dr.	KANG Yeong-Rok	Research Center, Dongnam Inst. of Radiological & Medical Sciences	Republic of Korea	Study on optimization of liquid scintillation counting during tritium measurements	Application in tritium studies
152	Mrs.	MARESOVA Diana	T. G. Masaryk Water Research Inst.	Czech Republic	Determination of very low level tritium volume activities in surface water and precipitation in the Czech Republic	Application in tritium studies
158	Dr.	FENG Lin	Third Institute of Oceanography	China	Determination of the baseline organically bound tritium (OBT)	Application in tritium studies
234	Dr.	SUGIHARA Shinji	Radioisotope Center, Kyushu Uni.	Japan	OBT measurement in annual ring of tree, Japan	Application in tritium studies
246	Ms.	GAIGALAITÉ Lina	Center for Physical Sciences and Technology (F TMC)	Lithuania	The decade tritium monitoring results in groundwater near a radioactive waste repository	Application in tritium studies
294	Dr.	KANG Yeong-Rok	Dongnam Institute of Radiological & Medical Sciences (DIRAMS)	Korea	Study on optimization of liquid scintillation counting during tritium measurements	Application in Tritium studies
289	Dr.	MÖBIUS Siegurd	German Society for Liquid Scintillation DGFS e.V.	Germany	Simple LS Arrangement for Education and Measurement of Natural Radionuclides in Water Samples of Madagascar and Sri Lanka	LSC for natural radionuclides measurement and other topics
107	Dr.	Juergen Wendel	German Society for Liquid Scintillation DGFS e. V.	Germany	DGFS : 15 Years of Research, Education and Training in Liquid Scintillation – Heading for More	LSC for natural radionuclides measurement and other topics
112	Dr.	NIKOLOV Jovana	University of Novi Sad, Faculty of Sciences, Dep. of Physics	Serbia	Development of direct LSC method for biogenic fraction determination in fuels	LSC for natural radionuclides measurement and other topics
159	Mr.	WANG Kaifeng	Peking University	China	Extraction and Characterization of Groundwater Colloids	LSC for natural radionuclides measurement and other topics
183	Mr.	NISTI M. B.	IPEN - CNEN/SP	Brazil	Evaluation of the LSC counting time for determination gross alpha and gross beta activity, ^{3}H and ^{90}Sr/^{90}Y	LSC for natural radionuclides measurement and other topics
186	Prof.	MIETELSKI Jerzy-Wojc	The Henryk Niewodniczański Inst. of Nuclear Physics	Poland	Secondary electron spectra measured in modified LSC vial	LSC for natural radionuclides measurement and other topics
206	Mr.	MASHABA Machel	North West University	South Africa	Gross Alpha - Beta Measurements of Water Samples from Wonderfonteinspruit Catchment Area in the Gauteng Province South Africa using LSC	LSC for natural radionuclides measurement and other topics
253	Mr.	ABRANTES João	Instituto Superior Técnico, Uni. de Lisboa	Portugal	Suitability of gross alpha region of interest for the determination of gross alpha and gross beta activities in water by liquid scintillation counting	LSC for natural radionuclides measurement and other topics
273	Dr.	TORO Laszlo	Matefin Ltd.	Romania	Limitations of the LSC technique in alpha spectrometry of transuranium elements	LSC for natural radionuclides measurement and other topics
299	Mrs.	CHO sooyoung	Korea Institute of Geoscience and Mineral Resources(KIGAM)	Korea	^{222}Rn concentration in groundwater of geological rock types in South Korea using LSC	LSC for natural radionuclides measurement and other topics
307	Prof.	TODOROVIC Nataša	University of Novi Sad, Faculty of Sciences	Serbia	Establishment of rapid LSC method for direct alpha/beta measurements in waters	LSC for natural radionuclides measurement and other topics

Monday

1 May 2017

1. **Opening Session**
2. **Session: New development on LSC instrumentation methodology, scintillator and spectrum analysis-1**
3. **Session: LSC in neutrino and neutron detection**
4. **Session: Application in environmental radioactivity-1**
5. **Hidex Workshop: Serving LSC Community, How to Get the Most out of Your TDCR triple Coincidence Detector**

Radionuclide metrology using LSC:
Current status and limitations

Karsten Kossert

Physikalisch-Technische Bundesanstalt (PTB), Bundesallee 100, 38116 Braunschweig, Germany

Corresponding author's e-mail: karsten.kossert@ptb.de

Liquid scintillation counting plays a key role for accurate determination of the activity and nuclear decay data in radionuclide metrology. In particular the TDCR (triple-to-double coincidence ratio) method and the CIEMAT/NIST efficiency tracing technique can be considered as great success since they were applied to measure the activity of a number of radionuclides with low uncertainties in the past 3 decades. The methods are complemented with 4πβ(LS)-γ- and 4πβ(Č)-γ coincidence counting as well as the TDCR-Čerenkov method.

The talk gives a brief overview about the methods including uncertainty considerations, and summarizes recent progress achieved within the ICRM community. This comprises development of new measurement systems, electronics and extensions of models to compute counting efficiencies.

In addition, we discuss several challenges and difficulties which define current limitations of the methods. This comprises

- the accurate computation of emission spectra for beta and electron-capture isotopes
- the ionization quenching function and related model dependence
- instrumentation and related limitations.

Six decades of environmental-radioactivity measurements

Sven P. Nielsen

DTU Nutech, Frederiksborgvej 399, 4000 Roskilde, Denmark

Corresponding author’s e-mail: spni@dtu.dk

Studies of environmental radioactivity were initiated in 1956 at the Research Establishment Risø located at Roskilde, Denmark. Availability of commercial equipment for such studies was limited which prompted development of instruments for low-level counting. Chemical analyses of environmental samples were combined with measurements of radioactivity and allowed early identification of fission products due to global fallout from nuclear weapons testing.

A brief review is given on the development and use of instruments for low-level counting which combined with chemical analyses have been successful over the years in providing reliable data on levels of environmental radioactivity and assessments of radiological risks. The sources of man-made radionuclides that have been considered include global fallout from nuclear weapons testing during the 1960’s, local radioactive pollution from an accident in Greenland at Thule in 1968, and fallout from the accident in Chernobyl in 1986 and in Fukushima in 2011.

The comparison of scintillation properties of YAP:Ce, YAG:Ce and ZnO:Ga powders as a potential substitution of LSC cocktail

Jiri JANDA

University of Defence, NBC Defence Institute, Vita Nejedleho, Vyskov, CZ

The work deals with the possibility of replacement of traditional liquid scintillation cocktail by scintillation powder with grain size up to 30 μm. Based on previous research, YAP:Ce, YAG:Ce and ZnO:Ga powders were selected. The basic scintillation parameters were investigated, such as geometry, amount of scintillator, efficiency and activity linearity dependences as well as auxiliary parameters like afterglow, temperature dependency and repeatability usage. All parameters were simultaneously measured using AquaLight as a scintillation cocktails representative.

On-line LS Spectroscopy of Super-heavy Elements

Jon Petter Omtvedt [1], Yukiko Komori [2], Solveig Wulff [1], Hiromitsu Haba [2]

[1] University of Oslo, Department of Chemistry, PO Box 1033 – Blindern, NO-0315 Oslo, Norway
[2] Nishina Center for Accelerator-Based Science, RIKEN, Wako, Saitama 351-0198, Japan

Corresponding author’s e-mail: j.p.omtvedt@kjemi.uio.no

Exploration of the Super-heavy Elements (SHEs) is one of today’s research frontiers – it is exploration into mostly unknown and uncharted territory. New elements, much heavier than those normally found in nature, might have unexpected properties or behaviors. Even though sophisticated models and powerful computers enable us to predict, to a certain degree, both physical and chemical properties of unknown elements and nuclei, experimental data on SHEs is paramount to improve their predictive power. In particular parameters which effect are difficult to observe for light elements, but nevertheless important for accurate description of their behavior, can be “stress tested” when applied to SHEs. The typical example is of course the relativistic part of the atomic wave-function that describes chemical bonding. Due to the extreme mass of the SHEs, "relativistic effects" are much more dominant for the SHEs and thus provide an ideal testing ground. Experimental verification of theoretical predictions will thus not only verify that the model is correct for a given (super-heavy) element, but increase the validity of the model for all elements.

Chemical studies of SHEs, more accurately described as the transactinide elements with atomic numbers $Z \geq 104$, have been performed on elements up to hassium (Z=108) and on copernicium (Cn, Z=112) and flerovium (Fl, Z=114). See e.g. [1] for a comprehensive review. Such experiments are extremely difficult due to the very low production rates of around one atom per minute for rutherfordium (Rf, Z=104) and down to less than one atom per week for Fl (chemistry at the "one-atom-at-a-time" scale). In addition the isotopes of these elements are all radioactive and the most accessible ones decay in less than a minute. As the elements become heavier their isotopes generally decay faster. For this reason most chemical studies have been performed in gas phase, as the transfer from the target to the chemistry apparatus and finally to detection are easier and faster than for experiments performed in aqueous phase. Therefore, only elements rutherfordium (Rf, Z=104) and dubnium (Db, Z=105) have been studied and detected in aqueous solution since the current technology is not fast enough to handle half-lives shorter than around 30 s. In addition, seaborgium (Sg, Z=106) was studied in aqueous phase by observing the decay products of 8.5 and 14.4 s ^{265}Sg [2], see [1] for a review.

The limiting factor for aqueous phase studies is the detection method, since dry samples have to be prepared for detection by solid state detectors and this is a slow and complicated process when the starting point is an aqueous solution. This process typically takes 30-40 s. Therefore an alternative detection method using Liquid Scintillation (LS) for aqueous phase studies was developed. This system was combined with the very fast liquid-liquid extraction (LLX) system SISAK and successful detection of the 4-s ^{257}Rf nuclide was demonstrated in several experiments [3,4]. The SISAK system constitutes specially constructed centrifuges with very small hold-up volumes and continuous flow. However, the system is very complex and complicated to maintain running for days on end, as required by SHE experiments to obtain needed statistics. In addition, the fast transport through the SISAK system requires high flow rates (around 0.5 mL/s for each phase), which produces significant amounts of waste when it runs continuously for extended periods.

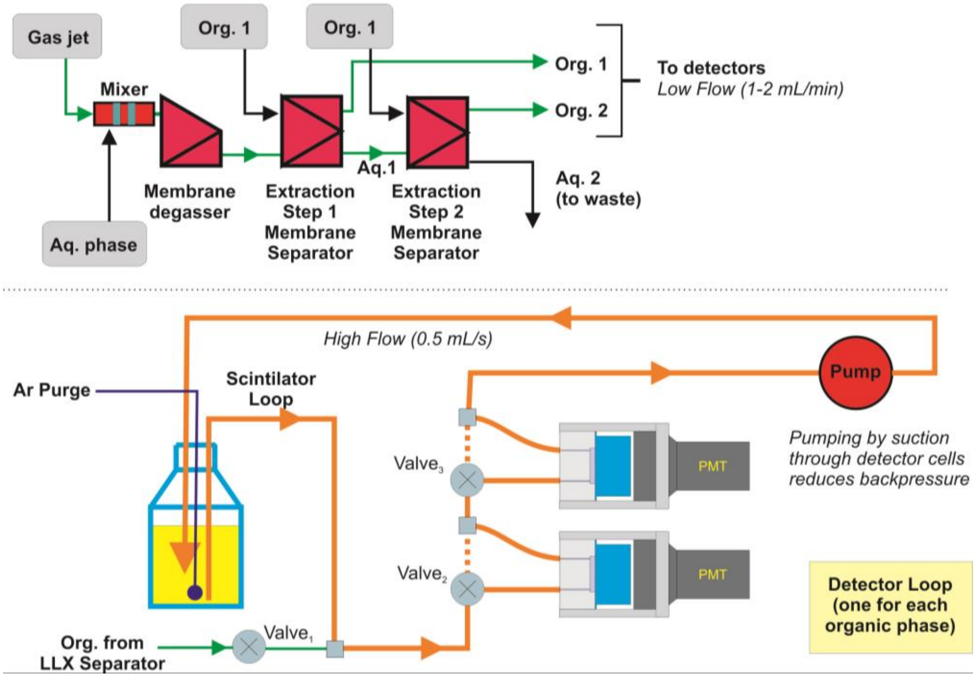

Fig. 1 Schematic setup of low-flow rate liquid-liquid separation system with high flow rate LS detection system.

Recently a new method for using the LS detection system developed for SISAK has been demonstrated. In this setup a new LLX system [5] based on membrane separation and flow rates in the mL/min range (a factor of 30 less than what was used for the SISAK system) is combined with the old LS detection system. However, the scintillator is now run in a loop (with a delay tank) as illustrated in Fig. 1. Thus, the fast transport is maintained but the generation of waste significantly reduced.

In addition, the small amount of organic phase from the membrane LLX system compared to the amount of scintillator cocktail significantly reduces problems with quenching. This removes the need for "on-line" quench monitoring and the complicated feedback loop [3] in the spectroscopy system to perform real-time quench correction (which was needed to correctly identify "mother" events in real time in order to close a detection cell to identify correlated daughter-decays).

At LSC2017 test results and outlook for SHE experiments with this new system will be presented.

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A performance comparison of two liquid scintillation counters from PerkinElmer, Inc.

Xiao-gui FENG [1)], Guo-hua JIANG [2)], Jian-hua HUANG [2)], Jian-yu DU [3)],
Qian-ge HE [1)], Jian-chen WANG [1)], Jing CHEN [1)]

[1)] Institute of Nuclear and New Energy Technology, Collaborative Innovation Center of Advanced Nuclear Energy Technology, Tsinghua University, Beijing 100084, PR China
[2)] Analytical and Testing Center, Beijing Normal University, Beijing 100875, PR China
[3)] PerkinElmer, Beijing 100015, PR China

Corresponding author's e-mail: fengxiaogui@tsinghua.edu.cn

PerkinElmer has developed a new liquid scintillation counter (LSC), Quantulus GCT 6220, which features its Guard Compensation Technology (GCT) and is intended to replace the old well-known Quantulus 1220. The new and the old LSCs have been compared on their basic performances, i.e. background count rate, counting efficiency, and α/β discrimination, by determining the radioactivity of the radionuclides of ^{3}H, ^{14}C, ^{241}Am, ^{90}Sr/^{90}Y, ^{60}Co, and ^{137}Cs. For the new LSC, the count in β-MCA (multi-channel analyzer) decreases with the increase of GCT level. If the factory preset "GCT high" is applied, the new LSC exhibits lower background count rate in β-MCA than the old one, but higher in α-MCA because the GCT has no effect on the count in α-MCA. As for the counting efficiency, which LSC is higher depends not only on the LSC setting, but also on the decay properties of the radionuclide to-be-determined. The results of α/β discrimination vary with the quench level of the samples: the new LSC is better for high quenched samples, while the old one is better for low quenched samples.

Design and performance of a miniature TDCR counting system

K. Mitev[1], P. Cassette [2], V. Jordanov [3], H.R. Liu [4], Ch. Dutsov[1]

[1] Sofia University “St. Kliment Ohridski”, Faculty of Physics, 5 James Bourchier Blvd, 1164 Bulgaria
[2] CEA, LIST, Laboratoire National Henri Becquerel, LNE-LNHB, Gif-sur-Yvette F-91191, France
[3] LABZY, LLC, Santa Fe, New Mexico, USA
[4] National Institute of Metrology, Division of Ionizing Radiation, Beijing, 100029, P.R. China

Corresponding author’s e-mail: kmitev@phys.uni-sofia.bg

The objective of this work is to present the design and first tests of a miniature Triple-to-Double Coincidence Ratio (TDCR) system developed at the Sofia University “St. Kliment Ohridski”. The system (hereafter referred to as TDCR-SU) is intended for absolute activity measurement of pure beta-emitters and ^{222}Rn dissolved in liquid scintillation cocktails. It consists of a TDCR counter module equipped with dedicated front-end electronics and a digital pulse-height analyzer (hereafter referred to as nanoTDCR). Similarly to the ENEA portable TDCR system [1], the counter has three Hamamatsu R7600-200 square photomultiplier tubes (PMTs), but has cylindrical optical chamber in which the PMTs are fixed on a certain distance from the LS vial in order to achieve full solid angle view of the vial by them (see Fig. 1). The optical chamber of the vial is made of Polytetrafluoroethylene (Teflon®) and the housing is made of Polyoxymethylene (POM). The nanoTDCR module is dedicated to TDCR measurements and was developed by the labZY company. By design, it has similar functionalities as the MAC3 TDCR counting module [2] with some additional functions like: individual extendible dead-time time in each channel; capability of simultaneous coincidence counting with two different extendable dead-times; capability of simultaneous coincidence counting and spectrum acquisition from a PMT, etc. The nanoTDCR consumes about 1W of power and can be powered directly from USB computer ports. The nanoTDCR module weighs 135g and has compact form factor (92mm x 38mm x 25mm). A detailed description of the nanoTDCR’s design and functionality is beyond the scope of this work and will be given elsewhere.

Fig. 1. Description of the TDCR-SU system. Left to right: Cross-sectional front and top views of the counter; the optical camber; the housing and the counter with a mounted PMT.

As a preliminary test, the nanoTDCR counting module was first compared to the acquisition system of the primary TDCR counter (RCTD1) of the Laboratoire National Henri Becquerel (LNHB), including a MAC3 module. The logical outputs of the constant fraction discriminator of this acquisition system were connected to the nanoTDCR inputs and simultaneous counting of 4 different ^{241}Am sources with activities in the 480 – 4600 Bq range were performed. The comparison between the raw coincidence results of the two acquisition systems is shown in Table 1, where excellent agreement can be observed. The relative differences are less than 0.3%, and can be attributed to the small differences of the live times of the two systems.

Table 1. Comparison between the MAC3 and nanoTDCR counting modules

RCTD1 with Source:	Coincidences AB, s^{-1}			Coincidences BC, s^{-1}			Coincidences AC, s^{-1}			Coincidences ABC, s^{-1}		
	MAC3	nanoTDCR	Δ, %	MAC3	nanoTDCR	Δ, %	MAC3	nanoTDCR	Δ, %	MAC3	nanoTDCR	Δ, %
Am-241-NO.1	490	490.5	-0.11	490	490.2	-0.04	490	490.2	-0.03	490	490.0	0.00
Am-241-NO.2	1517	1519.7	-0.18	1516	1519.4	-0.22	1516	1519.3	-0.22	1516	1519.1	-0.21
Am-241-NO.3	2557	2563.0	-0.23	2557	2562.6	-0.22	2557	2562.6	-0.22	2557	2562.3	-0.21
Am-241-NO.4	4604	4599.7	0.09	4604	4599.4	0.10	4604	4598.8	0.11	4604	4599.1	0.11

In a second benchmark comparison the four ^{241}Am sources were measured with the TDCR-SU system and the LNHB's primary TDCR system (RCDT1). An excellent agreement between the two counting systems was observed with differences smaller than 0.3% (see Table 2). There is no systematic trend in the differences, which indicates a linear response of the TDCR-SU system for counting rates in the interval 480 – 4600 s^{-1}.

Table 2. Comparison between the LNHB's RCTD1 counter and the TDCR-SU system

Source:	Logical sum of the net double coincidences (D) , s^{-1}			Net triple coincidences (T) , s^{-1}			Ratio T/D		
	RCTD1	TDCR-SU	Δ, %	RCTD1	TDCR-SU	Δ, %	RCTD1	TDCR-SU	Δ, %
Am-241-NO.1	487	486.4	0.12	487	486.1	0.18	1.0000	0.9994	0.06
Am-241-NO.2	1513	1515.2	-0.15	1513	1514.1	-0.07	1.0000	0.9993	0.07
Am-241-NO.3	2553	2559.5	-0.25	2554	2557.6	-0.14	1.0004	0.9992	0.11
Am-241-NO.4	4600	4591.2	0.19	4601	4588.0	0.28	1.0002	0.9993	0.09

In the third test the performance of the LNHB's RCTD1 counter and the TDCR-SU system are compared by primary measurements of ^{3}H, ^{14}C, ^{63}Ni and ^{55}Fe sources (see Table 3). The activities of the ^{3}H, ^{14}C and ^{63}Ni sources are calculated with the TDCR07c [3] program using the same cocktail data and kB values. The ^{55}Fe activities are calculated with the Fe55_6 code from LNHB. This code uses a KLM disintegration scheme for Fe-55 decay and a Monte Carlo method for the evaluation of uncertainties. Excellent agreement between the two systems is observed for the ^{3}H and ^{14}C sources within the estimated uncertainties and a very good agreement is found in the case of the ^{63}Ni source. The agreement for the ^{55}Fe source is not as good as in the previous tests, but the results are still compatible if expanded (k=2) uncertainties are considered. It is worth noticing that the uncertainty of the TDCR-SU measurement is too large to allow an unambiguous comparison.

Table 3. Comparison between the LNHB's RCTD1 counter and the TDCR-SU system

Source	**Activity measured by RCTD1 , Bq**	**Meas #**	**Activity measured by TDCR-SU, Bq**	$\Delta=\frac{A_{RCTD1}-A_{TDCR-SU}}{A_{RCTD1}}100$, **%**
H-3 in toluene-based LSC	1010.7±2.1 (0.21%)	1	1004.9 ± 5.0 (0.50%)	0.57
		2	1010.9 ± 5.1 (0.50%)	-0.02
C-14 in toluene-based LSC	1728.2± 2.4 (0.14%)	1	1738.2 ± 4.7 (0.27%)	-0.58
		2	1731.3 ± 4.7 (0.27%)	-0.18
Ni-63 in Ultima Gold LSC	1325.9± 4.1 (0.31%)	1	1313.9 ± 4.9 (0.37%)	0.91
Fe-55 in Ultima Gold AB LSC	1493.0±7.4 (0.5%)	2	1369 ± 64 (4.7%)	8.3

The first tests of the TDCR-SU are promising and show that it could be an effective TDCR counting system which can be used for primary standardization of pure-beta radionuclides. Further developments on the potential uses of the nanoTDCR counting module and on the application of the system for the absolute measurements of ^{222}Rn dissolved in liquid scintillation cocktail are also discussed in the paper.

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New pyrazoline fluorophores for more efficient organic scintillators

Valery N. Bliznyuk[1], Ayman Seliman[1], Alexander A. Ishchenko[2], Nadezhda A. Derevyanko[2], Timothy A. DeVol[1]

1) Department of Environmental Engineering and Earth Science, Clemson University, Clemson, SC 29634-0919 USA

2) Institute of Organic Chemistry, National Academy of Science of Ukraine, 5 Murmanskaya Str., Kiev 02660, Ukraine

Corresponding author's e-mail: vblizny@clemson.edu

The synthesis, spectroscopic and scintillation properties of three new pyrazoline core based fluorophores are presented. Various optical and physical properties of the fluorophores dissolved in organic solvents as well as solid polyvinyltoluene (PVT) resin matrix of different porosities were studied. These properties include but not limited to molar absorptivity, absorption and emission wavelengths as well as quantum yield (QY) relative to a reference material. In solution these fluorophores exhibited ~10% higher photoluminescence relative to existing commercially used fluorophores (e.g. 9,10-diphenylanthracene). When the synthesized fluorophore compounds were dissolved in PVT matrices, they showed at least three-times higher photoluminescence and radioluminescence compared to existing analogs at the same concentration. The effect of luminosity enhancement allows for significant reduction in fluorophore concentration in plastic scintillator materials without jeopardizing their scintillation properties. In addition, the applied synthetic route allows for functionalization of the fluorophores with a vinyl group for further covalent bounding to the PVT chains, which dramatically improves chemical stability of the system simultaneously improving the photoluminescence quantum yield. Possible mechanisms of the enhanced scintillation properties are discussed along with additional spectroscopic characteristics of the fluorophores.

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Effect of sodium salicylate on the determination of Pb-210/Bi-210 and Ra-228/Ac-228 by Cerenkov counting

Yadong Wang, Lijuan Song, Xiongxin Dai

China Institute for Radiation Protection, Street Xuefu 102, Taiyuan, Shanxi 030006, China

**Corresponding author's e-mail: wangyadong24@163.com*

Abstract:

High energy electrons produced from beta decay can create Cerenkov light when moving in a dielectric and transparent medium. Since Cerenkov counting acts as natural discrimination for alpha particles and low-energy beta particles, some potential radioactive impurities or progenies would not interfere with the measurement. There are many applications of Cerenkov counting technique in the determination of high energy beta-emitting radionuclides, such as ^{32}P, ^{89}Sr, ^{90}Sr/^{90}Y, ^{228}Ra/^{228}Ac, ^{210}Pb/^{210}Bi. In this work, we are developing a Cerenkov counting technique for the determination of ^{210}Pb from ^{210}Bi and ^{228}Ra from ^{228}Ac at equilibrium using a Hidex 300 SL TDCR liquid scintillation counter.

Due to the nature of Cerenkov radiation and instrumental limitations, detection efficiencies of ^{210}Bi and ^{228}Ac by Cerenkov counting are generally quite low (~20%). Water-soluble wavelength shifting compounds, such as sodium salicylate, have been used to improve the detection efficiency of Cerenkov photons. In this study, we found that the addition of sodium salicylate could significantly increase the counting efficiencies of ^{210}Pb/^{210}Bi and ^{228}Ra/^{228}Ac. Furthermore, a sharp increase of the detection efficiency for the alphas from ^{210}Po was observed with the addition of sodium salicylate, implying that scintillation light rather than Cerenkov photons from the alphas has been produced in the presence of sufficiently high concentration of sodium salicylate. More details about the effects of sodium salicylate on the counting of ^{210}Pb/^{210}Bi and ^{228}Ra/^{228}Ac will be presented.

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Water-based and Metal-doped Liquid Scintillator for Neutrino Physics

Minfang Yeh

Brookhaven National Laboratory, Upton, NY11973, USA
yeh@bnl.gov

The pure liquid scintillator detector is known to have high light yield and long attenuation length with extended chemical stability in many years of operation for low-energy neutrino detection. However its high cost, less material compatibility, and specific liquid handling often raise special concerns, particularly the chemical safety in the confined space of underground laboratory. The water-based liquid scintillator (WbLS) is of great interest from scientific community for multi-disciplinary application, in paticular the large-scale physics experiments, such as nucleon decay, geo-neutrino, solar-neutrino, etc., due to its enhancements in chemical safety, metal-loading ability, cost-efficiency and unique capability of Cherenkov and scintillation detections. The principal of WbLS has been demonstrated at bench-top scale with a variety of organic scintillators doped with different metallic isotopes in water. To make feasible WbLS of many tens of kiloton as the target mass for future neutrino detectors, extensive studies in chemical stability, mass-producible manufacture, and online circulation are ongoing. In this talk, the preparation, performance, and technical challenge of WbLS will be presented.

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Boron-10 and Lithium-6 Loaded Scintillator for Neutron Detection

Xiongxin Dai

China Institute for Radiation Protection, 102 Xuefu Street, Taiyuan, China

* *Corresponding author's e-mail: daixx@yahoo.com*

Abstract:

Neutron detection has broad applications in many fields for various purposes. For instance, in nuclear instrumentation, monitoring neutron flux is critical for proper operation of a nuclear reactor; in homeland security and defence, neutron detection can be used to trace illicit special nuclear materials (SNM); for radiation safety, neutron detectors and neutron dosimeters are used for monitoring hazardous radiation associated with neutron sources, space travel, accelerators and nuclear reactors etc; neutron imaging has also applied in material science research to characterize the morphology of materials. Particularly, there is a need for detection of low flux of thermal neutrons in an environment with a relatively high background of gamma rays. Helium-3 based neutron detectors have been extensively used for this purpose because of their good neutron-to-gamma discrimination capability. However, due to a global shortage of He-3, it is necessary to develop an alternative neutron detecting material for He-3 replacement.

Both boron-10 and lithium-6 have large cross sections for thermal neutron capture. The $^{10}B(n,\alpha)^{7}Li$ reaction has a Q-value energy of 2.79 MeV and produces either a 1.78 MeV alpha (6.3% branching ratio) or a 1.47 MeV alpha with a 0.477 MeV gamma (93.7% branching ratio); while the $^{6}Li(n,\alpha)^{3}H$ reaction has a Q-value energy of 4.78 MeV and produces a 2.05 MeV alpha and a 2.73 MeV triton. Loading an organic scintilators with B-10 and Li-6 can be achieved by dissolving a boron/lithium compound directly into the scintillation solvent or by incorporating a medium containing a B/Li compound into the scintillator, where the medium does not dissolve into the solvent. Since the charged particles produced from the neutron capture reactions of B-10/Li-6 completely deposit their energy within the scintillating medium, their signals can be well separated from gamma background using pulse-shape-discrimination techniques. To ensure high performance and applicability of the scintillator, it needs to have good stability, high light yield, good optical transparency, low toxicity and high flash point. In this work, new linear alkylbenzene (LAB) based scintillators, loaded with boron or lithium, have been developed for neutron detection. More details about development and performance of the scintillators will be presented.

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Phase stability and lithium loading capacity in a liquid scintillation cocktail

Denis E. Bergeron [1], H.P. Mumm [1], M. Tyra [1]

[1] Physical Measurement Laboratory, National Institute of Standards and Technology, 100 Bureau Dr., Gaithersburg, MD 20899, USA
Corresponding author's e-mail: denis.bergeron@nist.gov

Antineutrinos can be detected in a liquid scintillation (LS) scheme via the inverse beta decay mechanism:

$$\bar{\nu}_e + p \rightarrow e^+ + n$$

where $\bar{\nu}_e$ represents the antineutrino, p is a proton, e^+ is a positron, and n is a neutron. An organic scintillant provides the proton-rich target for good antineutrino detection efficiency and discrimination. Dopants with high neutron cross-section such as Gd or ^{6}Li can further improve the performance of LS cocktails. By now, many formulations have been reported combining good detection efficiency with desirable optical properties and pulse-shape discrimination to reduce backgrounds from gamma-rays and neutrons [see, e.g., 1-3].

As the next generation of neutrino detectors is being designed to probe outstanding questions surrounding neutrino oscillation and reactor spectra, there is a need for scintillants that can be easily and inexpensively prepared in very large quantities (thousands of liters) for use in large detectors that will operate for years [2-3]. We have conducted an investigation of some off-the-shelf scintillants, seeking to achieve maximum loading with lithium while preserving good optical and pulse-shape discrimination properties.

Loading an organic scintillant with large quantities of aqueous material requires a good surfactant mix, and the commercially available scintillants that utilize alkylphenol ethoxylates promise high loading with good optical transmission and high scintillation efficiency. These were the focus of our studies.

Here, we present the results of a series of experiments designed to distinguish microemulsions (or, synonymously, reverse-micellar solutions) from emulsions. For high energy physics applications demanding long-term scintillant stability, the gradual phase separation characteristic of emulsions is unacceptable. Microemulsions are isotropic mixtures that exhibit long-term phase stability, so that lithium trapped in nano-sized reverse micelles should remain suspended uniformly in the scintillant over the long timescales required.

Using optical and Compton spectrum quenching techniques [4], we attempted to optimize the transmission of optical scintillation photons while guaranteeing uniform lithium distribution by working in the microemulsion phase. We prepared samples with a wide range of lithium chloride additions and monitored quench indicating parameters (QIPs) over time. We demonstrated that phase separation was easily detected with QIP monitoring. We used centrifugation to effectively accelerate phase separation of emulsions, identifying compositions that might appear acceptable at first, but that would be prone to instability over a long-term experiment (Figure 1). Thus, we arrived at a recipe for a cocktail with optimal—in terms of detection efficiency and long-term stability—lithium loading.

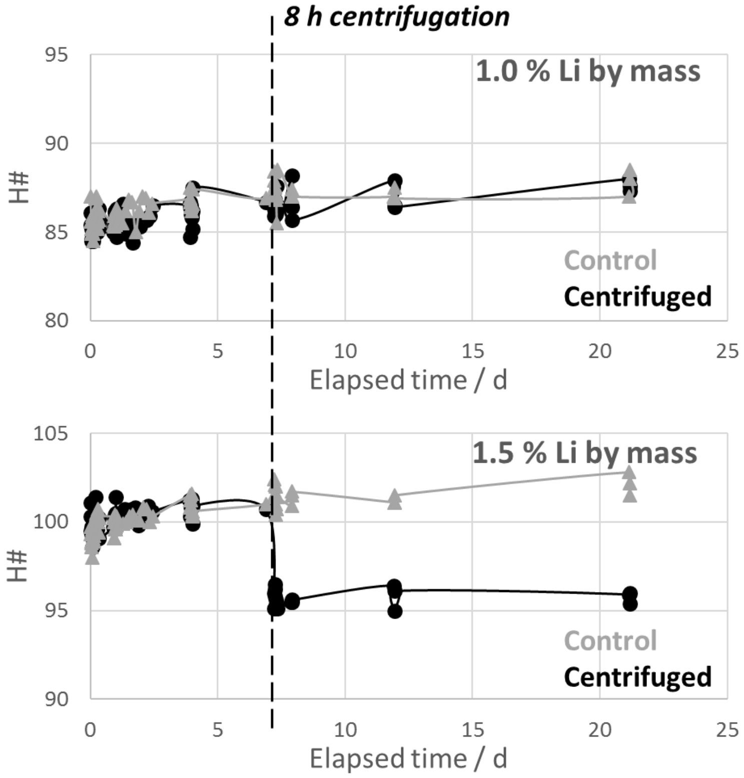

Fig. 1 QIP (Horrock's number, H#) measurements contrasting the stable cocktail with 1.0 % by mass lithium with the emulsion containing 1.5 % lithium. The emulsion undergoes phase separation upon centrifugation, while the microemulsion does not.

We continue to investigate and characterize the properties of the cocktail formulation, with ongoing dynamic light scattering, pulse-shape discrimination and transmission measurements, and efficiency studies. The cocktail has also been used in a recently commissioned neutron spectrometer at NIST.

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The Deuterium-Deuterium neutron time-of-flight spectrometer at EAST tokamak

T. Fan1), L. Ge1), X. Peng1), Z. Hu1), T. Du1), Y. Zhang1), J. Sun1), Z. Chen1), X. Li1), G. Zhong2), G. Gorini3), J. Kallne4)

1) Institute for Heavy Ion Physics, School of Physics, Peking University, Beijing, 100871, China
2) Institute of Plasma Physics, CAS, Hefei, China
3) TIFP-CNR and Department of Physics, University of Milano-Bicocca, Milano, 20126, Italy
4) Department of Engineering Sciences, Uppsala University, Uppsala, 75015, Sweden

Corresponding author's e-mail: tsfan@pku.edu.cn

Some developments on neutron measurement techniques are required to meet diagnostic challenges, including very high levels of neutron and gamma fluxes, nuclear heating, and harsh environmental conditions, in future fusion devices or reactors, such as JT60-SA, CFETR, ITER and DEMO.

The prototype neutron time-of-flight enhanced diagnostics (TOFED) spectrometer has been developed for deuterium plasma operation in EAST in order to provide the essential information on ion temperature, fuel ion composition and the neutron yield rate of the plasma related to fusion power and also understand the behavior of fast ions of external auxiliary power. The TOFED design, where the ring of second plastic scintillators (S2) is split into two spherical zones, is shown to enhance the discrimination capability and will provide fusion neutron spectra with reduced admixture of multiple scattering events, which is essential for increasing the sensitivity to weak components in the neutron emission. The prototype TOFED spectrometer consists of 32 S2 plastic scintillators and a five-layered detector (S1) assembly coupled to photomultiplier tubes. A new fully digital data acquisition system with on-board CFD timing function has been adopted and can provide a time resolution <500 ps, compatible with high count rate capability up to about 1 MHz/channel of the prototype TOFED spectrometer. TOFED is expected to have high energy resolution of 6.6% for deuterium-deuterium neutrons.

On the other hand, the preliminary implications of measuring the neutron emission on EAST together with the experimental challenges are presented, and limitation of this system and ways of further improvement are suggested.

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Liquid Scintillator Neutron Detection System for Fast-ignition

Hongjie Liu [1,2], Yuqiu Gu[1,2], Bo Cui[1], Zenghai Dai[1], Leifeng Cao[1,2]

1) Research Center of Laser Fusion, CAEP, No.64 Mianshan Road, Mianyang, 612900, China
2) Science and technology on plasma physics laboratory, No.64 Mianshan Road Mianyang, 621900, China

Corresponding author's e-mail: buyijie@163.com

Scintillator detectors in inertial confinement fusion experiments are predominantly used to measure neutron yield and ion temperature of the primary fusion reactions. The detection of neutrons in fast-ignition experiments is very challenging since it requires the neutron detection system to recover within 100 ns from a high background orders of magnitude stronger than the signal of interest. Liquid scintillator with different compositions was investigated. We present several designs of liquid scintillator using the Geant4 Code and the X-Lab Code. Our liquid scintillator is based on PPO, dissolved in xylene and enriched with molecular O_2. The detector consists of a 2-3 liters volume of liquid scintillator coupled to a gated MCP. A small air bubble allows for thermal expansion of the liquid without a significant pressure increase. The gating performance under high-intensity γ rays was experimentally checked. The typical flight time spectrum of the neutrons from (p,n) reaction driven a PW laser is shown in Fig.1. The neutron yield in the fast ignition experiments on Shenguang-II laser facility was successfully measured using this detector. In fast ignition experiments, a PW laser beam was employed to produce high intense electron beams to ignite the compressed fuel. Our neutron detection system could suppress the background signal and eliminate the afterglow present in conventional plastic scintillators.

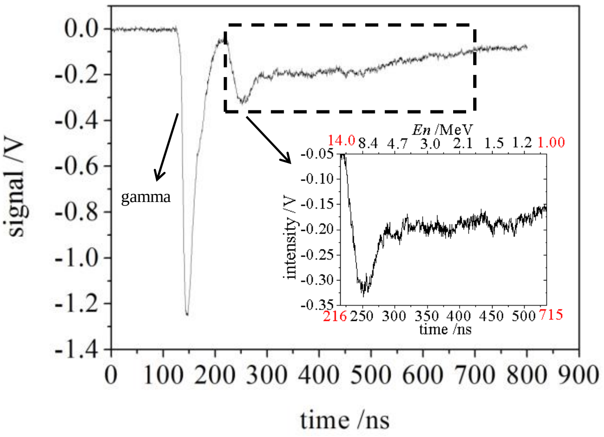

Fig. 1 Neutron flight time spectrum obtained in PW laser

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Synthesis and Characterization of Organic Tellurium Compounds for SNO+ Liquid Scintillator

Lianpeng Tian

(For the SNO+ Collaboration)

Queen's University, Department of Physics, Engineering Physics & Astronomy,

Kingston, ON K7L 3N6, Canada

[*] *Corresponding author's e-mail: lt63@queensu.ca*

Abstract:

The SNO+ experiment is a multi-purpose neutrino experiment. The primary objective of SNO+ is to search for the neutrinoless double-beta decay of ^{130}Te. In Phase I, the detector will be filled with 0.5% natural tellurium in 780 tonnes of liquid scintillator, corresponding to approximately 1330 kg of ^{130}Te. In order for the SNO+ neutrinoless double beta decay search to be successful, a technique is needed to dissolve purified tellurium in linear alkyl benzene (LAB) scintillator. The technique should result in a liquid scintillation cocktail that is stable, has good light yield and optical transmission properties, and maintains the expected low levels of intrinsic radioactive background.

The SNO+ team is developing a telluric acid diolization process to synthesize organic tellurium compounds that can be dissolved in LAB and loaded into acrylic vessel of the detector. The raw materials used for this so-called TeBD synthesis process are telluric acid (TeA), ultra-pure water (UPW), and 1, 2-butanediol (BD). The main objective of this work is to investigate optimal synthesis parameters (e.g., initial TeA:BD ratio and reaction temperature) for the production of LAB-soluble organic tellurim complexes with good quality. For characterization of the organometallic tellurium complexes, the produced TeBD mixture was analyzed by mass spectrometry (MS), NMR spectroscopy, X-ray fluorescence (XRF), ultraviolet-visible absorption spectroscopy (UV-Vis), and Karl Fischer Titration. The synthesis reaction mechanism and the TeBD structures will be discussed. Scaleup efforts of tellurim loading at different process development steps will also be briefly covered.

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A Fast-neutron Coincidence Collar Using Liquid Scintillators for Fresh Fuel Verification

Jonathan S. Beaumont [1)], Tae Hoon Lee [1)], Mikhail Mayorov [1)], Carlo Tintori [2)], Francesco Rogo [2)], Bruno Angelucci [2)], Matteo Corbo [2)]

[1)] International Atomic Energy Agency, Vienna International Centre, PO Box 100, 1400, Vienna, Austria

[2)] CAEN S.p.A, Via Vetraia, 11 Viareggio LU, 55049, Italy

Corresponding author's e-mail: j.beaumont@iaea.org

The fast neutron collar (FNCL) is a safeguards instrument developed by the IAEA for non-destructive assay of fresh fuel assemblies (FFAs) containing low-enriched uranium. The FNCL employs active neutron coincidence counting (NCC) to measure an induced fission rate in FFAs, and from this calculate the ^{235}U content. The FNCL has been designed for modern FFAs containing burnable neutron poisons such as gadolinium, commonly included in fuel to allow reactors to operate for longer periods without refuelling. Such fuels present complications for verification by traditional NCC instruments using moderated ^{3}He thermal neutron detectors, such as the uranium neutron collar (UNCL), due to high absorption of thermal neutrons by burnable neutron poisons.

Until recently, this problem has been solved through two main approaches. The first was to apply a correction factor to the measurement which relied on a declaration of gadolinium content in the FFA. This could not be independently verified and therefore presented the possibility that nuclear materials could be diverted. The second was to use a cadmium lining within the instrument to remove thermal neutrons from the interrogation flux, thus significantly reducing the dependence of the measurement on gadolinium poisons. This also led to suppression of the induced fission rate and, to compensate, the measurement times had to be extended to between two and three hours.

The FNCL offers an alternative solution to the above problem by utilising low-hazard EJ-309 liquid scintillation detectors and an integrated data acquisition system (CAEN S.p.A) to measure fast coincident neutrons above a threshold of 0.5 MeV. This leads to a minimal dependence on gadolinium poisons, removing the need for a correction. Additionally, no neutron thermalisation is required for detection, thereby allowing the coincidence gate to be reduced from microseconds to nanoseconds. This virtually eliminates accidental neutron counts, the major source of measurement uncertainty in traditional NCC. Subsequently, the measurement times can be greatly reduced. The final stages of FNCL development and field test measurements of FFAs with a variety of ^{235}U enrichments and gadolinium content are presented.

Next-Generation, Micron-order Resolution Neutron Imaging with Gel Scintillator Filled Glass Capillary Arrays

Xiaodong Zhang 1), Xiongxin Dai 2), Chengliang Wan 1), Hao Qiao 1)

1) School of Nuclear Science and Technology, Lanzhou University, Lanzhou, Gansu 73000, China
2) China Institute of Radiation Protection, Taiyuan, Shanxi 030006, China

Neutron imaging devices with a spacial resolution in the range of microns are of great interests in the application of advanced material research, such as the characterizations of the lithium-ion diffusion profile in the electrode of lithium batteries, the voids and cracks in spent nuclear fuels, and the deuterium-tritium (DT) reaction profile of DT filled capsules in inertial confinement fusion experiments. But the spatial resolution of the state-of-the-art devices with thermal and cold neutrons is around 20 microns and the resolution of these with fast neutrons is even worse and in the order of hundred microns. That is due to the combined projected-ranges of secondary particles produced by the neutron absorption reactions for thermal and cold neutrons and the projected-range of recoiled protons by fast neutrons. New neutron imaging devices made of a lithium-6 doped gel scintillator (for thermal and cold neutron detection) or gel scintillator (for fast neutron detection) filled micron-order glass capillary array and a cooled low readout noise (1.8 e^-), lowest light sensitivity (2.23 photons), high quantum efficiency (70%@600nm) scientific CMOS camera (BIC-C40D) are under developing in our research groups. Simulations expect that the spatial resolution for the thermal and cold neutron imaging system is about one micron and about less than 10 microns for the fast neutron imaging one. The experimental characterizations of these devices will be done at the beginning of the coming year in the Institute of Nuclear Physics and Chemistry and the Center of Laser-driven Nuclear Fusion, China Academy of Engineering Physics, respectively. Both simulations and experimental results will be presented and discussed in this conference.

IAEA's ALMERA Effort Towards Harmonization of Radioanalytical Procedures: Development and Validation of a Rapid Procedure for Simultaneous Determination of ^{89}Sr and ^{90}Sr in Soil Samples Using Cerenkov and Liquid Scintillation Counting

A. Pitois

International Atomic Energy Agency, IAEA Environment Laboratories, Seibersdorf, Austria

Corresponding author's e-mail: a.pitois@iaea.org

The International Atomic Energy Agency (IAEA) coordinates and provides analytical support to the worldwide network of Analytical Laboratories for the Measurement of Environmental Radioactivity (ALMERA). The goal of ALMERA is to provide timely and reliable measurement results of environmental radioactivity in routine monitoring and emergency situations. Finding and choosing the most adequate analytical procedures for environmental monitoring can be a challenge for laboratories due to a wide variety of technologies available and the rapid developments in this field. To respond to this challenge, the IAEA has included within its ALMERA activities, the development and validation of a set of procedures for the determination of natural and anthropogenic radionuclides in environmental samples, both for routine [1-3] and emergency [4-6] environmental monitoring. Such tested and validated analytical procedures are essential tools for the production of reliable and comparable environmental radioactivity measurements in routine and emergency situations, and are contributing to methodological harmonization.

Partly prompted by the interest for high throughput procedures following the analytical burden of laboratories after the Fukushima Dai-Ichi nuclear power plant accident, a rapid procedure for the simultaneous determination of ^{89}Sr and ^{90}Sr in soil samples was developed and validated within the ALMERA network. The development and validation of the procedure relied on a collaborative effort of network laboratories and followed a thorough methodology [7] involving literature survey, selection, testing and validation.

An expert group consisting of 4 ALMERA member laboratories having an extensive and well-recognized expertise in radiostrontium analysis and of the IAEA Environment Laboratories was set up to develop the procedure. The suitability of the procedure for a wide range of ^{89}Sr/^{90}Sr ratios, its rapidity, the efficiency of the chemical separation and its potential use in a wide range of laboratories were considered as the primary requirements for the development of the procedure. For flexibility of use in laboratories worldwide, all sample digestion techniques, i.e. fusion, microwave oven digestion, leaching and full digestion on hot plates using a mixture of acids, were considered. However, the fusion technique, if available, is generally preferred in emergency situations for time savings. The procedure is based on the chemical separation of radiostrontium from interfering elements using co-precipitation and extraction chromatography steps followed by sequential counting in Cerenkov and liquid scintillation counting modes. The procedure can be carried out within 12 hours when using the fusion technique to digest the sample. The efficiency of the radiochemical separation is 70-80%. Typical measurement spectra of ^{89}Sr and ^{90}Sr in Cerenkov and liquid scintillation counting modes are shown in Fig. 1.

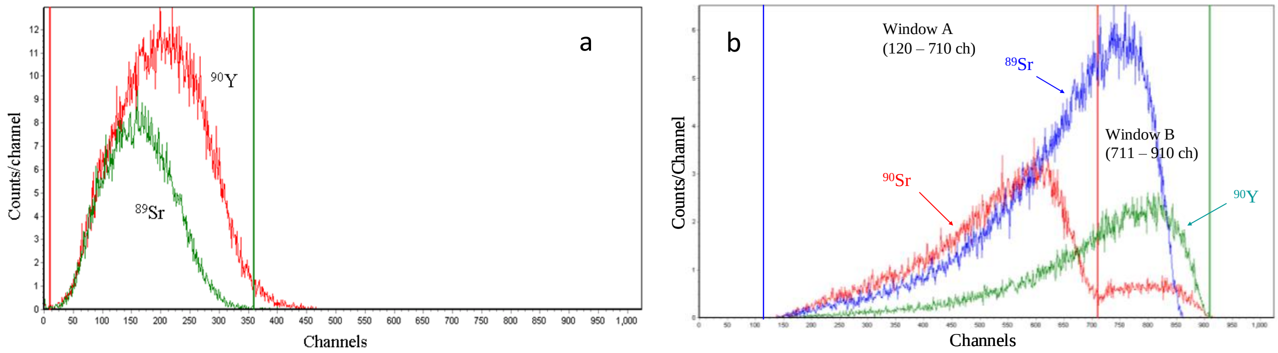

Fig. 1 Typical measurement spectra of ^{89}Sr and ^{90}Sr in a) Cerenkov mode; b) Liquid Scintillation Counting mode

The procedure was tested and validated in terms of repeatability, reproducibility and trueness (relative bias) in accordance with ISO guidelines. Ten laboratories from the ALMERA network participated in the validation procedure by performing reproducibility tests. For this purpose, five validation samples with properly selected properties, i.e. soil sample with three different ^{89}Sr/^{90}Sr ratios, i.e. 5, 10 and 20, with lower ^{89}Sr and ^{90}Sr massic activities, i.e. hundreds Bq/kg range, and with heat treatment to promote the presence of embedded radiostrontium, were prepared at the IAEA Environment Laboratories and shipped to the participating laboratories in order to check the robustness of the procedure at different ^{89}Sr/^{90}Sr ratios, its sensitivity and to test the sample digestion techniques.

In total, 230 measurement data were obtained in this comprehensive validation campaign, ensuring robustness and confidence in the measurement results. Outlier tests, i.e. statistical tests on distribution and tests on relative bias, detected 7% outlier data, which were removed from further analysis. An overall good agreement of the individually reported measurement results with the target values was observed for all investigated ^{89}Sr/^{90}Sr ratios and the ^{89}Sr/^{90}Sr ratios were well estimated, demonstrating the robustness of the procedure. Good agreement between measured and target values was also obtained at the lower massic activities range, demonstrating the sensitivity of the procedure. The limitations in terms of digestion techniques were assessed. The fusion, microwave oven and full digestion techniques provided a good agreement between measured and target values/ratios for all validation samples. The use of the leaching technique led to an underestimation of approx. 10%, which could still be acceptable in emergency response. Those results are illustrated in Fig. 2 for a ^{89}Sr/^{90}Sr ratio of approx. 5. However, when the presence of embedded radiostrontium is promoted by heat treatment of the sample, leaching led to an underestimation of more than 25%, making this digestion technique not fit for the purpose. The ^{89}Sr/^{90}Sr ratio remain however well estimated.

In conclusion, a simple and rapid analytical procedure for direct and simultaneous determination of ^{89}Sr and ^{90}Sr in soil samples was developed and validated in this study. The procedure can be used in a large number of laboratories worldwide for the rapid screening of soil samples contaminated by elevated levels of radiostrontium in emergency situations. Such tested and validated analytical procedure applicable worldwide for radiological emergencies contributes to methodological harmonization, leading to enhanced comparability of environmental radioactivity measurement results in emergency situations.

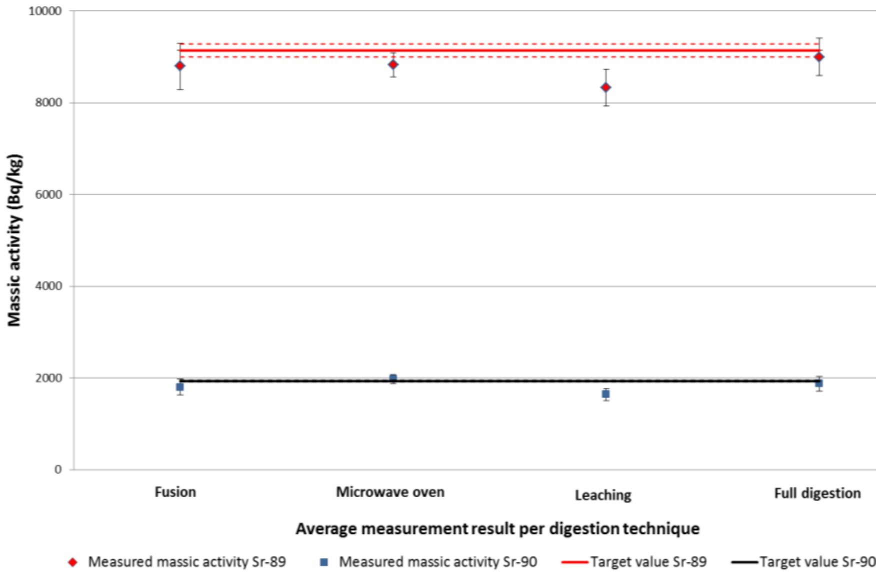

Fig. 2 Measured ^{89}Sr and ^{90}Sr massic activities as a function of the sample digestion technique for a validation soil sample of ^{89}Sr/^{90}Sr ratio ~5 (no heat treatment applied to the validation sample)

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Determination of radiostrontium in seawater using automated separation system at routine and emergency situation

Hyuncheol Kim, Yoonhee Jung, Kun Ho Chung, Jong Myung Lim, Mun Ja Kang

Environmental Radioactivity Assessment Team, Korea Atomic Energy Research Institute, Korea,
111 Daedeok-daero 989 beon-gil, Yuseong,gu, Daejeon, 34057, Korea

Corresponding author's e-mail: hckim3@kaeri.re.kr

Radiostrontium is an important radionuclide with environmental radioactivity monitoring aspects due to its high fission product and similar chemical behavior with Ca. National authorities have monitored ^{90}Sr in seawater for national public health safety and many laboratories have done it. Global fallout level of ^{90}Sr in seawater is very low, ~ 1 mBq kg^{-1}, so the analysis of it needs a large amount of seawater, It is time consuming and labor intensive work. For emergency situation such as nuclear accident, rapid analysis of ^{90}Sr in seawater is needed for public health safety. In this case, the amount of seawater can be reduced by 10 times lower than that of routine analysis, due to relative higher level of ^{90}Sr than normal case. However, a lot of samples should be analyzed for reliable decision making. It is time consuming and labor intensive work, also.

We have developed the fast analytical method for ^{90}Sr in seawater using automated separation system, which are two systems. One is called ACE-LAS (Automated ConcEntration for Large Amount of Sample) and applicable for routine analysis. The other is called ASRS (Automated Sequential Radionuclides Separator) and is applicable for emergency situation. Its main principle is extraction chromatography, such as cation exchange resin, DGA, and Sr resin. Two automated systems basically share the operating software and same mechanical characteristics. Only difference of them is the size, which can treat a large amount of sample or not.

For routine analysis, ^{90}Sr in seawater is concentrated through cation exchange resin, and then extracted it from the column. And then, it is waited for 3 weeks until being equilibrium with ^{90}Y. ^{90}Y is separated from sample using DGA resin and analyzed by LSC. It can treat 8 samples at the same time, and about 6 L of seawater can be applied for each column. For emergency situation, about 100 ml of seawater is passed through cation exchange column and, extracted ^{90}Sr from the column using 8 M HNO_3. Then, it flows sequentially into Sr resin column, then ^{90}Sr is extracted. Above operations are controlled by computer, so it can save the analytical time and labor.

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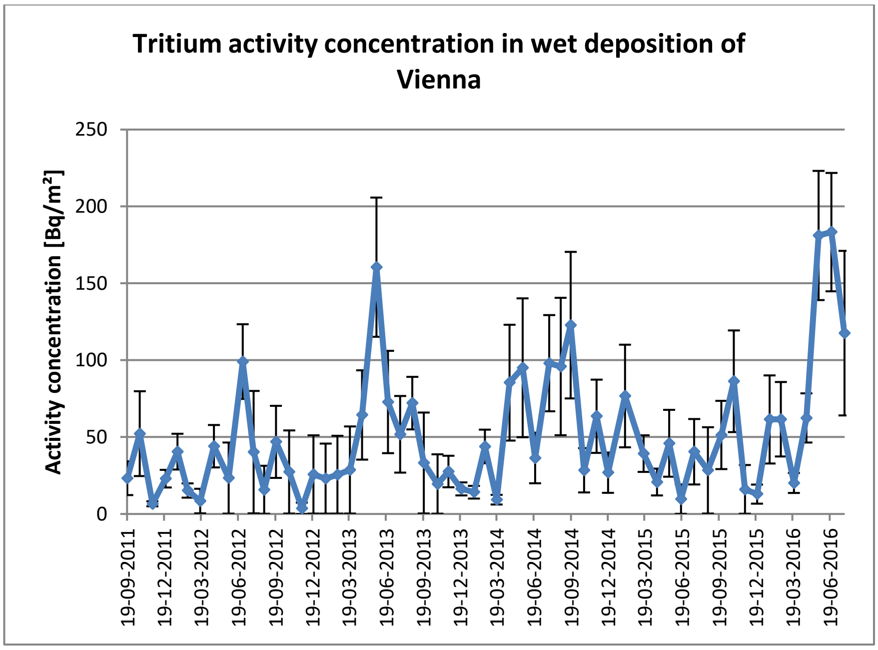
Tritium and Gross Alpha and Beta Measurements performed for the Official Radioactivity Monitoring in Austria

Claudia Landstetter, Bernd Hiegesberger, Christian Katzlberger

Austrian Agency for Health and Food Safety Ltd., Spargelfeldstrasse 191, 1220 Vienna, Austria

Corresponding author's e-mail: claudia.landstetter@ages.at

The main task of the Division for Radiation Protection is to provide professional support for the Ministry of Health and Women's Affairs and the Ministry of Agriculture, Forestry, Environment and Water Management to ensure public protection from radioactivity. To do this, the Division carries out a variety of projects in fields such as emergency planning and food and water monitoring. The official radioactivity monitoring in Austria (environment media and foods) is carried out according to the EURATOM Treaty, Austrian Radiation Protection Act (StrSchG) and according to bilateral, as well as multilateral agreements. Among many other radionuclide determinations Tritium measurements are conducted in e.g. wet deposition samples, surface water samples and drinking water samples. Additionally gross (α+β) measurements are conducted in surface water samples and drinking water samples. Figure 1 shows the activity concentration of Tritium in wet deposition of Vienna from September 2011 till now. As the activity concentration is given in Bq/m³ the values correspond to the amount of rain during these sample periods. The sample period is one month.



In wet deposition samples and in surface water samples a decrease of the Tritium activity concentration over the years can be seen. During the occurrence of floodwaters resuspension of sediments can lead to higher K-40 concentrations. Therefore the results of gross (α+β) measurements in surface water samples can be increased. These measurements are performed using the liquid scintillation counter Quantulus 1220. This presentation shows the measurement methods, the collected data of the last 5 years and difficulties encountered because of e.g. various cocktail compositions and ventilation conditions.

Rapid analysis of ^{90}Sr and ^{99}Tc in low active effluent using extraction disks: the advantages of the use of liquid scintillation counting

Braekers D. 1), Doumont C. 1), Di Pasquale S. 1), Deconninck B.1)

1) Institute for radioelements IRE Elit, 1 avenue de l'espérance, B-6220 Fleurus, Belgium

Corresponding author's e-mail: damien.braekers@ire-elit.eu

Long lives pure beta nuclides such as ^{90}Sr and ^{99}Tc are important isotopes in radiological monitoring programs due to their potential long term effects on the environment. One of the main potential releases in the environment is the liquid effluents discharged from nuclear waste reprocessing plants. In this framework, the use of solid phase extraction disks (e.g. technetium and strontium 3M Empore^{TM} rad disks) for the rapid determination of ^{90}Sr or ^{99}Tc in the water samples has proved its effectiveness (Ref 1).

However, the use of these extractions membranes for such complex matrix like effluent samples requires particular attention especially regarding the possible unusual interferences. They could have a strong impact on the chemical yield of the extraction process or on the measurement itself if it's a radioactive interference. In the present work, we show two examples of theses interferences and how liquid scintillation counting (LSC) is effective to work around .

The Tc-99 extraction disks are known to be sensitive to other radionuclides such as Ru-103, Ru-106 or Pu-241 but there are limited information about the interference of Cl-36 on Tc-99 measurement (Ref. 2). By using the LSC, this interference has been detected in the B1 sample from the NPL 2012 proficiency test. The impact of the Cl-36 isotope with the Tc-99 result can be reduced by adjusting the experimental extraction conditions.

Secondly, the extraction yield of strontium is strongly dependent with the chemical composition of the sample. It's usually recommended to determine the chemical yield for each isotope especially if the nature of the sample is complex and changing over the time. In this case the ^{85}Sr isotope can be used as an internal tracer for the chemical yield determination. The detection is again improved by the use of LSC as the beta counting windows can be adjusted simultaneously on the ^{85}Sr low energy emission for the chemical yield determination and on ^{90}Y emission for the ^{90}Sr activity determination (see Fig. 1).

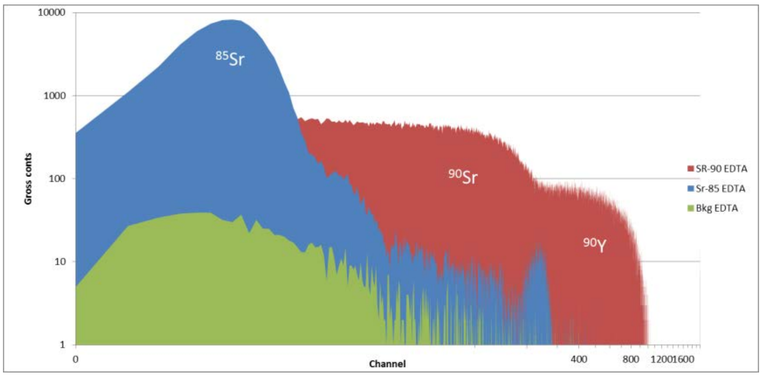


Fig. 1 The LSC spectra of ^{85}Sr and ^{90}Sr

In conclusion, the liquid scintillation counting allows easily the detection of interference's isotopes in the analysis of ^{99}Tc in liquid effluents by extraction using Technetium Empore^{TM} Rad disk. For the rapid analysis of ^{90}Sr using strontium extraction disks, the use of an internal ^{85}Sr tracer combined with LSC is very effective for the chemical yield determination of the strontium extraction process.

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Gross Alpha – Beta Measurements of Water Samples from Wonderfonteinspruit Catchment Area in the Gauteng Province South Africa using LSC

Mashaba, M. 1), Kotze, D. 2), Tshivhase, V.M. 1), Faanhof, A. 1, 2)

[1] Northwest University, Centre of Applied Radiation Science & Technology (CARST), Mafikeng, South Africa.

[2] The South African Nuclear Energy Corporation SOC Limited, Pretoria, South Africa.

Corresponding Author's email: mashabamac@gmail.com

Liquid scintillation counting (LSC) offers an attractive method for fast screening of water samples contaminated with NORMs. The determination of gross α-β emitters with the LSC method requires an accurate PSA setting under various quenching conditions. The appearance of quenching in the sample affects not only counting efficiency but the α-β discrimination as well. The aim of the research is to evaluate the potential of a low-background liquid scintillation system with advanced spectrometry capabilities, the Quantulus 1220^{TM}, to be used directly for the determination of the gross α- and β-activities in environmental water collected from the Wonderfontein catchment area (WCA) without extensive sample preparation. The results were verified by element specific separations (U, Ra, Pb/Po and Th) followed by α-spectrometry at the radionalytical laboratories of the South African Nuclear Energy Corporation (Necsa). An acceptable correlation was obtained between LSC and α-spectrometry. The method has demonstrated that LSC is able to produce reliable result for measuring α/β-activity in water samples. The method developed offers an attractive and cost-effective alternative.

Tuesday

2 May 2017

1. **Session: LSC in Nuclear chemistry**
2. **Session: Radionuclides metrology using LSC, standardization and quality assurance-1**
3. **Session: New development on LSC instrumentation methodology, scintillator and spectrum analysis-2**
4. **Poster session-1**
5. **PerkinElmer Workshop: Advances in Ultra low level detection and alpha beta separation**

Rapid on-site screening of aqueous waste streams using dip stick technology & liquid scintillation counting

Phillip E. Warwick and Ian W Croudace

GAU-Radioanalytical Laboratories, Ocean & Earth Science, University of Southampton, National Oceanography Centre, European Way, Southampton SO14 3ZH, United Kingdom

Screening of decommissioning wastes for non-gamma emitting radionuclides typically relies on time-consuming radiochemical analysis by expert laboratories remote from the decommissioning site. Such characterisation is usually targeted at specific radionuclides and involves chemical separation in purpose-designed analytical facilities prior to radiometric analysis. Additional time is required to arrange transport of potentially radioactive materials from the site which in turn requires some prior knowledge of the potential radionuclide levels present. There are significant advantages to developing a more rapid screening approach which can be deployed on site for the initial identification and quantification of radionuclides, permitting more time and cost-effective screening of wastes.

The project aims to develop a purpose-designed extraction / detection system that can be deployed by non-specialists either on-site or as part of a mobile laboratory. The system comprises three main components; (1) an optimised compact extraction system for recovery of radionuclides from the waste form; (2) an extraction test strip designed to recover the radionuclides from the waste digest; (3) a scintillation-based detection system capable of quantification of alpha, low energy beta and high energy beta emitting radionuclides.

Data are presented on the preliminary evaluation of the extraction / detection system for the measurement of ^{90}Sr in aqueous wastes. An assessment of system response, sensitivity, robustness and ease of deployment is presented along with a review of how the system can be developed for more widespread application.

Validation of radiochemical methods for the determination of difficult-to-measure nuclides using LSC

Nóra Vajda [1)], Zsuzsa Molnár[1)], Edit Bokori[1)], Szabolcs Osváth[2)], Dénes Párkányi[3)], Mihály Braun [4)]

[1)] RADANAL Ltd., Konkoly-Thege M. út 29-33, Budapest, 1121, Hungary
[2)] National Research Directorate for Radiobiology and Radiohygiene, National Public Health Center, Anna utca 5., Budapest, 1221, Hungary
[3)] Centre for Energy Research, Hungarian Academy of Science, Konkoly-Thege M. út 29-33., Budapest, 1121, Hungary
[4)] ISOTOPTECH Zrt., Bem tér 18/C, Debrecen, 4026, Hungary
Corresponding author's e-mail: vajdanor@gmail.com

A number of research programs have addressed development of analytical techniques for the determination of difficult-to-measure (DTM) radionuclides for the comprehensive characterization of radioactive wastes. DTM nuclides are of low-abundance, long half-life, they emit either α particles, or electrons (β, Auger) or X-rays that all suffer significant self-absorption. Liquid scintillation is an ideal tool to avoid absorption and assure high detection efficiency, but its use is limited by the poor energy resolution. The biggest challenge in the analyses is the identification of the small amounts of DTM nuclides and confirmation of the absence of interferences/impurities. In order to obtain correct results i) high requirements towards the selectivity of the separation have to be met, ii) isotopic interferences have to be taken into account, iii) the methods have to be carefully validated. For the latter purpose, various techniques such as measurement of reference materials, comparison with other methods, inter-laboratory comparisons, systematic assessment of the influencing factors, assessment based on scientific understanding of the principles can be used and have to be used according to ISO 17025[1].

RADANAL has been involved in the development of methods for the determination of a couple of β-decaying DTM nuclides, such as ^{90}Sr, ^{63}Ni, ^{93}Zr, ^{151}Sm. The methods are based on radiochemical separations followed by LS spectrometry. Extraction chromatography (EC) as a major tool is generally adequate to obtain high selectivity. The procedures for Sr, Ni, Zr, and Sm separations - developed at our laboratory - are based on EC using Sr resin, DMG resin, UTEVA resin and DGA resin[2], respectively in combination with further purification procedures. Chemical recoveries using properly selected tracers (e.g., ^{95}Zr, ^{140}La) or carriers (Sr, Ni, Zr, La) and decontamination factors for the major interferences have been determined.

Isotopic interferences in ^{90}Sr, ^{63}Ni, ^{93}Zr, ^{151}Sm determination (^{89}Sr, ^{91}Sr, ^{92}Sr, ^{59}Ni, ^{95}Zr, ^{154}Eu, ^{155}Eu, ^{147}Pm, respectively) have been taken into correction by careful spectrum evaluation, and performing further purification.

For method validation, the *use of reference materials* and participation in *interlaboratory exercises* are realistic choices only in case of a few well-known radionuclides (^{90}Sr, occasionally ^{63}Ni). For the more "exotic" nuclides method validation can be based on the application of an *independent analytical technique*. Inductively coupled plasma mass spectrometry (ICP-MS) is an excellent tool for detection of relatively long-lived radionuclides, such as ^{93}Zr and there is a chance to use it for radionuclides that have half-lives down to around 100 years (^{151}Sm). *Standard addition* is a good technique for “quench independent” efficiency calibration (^{63}Ni). *Isotope dilution technique* is appropriate for calculating the accurate quantity (activity) of the analyte (^{93}Zr) when validation is done via ICP-MS measurement. But neither of the latter two techniques is able to reveal possible contamination in the source. *Scientific understanding of the principles* can help improve the performance of the results. As such principle, the law of radioactive decay is used to check the half-life of the analyte (identify ^{90}Sr via the ingrowth of its ^{90}Y progeny). Another example is the comparison of measured activity/mass ratios with theoretical values. We compared the measured ^{93}Zr/stable Zr mass ratio with the calculated ones in case of Zr fuel cladding in order to confirm the trueness of the analytical results.

In the presentation, the methods developed for the determination of ^{90}Sr, ^{63}Ni, ^{93}Zr, ^{151}Sm in radioactive wastes will be briefly discussed and emphasis will be given to the various aspects of method validation. Examples for good and bad practices will reveal the need for competent method validation.

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Rapid Method for the Determination of Sr-90 in Steel and Concrete Samples

Sherrod Maxwell (1) and Ralf Sudowe (2)

The name of the second institution, address, post code, country

(1) Savannah River Nuclear Solutions, LLC, Building 735-B, Aiken, SC 29808, USA
(2) Colorado State University, Fort Collins, CO 80523

Corresponding author's e-mail: sherrod.maxwell@srs.gov

The rapid measurement of Sr-89, 90 in steel and concrete samples is important in the event of a radiological emergency such as an RDD (Radiological Dispersive Device), a nuclear accident and for the timely assay of nuclear facility decommissioning samples. A rapid acid digestion with and without hydrofluoric acid to enhance dissolution of refractory particles on the surface of the steel samples will be presented, coupled with a rapid extraction chromatography techniques to purify Sr-89,90 and/ or Y-90. The preconcentration method was designed to remove large amounts of iron resulting from steel digestion, facilitating subsequent column purification methods. A single matrix removal step is employed with effective removal of chemical interferences and high chemical yields. Results with refractory soil added will also be presented, along with a alkaline fusion alternative to digest mixed steel and concrete samples. Sr-89, 90 isotopes are measured by either gas flow proportional counting or liquid scintillation counting. The sample preparation method takes less than 8 hours.

For older facilities, only a ^{90}Sr assay may be needed during decommissioning activities. In this case, the ^{90}Sr daughter, ^{90}Y, is assumed to be in secular equilibrium with ^{90}Sr, a reasonable assumption in a solid matrix like steel or concrete. Steel or mixed steel/concrete sample aliquots can then be assayed immediately after total dissolution and rigorous purification of the ^{90}Y, eliminating the time normally needed for ingrowth of ^{90}Y following isolation of $^{89/90}$Sr.

A new bomb-combustion system for tritium extraction.

Richard Marsh [1, 2], Ian Croudace [1, 2], Phil Warwick [1, 2]

[1] Raddec International Ltd
[2] GAU-Radioanalytical, University of Southampton.

Corresponding author's e-mail: richard.marsh@noc.soton.ac.uk

Quantitative extraction of tritium from a sample matrix is critical to efficient measurement of the low-energy pure beta emitter. Pyrolysis using a tube furnace has been adopted as an industry standard approach for the liberation of tritium (Warwick et al., 2010) from a wide range of materials however pyrolysis of organic-rich materials can be problematic. Practically the mass of organic rich sample combusted must be limited to <1 g to minimise the possibility of incomplete combustion. This can have an impact on both the limit of detection that can be achieved and how representative the subsample is of the bulk material, particularly in the case of heterogeneous soft waste. Raddec International Ltd, in conjunction with GAU-Radioanalytical, has developed a new high-capacity oxygen combustion bomb (the Hyperbaric Oxidiser; HBO_2) to address this challenge. The system is based around a 5-litre combustion vessel and purpose designed product gas handling system (Croudace et al., 2016). The system is capable of quantitatively combusting samples of 20-30 g under an excess of oxygen, facilitating rapid extraction of total tritium from a wide range of orphan waste materials including mixed soft waste, plastic, rubber and oil. The large sample capacity also enables improvements to the limit of detection that can be achieved making the HBO_2 a useful tool for the analysis of biota samples for environmental monitoring purposes. ^3H is recovered as tritiated water vapour from the HBO_2 combustion chamber under vacuum. The vapour is trapped as ice in vacuum-grade glass tubes held in a Raddec cryoelectrical system. After thawing, the combustion water can be taken for direct analysis by Liquid Scintillation Counting without the need for further purification or pre-treatment. This presentation offers new data arising from operational experiences using the HBO_2 for waste characterisation of materials derived an experimental fusion reactor site. Inter-comparison and validation data against established pyrolysis techniques are also presented.

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Application of LSC for ^{3}H, ^{14}C, ^{36}Cl, ^{41}Ca and ^{63}Ni determination in various matrix from nuclear waste

R. Brennetot, M. Giuliani, C. Colin, E. Laporte, S. Guégan, J. P. Degros, A. Masset, P. Deloffre, C. Mougel

Den - Service d'Etudes Analytiques et de Réactivité des Surfaces (SEARS), CEA, Université Paris-Saclay, F-91191, Gif-sur-Yvette, France

Corresponding author's e-mail: rene.brennetot@cea.fr

Waste management is a key issue for nuclear industry. In France, ANDRA (the national agency for nuclear waste management) is in charge of sizing, building and operating waste repositories. In order to assure the security of these facilities, a list of radionuclides and their maximum associated activities has been defined as acceptance criteria by ANDRA. From this list, some are readily and easily measured while many, such as pure beta emitters are difficult to measure, requiring long radiochemical processes. In addition, these radionuclides have to be measured from various matrices such as liquid effluents, resin, concrete or metals [1-2].

For more than twenty years, the LASE (Operator Support Analysis Laboratory) has been developing and implementing radiochemical methods devoted to the determination of radionuclides in various nuclear waste. Within the French UNGG nuclear power plant dismantling framework, a huge amount of radioactive graphite (activated and potentially contaminated) will be produced in the future. Their radioactive level depends on their location and needs to be well characterized to ensure the safety of the repository that will be chosen in future years. Different radiochemical separations have been developed in the laboratory in order to be able to measure beta emitters by Liquid Scintillation Counting (LSC) in graphite. Examples for measurement of ^{3}H, ^{14}C, ^{63}Ni and ^{36}Cl in graphite at different activity levels from kBq/g to few Bq/g will be presented [1]. Extraction after combustion has been developed for ^{3}H, ^{14}C and ^{36}Cl while acid digestion is preferred before LSC measurement for ^{63}Ni. To validate, to improve and to compare radiochemical separations with others European laboratories, our laboratory participate in 2010 to carbowaste program and our results will be presented.

A second matrix of interest, concrete, has been investigated. An associated specific treatment has been developed in order to extract and separate radionuclides. Concrete can come from buildings, immobilized waste (such as resin or sludges for example) or structure materials close to the core of nuclear installations. Appropriate procedures have been developed in order to measure ^{3}H, ^{14}C in concrete for LLW or ILW repository. For these determinations, pyrolysis method for extraction and LSC for measurement have been developed and validated with homemade material and will be presented. A complete procedure has been developed for ^{41}Ca separation and measurement by LSC or AMS for very low level in concrete and will be presented.

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Determination of technetium-99 in UO$_2$ samples using liquid extraction separation and LSC measurement

Keliang Shi [1)], Zhiwei Niu [1)], Wangsuo Wu [1)]

[1)] Radiochemistry Lab, School of Nuclear Science and Technology, Lanzhou University, Lanzhou 730000, China

Corresponding author’s e-mail: shikl@lzu.edu.cn and wuws@lzu.edu.cn

Technetium-99 (^{99}Tc) is a long-lived radionuclide which is produced through thermal neutron fission reactions of ^{235}U and ^{239}Pu. Because of the long half-life (2.13×10^5 years), high fission yield (about 6%) and high mobility (weak sorption of TcO_4^- to geological media), ^{99}Tc has been regarded as one of the most imoprtant radionuclides for safety assessment of environmental radioactivity and repository of nuclear waste. The present work mainly focused on the method for determination of ^{99}Tc in UO$_2$ samples. ^{99}Tc was separated from high level of uranium through 2,6-dimethylpyridine extraction in alkali media and measured by liqiud scintillation counting. With the application of proposed procedure, a mean recovery of >94% for ^{99}Tc was achieved for 0.2 g UO$_2$ samples (^{99}Tc in µg level), reaching a detection limit of 10^{-9}g ^{99}Tc/gUO$_2$ (see Table 1). The RSD of the analytical method is better than 2% for the measurement of six duplicates. Our results show that the developed method can be used for the determination of ^{99}Tc in nuclear waste containing high level of uranium.

Table 1 The analytical results for ^{99}Tc spicked in UO$_2$ samples (0.25 µg ^{99}Tc added)

Sample number	1	2	3	4	5	6
Counts (cps)	226	230	237	226	229	229
^{99}Tc measurement (µg)	0.235	0.239	0.246	0.235	0.238	0.238
Recovery (%)	94.4	95.9	98.8	94.3	95.6	95.5
RSD			1.76 %			

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PSresins for the characterization of nuclear wastes: application to ^{126}Sn and ^{63}Ni

Eduard Pelay 1), Ariadna Ferradal 1), Ignasi Villarroya 1), Héctor Bagán 1), Alex Tarancón 1,2), José F. García 1)

1) Department of Chemical Engineering and Analytical Chemistry, University of Barcelona, Martí i Franquès, 1-11, ES-08028, Barcelona, Spain
2) Serra-Húnter Program, Generalitat de Catalunya, Barcelona Spain

Corresponding author's e-mail: hector.bagan@ub.edu

After some decades working, the number of nuclear power plants and other radioactive installations that need to be decommissioned is increasing. In this scenario, it is appearing high amounts of nuclear wastes that need to be characterized before any decision regarding their disposal procedure is taken. In these wastes, the gamma emitters can be easily quantified due to the high selectivity of their measurement but the alpha and beta emitters present some difficulties on its measurement.

Liquid scintillation is the most common technique for the measurement of beta emitters, and it is also widely applied for the determination of alpha emitters due to its high detection efficiency for such particles. However, one of its main drawbacks is the lack of selectivity because the spectra of the different radionuclides are always partially or completely overlapped. This makes necessary to separate the individual radionuclide before any measurement by using different procedures as liquid extraction, precipitation or chromatographic extraction.

Plastic scintillation in form of microspheres (PSm) is an alternative technique for the measurement of alpha and beta emitters. One of its main advantages is their solid surface that allows the inclusion of strategies for the selective separation of radionuclides. The PSresins follow this idea unifying separation and measurement preparation in a single step by incorporating to the PSm the specific extractant for each radionuclide considered. The PSresins permit the separation, preconcentration and direct measurement of the radionuclides, saving manpower, time and reagents.

Different PS resins were developed in our research group in the recent years, and more recently two resins focused on the characterization of ^{126}Sn and ^{63}Ni activity present on nuclear wastes.

^{126}Sn is a long-lived fission product generated in nuclear reactors, and even when it is not one of the main contributors to the initial waste activity, it is becoming of interest in the decommissioning of nuclear facilities for its behavior and relevance in long term storage. For the preparation of this PSresin, a know extractant, tributyl phosphate dissolved in dodecane, was selected. In this case, a new immobilization procedure based on the impregnation of the PSm was studied. Also, due to the nature of the extractant, the PSm support can be affected by the extractant, therefore, a new PSm support, prepared with crosslinker (C-PS), was used. This C-PS has a three dimensional chain structure that makes the polymer resistant to organic and aggressive media and therefore inert to the extractant studied. Different proportions of tributyl phosphate and dodecane were tested for the preparation of the PSresin and compared in terms of Sn retention and break volume of the column. The optimum proportion was selected and the retention of interferences as well as the analysis of ^{126}Sn solutions was studied.

The other isotope studied, ^{63}Ni, is produced by neutronic activation in the nuclear reactors and its determination is an important part in the decommissioning of nuclear power plants. For the preparation of this resin, three different strategies were used and compared to evaluate their potential application and limitations for PSresins preparation. The first one was the standard procedure used for the other PSresins, the immobilization of a selective extractant by solvent evaporation. In this approach, two extractants were selected, dimethylglyoxime, commonly used for the separation of nickel, and (2-1H-imidazol-2-yl)pyridine, as an innovative extractant. The second strategy used was the preparation of an imprinted polymer. This technique permits to obtain a polymer with selectivity to the target element using as a raw material a nonselective complexant. The polymer obtained following this strategy would have the advantage of not have break volume or leaching of the extractant. The final approach used was the covalent bound of the selective extractant (in this case di-(2-picoly)amine) to the polymer. This option still uses a selective extractant, but in this case, is bound to the PSm and therefore the resin obtained does have neither break volume nor leaching of the extractant. The three approaches were compared in terms of Ni retention, selectivity again its common interferences (Co and Fe) as well as detection efficiency of the PSresin. With these results, the best approach for the resin preparation was selected to development the final PSresin.

Adaptation of PTB's analytical modelling for TDCR-Cherenkov activity measurements at LNE-LNHB

C. Thiam, C. Bobin, J. Bouchard

CEA, LIST, Laboratoire National Henri Becquerel (LNE-LNHB), Bât. 602, PC 111, 91191 Gif-sur-Yvette Cedex, France

Corresponding author's e-mail: cheick.thiam@cea.fr

For several years, the TDCR method (Triple to Double Coincidence Ratio) based on Cherenkov effect has been investigated as an alternative to conventional liquid scintillation (LS) counting, in particular for activity measurements of high-energy β-emitters. Its interest lies in the capability to directly carry out Cherenkov counting with aqueous samples (i.e. without scincillation cocktail) thus making source preparation easier. Despite lower detection efficiencies compared to LS counting, the threshold effect can be advantageously used in specific cases to discriminate the radionuclide to be standardized from potential impurities that emit energies under the Cherenkov threshold (~ 260 keV in aqueous solutions). For instance, activity measurement of ^{32}P (E_β max ~ 1710 keV) in aqueous solutions is not affected by the usual impurity ^{35}S (E_β max ~ 167 keV).

The activity determination needs a light-emission modelling to establish a mathematical relation between the experimental TDCR value and the detection efficiency of double coincidences. For that purpose, an analytical TDCR model modified for Cherenkov counting was first investigated at PTB for activity measurements using polyethylene vials [1]. This analytical TDCR-Cherenkov model is based on the Frank and Tamm theory [2] to calculate the mean number of photoelectrons detected in photomultipliers (PMTs). It takes also into account the specific physical properties related to the Cherenkov emission: the continuous spectral bandwidth between UV and visible light, the energy threshold depending on the aqueous medium and the anisotropy of light emission. PTB's modelling was adapted at LNE-LNHB by considering the specific features of the TDCR counter dedicated to Cherenkov counting in our lab. The XP2020Q PMTs have a spectral sensitivity comprised between 160 nm and 600 nm. The quantum efficiency as a function of wavelength was also included in the model (~ 24 % in the 300-400 nm wavelength region). This work was initiated in the framework of the project of the European Metrology Research Programme (EMRP) MetroMRT (Metrology for molecular radiation therapy). The objective was the implementation of an analytical modelling enabling the application of TDCR-Cherenkov counting in the case of the standadization of ^{90}Y-labelled resin microspheres (SIR-Spheres, Sirtex) after dissolution [3].

The present work shows the first results obtained for the validation of the analytical modelling developed at LNE-LNHB in the case of pure β-emitters, ^{89}Sr (E_β max ~ 1495 keV) and ^{90}Y (E_β max ~ 2280 keV). The activities obtained by TDCR-Cherenkov measurements are in good agreement, within 0.5 %, with the expected value given by conventional LS TDCR counting. The analytical model was also applied for the activity measurement of ^{68}Ge/^{68}Ga. In that case, only interactions given by β^+ events resulting from ^{68}Ga (E_β max ~ 1899 keV) are counted because electron-capture events from ^{68}Ge have lower energies (maximal values of about 10 keV) than the Cherenkov threshold in aqueous solutions. The results are compared to those obtained using the

$4\pi\beta-\gamma$ coincidence method (based on LS counting in the $\beta-$channel) in the framework of an international comparison conducted by NIST. The analytical modelling is also compared to the stochastic approach developed using the Monte Carlo code Geant4 [4].

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Primary activity standardization of ^{134}Cs

Karsten Kossert(1), Justyna Marganiec-Gałązka(1), Ole J. Nähle(1)

(1) Physikalisch-Technische Bundesanstalt (PTB), Bundesallee 100, 38116 Braunschweig, Germany

Corresponding author's e-mail: karsten.kossert@ptb.de

Cesium-134 was measured by means of a new coincidence counting system which is equipped with a liquid scintillation detector to count β particles and an NaI crystal to detect γ-rays which are emitted simultaneously. In order to validate this new technique, additional measurements were carried out with an established 4πβ-γ-coincidence counting system with a proportional counter in the β channel.

The coincidence counting experiments were complemented with measurements in further liquid scintillation counters with two and three photomultiplier tubes. The counting efficiencies for these systems were determined using CIEMAT/NIST efficiency tracing and the triple-to-double coincidence ratio (TDCR) method, respectively.

The results of all four methods are in good agreement and the relative standard uncertainty of the combined result was found to be 0.25%. A standardized ^{134}Cs solution was submitted to the Bureau International des Poids et Mesures (BIPM) to be measured in the ionization chambers of the International Reference System (SIR) for comparison purposes.

The pros and cons of the individual methods are discussed.

Activity determination of ^{88}Y by means of 4π(LS)β-γ coincidence counting

Justyna Marganiec-Gałązka [1)], Ole J. Nähle [1)], Karsten Kossert [1)]

[1)] Physikalisch-Technische Bundesanstalt (PTB), Bundesallee 100, 38116 Braunschweig, Germany

Corresponding author's e-mail: justyna.marganiec@ptb.de

Yttrium-88 decays by electron capture and β^+ emission and is an important isotope for the calibration of γ-spectrometry systems due to the high-energetic γ-ray-emissions at 898 keV and 1836 keV, respectively. Precise activity measurements of ^{88}Y solutions are a prerequisite to prepare the required activity standards of this radionuclide. However, an accurate determination of the activity is not trivial due to the low energies of X-rays and Auger electrons which are emitted as a consequence of the electron capture process.

In this work, the activity concentration of an ^{88}Y solution was measured using a new liquid scintillation (LS) based 4πβ-γ coincidence counting system constructed at PTB. The system comprises a TDCR counter as β-channel, a NaI scintillation detector as γ-channel and an automated sample changer system. Efficiency variation was carried out with the aid of chemical quenching as well as by means of grey filters (neutral density filters: from 0.15ND to 1.2ND) which were placed around and underneath the vials. Since the β-channel can either be defined as the detected double coincidences (D) or the detected triple coincidences (T) various options for the extrapolation exist. In additional, several fit functions were compared. The efficiency was finally extrapolated to a β-efficiency of 100%.

Since the 4π(LS)β-γ coincidence counting has never been used before for this isotope at PTB, additional validation measurements were made with the well established coincidence counting technique using a proportional counter in the β channel. The results of both coincidence methods were found to be in excellent agreement with a relative deviation of less than 0.1%.

A thorough uncertainty consideration reveals a rather large uncertainty of the result from the 4π(LS)β-γ coincidence counting measurement. The major uncertainty component that contributes to the overall relative uncertainty of about 0.5%, is assigned to the fitting procedure. Possibilities to reduce the uncertainty in the future will be discussed.

Standardization of Na-22 by the CIEMAT/NIST method

LIU HaoRan [1)], LIANG JunCheng [1)], YUAN DaQing [2)]

[1)] National Institute of Metrology, Division of Ionizing Radiation, Beijing, 100029, P.R. China

[2)] China Institute of Atomic Energy, Department of Nuclear Physics , Beijing , 102413, China

Corresponding author's e-mail: liuhr@nim.ac.cn

1. Introduction

^{22}Na decays predominantly to the 1275 keV level of ^{22}Ne by beta plus and electron capture, a very small fraction (0.056%) disintegrates to the ground state of ^{22}Ne [1]. The CIEMAT/NIST method was chosen to measure the activity concentration of ^{22}Na solution, with ^{3}H as the tracer. Seven samples with the radioactive solution and one blank sample without active solution were prepared in the same way. All the samples were measured in a Tricarb-3100 counter, with tSIE as the quench-indicating parameter.

2. Efficiency calculation

The efficiencies of the ^{3}H and ^{22}Na were calculated by the program CN2003[2]. The counting efficiency of the electron-capture transition to the 1275 keV level of ^{22}Ne was calculated by means of the KLM atomic rearrangement model. The contribution of this branch to the overall counting efficiency is very small[3]. The calculated detection efficiencies of ^{22}Na versus ^{3}H are shown in Fig. 1.

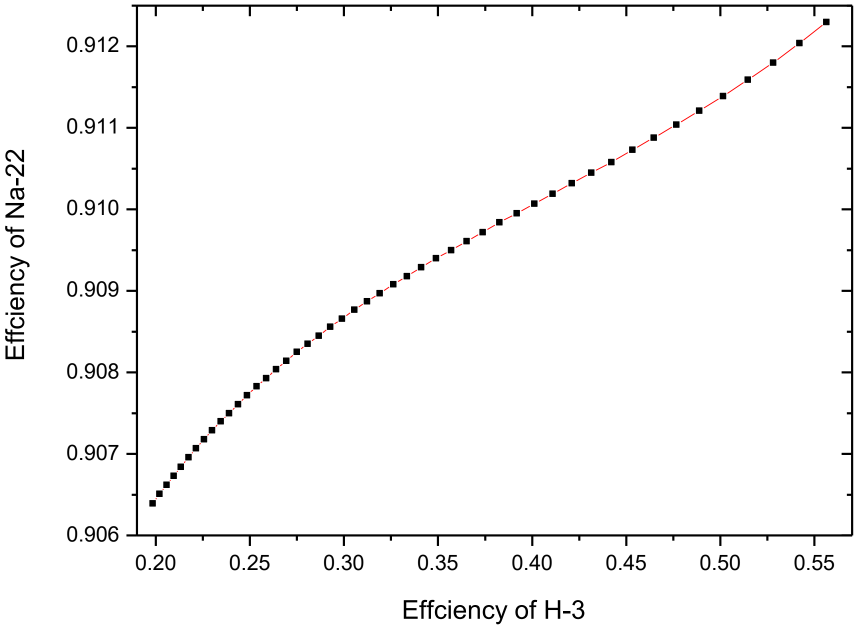

Fig. 1 The calculated detection efficiencies of ^{22}Na versus ^{3}H

3. Results and discussions

The activity concentration of seven samples are shown in Fig.2. It was noticed that the tSIE values of sample N_1 and N_2 were out of the tSIE range of quench correction curve. The activity concentration obtained by applying the extrapolation method, sample N_1 and N_2, were significantly deviated from the results of the interpolation method, sample N_3 to N_7. It implies the extrapolation method is not applicable here. The final result only considered the results of sample N_3 to N_7, which indicates good agreement between each other. The mean activity concentration is 296.6 kBq.g^{-1} on the reference date of 10 March 2012 (2 h UTC), with relative standard deviation of 0.02 %.

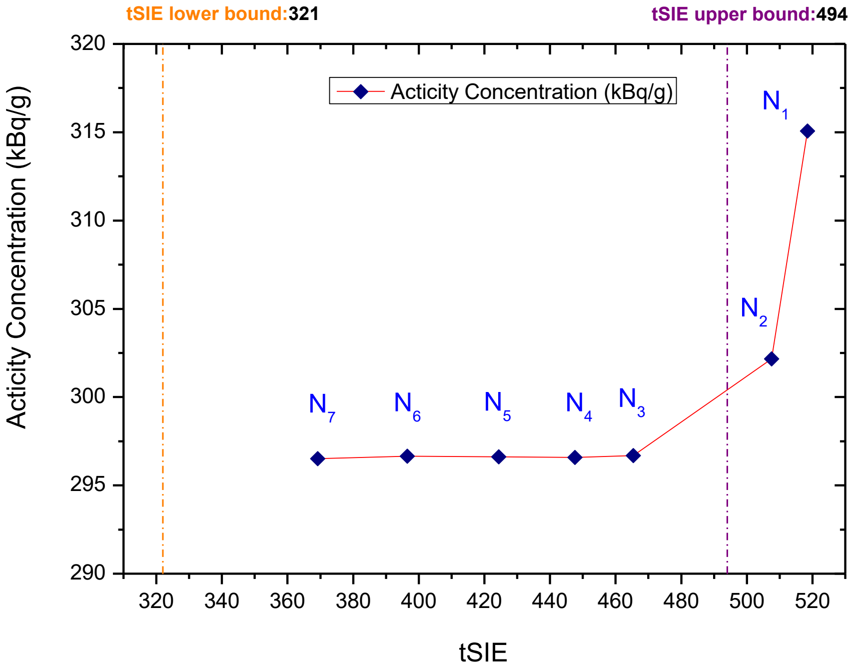

Fig. 2 Activity concentration of ^{22}Na solution

The uncertainty associated with tSIE and kB values were taken into account in the final uncertainty budget. It was observed that the experimental tSIE values were affected by Compton spectrum of gamma rays from ^{22}Na source. As shown in Table 1, the tSIE values of N_1 (unquenched sample) and N_2 (quenched sample), are much higher than unquenched blank sample. It indicates that the tSIE values of ^{22}Na samples are shifted by themselves. An estimate has been made by taking into account the tSIE difference between the sample blank and N_1 as the tSIE shift. The relative deviation of efficiencies of ^{22}Na by tSIE shift effect is less than 0.04%.

Table 1 The tSIE values of blank and ^{22}Na samples with different amount of quench agent

Sample No.	CH_3NO_2(μl)	tSIE
Blank	0	491.8
N_1	0	518.5
N_2	20	507.5
N_3	50	465.4
N_4	70	447.6
N_5	100	424.4
N_6	150	396.5
N_7	200	369.2

When applying the Birks formula for ionization quench correction, the ionization quenching parameter kB is not an independent parameter, but depends on the selected stopping power. Therefore, it is necessary to evaluate the influence of different kB values for efficiencies calculation. The influence of kB values for efficiencies of ^{22}Na is shown in Fig.3. There is a very small difference of using different kB values, within 0.03% in the whole interval.

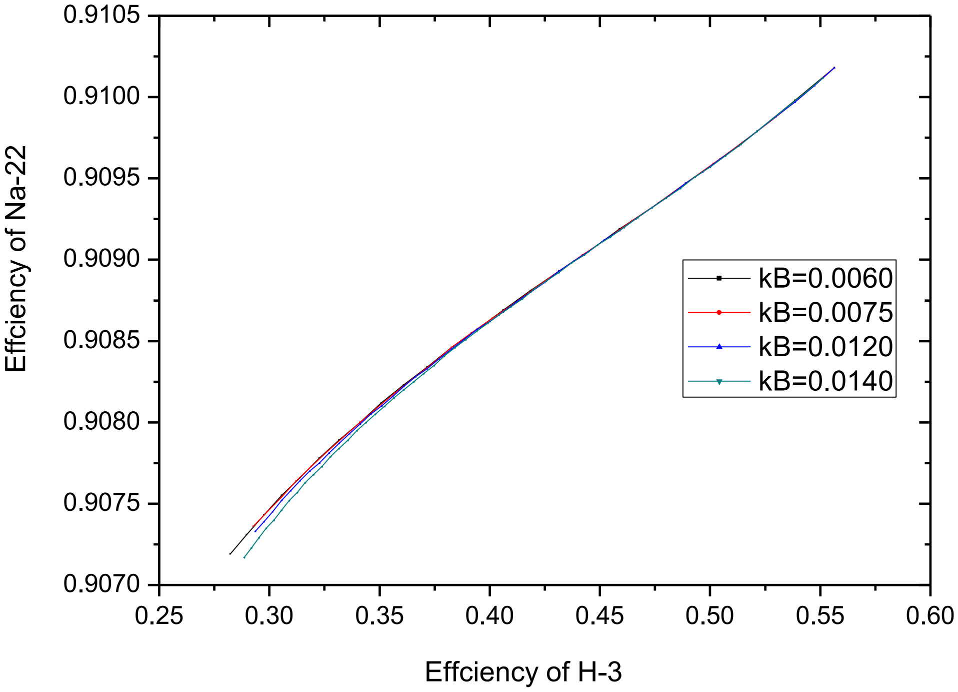

Fig. 3 The influence of kB values for computed efficiencies of ^{22}Na

4. Summary

A solution of ^{22}Na was standardized by CIEMAT/NIST method in terms of specific activity. The mean activity concentration is 296.6 kBq.g^{-1} on the reference date of 10 March 2012 (2 h UTC). A tSIE shift was observed. The influence of tSIE shift, as well as kB values were evaluated and taken into account in the final uncertainty budget.

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The use of recommended decay data from the DDEP for activity determinations using LSC

Mark A. Kellett, Christophe Dulieu, Xavier Mougeot (1)

(1) CEA, LIST, Laboratoire National Henri Becquerel (LNE-LNHB), Bât. 602 PC111, CEA-Saclay 91191 Gif-sur-Yvette Cedex, France

Corresponding author’s e-mail: mark.kellett@cea.fr

The need for the consolidated use of equivalent decay data when making absolute activity measurements using LSC was identified by the Consultative Committee for Ionizing Radiation, Section II: Measurement of radionuclides, CCRI(II), a number of years ago. The Decay Data Evaluation Project (DDEP), whose mission is to provide recommended decay data to the metrology community, provides the necessary data, and so their use in metrological measurements was approved by the CCRI(II).

The DDEP was originally created as a collaboration between two national metrology institutes: the French Laboratoire National Henri Becquerel (LNE-LNHB) and the German Physikalisch-Technische Bundesanstalt (PTB) in 1995, but quickly gained momentum when other institutes from around the world, particularly the US, joined. Since its formation, the evaluators within the DDEP have provided recommended decay data for over 220 radionuclides, of particular importance in detector calibration and activity standardisation.

Furthermore, enhancements in the calculation of beta spectra undertaken at the LNE-LNHB using the *BetaShape* code [1] have been shown in [2] to improve the compatibility between the triple-to-double coincidence ratio (TDCR) method and the CIEMAT/NIST method in the absolute activity determination of ^{63}Ni. The improved calculations better predict the low energy part of the beta spectrum, where the scintillation yield is highest, which is of particular importance for radionuclides such as ^{63}Ni, which have a relatively low beta spectrum end-point energy, i.e. ~67 keV in this case.

A current European Metrology Programme for Innovation and Research (EMPIR) project, *MetroBeta*, aims to further improve the calculation of beta spectra by the inclusion of additional corrections and nuclear structure effects, which in turn will be compared with measurements made by the use of innovative cryogenic detectors – metallic magnetic calorimeters (MMCs).

In order that the DDEP recommended data can be easily accessed by all, the LNE-LNHB provide a dedicated website (http://www.nucleide.org/NucData.htm) and have developed the online tool LARAWEB to facilitate their use (http://www.nucleide.org/Laraweb/) [3]. The decay data evaluations and schema are also published through the Monographie BIPM-5 series [4] of the *Bureau International des Poids et Mesures* (BIPM), as well as a pocket guide, entitled the Mini Table of Radionuclides [5].

Further details on the need for the consistent use of recommended decay data for all measurements using the LSC technique will be given and their availability will be highlighted.

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First absolute standardization of ^{18}F at NMISA via 4π (LS)β-γ counting for SIRTI comparison

M.W. van Rooy [1)], M.J. van Staden [1)], B.R.S. Simpson [1)], J. Lubbe [1)]

[1)] NMISA, 15 Lower Hope Road, Rosebank, 7700, Cape Town, South Africa

Corresponding author's e-mail: mvrooy@nmisa.org

During 2015, the National Metrology Institute of South Africa (NMISA) performed its first absolute standardization of a ^{18}F solution. The method employed 4πβ-γ coincidence counting using liquid scintillation (LS) in the 4π channel. The standardization formed part of the BIPM.RI(II)-K4.F-18 International Reference System Transfer Instrument (SIRTI) comparison which took place one month later, for which NMISA was only the fourth participant thus far.

Fluorine-18 is used during positron emission tomography (PET) scans. Literature indicates that ^{18}F is a simple radionuclide to standardize due to its straightforward decay scheme and high beta-plus energy. We present a more complete detection efficiency analysis of the 4πβ-γ counting method than that currently found in the literature. Experimentally, the biggest challenge is measurement logistics due to its short half-life of 1.8 hours, which was overcome at NMISA by using a fifteen channel double- and triple-coincidence unit for simultaneous measurements at various electronic discrimination levels [1], to allow for activity determination through the extrapolation technique. Because the sources needed to be counted on the day of preparation, it is interesting to note that those counted first had an effiency of only 85 % while the last measurement gave 95%. This was probably due to the scintillator being slightly cloudy at first and clearing during the day. Nevertheless the extracted source activity concentrations were comparable to one another. Our primary standardization resulted in a standard uncertainty of 0.34 % with the main contributions coming from efficiency extrapolation and the uncertainty in the beta-plus branching ratio, both being 0.2 %.

The result of our primary standardization was used to determine a calibration factor for the NMISA secondary standard ionization chamber for ^{18}F. This factor was used during the SIRTI comparison. The NMISA SIRTI equivalent activity is 0.34 % higher than the key comparison reference value (KCRV) and in agreement within one standard deviation.

In addition to the extrapolation method, we analyzed the partial 4π(LS)β-γ measurement data via a non-extrapolation method [2, 3, 4] based on a detection efficiency analysis. This indicates that the measurand C/G samples both the LS positron efficiency and the efficiency of the annihilation photons interacting with the scintillator. Equating the measurand value to theoretical formulae based on the figure-of-merit, these (double-coincidence) efficiencies can be determined from a knowledge of the theoretical spectral shapes. This then allows for the ^{18}F activity to be determined from data collected corresponding to only the lowest discrimination level, advantageous if apparatus is not available for multi-discrimination data acquisition. Initial results from various source measurements with C/G ranging from 85 % to 95 % indicate non-extrapolation results that are 0.13 % to 0.35 % lower than the extrapolated results. The average non-extrapolation result translates into a SIRTI equivalent activity which is closer to the KCRV compared to the extrapolation method.

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COMPUTATIONAL MODELING OF ORGANIC FLUOR MOLECULES

Nathaniel J. Nicholsa, Lindsay C. Shuller-Nickles, Timothy A. DeVol, Ayman F. Selimana,

Environmental Engineering and Earth Sciences, Clemson University, Anderson, SC 29625, USA

Corresponding author's e-mail: njnicho@g.clemson.edu

The goal of this research is to benchmark the computation parameters necessary for accurately modeling the fluorescence process of seven organic scintillating molecules (2-(1-naphthyl)-5-phenyloxazole (αNPO), 2-(1-naphthyl)-4-vinyl-5- phenyloxazole (vNPO), 5-(4-Bromophenyl)-3-(4-ethylphenyl)-1-phenyl-4,5-dihydro-1H-pyrazole (PZ1), 3-(4-Ethylphenyl)- 5-(4-vinylphenyl)-1-phenyl-4,5-dihydro-1H-pyrazole (vPZ1), and 3-(4-Ethylphenyl)-5-(4-fluorophenyl)-1-phenyl-4,5-dihydro-1H- pyrazole (PZ2), 2-(4-tert-butylphenyl)-5-(4-biphenylyl)-1,3,4 oxadiazole (t-butylPBD), and 2-[4-(4'- vinylbiphenylyl)]-5-(4-tert-butylphenyl)-1,3,4-oxadiazole monomer (vPBD)). Organic fluorophores are essential in scintillation detection, as they emit detectable photons in response to energy deposition from ionizing radiation. Optimal detection efficiency occurs with emitted photons at the maximum response wavelength of the photodetector, which for a conventional photomultiplier tube is 420 nm. The αNPO, vNPO, PZ1, vPZ1, and PZ2 fluorophores were examined experimentally in previous studies1, 2 and don't require additional wavelength shifters. We report on the performance of four exchange-correlation functionals (B3LYP, M06-2X, CAM-B3LYP, wB97X-D) for each fluor molecule in toluene and cyclohexane solutions. While B3LYP is widely executed, studies have shown that the use of functionals with long-range corrections (M06-2X, CAM-B3LYP, wB97X-D) produce more accurate results. This research has identified the M06-2X functional as the best functional with respect to locating distinct absorption and emission features throughout the entire fluorescence spectra. The present research also shows that the implicit solvation model does not distinguish specific chemical effects from the interaction of solvent molecules and fluorophore molecules. Therefore, an explicit form of modeling solvent effects is being explored.

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Development of multifunctional digital pulse processing module for particle identification in liquid scintillator

H.J. Kim [1), M.J. Kim [1), J.Y. Lee [1), S. C. Kang [1)

1) Department of Physics, Kyungpook National University, Daegu 41566, Korea

Corresponding author's e-mail: hongjoo@knu.ac.kr

Among application of liquid scintillators, particle identification method can be applied to α/β separation and neutron detection [1]. The typical electronic module for particle detection consists of preamp, shaping amplifier, a multichannel analyzer, an analog to digital converter (ADC), a time to digital converter and a scalar module [1]. New systems based on the digital pulse processing technique have been developed and new architecture is much simpler than previous types [2]. Pulses are sampled immediately after the detectors or preamplifier stages and digitized signals are processed afterwards. Usually, a flash analog to digital converter (FADC) with a high sampling rate is used for the signal digitization and digital signal processor (DSP) and/or field programmable gate array (FPGA) is used for fast signal processing. This method can be applied to the particle identification, neutron and gamma separation, radiation detection, and the nuclear spectroscopy.

We developed a stand-alone multifunctional digital pulse processing module (MDPPM) based on FADC and FPGA technique. The MDPPM module consists of programmable gain amplifier, four 100 MSa/s interleaved FADC which provides a 400 MSa/s FADC with 10 bit resolution, FPGA digital signal processor, data buffer memory and TCP/IP interface. Total pulse width for charge integration can be varied from 20 to 1270 ns by the register setting. For tail charge integration, the delay of peak to tail start time can be adjusted from 0 to 1000 ns. A software threshold setting can be applied to trigger an event by a self-trigger algorithm. It has programmable internal gain from 0.7 to 10 and either positive or negative polarity of signal can be selected by software. Data buffer consists of dual buffers with 2048 depth which minimize dead time for data taking. Data can be transferred by Ethernet 10T/100T interface and maxim data transfer rate is 3 Mbytes/s. Digital pulse shape analysis (DPSA) method is implemented by digital charge comparison (DCC) method [3] which uses total to tail charge for comparison by FPGA logic. This module has the functions of a pulse area-sensing ADC, a partial pulse area-sensing ADC for DCC, a time tag by a digital constant fraction discrimination method. Particle separation between α/β or n/γ can be achieved with liquid scintillator using DPSA method. Also time tagging information of triggered samples is recorded with every 10 ns interval and it can be used for short-lived decay product or thermal neutron capture using delayed coincidence technique (DCT).

Two performance test results will be reported with conventional LSC. Using DCT and DPSA method, isotopes having a relatively short half-life could be selected out by their characteristic energy and decay-time distributions for the sample. To estimate activity of Radon (^{222}Rn), the sub-chain ^{214}Bi (Q_β = 3.27 MeV) → ^{214}Po (Q_α = 7.83 MeV, $T_{1/2}$ = 164 μs) can be used to estimate radon level in the time and energy interval by DCT with β and α requirement, where DPSA is used to identify α. Ultima Gold AB was used for the detection radon decay product in Tab water sample (approximately 100 mL in 700 mL UG-AB sample) in the one liter of SUS container. Fig. 1 left shows measured half-life of ^{214}Bi. Measured half-life of 170 ± 34 μs is consistent with expected with half-life of 164 μs. Fig. 1 right shows capability of β and α separation with DPSA method. . With UG-AB mixed with Tab water sample, we measured 0.03±0.004 Bq/L of low radon level with 12 hours data taking.

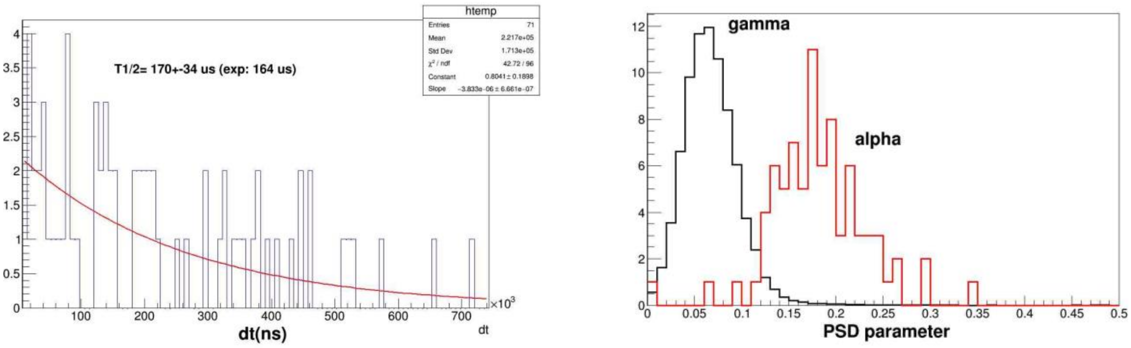

Fig. 1 Decay time distribution of ^{214}Bi (left) and DPSA of α/β (right)

A large size (20 liter) of liquid scintillator, which contains 0.5 % gadolinium, is used for the time tag study. Fast neutrons interact with the detector and produces proton recoils. Some of them lose total energy in the detector, thermalizes and eventually captured on gadolinium, which de-excites by gamma emission from an 8 MeV excited states. The neutron capture time in gadolinium can be extracted by a time correlation between recoiled proton and gamma emissions by captured neutrons in the gadolinium. Using the time tag of two successive events above the threshold, the time difference between two events can be extracted. A ^{252}Cf neutron fission source is used for the neutron tagging study and we show that we can extract thermal neutron signal from gamma background environment.

The MDPPM can be used for the various signal processing of LSC for particle identification such as radium and radon detection, short-lived decay product measurement, thermal and fast neutron detection, and radiation detection.

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An influence of the TDCR system settings on the radionuclides standardization

T. Ziemek 1), A. Jęczmieniowski 1), R. Broda 1) , E. Lech 1) , A. Listkowska 1)

1) National Centre for Nuclear Research Radioisotope Centre POLATOM, Andrzeja Sołtana 7, 05-400 Otwock - Świerk, Poland

Corresponding author's e-mail: tomasz.ziemek@polatom.pl

The triple-to-double coincidence ratio (TDCR) method [1] is applied at the NCBJ RC POLATOM for pure β-emitters standardization in two LS-counters: the TDCR system [2] and the 4π(LS)-γ coincidence system with a TDCR detector in the beta channel (referred to as TDCRG) [3]. Radionuclide activities in both counters are calculated assuming the Poissonian statistics of emitted light and the semi-empirical Birks function for describing the non-linearity of light emission as a function of energy released in the scintillator [4]. The threshold was set below 1-electron peak in both systems. Working points at the plateau of PMTs HV-characteristics were selected.

The systems functioning and stability was tested by ^{3}H and ^{14}C control sources measurements. Sources prepared in 20 mL high-performance PerkinElmer glass vials filled-in with 10 mL Ultima Gold LS-cocktail were measured in both counters during 6 hours each. Activities were calculated with regard to the source activity independence of the detection efficiency (referred to as the zero-slope criterion) for a selected ionization quenching parameter *kB* value. A non-acceptable difference of 2.2% between calculated specific activity values of ^{3}H source at the TDCR and TDCRG system and 0.2% of ^{14}C source respectively was observed. Various *kB* parameter values had to be selected in each case. Differences obtained with a one selected *kB* parameter value of 0.012 cm MeV^{-1} were of 0.9% for ^{3}H source and 0.6% for ^{14}C source respectively. Supposed existed irregularities in system settings had to be removed.

The threshold setting in both counters was checked. An additional measurements of the ^{3}H source were performed in the TDCRG counter with various threshold setting in the range from 25 mV to 250 mV. No influence the threshold setting on the difference between results obtained in the two systems was observed.

The plateau of ET Enterprises 9214B PMTs HV-characteristics of the TDCRG counter showed a positive slope. Working point at the plateau was increased from 2000V to 2200V resulted in 2.5% increase in counting rates. New series of ^{3}H, ^{63}Ni and ^{14}C sources in Ultima Gold scintillator in frosted and glass vials were measured in both systems and activities were calculated considering the zero-slope criterion with *kB* = 0.010 cm MeV^{-1}. An agreement of the measurement results better than 0.2% was obtained in both systems except of ^{63}Ni and ^{14}C sources in glass vials measured in the TDCR system. The last values were higher than the mean activity value of about 0.4% and 0.6% respectively. A significant influence the PMTs working point selection on the calculated activity value was demonstrated.

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Design and development of a miniaturized detector for radionuclide determination in automated flow systems

Oscar Pozo [1], Antoni Borràs [1], Tomeu Servera [2] , Alvaro Pineda [2] , Victor Cerdà [3] , Laura Ferrer[1]

[1] Environmental Radioactivity Laboratory (LaboRA), University of the Balearic Islands, Cra de Valldemossa km 7.5, E-07122, Palma de Mallorca, Spain.
[2] IC-Malaga, 07340 Alaró, Spain.
[3] Environmental Analytical Chemistry Laboratory, University of the Balearic Islands, Cra de Valldemossa km 7.5, E-07122, Palma de Mallorca, Spain.

Corresponding author’s e-mail: laura.ferrer@uib.es

The use of radioactive isotopes has dramatically increased in the last years. They are extensively used in nuclear power plants, medicine, industry and research. The availability of portable miniaturized automatic systems with the capacity to perform determinations of specific radionuclides in environmental samples is of capital interest for fast response in case of emergency events.

A miniaturized Liquid Scintillation Counter has been developed to detect the presence of beta and alpha emitting radionuclides in aqueous samples. The sample is mixed with scintillation liquid which emits a unique visible light pulse for each desintegration. The detection of these light pulses is performed by a couple of independent Hamamatsu R7600-U Photomultiplier Tubes (PMT) working in coincidence to avoid the detection of dark counts.

The Ultima Flo-M liquid scintillation cocktail has been selected because of its low viscosity, a requirement for optimal flow systems working conditions, and the optimal sample to scintillation liquid ratio is set to1:2.

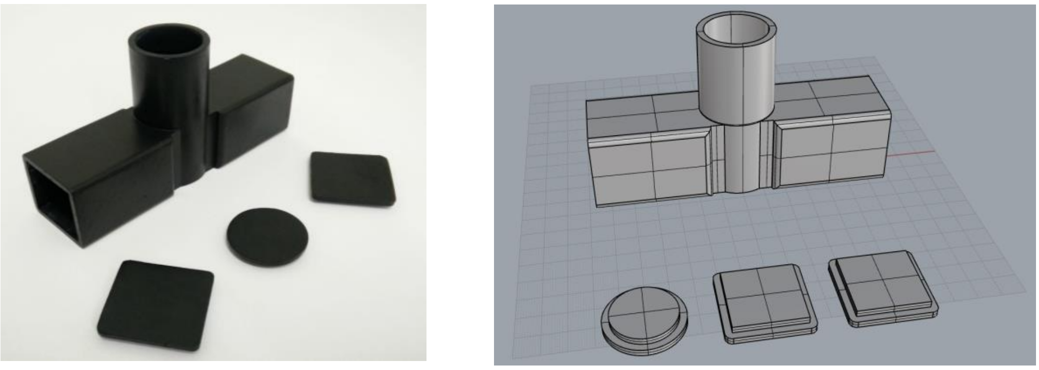

Fig 1. 3D printed black box to integrate the liquid scintillation vial and two PMT.

A specific 3D-printed module has been designed to integrate the LS vial and both PMT, allowing to simultaneously isolate these components from outer light sources and to optimize the geometry in order to increase the detection efficiency.

Specific modules have been designed to independently control the PMT power supply and their corresponding readout.

The final goal is to couple this miniaturized sensor to an automatic extraction and preconcentration system with a unified computer controlled system for the detection and flow systems. Using selective resins, this system will allow to detect specific radionuclides in different matrices of interest.

Use of active scintillating targets in nuclear physics experiments - measurement of spontaneous fission

Gilbert Bélier1) , Jean Aupiais1), G. Sibbens2), A. Moens2), D. Vanleueuw2)

1) CEA, DAM, DIF, DPTA Centre du Grand rue, 91297 Arpajon, France
2) Europan Commission, Joint Research Centre, IRMM, Retieseweg 111, 2440 Geel, Belgium

Corresponding author’s e-mail: gilbert.belier@cea.fr

The Liquid Scintillation Spectrometry technique has advantages that can be used in nuclear physics experiments. Compared to ionization chambers for example, liquid scintillation provides very high detection efficiencies. In the context of studies on the nuclei fission process this is a definitive advantage where count losses have to be known very precisely. We will present the general features of a scintillating target used in nuclear physics experiments, and the special developments that were performed. Particularly the response of a few homemade and commercial scintillators to fission events will be presented. We will detail simulations that were performed, in order to precisely determine the detection efficiency to fission events and also to α decays [1]. Finally we will present preliminary results obtained in the measurement of the spontaneous fission (SF) of ^{240}Pu and ^{242}Pu. It will be shown that a precise count of SF and alpha decay events can be made, together with a precise count of pile-up events (Figure 1). It will be shown that very precise ratio of the SF over α-decay half-lives can be made, and that the main limitation is due to the sample impurities knowledge.

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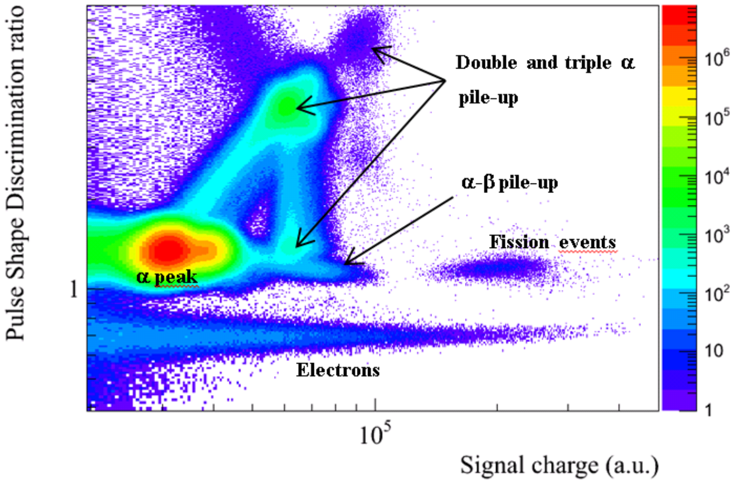

Figure 1 Identification histogram: Pulse Shape Discrimination ratio against the total charge of the signal. The ratio is defined as the charge of the slow component in the signal over the charge of the fast component.

Fabrication and Characterization of Low Afterglow Liquid Scintillator

Liu Shenye, Chang Guanjun, Yang Junxiao, Huang Yawen, Cao Ke, Su Ming and Yu Bo

Research Center of Laser Fusion, Chinese Academy of Engineering Physics, P.O. Box 919-986, Mianyang, Sichuan 621900, Peopl’s Republic of China

Southwest University of Science and Technology，Mianyang，Sichuan，6211010，People’s Republic of China The name of the first institution, address, post code, country

A fast liquid scintillator with very low afterglow has been Fabrication for neutron detecting with high intense Gama background in ICF experiments. The optimum fluors and solvents have been now firmly established finally, after we have modeled with molecular theory and tested various combinations of fluors and solvents by experiments. We have chosen that the first scintillator are 2,5-Diphenyloxazole(PPO), the secondary scintillators are p-bis(o-methyla-tyryl)-benzene (bis-MSB), and the solvents are para-xylene. The fluorescence yields of the various liquid scintillators have been measured by ultraviolet light exciting. We have made sure the optimum mixture is 0.01g/mL PPO and 0.0005g/mL bis-MSB. For measuring the glow curves of scintillators, we have measured the glow curves of the liquid scintillators by use of the time-correlated single photon counting technique (TCSPC). The light intensity irradiated by the scintillators is characterized with dynamic range of several orders of magnitude. In this paper we introduce the experimental results. The results have shown that the relative light yield (compare to NE213) is 88%, the after-glow is decreased with a range of five orders of magnitude in the time of 100ns for the fabricated liquid scintillators. The Low Afterglow Liquid Scintillator with gated photomultiplier can be used to detect the neutron produced by fusion with intense Gama radiation in the ICF implosion experiments.

Combination methods for rapid determination of mixtures of alpha and beta emitters in water samples

Jordi Fons-Castells, Montserrat Llauradó

Laboratory of Environmental Radioactivity of the University of Barcelona, Martí i Franqués 1-11 3th floor, Barcelona 08028 Spain.

Corresponding author's e-mail: jordi.fons@ub.edu

From gross alpha and gross beta determination by LSS, spectra of the entire alpha and beta emitters in the sample was obtained. However, usually this signal is integrated to report gross alpha and gross beta, and hence it lose specificity. There are in the literature several procedures for the deconvolution of LS spectra; conctructing PLS models [1] or fitting the spectrum of the sample to a linear combination of experimental standard spectra [2], to a tailed Gaussian functions [3] or to a Fourier series [4]. In this work PLS calibration was used to quantify several radionuclides from the same spectra. To diminish problems of interference, other techniques were used to discard or identify some of them in the sample. The radionuclides discarded with this techniques were excluded on the PLS model construction, to improve its selectivity. The radionuclides studied were the gathered in the directive EURATOM 2013/51.

The methods used were; simultaneous determination of gross alpha and beta by LSS with and without evaporation (in order to improve the detection limits in the first case, and to measure ^{3}H and ^{14}C in the second), measurement by gamma spectrometry in 500 mL of sample and determination of ^{226}Ra, ^{228}Ra and ^{210}Pb by means of radium RAD disk by LSS measurement.

Gross alpha and beta. Concentration method (LSS$_{conc.}$)

A 100 mL aliquot of sample was evaporated to dryness. When the precipitate obtained was cooled to room temperature, it was dissolved in 10 mL of deionised water acidified by HCl to pH ≈ 1.5. Then, an 8 mL aliquot of the evaporated sample was mixed with 12 mL of Ultima Gold AB in a PE vial. The sample was then measured for 400 min in a Quantulus. It is recommended that the vial should remain in the counter for 2 h before counting to avoid photoluminescence phenomena. The optimal PSA for this determination was set as 100 using ^{236}U and ^{40}K as calibration standards. Quenching was been controlled by means of SQP[E] and all the measurements were in the rage of 800 ± 10. [5]

Direct measurement by LSS (LSS$_{dir.}$)

An aliquot of 8 mL of sample, just filtered if suspended solids were observed, was mixed with 12 mL of UGAB in a PE vial. The measurement was performed in the same way that LSS$_{conc.}$.

High resolution gamma spectrometry

A sample aliquot of 500 mL was transferred in a calibrated geometry and measured for 5 hours high resolution gamma spectrometer of high purity germanium CANBERRA BEGe 3830.

Separation by means of radium RAD disk and LSS measurement

The radium RAD disk was prepared in a vacuum filtration system. RAD disk was conditioned with 20 mL of 2 M HNO_3, then 1 L sample acidized to pH<2 with concentrated HNO_3 was filtered through the RAD disk. Afterwards, ^{210}Pb was eluted with 7 mL of diammonium hydrogen citrate (DHC) 0.05 mol L^{-1} at pH 5.75. An aliquot of 5 mL of the eluted fraction were mixed with 15 mL of Optiphase Hisafe III in a PE scintillation vial and was counted for 100 min in a Wallac QUANTULUS 1220. On another hand, after the elution of ^{210}Pb, the RAD disk was transferred to another vial and was counted for 100 min after 8 hours in order to avoid misclassification and shifting of the spectra. In all cases the flow rate was lower than 50 mL min^{-1} [6].

PLS models construction for $LSS_{conc.}$ and $LSS_{dir.}$ methods

For $LSS_{dir.}$ standards of 13 RN's (^{3}H, ^{14}C, ^{40}K ^{60}Co, ^{90}Sr/^{90}Y, ^{134}Cs, ^{137}Cs ^{210}Pb, ^{226}Ra, ^{228}Ra, ^{nat}U, $^{239+240}$Pu and ^{241}Am) were analyzed at three levels of activity by triplicate. For $LSS_{conc.}$ just 11 RN's were measured because ^{3}H and ^{14}C were removed from the sample when this method is used, due to the evaporation to dryness. From all the analisis two spectra, an alpha and beta were obtained. In order to minimize the computation time and to reduce the complexity of the model, region of interest (ROI) for alpha (454-915) and beta (75-895) spectra were selected. Afterwards, each spectrum was smoothed using a Savitzky-Golay filter with polynomial order 1 and a window width of 31 channels. For each set of spectra ($LSS_{conc.}$ and $LSS_{dir.}$) matrixes that joins alpha and beta spectrum for each sample were construct. Before the PLS model construction this matrix were mean centered. Additionally, for both methods a matrix with the standard activities of each sample was calculated. In this way, we have two matrixes for each method; the first one which contains the activity of all the RN's in each standard solution, and a second one with its alpha and beta spectrum smoothed and mean centered. With both matrixes it is possible to construct different PLS models using the rows of the RN's of interest for each scenario. [7]

Application of the methods

In Fig 1. a flowchart of the procedure of application of the studied methods is shown.

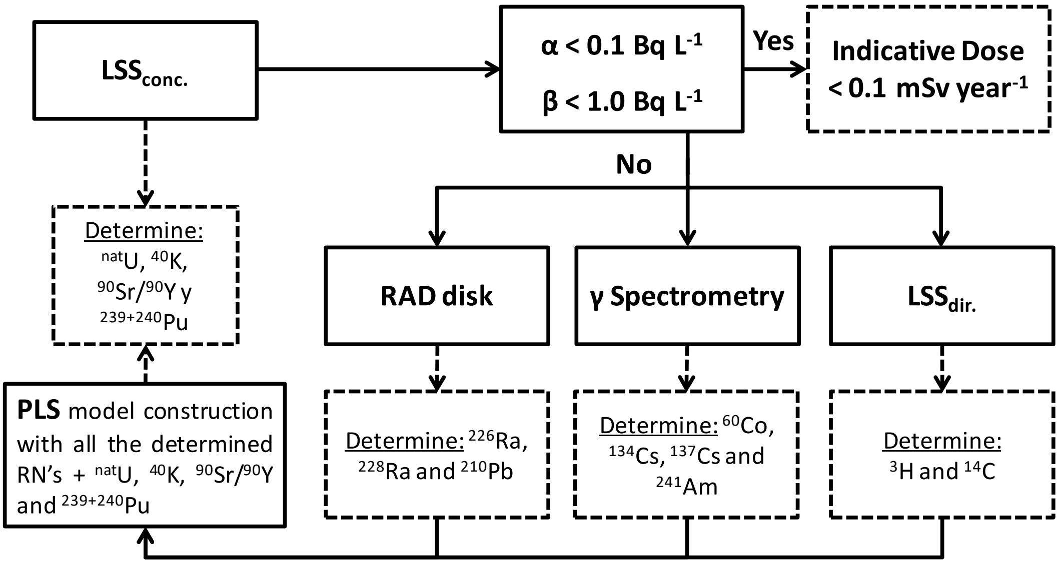

Fig. 1 Application flowchart of different methods and PLS model construction

The first step is perform the simultaneous determination of gross alpha and gross beta by $LSS_{conc.}$. If the results are below the 1.0 Bq L^{-1} and 0.1 Bq L^{-1} for gross beta and gross alpha respectively, it can be assume that the indicative dose is also below 0.1 mSv y^{-1} and hence, no further investigation is need. Otherwise, gamma spectrometry measurement, gross alpha and gross beta without evaporation and determination of ^{226}Ra, ^{228}Ra and ^{210}Pb using radium RAD disk were performed. With the information obtained with these determinations, the RN's that had to be included in the model for the determination of each sample were selected. From the alpha and beta spectra obtained from $LSS_{conc.}$ by means of the constructed model, ^{nat}U, ^{40}K, ^{90}Sr/^{90}Y and $^{239+240}$Pu can be determined.

Results

As an example, IAEA-TEL-2014-03 sample 3 quality control material was analyzed following the above described application flowchard. The results obtained are shown in Table 1. The technique used to determine each radionuclide is shown in shading.

Table 1. Activity determined and certified by the organizer and relative bias for IAEA-TEL-2014-03 sample 3 quality control material.

RN	**γ Spec. (Bq L^{-1})**	**RAD disk (Bq L^{-1})**	**$LSS_{conc.}$ (Bq L^{-1})**	**Certified (Bq L^{-1})**	**Rel. Bias (%)**
^{40}K	< 10.7	-	< 0.35	-	-
^{90}Sr	-	-	22.6 ± 2.3	24.5 ± 0.2	-8 %
^{134}Cs	23.2 ± 0.4	-	31.6 ± 3.2	26.3 ± 0.2	-12 %
^{137}Cs	19.3 ± 0.3	-	15.2 ± 1.5	19.6 ± 0.1	-2 %
^{226}Ra	-	19.4 ± 0.6	15.2 ± 1.5	17.9 ± 0.1	8 %
^{nat}U*	-	-	6.3 ± 0.6	5.48 ± 0.04	14 %
^{241}Am	21.3 ± 0.3	-	17.0 ± 1.7	20.0 ± 0.1	6 %

*Sum of ^{234}U and ^{238}U

The activity of γ-emitters (^{134}Cs, ^{137}Cs, ^{241}Am) was reported from the result of γ spectrometry, as well as the result of ^{226}Ra was reported from RAD disk analysis ^{90}Sr and ^{nat}U were reported from the spectrum of $LSS_{conc.}$ quantified by PLS model. The relative bias between the shading values and the certified by the organizer is in all the cases below 15 %.

The proposed strategy was proved useful for the determination of mixtures of several alpha and beta emitters. For this reason, it would be interesting to apply the proposed strategy, maybe including other methods, to further scenarios like NORM materials.

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Multiple Beta Spectrum Analysis Method Based on Spectrum Fitting

UkJae Lee, Jun Woo Bae, Hee Reyoung Kim

Ulsan National Institute of Science and Technology, Banyeon-ri, Eonyang-eup, Ulju-gun, Ulsan, 44919, Korea
Corresponding author's e-mail: dldnrwp@unist.ac.kr

Introduction

Beta spectrum has a wide range of energy distribution by beta-decay with neutrino[1]. When the sample containing mixed beta emitting nuclides is measured, it is difficult to distinguish each nuclide due to overlapping of spectrums[2]. In the present study, the analysis of the beta spectrum slope based on the curve fitting is suggested to separate the spectrums from the mixed sources.

Nuclide definition and separation

Most different beta nuclides has different maximum energy except for ^{129}I (E_{max} = 0.154 MeV) and ^{14}C(E_{max} = 0.156 MeV) which has overlapped beta spectrum each other as shown in Table 1. The maximum energy(E_{max}) of beta nuclides was compared by applying the concept of relative difference which was defined as (higher energy - lower energy) / higher energy * 100 between two nuclides. All beta nuclides represented in Table 1 showed a relative difference of more than 5%. Spectral separation among the mixed spectrums with different beta nuclides can be implemented from the comparison between maximum energy and energy less than 5% of the maximum energy.

Table 1 The maximum energy per major beta nuclides

	E_{max}(MeV)	difference	Relative difference		E_{max}(MeV)	difference	Relative difference
^{3}H	0.01859	0.04841	72.25	^{90}Sr	0.546	0.141	20.52
^{63}Ni	0.067	0.089476	57.18	^{85}Kr	0.687	1.022	59.80
^{14}C	0.156476	0.010524	6.30	^{32}P	1.709	0.573	25.11
^{35}S	0.167	0.127	43.19	^{90}Y	2.282	1.258	35.54
^{99}Tc	0.294	0.252	46.15	^{106}Rh	3.540		

When the A is the total count and CR_E is count ratio at energy E, the number of counts per specific energy C_E is defined as C_E=A×CR_E. The count rate of E_{max} and $E_{max95\%}$ channel is assumed to be generated by single radionuclide based on energy library of each nuclides. The E_{max} value is used for defining the kind of nuclides. It means the shape of spectrum can be defined by using E_{max}. $E_{max95\%}$ is used for the defining the magnitude of spectrum. The counts per specific energy, C_E, is assumed that it is proportional to product of count ratio and activity. Therefore, the count per specific energy is calculated by multiplying the value of ratio of $E_{max95\%}$ by count ratio per energy.

Referring to ICRU, the information about the count ratio per the energy of ^{32}P, ^{90}Y and ^{106}Rh was used for spectrum preparation [3]. Fig. 1 showed count ratio per the energy of each radionuclide where the blue, red and yellow were ^{32}P, ^{90}Y and ^{106}Rh, respectively.

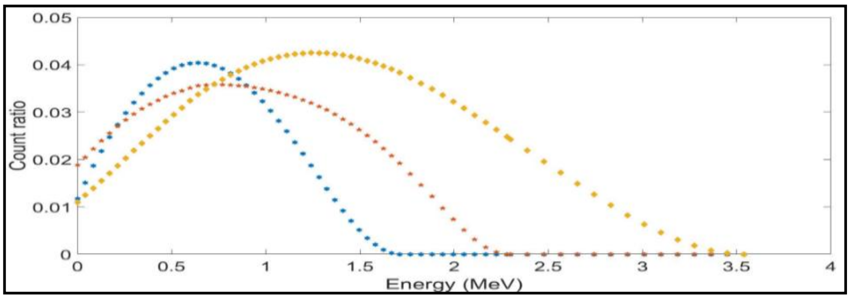

Fig.1. Prepared interpolated spectrum data for $\boldsymbol{^{32}P}$, $\boldsymbol{^{90}Y}$ and $\boldsymbol{^{106}Rh}$

Result of separation based on spectrum fitting method

The process of separation is as follows. First process is the definition of the ratio of ^{106}Rh by using the ratio of $E_{max95\%}$ value because ^{106}Rh has the highest maximum energy. Next step is multiplying the ratio of $E_{max95\%}$ value to the spectral function of ^{106}Rh which is based on count ratio per energy and deducting the value of counts per specific energy from mixed spectrum data. Then, mixed spectrum of ^{32}P, ^{90}Y remains. The same process is repeated for ^{90}Y. This way, the spectra of all the nuclides are separated from mixed beta spectrum and the each beta nuclide is recognized.

Table 2 represented the result of application of the proposed method to mixed spectrum data in various ratios of mixed nuclides. The value of 'Standard' is the total count of each nuclides based on ICRU and the value of 'Fitted' is the total count calculated by fitting function, where 'Error' is calculated by (Standard – Fitted)/Standard. The maximum error of 2.0138% was obtained and spectrum fitting method is expected to apply to the separation of multiple spectra including various kinds of beta nuclides.

Table 2 The application result of the proposed method to mixed spectrum data

	Mixed Ratio (^{32}P:^{90}Y:^{106}Rh = 1:1:1)			Mixed Ratio (^{32}P:^{90}Y:^{106}Rh = 1:2:2)		
	Standard	Fitted	Error (%)	Standard	Fitted	Error (%)
^{32}P	1	1.0053	0.5333	0.6	0.6121	2.0138
^{90}Y	1.3227	1.3169	0.4353	1.5872	1.5803	0.4353
^{106}Rh	1.8648	1.8667	0.1013	2.2378	2.2401	0.1013
	Mixed Ratio (^{32}P:^{90}Y:^{106}Rh = 2:2:1)			Mixed Ratio (^{32}P:^{90}Y:^{106}Rh = 2:1:2)		
	Standard	Fitted	Error (%)	Standard	Fitted	Error (%)
^{32}P	1.2	1.2051	0.4250	1.2	1.1988	0.0985
^{90}Y	1.5872	1.5843	0.3684	0.7936	0.7891	0.5690
^{106}Rh	1.1189	1.1200	0.1013	2.2378	2.2401	0.1013

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A method for ^{237}Np determination with liquid scintillation counting in the experiment of neptunium sorption onto bentonite

Li Ping, Liu Zhi, Guo Zhi-jun, Wu Wang-suo

Radiochemistry Laboratory, Lanzhou University, Lanzhou 730000, China

Among various analytical methods developed and used for the determination of neptunium (alpha spectroscopy, Neutron Activation Analysis (NAA), High Resolution γ-Spectroscopy (HRGS), Inductively Coupled Plasma Mass Spectrometry (ICP–MS), luminescence method and Liquid Scintillation Counting (LSC) etc.), The LSC method does not require extensive sample preparation and is very sensitive for alpha-decaying radionuclides such as ^{237}Np [1]. However, direct determination of ^{237}Np from its β-active daughter ^{233}Pa is difficult.

In this study, a method was set for the determination of ^{237}Np with liquid scintillation counting (LSC), the α/β discrimination is carried out with the function of pulse shape analysis (PSA) without a pure α emitter or a pure β emitter. Before we use the PSA technique to discriminate α/β, an approach was developed to set the optimum PSA by measuring a mixed α/β emitters sample and a background sample. The mathematic treatment of neptunium peak indicated that at the selected PSA-level (38) we detected ~86% of the total α emission [2]. It is suitable for the sample determination in the sorption experiments at this PSA-level with LSC. Moreover, we confirmed that, at m/V(bentonite) = 0~10g/L the suspension in sample did not influence LSC determination obviously. Thus, we could detect ^{237}Np samples containing bentonite suspension directly with LSC by setting PSA-level=38.

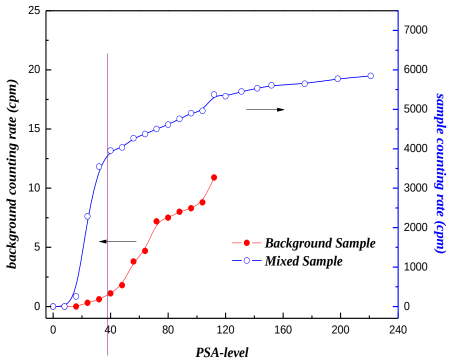

Fig.1 Count rate of a mixed sample and a background sample as a function of the PSA-level used.

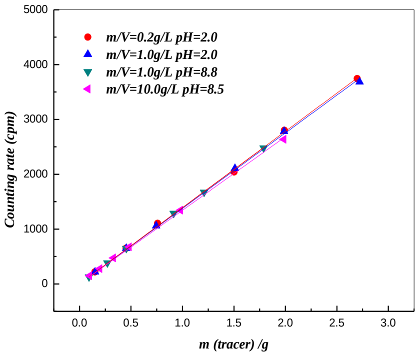

Fig. 2 Influence of bentonite on LSC measurement at a PSA-level=38.

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Tuesday

2 May 2017

Poster session-1

TEFLON VIALS FOR PRECISE C-14 IN BENZENE MEASUREMENTS BY LSC TECHNIQUE.

Vadim Skripkin[1] and Mykhailo Buzynnyi[2]

[1]Institute of Environmental Geochemistry NAS of Ukraine, 34a Palladin ave., 03680, Kyiv, Ukraine
[2]The O.M.Marzeev Institute of Public Health NAMS of Ukraine, 50, Popudrenko str., 02094, Kiev, Ukraine

Corresponding author: Michael Buzinny, mbuz@ukr.net

LSC equipment developers and manufacturers are using vials of Teflon due to their extreme performances - high counting efficiency and low background. As for measurements of radiocarbon, most applicable range of inner volume is of 0.3 to 7 ml. Practical aspects of Teflon vial for C14 use are benzene leakage and vial durability.

We had developed and well tested Teflon vials using Teflon and titanium as materials. Thinking about possible sorption, we avoid using of other materials. To control counting efficiency we avoid presence of any “dead zone” inside. Both, flask and cap are produced of Teflon, when metal base and pin valve are produced of titanium. Each vial uses spline Teflon to Teflon coupling and metal washers. We produce vials with volumes of: 0.8, 1.5, 2.5, 4.0 and 7.0 ml.

After filling with benzene sample, vial is covered with cap. Then walls of vial is lightly compressed to reduce air in vial, and after that pine valve located in cap finally closes inner volume of vial. This, last one, keeps vial under minor vacuum and minimizes benzene leakage.

Background count rate corresponds one of other Teflon vial of the same volume measured in similar counting condition. Counting efficiency, measured for some vials (2.5ml and 4.0ml) is higher because of better light collection achieved by optimal ratio of height and diameter of flask. As we had estimated, leakage of benzene is below 0.1% for one months of storage for series of several vials.

Evaluation of several liquid & solid scintillators in terms of pulse shape discrimination for fission studies

Jean Aupiais 1), Gilbert Bélier 1), Sylvain Vayre 1), Matthieu Hamel2)

1) CEA, DAM, DIF, F-91297 Arpajon France
2) CEA, DRT, LIST, F-91190 Saclay, France

Corresponding author's e-mail: jean.aupiais@cea.fr

The Liquid Scintillation Spectrometry technique has the advantages of providing very high detection efficiencies. In the context of fission studies, this advantage allows precisely quantifying the count losses. However, the fission studies with liquid or solid scintillators as detectors require an optimization of the Pulse Shape Discrimination (PSD) to separate fission fragments from other interfering events (protons, alpha and beta particles, neutrons). In particular, such scintillating mixtures have to present specific properties such as the capability of including high amount of actinides and to be able to preserve its fluorescence properties in case of high particles flux. We will present various tests of scintillating mixtures (including home made solid scintillators) in terms of PSD. Among them, the commercial scintillators EJ-309 presents good PSD properties and higher fluorescent yield compared to a DiIsopropylNaphthalene-based scintillating mixture (DIN) (see Figure 1). Among liquid scintillators, a special attention was paid to free-hydrogen liquid-based scintillators for improving the separation from the fission fragments. The resistance of a detector against the irradiation is also an important criterion. Several mixtures containing about 0.3 mg of 235U were tested in terms of robustness against high flux neutron irradiation: DIN, EJ-309, Alphaex^{TM} and EJ-313. An example of PSD is depicted in the Figure 2. We observed no decrease of the light emission yield of EJ309-based scintillating mixture whereas a decrease of 8.8 % (for 2.286 10^{11} fissions), 34.1 % (5.182 10^{10} fissions), and 57.9 % (2.177 10^{10} fissions) is noticed for DIN, EJ-3013 and Alphaex, respectively.

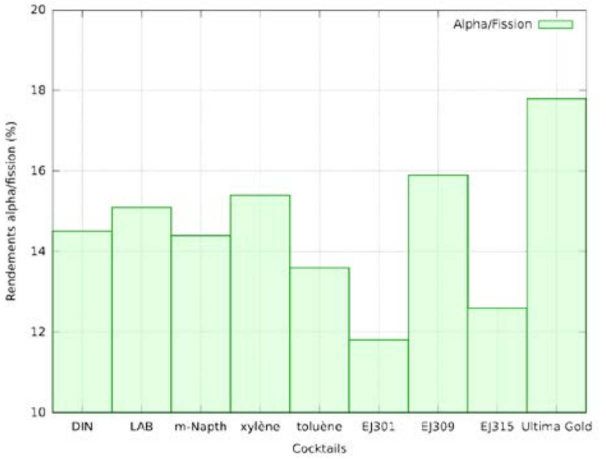

Fig. 1 Variation of the light efficiency ratio alpha/fission for several solvents.

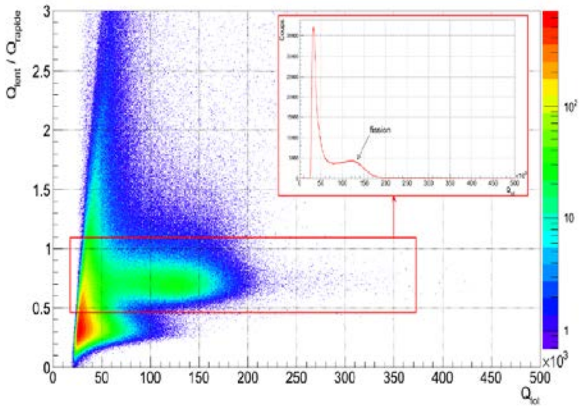

Fig. 2 Event histogramm for DIN.

LSC Counting - Do's and Don'ts

James Thomson

Meridian Biotechnologies Ltd., Unit 1 Millennium Way, Chesterfield,Derbyshire, S41 8ND

Email: james@meridian-biotech.com

The use of LSC cocktails is fraught with many pitfalls for the unwary and this paper will hopefully educate both the experienced and inexperienced in the art of sample preparation and counting. The sequence of events from sampling, through sample preparation and on to LSC analysis is covered.
Suggestions and advice is given on sampling techniques; correct cocktail selection to overcome problems such as capacity, stability, colour formation and luminescence interferences and finally recommendations on how to keep your LSC counter in optimal condition.

Determination of biogenic component in liquid fuels by the ^{14}C method and direct LSC measurement

Ines Krajcar Bronić, Jadranka Barešić

Ruđer Bošković Institute, Bijenička c. 54, 10000 Zagreb, Croatia

Biogenic materials itself or as blends to fossil fuels are presently often used for energy production and/or transport. One of the methods for determination of the fraction of the biogenic component in any type of fuel is the ^{14}C method, which is based on different content of ^{14}C in biogenic and in fossil component: while the biogenic component reflects the modern atmospheric ^{14}C activity, no ^{14}C is present in fossil fuels. A technique of direct LSC measurement of the ^{14}C content in liquid fuels is simple and fast because it does not require any sample pre-treatment. However, its main disadvantage is caused by different liquid colours that change quenching properties and measurement efficiency. Recently we proposed a new evaluation technique that takes advantage of different quenching properties of various liquids of different colours. By utilizing various modern organic liquids (various brands of domestic oils, benzene, ethylene) we constructed the modern calibration curve that relates their count rates and SQP (standard quench parameter) values. A background calibration curve was constructed by using various ^{14}C-free liquids. All samples were prepared in low-potassium glass vials with the UltimaGold F scintillator (10 mL of a sample and 10 mL of UGF). Measurement was performed by LSC Quantulus 1220 and spectra were evaluated in the window 124 – 570 channels. We suggest that the data evaluation method could be used for determining the biogenic fraction in various types of organic liquids, including liquid fuels of unknown chemical composition. Here we present further studies of the applicability of the proposed method. We have prepared mixtures of fossil fuels and some biogenic liquids in a nominal concentration range of the biogenic component from 0 % to 100 %. Test will be made to check if the proposed method depends on the qualitative composition of the mixture, i.e., on the fossil matrix or the biogenic additive type. The sensitivity and limitations of the method will be discussed.

Development of the ‘WILMA’ LSC platform for the online monitoring of radionuclides in air and water

Tom Deakin [1,2], Daniel M. K. Schofield [1]

[1] LabLogic Systems Ltd., Paradigm House, 3 Melbourne Avenue, Broomhill, Sheffield, S10 2QJ. UK.

[2] The University of Sheffield, Department of Physics and Astronomy, Hicks Building, Hounsfield Road, Sheffield, S3 7RH. UK.

Corresponding author’s e-mail: tdeakin@lablogic.com

The regular monitoring of air and water samples in and around civil nuclear sites (both active and during the process of decommisioning) is of key importance, both in terms of ensuring that any possible contamination resulting from leaks or spills is detected in a timely fashion and to satisfy the regulatory and environmental demands of government bodies such as the UK Environment Agency (EA) and Office for Nuclear Regulation (ONR). Currently, the periodic manual sampling, analysis and reporting process is resource heavy in terms of both the cost and time (sometime weeks) required to obtain, analyse and collate the data arising from measurements of manually-acquired samples.

Here, we present on the design, development and evaluation of an innovative radioactivity monitoring system, based on LabLogic’s ‘WILMA’, a system developed in conjunction originally with the US EPA [1]. The system combines a low-level, liquid scintillation-based radioactivity detector and an automated fluid handling system that enables small samples (mL) to be collected from a supply point and mixed with scintillation cocktail. The mixed sample is analysed in a measurement chamber, where the radioactive count rate of the water sample is obtained, along with an MCA spectrum, to determine the elemental origin of any possible contamination. Following automated flushing and washing cycles, the complete measurement cycle can then be repeated to give up to four analyses per hour, over periods in excess of 30 days without human intervention. If the count rate for a particular sample exceeds a pre-determined threshold limit, an alarm is raised by the system that results in an e-mail being sent to an operative who can then investigate the issue further. The potentially contaminated sample is diverted to a separate container automatically, which can be removed for further analysis.

Recent work has focused on the system development for a number of different applications in the civil nuclear and environmental monitoring sectors:

1) Field-based groundwater analysis funded via an Innovate UK feasibility study R&D grant and undertaken in conjuction with the Land Quality team at Sellafield Ltd. in the UK. This extension of the WILMA programme sought to develop a system that aims to increase the throughput of groundwater sampling and analysis to provide full radiometric characterisation in real-time, as well as temporal trend analysis. The novel development described in this work focuses on the addition of features to satisfy the rigorous demands required for groundwater monitoring, including: direct

sample acquisition from boreholes with sampling depths up to 50 m; sample pre-treatment and filtration; improvements to the lower limit of detection to enable more accurate quantification of key environmental radionuclides and a specific software package to automate the data acquisition and analysis processes. In addition, the requirements for remote operation, requiring high reliability, low power consumption and secure wireless data transfer, as well as resistance to fluctuations in the ambient environment over several weeks' continuous operation, have been factored into the design. The prototype system has now been developed and a comprehensive evaluation programme will take begin at key borehole locations on the Sellafield site during the spring of 2017, the preliminary results from laboratory testing with representative groundwater samples will be presented here (Figure 1).

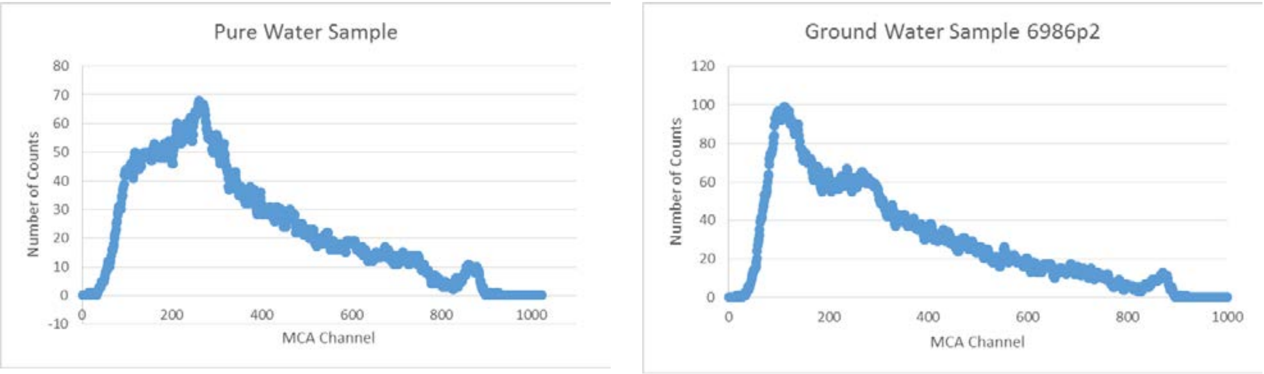

Fig. 1 TDCR LSC spectra for pure water control and borehole groundwater samples.

2) Integration of the fluid handling and LSC modules with an automated air sampler and bubbler system for ^{3}H and ^{14}C analysis in air samples (Figure 2). The development of the WILMA platform for this application has taken the form of two strands: integration of a ^{3}H air sampler and bubbler for waste repository sample collection and analysis in collaboration with the National Physical Laboratory as part of the EMRP Euramet 'MetroDECOM' programme [2], where a complete system is undergoing characterisation testing; and development and supply of four complete systems for ^{3}H stack monitoring at a site being decomissioned in Italy. Results from both of these case studies will be presented here.

Fig. 2 Integrated tritium air sampler/bubbler and LSC system.

3) On-line monitoring of NORM water samples as part of the water treatment process at a site in Western Germany. As a further demonstration of the flexibility of the WILMA system, a small-footprint ‘benchtop’ WILMA system was demonstrated at a German water treatment works during the Summer of 2016. For this application, the focus was on the characterisation of enhanced levels of natural Rn and U over extended periods. The results and implications of this particular study (Figure 3) will be discussed.

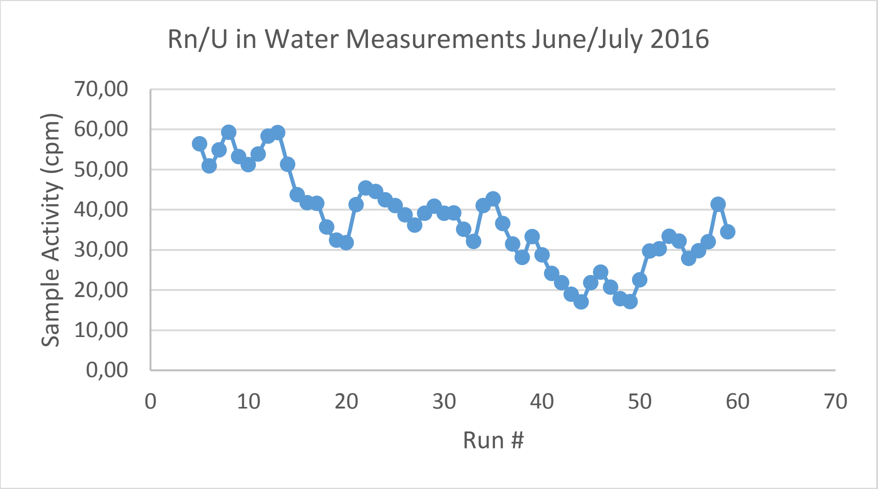

Fig. 3 Temporal data trend of background-corrected sample activity for Rn/U in water samples measured over a two-week period in Western Germany (5 hour count, 6 hourly sampling).

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A new compact TDCR-Cerenkov counter for the online measurement of ^{56}Mn activity

Benoit Sabot, Philippe Cassette , Cheick Thiam

CEA, LIST, Laboratoire National Henri Becquerel (LNE-LNHB), Bât. 602 PC111, CEA-Saclay 91191 Gif-sur-Yvette Cedex, France

Corresponding author's e-mail: benoit.sabot@cea.fr

The primary measurement method of emission rate of neutron sources is the manganese bath. In this method, the neutron source is placed in the center of a sphere full of manganese sulfate solution, activating manganese and, provided a detailed calculation of neutron interactions with the bath, the emission rate can be deduced from the activity of ^{56}Mn in the solution. This activity is generally measured by gamma spectrometry but this imposes the calibration of the bath, using a reference ^{56}Mn solution. As the half-life of ^{56}Mn is short (2.6 h), this requires the use of a high-activity source, made by irradiation of manganese in a nuclear reactor. In order to avoid such difficulty, we developed in the past a new online primary measurement method, based on Cerenkov-gamma coincidence counting [1]. One drawback of this method is that, because of the geometry of the counter, the counting rate of the gamma detector is quite low, imposing long measurements to get good counting statistics. Here we describe a new approach of such online primary measurement system, but with a higher overall detection efficiency, based on a 4-π Cerenkov counter. The manganese solution is circulating in a transparent quartz cell, facing three compact photomultiplier tubes (PMT) placed 120° apart (Figure 1).

Fig. 1 View of the TDCR-Cerenkov optical chamber

The interaction of high-energy beta electrons with the solution and the quartz cell induces the emission of Cerenkov light. This light is detected by the three PMT and fast double and triple coincidence are recorded, allowing the determination of the activity of the solution using the Triple to Double Coincidence Ratio (TDCR) model. This model is an adaptation of the classical liquid scintillation TDCR method [2], by considering a Cerenkov light emission model and a parameter taking into account the anisotropy of the light emission. The optical chamber and the external housing of the counter are made in PLA by 3D printing. The overall precision of such printing process is well suitable for this application and allows a very flexible optimization of the counter.

The anodes of the PMT's are connected through fast preamplifiers and constant fraction discriminators to a dedicated FPGA electronic module developed at LNHB. This module allows the recording of individual, double and triple coincident pulses, together with timing signals from live-time clocks. The module is connected by an USB link to a personal computer. The activity of the solution in the cell is calculated from the coincidence counting rates, using the Cerenkov-TDCR model initially developed by Kossert [3] and modified at LNHB. More details on this model are given in another paper submitted to this conference [4].
The paper will give details on the conception and optimization of this new TDCR-Cerenkov counter. It will also report the first measurement results obtained during the measurement of the neutron emission rate of an AmBe source, in comparison with off-line activity measurements. Advantages and drawbacks of the TDCR—Cerenkov counter, versus the Cerenkov-gamma coincidence measurement system are also discussed.

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Improved counting efficiency determination by core function modelling in triple to double coincidence ration counter

Sini Raitanen [1),]Timo Oikari [1)], Risto Juvonen [1)], Ville Haaslahti [1)]

[1)] Hidex Oy, Lemminkäisenkatu 62, 20520 Turku, Finland

Corresponding author's e-mail: ville.haaslahti@hidex.com

Triple to double coincidence ratio with liquid scintillation counting is an established method in radionuclide metrology. More recently a commercial TDCR counter was introduced where TDCR was used as direct counting efficiency determination factor. Now the authors present an improved mathematical equation modelling for counting efficiency determination using triple to double coincidence factor.

Ratio of real counting efficiency was compared to direct TDCR method, external ^{152}Eu standard method based on spectral end point (QPE) and core function modelling improved TDCR method.

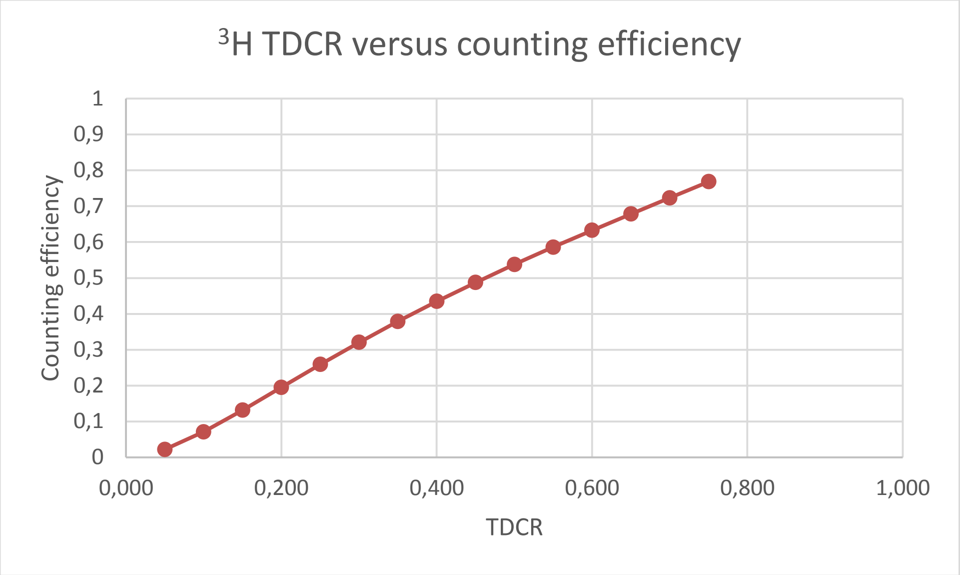

Fig. 1 3H TDCR versus the modelled counting effiency

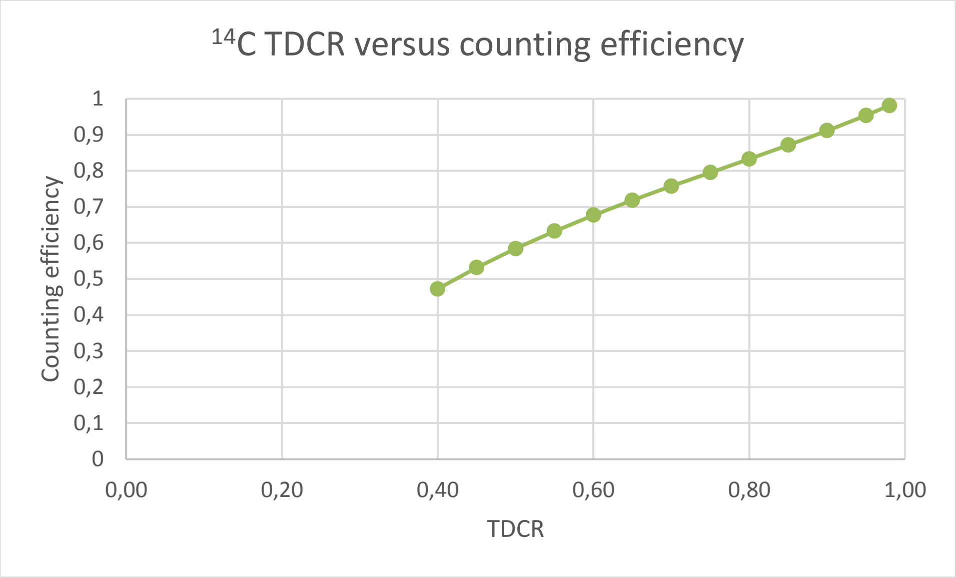

Fig. 2 14C TDCR versus the modelled counting efficiency

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Luminescence free liquid scintillation counting method

Timo Oikari [1)], Ville Haaslahti [1)], Sini Raitanen [1)], Risto Juvonen [1)]

[1)] Hidex Oy, Mustionkatu 2, 27800 Turku, Finland

Corresponding author's e-mail: risto.juvonen@hidex.com

Luminescence is one of the major interferences in scintillation counting causing increase of the background and variation in the results. Regardless of the origin, luminescence is low energetic and yields in emission of single photons. As such it's the lowest energetic true photon emitting phenomena in the samples.

As standard procedure, scientists are waiting for hour to several days for luminescence decay. One can also subtract Chemiluminescence CPM from the results but as the luminescence count rate is relatively low and variable this may yield in high uncertainty of the results. Very often samples are counted repeatedly and outliers are removed. One can also set a luminescence free energy window above luminescence region. With conventional counters this works relatively well with higher energy isotopes like 14C but yields in too low counting efficiency for 3H. With triple coincidence counters Hidex 300 SL and 600 SL, the counting efficiency is higher allowing luminescence free methods also for 3H in water. The feature is called Luminescence Free Counting Mode.

Figures 1 and 2 illustrate the interference of luminescence in all beta counts (including double and triple betas) and triple beta counts only. Luminescence do not interfere with triple beta counts even in these highly luminescent 14C samples.

Fig. 1 Spectra of all betas (black, high luminescence peak) and triple betas only (purple, no luminescence)

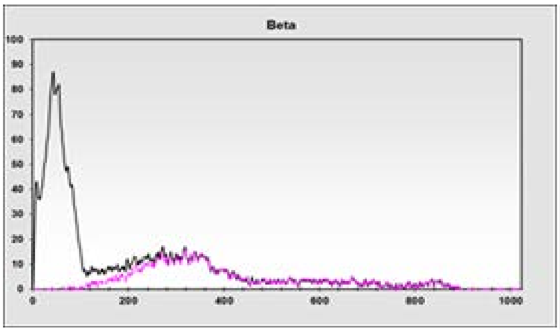
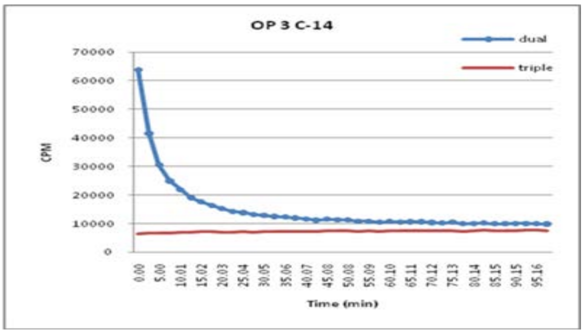


Fig. 2 Luminescence decay of double betas (blue, luminescence interference) and triple betas only (red, no luminescence)



Conventional methods (decay of luminescence and removal of outliers) were compared to luminescence free counting method for measurement of 3H in water (Table 1).

Table 1. 3H performance comparison of 300 SL to Wallac Quantulus

- 8+12 ml, 4 h measurement time, 3 s uncertainty
- Samples were stabilized for 20 h before the start of the measurement

	Normal mode	Lum free mode	Quantulus
Bg	4.7 CPM	2.8 CPM	0.5 CPM
Eff	37 %	24 %	25 %
Ld	2.4 Bq/L	2.8 Bq/L	1.7 Bq/L
Outliers	High risk	Low risk	High risk

As conclusion the counting efficicncy is reduced but the detection limit is only slightly higher than that in normal count mode due to lower background count rate. As an advantage luminescence free mode allows more or less immediate start of the measurement after sample preparation and there is no need to detect and exclude outliers after the measurement. This yields in shorter measurement times in total and higher reliability of the results as the risk for outliers is reduced.

Development of a method to measure filter sample using plastic scintillator

Yuka Kato[1], Haruka Minowa[2], Yoshimune Ogata[3]

[1] Measuring Systems Engineering Dept., Hitachi, Ltd., 6-22-1 Mure Mitaka-shi Tokyo, 181-8622, Japan
[2] Radioisotope Research Facility, Jikei Univ., 3-25-8 Nishi-shimbashi, Minato-ku Tokyo 105-8461, Japan
[3] Radioisotope Research Center, Medical Branch Nagoya University, 65 Tsurumai-cho, Showa-ku Nagoya-city 466-8550, Japan

Corresponding author’s e-mail: yuka.kato.ot@hitachi.com

A simple, quick and safety analysis method to measure radiostrontium in seawater is tried to be developed. The conventional method to analyze radiostrontium is time-consuming complicated procedures with deleterious substances. And the sample is measured via a low-background 2π gas flow counter or a liquid scintillation counter. Latter method generate organic liquid waste.

The chemical separation of radiostrontium in seawater was performed by collecting it on a filter as a carbonate precipitate by means of a cation exchange method. Details of the chemical separation procedure is described in another study of ours.

In this study, a new method to measure a filter adhering radiostrontium is described. The filter with radiostrontium was sealed by polyethylene film to prevent contamination. Plastic scintillator (EJ-200, Eljen Technology) was formed to cylindrical shape with 47 mm in diameter and 15 mm in thickness, because the thickness of 15 mm is enough to absorb the high energy beta-rays emitted from ^{90}Y. An 145-mL plastic bottle (POLYVIALS^{R} 145, Zinsser Analytic GmbH) was horizontally cut to two pieces. An acrylic pipe was put in the bottom piece and a plastic scintillator was on it. Then the filter with sample was on it and another plastic scintillator was on it to sandwich the filter. Finally, upper piece of the bottle was put on them to cover them. The acrylic pipe was a spacer to

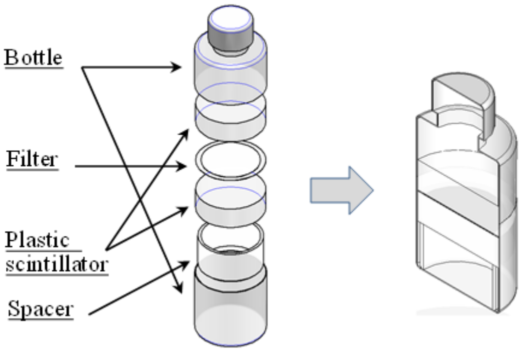


Fig. 1 Structure of the plastic scintillation bottle.

Fig. 2 The liquid scintillation analyzer.

make the height of the filter to the center of the sensitive position of PMTs. The assembly of the plastic scintillation bottle is illustrated in Fig. 1. The bottle containing the filter sample was measured with a low-background liquid scintillation analyzer (AccuFLEX LSC LSC-LB7, Hitachi, Ltd.) shown in Fig. 2. The liquid scintillation analyzer equipped with three PMTs and guard detectors. This system enables to measure bottles up to 145 mL in volume. In addition, it equipped with a new BG optimization function for reducing BG.

To estimate the counting efficiency, certain amount of ^{90}Sr standard solution was drop-deposited on a filter as same as the one used at the chemical procedure, and dried up at room temperature. The filer was laminated with thin polyethylene film, mounted on the plastic scintillator bottle, and measured with AccuFLEX LSC LSC-LB7.

The counting efficiency was estimated to 75% at ^{90}Sr-^{90}Y radiochemical equilibrium. The background was 0.2 cps. Then the minimum detectable concentration (MDC) toward the counting time of this method started with 200 mL of seawater is calculated as in Fig. 3. The MDC for 50 minutes counting was estimated as 0.1 Bq L^{-1}.

The MDC is sufficient to estimate the radiostrontium concentration, because it is one hundredth of the guidance level of radiostrontium concentration provided by WHO, 10 Bq L^{-1} 1).

In this study, a filter adhering the radiostrontium as precipitate was used. There is another method to collect strontium by means of solid phase extraction disk (Empore^{TM} Strontium RadDisk, 3M). Also the disk can be sealed with polyethylene film and it can be mounted on our plastic scintillation bottle. The method was experimentally performed by us and was successfully applied.

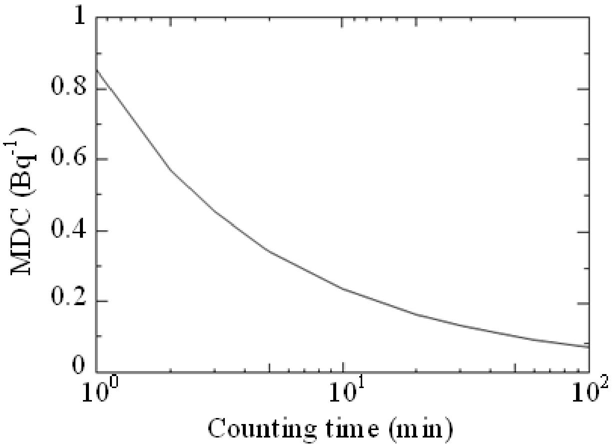


Fig. 3 The MDC of this method toward the counting time.

The new counting method using the plastic scintillation bottle can be applied to measure other beta nuclides emitting relative high energy beta-rays, such as ^{32}P, etc2). One of the advantages of this method is not to generate the radioactive organic liquid, which brings troublesome disposal. Furthermore, when using a gamma-ray sensitive solid scintillator on behalf of the plastic scintillator, one can measure gamma emitter. Single liquid scintillation analyzer can be used for plastic scintillation counting and gamma-ray counting as well as liquid scintillation counting.

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Approach to the uncertainty estimation of the ^{89}Sr/^{90}Sr determination by LSC

Eva Kabai (1)

(1) Federal Office for Radiation Protection, Ingolstaedter Landstr. 1, 85764 Oberschleissheim, Germany

Corresponding author's e-mail: ekabai@bfs.de

The uncertainty of the liquid scintillation measurement of ^{89}Sr/^{90}Sr in different environmental and food samples was evaluated. The complete uncertainty budget was calculated using the tool UncertRadio, including the spectral evaluation procedure, the determination of the counting efficiency, the quenching curve, the chemical yield and the counting itself. The approach allows to determine the contribution of each parameter to the total uncertainty for both isotopes ^{89}Sr and ^{90}Sr individually. Table 1 summarizes the results for the most critical parameters, using a radiochemical analysis of ^{90}Sr in milk as example. Additionally, details are given for the calculation of the minimum detectable activity and the decision threshold applying the same approach.

Table 1 Example of the results of the uncertainty analysis for ^{90}Sr in milk

Symbol	**Description**	**Unit**	**Parameter value**	**Parameter standard uncertainty**	**Relative contribution to uncertainty (%)**
eta	Chemical yield	-	9.23E-01	1.85E-02	30.5
m	Sample mass	kg	1.46E-01	1.46E-03	7.6
f_A	Dry/fresh weight ratio	-	9.79E-01	9.79E-03	7.6
N_b	Number of counts (sample)	imp	1.69E+04	3.37E+02	31.0
k_b	Correction factor (sample)	-	9.39E-01	9.39E-03	7.8
$A_{\text{Sr tracer}}$	Sr tracer activity	Bq	2.00E+01	2.00E-01	7.6
k_{STD}	Correction factor (standard)	-	9.39E-01	9.39E-03	7.6

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Assignment of reference value for ^{90}Sr in black tea powder used as TAEK proficiency test sample

Gülten ÖZÇAYAN, Nazife ASLAN

Turkish Atomic Energy Authority, Sarayköy Nuclear Research and Training Center,
Radiation Metrology Division, 06983 Saray, Kazan, Ankara-TURKEY

Corresponding author’s e-mail: gulten.kahraman@taek.gov.tr

Because of the fact that radioactivity measurements play an important role in radiological protection and human health, it is essential to obtain accurate and reliable radioactivity values. Proficiency tests (PTs) are one of the tools for regularly assessing the quality of the analytical data produced by different laboratories. For that reason, second proficiency test on ^{137}Cs, ^{40}K and ^{90}Sr massic radioactivity measurements in Turkish black tea powder samples have been organized by Sarayköy Nuclear Research and Training Center (SANAEM) of Turkish Atomic Energy Authority (TAEK). The present study summarizes the method used for assigning the reference value and the evaluation of ^{90}Sr results reported by the participant laboratories. The method described in this work was based on the radiochemical separation of ^{90}Sr using strontium resin and measurement with Perkin Elmer 1220 Quantulus ultra low-level liquid scintillation counter. The average activity of ^{90}Sr was measured as 155 ± 16 Bq kg^{-1} dry matter (k=2) by liquid scintillation efficiency tracing (CIEMAT/NIST) method. The minimum detectable activity concentration for ^{90}Sr was calculated between 1.42 Bqkg^{-1} - 1.59 Bqkg^{-1} using 60 min. counting time, a counting efficiency of 99 % and chemical recovery of 78 %. Uncertainties arising from counting statistics, background, weighing, halflife, chemical recovery, efficiency and homogeneity were taken into account.

Only 5 out of 18 laboratories reported ^{90}Sr results due to the lack of source preparation setups or measurement systems. Evaluation of the reported results will be discussed in detail in the presentation. TAEK SANAEM plans to continue proficiency tests periodically to maintain and control quality assurance for the local laboratories.

Standardization of ^{170}Tm with CIEMAT/NIST efficiency tracing, TDCR and gamma spectrometric methods

Emin Yeltepe 1), Haluk Yücel 2), Karsten Kossert 3)

1) Turkish Atomic Energy Authority - Sarayköy Nuclear Research and Training Center
Saray M. Atom Cd. No: 27 Kazan Ankara 06981 Turkey
2) Ankara University Institute of Nuclear Sciences
Tandogan Cankaya Ankara 06100 Turkey
3) Physikalisch-Technische Bundesanstalt (PTB)
Bundesallee 100, Braunschweig 38116, Germany

Corresponding author's e-mail: emin.yeltepe@taek.gov.tr

In nuclear medicine, beta emitting radionuclides with high or medium energies are often used for therapy. For brachytherapy, pure beta emitters and beta emitters with low gamma emission probabilities are particularly preferred to ensure that the major portion of the dose is transferred to the affected region. Among these radionuclides, thullium-170 (^{170}Tm) has garnered interest for use in pulsed dose rate brachytherapy (PDRB) (Ballester et al., 2010 and Baltas et al., 2007) and bone palliative therapy (Das T., 2009). The high energy of the emitted betas ($E_{\beta,max}$ = 883.7 keV 18.3% and $E_{\beta,max}$ = 968.0 keV 81.6%), relatively long half-life (127.8(6) d) and low energy and intensity of the gamma rays (84.25 keV 2.48%) makes this radionuclide promising for future use in clinics. Literature survey shows that standardization studies of ^{170}Tm activity is scarce. This work presents the outcome of various activity standardization methods for ^{170}Tm which make measurements with low uncertainties possible. Such measurements are an important prerequisite for potential application of this isotope.

In this study, several mgs of high-purity (99.9% pure) Tm_2O_3 in a flame sealed-ampoule were irradiated in a high thermal neutron flux (at about 10^{12} n.cm^{-2}.s^{-1}) of Istanbul Technical University's 250 kW TRIGA Mark II Research Reactor for 5 minutes. The irradiated oxide sample was then dissolved by adding 2 mL HNO_3 and 1 mL HCl acids in an ultrasonic bath. A small part of the solution was taken for dilution. The ^{170}Tm produced via ^{169}Tm(n, γ)^{170}Tm reaction in the sample was diluted twice to reduce the activity level of about a couple hundreds of Bq in the counting vials. The rough activity values were estimated by assuming a counting efficiency of 100%. A total of 14 polyethylene vials were measured for 30 min periods. Measurements were performed in two Perkin Elmer Quantulus 1220 counters in AU-NBE and TAEK-SANAEM for comparison and a Hidex 300 SL TDCR counter (Metrological Version). CIEMAT/NIST efficiency tracing method was employed to determine the radionuclide efficiency for all three counting systems. To this end, tritium tablets with known activities were put into polyethylene vials with 15 mL of Ultima Gold® plus 1mL de-ionized water and 0 µl to 170 µL nitromethane was added as quenching agent to plot the ^{3}H efficiency versus SQP(E) parameter curve. CN2005 and MICELLE2 codes were used to calculate the radionuclide efficiency against ^{3}H efficiency. The TDCR method was also used for the standardization. The MICELLE2 code was used to plot the TDCR value versus double/triple counting efficiency.

Gamma ray spectrometry with a well-type, HPGe detector (44.8% relative efficiency, Canberra model GCW4023) was used as third activity determination method. The activities of were determined from the count rate of the 84.25 keV photopeak (P_γ=2.48(9) %) of ^{170}Tm for which the efficiency value was calculated with PENELOPE Monte Carlo code. Experimental efficiency determination around this energy was problematic due to the lack of suitable calibration sources in the laboratory, a plethora of X-rays in the calibration spectrum in this region due to uranium and thorium and fast change of the efficiency in this region (the knee of the efficiency curve is around 120 keV).

Table 1: Preliminary results of the measurements

Method	**Activity concentration (kBq/g) (ref. date 15.12.2015 15:30 UTC)**	**Uncertainty (1 σ confidence level) (kBq/g)**	**Relative uncertainty (%) (1 σ confidence level)**
CIEMAT-NIST (AU-NBE, QUANTULUS)	241.6	0.92	0.38
CIEMAT-NIST (TAEK-SANAEM, QUANTULUS)	242.7	0.93	0.38
CIEMAT-NIST (TAEK-SANAEM, HIDEX)	244.6	0.84	0.34
TDCR (TAEK-SANAEM, HIDEX)	245.6	0.74	0.30
HPGe Well	248.5	12.1	4.87

The relative standard uncertainty of the gamma spectrometric method (4.87% at 1 σ confidence level) is much higher than the uncertainty of the liquid scintillation counting methods. The major uncertainty contributions for gamma spectrometry are due to the uncertainty of the emission probability of the gamma ray and the uncertainty of the efficiency. Further studies may be conducted to produce more reliable nuclear data for ^{170}Tm, in particular the gamma-ray emission probability could be determined with better accuracy. Both CIEMAT/NIST and TDCR methods are quite robust for the standardization of ^{170}Tm because of the high energy of the betas and low gamma and X-ray emission. The methods and the uncertainty budgets will be discussed in detail in the presentation.

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Correlation in the Application of the Triple-to-Double Coincidence Method with Unequal Photomultiplier Tube Efficiencies

B. E. Zimmerman [1)]

[1)] Physical Measurement Laboratory, National Institute of Standards and Technology, 100 Bureau Dr., Gaithersburg, MD, 20899-8462 USA

Corresponding author's e-mail: bez@nist.gov

In the simplest application of the Triple-to-Double Coincidence Ratio (TDCR) method [1], where the photomultiplier (PMT) efficiencies for the three counting channels in a three-detector system are equal, the photon coincidence counting rates of triples, (R_T), and logical sum of doubles (R_D) are recorded so that a (usually linear or second-order polynomial) relationship can be established between the logical sum of doubles efficiency, ε_D, and a parameter $K = R_T/R_D$ through the use of a calculational model. For this case, the relative uncertainty on the activity of the sample being measured due to the uncertainties on R_T and R_D can be readily evaluated, even though the quantities are strongly correlated, because the measurement equation linking the input quantities to the measurand (i.e., the activity of the source) can be written as a relatively simple equation. For the linear case, the relative uncertainty on K is calculated according to the error propagation law as [2]:

$$\frac{u_K}{K} = \sqrt{\frac{u_T^2}{R_T^2} + \frac{u_D^2}{R_D^2} - 2\frac{u_{T,D}^2}{R_T R_D}} \qquad (1)$$

where u_D, u_T are the variances on the logical sum of doubles and triples rates, respectively, and $u_{T,D}$ is the covariance of R_T and R_D.

When the PMT efficiencies are not equal, the situation becomes much more complicated. In this case, it is necessary to also collect the counting rates in the three sets of doubles channels (R_{AB}, R_{BC}, and R_{AC}). These counting rates, along with R_T and R_D, all of which are strongly correlated, are used with the same type of model as discussed above to solve a set of three simultaneous equations with three unknowns in order to determine the individual PMT efficiencies. In this case, it is impossible to write the measurement equation in a closed form, making uncertainty analysis extremely difficult. While Monte Carlo methods can be used to estimate the individual variances, accounting for the covariances is a challenging problem that is rarely addressed.

One method that can be applied is to use a linear transform to create a new set of de-correlated variables that can be used in the efficiency (and uncertainty) calculations. One such transform is the Mahalanobis Transform [3]. Given a data matrix $\boldsymbol{A}$, in which the i rows represent repeated measurements of the five TDCR variables (R_{AB}, R_{BC}, R_{AC}, R_T, and R_D), a 5x5 covariance matrix, $\boldsymbol{S}$ can be calculated. A new matrix, $\boldsymbol{Z}$, can be calculated from

$$\boldsymbol{Z} = \boldsymbol{S}^{-1/2} \boldsymbol{A} (\boldsymbol{I} - (1/\mathrm{i})\, \boldsymbol{1}\, \boldsymbol{1}^{\mathrm{T}}) \qquad (2)$$

where $\boldsymbol{S}^{-1/2}$ is the square root of the inverse of $\boldsymbol{S}$, $\boldsymbol{I}$ is the identity matrix, and $\boldsymbol{1}$ is a row vector consisting of all ones, with dimension i. The term is parenthesis is called the *centering matrix* that when multiplied by $\boldsymbol{A}$ gives an i x 5 matrix in which the mean for each of the 5 TDCR variables is subtracted from each of the column elements. The matrix $\boldsymbol{Z}$ contains a new set of TDCR variables that are uncorrelated, with a mean of zero and unit variance. By reversing the centering process, an i x5 matrix of uncorrelated variables with the same mean as the original matrix, but which are now uncorrelated, is produced. These new variables can then be used in the usual TDCR analysis codes to calculate the individual PMT detection efficiencies, and hence determine ε_D.

For most cases, removing the correlations in the data results in very small differences (< 0.1 %) in the calculated activities. However, the differences in the variances on the calculated activities can be quite substantial. This paper will discuss the practical application of de-correlation transforms in the analysis of TDCR data using examples of data acquired using the National Institute of Standards and Technology (NIST) TDCR system. The examples will use code written in-house with MATLAB, but the method is also easily implemented in other languages, such as Mathematica, R, and Python.

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The Measurement of ^{160}Tb and ^{161}Tb by Liquid Scintillation Counting

J. Jiang and A. Davies

AWE plc, Aldermaston, Reading, RG7 4PR, UK

Corresponding author's e-mail: jun.jiang@awe.co.uk

Fission products and activation products in a Nuclear Forensics sample from a nuclear event are required to be separated, purified and quantified. The results should be provided accurately and promptly. ^{160}Tb and ^{161}Tb are activation and fission products respectively. They are important radionuclides to measure when analysing a Nuclear Forensics sample. The determination of ^{161}Tb is problematic due to its very low fission yield, short half-life, and difficult separation from other lanthanides. The analysis of ^{161}Tb was investigated in a previous study [1]. ^{160}Tb is the activation product of stable terbium isotope – ^{159}Tb. The measurement of both ^{160}Tb and ^{161}Tb in a sample is difficult because they cannot be chemically separated.

In this study, the analytical method for measurement of ^{160}Tb and ^{161}Tb was developed and applied to the analysis of two irradiated uranium samples during recent inter-laboratory comparison exercises. Lanthanides including terbium were separated from actinides, other fission and activation products using AG1x8 anion exchange resin (BioRad). Terbium was then separated and purified from other lanthanides using Triskem LN extraction chromatography resin [2]. The purified fraction containing ^{160}Tb and ^{161}Tb was repeatedly measured for consecutive days over a period of time using A Perkin-Elmer 1220 Quantulus Liquid Scintillation Counter. Program ”gnuplot” was used to calculate the count ratio of ^{160}Tb/^{161}Tb based on their half-lives. The counting efficiencies of ^{160}Tb and ^{161}Tb were determined using the CIEMAT/NIST (CNET) method. This paper will detail the method development of this study and also discussion of the results.

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Standardization of tritium with the TDCR instrument

Arūnas Gudelis, Lina Gaigalaitė, Paulius Butkus

Center for Physical Sciences and Technology (FTMC), Savanorių Ave. 231, Vilnius, LT-02300, Lithuania

Corresponding author's e-mail: arunas.gudelis@ftmc.lt

The triple-to-double coincidence ratio (TDCR) technique is a liquid scintillation counting method used for the standardization of beta-emitters as well as radionuclides decaying by electron capture mode. It was developed in late eighties of the last century [*Broda et al., 1988*]. Subsequently, the method underwent essential improvements in electronics [*Bouchard et al., 2000*] and became an irreplaceable tool of primary standardization [*Broda et al., 2007*].

Since 2007 it is used in the National metrology institute FTMC in Lithuania (Fig. 1). The sample "S" is positioned between three photomultipliers (Burle 8850), the results are analyzed with the software TDCRB-02. Within the trilateral comparison in 2013, tritium standardization results obtained by FTMC were compared to the CCRI(II)-K2.H-3 2009 ^{3}H international comparison results [*Cassette et al., 2016*] when the discrepancy of only 0.38% from CCRI(II) 2009 value was achieved.

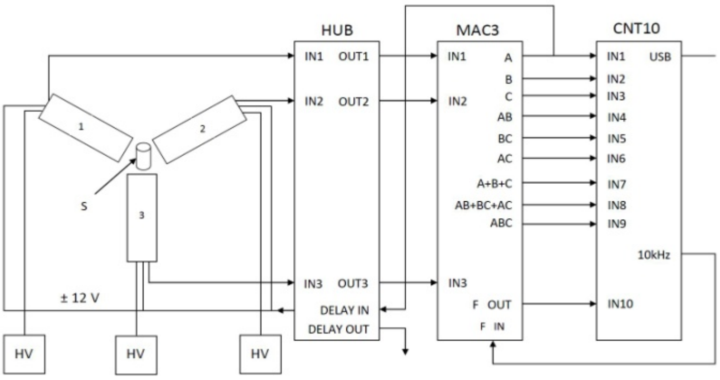

Fig. 1 The functional block diagram of the TDCR instrument

In 2016, tritium was standardized with the TDCR method. A certified tritiated water solution made in 2007 was mixed with the liquid scintillation cocktail Ultima Gold LLT and counted in the 20 mL polyethylene and glass vials. Different samples have been prepared for measurements at different runs by varying volume of the cocktail. The data were decay-corrected ($T_{1/2}$ = 12.312(25) y), the 0.38% value was taken as a measure for the systematic uncertainty, results were compared to values from the certificate. The results are shown in Table 1.

Table 1 Results of tritium standardization in the polyethylene vials

Measurement date	Massic activity (kBq/g) *k* = 1	
	TDCR	Certificate
2016-05-02	24.571 ± 0.094	25.057 ± 0.401
2016-05-17	24.517 ± 0.094	24.999 ± 0.400

The experimental results of different runs are in good agreement, they confirm high stability of the primary standardization instrument. It can also be seen that primary standardization results are in agreement within uncertainties with certificate values.

The application of the TDCR method for tritium standardization allows one to achieve standard uncertainties of the order of 0.5%. The further research is planned with the use of a digitizer technique.

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New calibration method for liquid scintillation counter using plastic scintillator

Yuka Kato 1), Etsuko Furuta 2)

1) Measuring Systems Engineering Dept., Hitachi, Ltd., 6-22-1 Mure Mitaka-shi Tokyo, 181-8622, Japan
2) Ochanomizu University, 2-1-1 Otsuka Bunkyo-ku Tokyo, 112-8610, Japan

Corresponding author's e-mail: yuka.kato.ot@hitachi.com

Performing calibration of liquid scintillation counter (LSC) with a commercially available check source, energy calibration is difficult unlike other radiation measuring instruments. Because the check source causes a slight quenching, it affects the shape of the beta-ray spectrum. Also, the degree of quenching of the check source is different for each manufacturer and manufacturing lot. So it makes more difficult to calibrate the energy of the LSC. The liquid scintillator used for the check source is toluene added PPO (primary scintillator) and POPOP (secondary scintillator). ^{3}H-labeled or ^{14}C-labeled toluene is added to a vial with this liquid scintillator and purge it with an inert gas to remove oxygen before sealing it with flame. However, the effects of chemical quenching cannot be removed completely, because of the causative agents such as residual oxygen and scintillator.

We have reported about the low energy beta radiation measurement using plastic scintillator (PS) of sheet type and pellet type [1] [2] [3]. Therefore, we focused on the character that those PS does not affected by chemical quenching. And we have developed a sample for energy calibration (calibration source) using the PS. We propose a new calibration method of LSC using the calibration source of PS.

In the experiment, we used two types of PS, BC-400 (Saint-Gobain, France) of sheet type and EJ-200 (G-teck Co., Japan) of pellet type. These PS is made by adding PPO and POPOP to Polyvinyl toluene, and it shows the maximum emission spectrum in the vicinity of 420nm in the same way as toluene. Sheet type was used in a pairs, and methionine labeled with each of ^{3}H, ^{14}C and ^{35}S (American Radiolabeled Chemicals, Ins., USA) was dried and sandwiched. Pellet type was packed in the low potassium glass ampoules, which were respectively added ^{3}H-labeld water (Amersham plc, UK), ^{3}H-labeled sodium acetate and ^{14}C-labeled sodium acetate (American Radiolabeled Chemicals, Ins., USA), and were sealed with flame. All measurement is performed with the liquid scintillation counter LSC-7200, LSC-8000, LSC-LB5B, LSC-LB7 (Hitachi, Ltd., Japan).

First, we have observed the spectrum of the ^{3}H, ^{14}C and ^{35}S using the PS of sheet type. Each beta-ray spectrum showed the maximum energy of near 18.6keV, 156keV and 167keV, the effectiveness of the calibration source was demonstrated. In addition, each spectrum showed ideal shape of beta-ray [4]. Next, the energy calibration of the LSC was carried out using PS of pellet type. LSC calibrated by energy with the pellet type PS showed good linearity energy. The result of the measurement showed the dependence with only the sample and not being affected by the difference in the model of the LSC. It is suggested that new calibration source of this method is effective in the energy calibration of the LSC.

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Determination of ^{210}Pb by liquid scintillation counting of ^{210}Pb and its progenies

Jung-Seok Chae, Yong-Jae Kim, Byung-Uck Chang
Korea Institute of Nuclear Safety

Corresponding author's e-mail: jschae@kins.re.kr

The analysis of ^{210}Pb is required in many fields for example NORM related research, geochronology, radioecology, and marine biogeochemistry. For determination of ^{210}Pb, various analytical methods have been used such as gamma-spectrometry by measuring the 46.5 keV photon emitted during ^{210}Pb decay, alpha-spectrometry through the determination of ^{210}Po after ingrowth, and the methods by measurement of ^{210}Pb or its immediate daughter ^{210}Bi by proportional counting. However, these methods show disadvantages such as poor counting efficiency, self-absorption in the sample for gamma-spectrometry and long waiting time for ingrowth of ^{210}Bi and ^{210}Po, respectively, for proportional counting method and alpha-spectrometry. Meanwhile, liquid scintillation counting method offers limit of detection comparable to those achieved by alpha-spectrometry and have no need to wait for ingrowth of ^{210}Bi or ^{210}Po.

Generally used liquid scintillation counting methods for ^{210}Pb determination involve liquid scintillation counting of ^{210}Pb after elimination of ^{210}Bi interference using double energetic window method or measurement of ^{210}Pb immediately after separation to minimize the effect of ^{210}Bi ingrowth. In this study a novel method for determination of ^{210}Pb by liquid scintillation counting using spectra regions of ^{210}Pb, ^{210}Bi, and ^{210}Po was established.

To prepare calibration standard of ^{210}Pb, extraction chromatography using Sr-resin was conducted. The chemical recovery was determined by gravimetric method with lead oxalate precipitation. The lead oxalate was dissolved in 1 mL of 6M HNO_3 and mixed with 15 mL of ULTIMA GOLD AB scintillator cocktail. The standard sample was measured repeatedly over time after chemical separation of ^{210}Pb from its progenies for estimation of gradually increasing detection efficiency caused by ingrowth of ^{210}Bi and ^{210}Po. Furthermore, the separated ^{210}Pb standard solution with varied quenching effects were prepared by adding different amount of CCl_4 to the standard solution for quenching correction of detection efficiency.

The measured counting efficiency of ^{210}Pb with its progenies was 79% at the time of 30 hours after separation and gradually increased with time. After 39 days from separation time, the counting efficiency was increased up to 171% by ingrowth of ^{210}Bi and ^{210}Po. The detail experimental results about quenching effect, method validation and optimization will be discussed during presentation.

The newly established method for ^{210}Pb determination can be used for analysis of the sample with extremely low activity concentration due to the increased detection efficiency. Moreover, this method allows accurate measurement during the ingrowth of ^{210}Bi and ^{210}Po after separation.

Application of LSC for measurements of concentrations of radon and thoron decay products in air

Stanislaw Chalupnik1), Krystian Skubacz1), Qiuju Guo2), Jian Zhang3)

1)Silesian Centre for Environmental Radioactivity, Central Mining Institute, Katowice, Poland
2)Peking University, Beijing, P.R. of China
3)National Institute of Metrology, Beijing, P.R. of China

E-mail: schalupnik@gig.eu

Liquid scintillation counting (LSC) is a measuring technique, broadly applied in environmental monitoring of radionuclides. One of the possible applications of LSC is the measurement of radon and thoron decay products. But this method is suitable only for grab sampling. The LSC method can be used for calibration of portable radon decay products monitors as the LSC method has the advantage to be an absolute one.

A comparison of the measurements of has been performed in the radon/thoron chamber of the Chinese National Institute of Metrology in Beijing. The method has been previously developed and tested in the Silesian Centre for Environmental Radioactivity with application of Triathler and Quantulus LS spectrometers. Measurements in the Chinese National Centre of Metrology have been performed with use of TriCarb 2770 and Hidex L-300 LS spectrometers. The results show a good compliance of the method’s transition to any of LS spectrometers without any additional calibration.

For long term measurements a different technique can be applied – monitors of potential alpha energy concentration (PAEC) with thermo luminescent detectors (TLD). The readout of TL detector shows directly potential alpha energy, with no dependence on equilibrium factor etc. This technique, which had been used only for radon decay products measurements, was modified to allow simultaneous measurements of radon and thoron PAEC. The TLD monitors have been checked in the radon/thoron chamber of NIM in Beijing, too.

DON'T TRUST YOUR MEMORY IN THE LAB – the implementation of a quality control program for LSC

Sofia Eriksson(1)

(1) Swedish Radiation Safety Authority, Dept. of Radiation Protection, SE-171 16 Stockholm, Sweden

Corresponding author's e-mail: sofia.eriksson@ssm.se

The radioanalytical laboratory at the Swedish Radiation Safety Authority (SSM) has implemented an internal quality control program for all their LSC methods. This poster demonstrates the process behind and the setting up of the internal quality system, and how it is used in the daily work.

In a research lab quality control is often overlooked and rather based on a scientist's experience with the instrument: scientists with long experience simply ”know” when the instrument is not measuring correctly or will rely on memory and knowledge of previous samples to recognize when something ”looks wrong”. An internal quality control program removes this person-dependency and it also removes the need to rely on memory. With internal quality control statistics will show you whether or not the instrument is reliable and hence whether or not you can use the results for an article or for your clients.

This poster offers a hands-on approach to setting up an internal control program for the LSC.

Simultaneous Analysis of Strontium and Cesium in a Single Process

TAN Hiong Jun Angela and LOO Howe Kiat

DSO National Laboratories, 12 Science Drive, Singapore 118225

Email: thiongju@dso.org.sg

Keywords: Cs-137, Sr-90, Cherenkov Counting, Mathematical Corrections

The typical analysis of Cs-137 is straight forward and simple using the gamma spectrometry. However, these measurements can be confounded by fluctuating background radiation when the shielding is insufficient. An alternative method is to measure Cs-137 using liquid scintillation techniques, via its beta emissions. Although the liquid scintillation technique involves laborious sample preparation to remove interfering radioisotopes, the advantages are high sensitivity and low sample quantity required for analysis. Without the use of additional Cs-resin, we improvised the current procedure for Sr-90 analysis for the measurements of both Sr-90 and Cs-137 simultaneously. Mathematical treatment of the measurement data was performed to obtain accurate quantification of the radioisotopes. This method is exceptionally useful for making quick hazard assessment when radioactive Cs and Sr are expected to be the major radio-contaminants e.g. releases during a nuclear fall-out incident. Our method had been successfully validated with test samples from IAEA Proficiency Tests - TEL-2014-03 and TEL-2015-03.

Application of DGA Resin for Yttrium Extraction during Radio-Strontium Analysis

LOO Howe Kiat, TAN Hiong Jun Angela

DSO National Laboratories, 12 Science Drive, Singapore 118225

Email: lhowekia@dso.org.sg

Keywords: DGA resin, Yttrium, Radio-Strontium

The measurement of Sr-90 by liquid scintillation and/or Cherenkov counting requires that all interfering radioisotopes are removed prior to counting. Sample clean-up procedures and extraction of the daughter isotope Y-90 are time consuming and tedious. Importantly, the quality of the results can only be ascertained through the analysis of the measured data; when quality is not acceptable, all the preparation efforts are wasted. To enhance the chances of obtaining good quality results, we developed a new method using Sr- and DGA- resins sequentially to achieve selective extraction of yttrium and strontium in radioactive materials at the same time for analysis. The method was used and validated in the recent IAEA Proficiency Test (TEL-2016-03), where the activity of Sr-90 in test sample #2 was determined with good accuracy and precision.

Preparation of Gadolinium Loaded Liquid Scintillator for Daya Bay Neutrino Experiment

ZHANG Zhiyong, DING Yayun, CAO Jun

Institute of High Energy Physics, The Chinese Academy of Sciences, Beijing 100049, China
Corresponding author's e-mail: zhangzhy@ihep.ac.cn

Daya Bay reactor anti-neutrino experiment was designed to measure the smallest mixing angle θ_{13} by detecting anti-neutrino created by Daya Bay and LingAo nuclear power plants. In order to measure $\sin^2 2\theta_{13}$ to 0.01 or better precision, the experiment uses eight identical three-layer homocentric cylindrical detectors deployed underground, each detector contains 40 tonnes of mineral oil as buffer, 20 tonnes of liquid scintillator as γ-ray catcher and 20 tonnes of gadolinium loaded liquid scintillator (Gd-LS) as target. The optical properties of Gd-LS have great influences on the anti-neutrino experiment. Ideal Gd-LS should have long attenuation length, high light yield, long term stability, and should be compatible with the material used to build the containers.

We developed a new Gd-LS recipe in which carboxylic acid 3,5,5-trimethylhexanoic acid was used as the complexing ligand to form organo-complex with gadolinium chloride, and 2,5-diphenyloxazole (PPO), and 1,4-bis[2-methylstyryl]benzene (bis-MSB) were used as primary fluor and wavelength shifter, respectively. The scintillator base was linear alkyl benzene (LAB). One hundred and eighty-five tons of 0.1% Gd-LS were successfully produced on schedule from Oct. 2010 to March 2011[1].

The Gd-LS liquid properties were well-characterized and essentially constant to within 1% between different storage tanks before filling the ADs. Good Gd-LS attenuation length and high light- yield met the quality assurance requirements. Equal portion of the scintillator (4 tons) from each storage tank was taken for filling each 20-ton AD to ensure identical functions of all eight detectors. The variation of Gd capture ratio among ADs was determined to meet the 0.1% design specification by measuring the neutron capture time. The long-term stability monitoring program for the Daya Bay scintillator, started three months after production, has been on going for more than 5 years and will be continuing along-side the Daya Bay data-taking. The long-term chemical stability of the Gd-LS had surpassed the previous reactor anti-neutrino experiments and led to a successful discovery of non-zero θ_{13}[2].

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Fast Neutron Multiplicity Detectors Based on Liquid Scintillators for uranium Mass determination

Li jinghuai1） ZhouHao1）Liu guorong1）Liang Qinglei1）

1）China Institute of Atomic Energe,Beijing 275（8）box，102413，China

Corresponding author’s e-mail:lijh0256@sina.com

Purpose of this work is to measure the mass of the uranium pellets with NDA(Non-Destructive Assay) method. ^{235}U atoms are activated to fission by two AmLi neutron sources and fission rate of the sample is related to the mass of ^{235}U. Six liquid scintillator detectors are used as the fast neutron multiplicity counter while a HPGe detector is used to determine the enrichment with relative intrinsic efficiency self-calibration technique.

Model of the liquid scintillators are BC501A with a diameter of 5 inches and a height of 2 inches. Two AmLi neutron sources are located at the bottom and top of the system. As liquid scintillators are sensitive to both neutron and gamma rays, an n/γ discrimination module MPD-4 with four channels is used to eliminate interference of gamma rays. Output signals of the MPD-4 are sent to the TDC（time-to-digital convertor）MCS6A which has a high time resolution.

With the fast neutron multiplicity measurement system based on scintillators(Figure 1), For pellet samples in which ^{235}U mass is no less than 10 grams, a precision better than 5% could be achieved in less than half an hour.

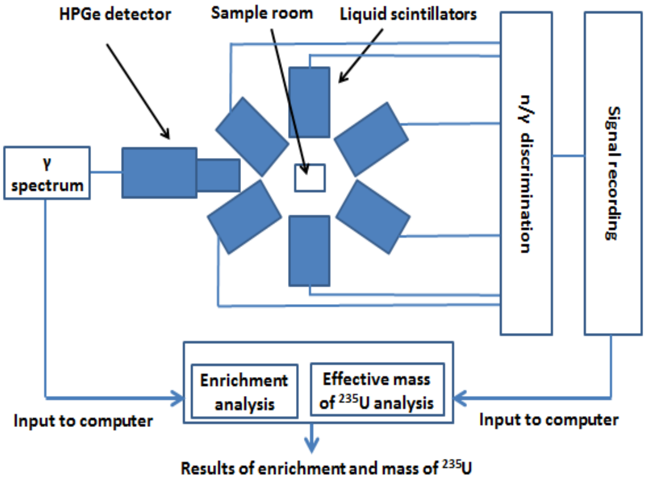

Fig. 1 Structure of the fast neutron multiplicity counter based on six scintillators

Simulation of neutron encode imaging with liquid scintillator filled capillary array

Leifeng Cao [1),2)], Jian Teng [2)],

[1)] Laser Fusion Research Center, China Academy of Engineering Physics, P. O. Box 919-986-6, 621900, China
[2)] Science And Technology on Plasma Laboratory, P. O. Box 919-986-6, 621900, China

Corresponding author's e-mail: leifeng.cao@caep.cn

Liquid scintillator filled capillary array has been suggested to be a detector for neutron encode imaging in laser driven inertial confinement fusion research. The aim is to get better spatial resolution than former used method. Primary simulations were performed with Monte Carlo method and it provided meaningful information for judging the feasibility. Further possible efforts for such an issue were also discussed.

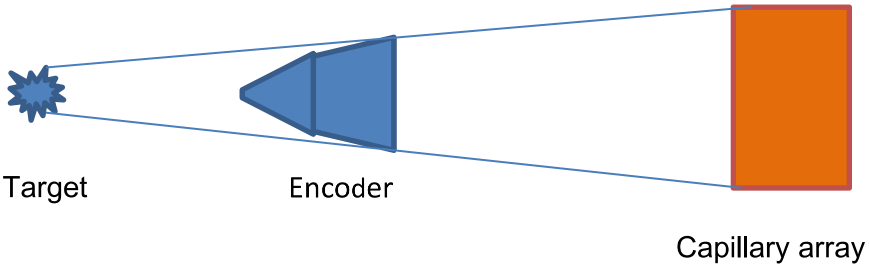

Fig. 1 Scheme of the system

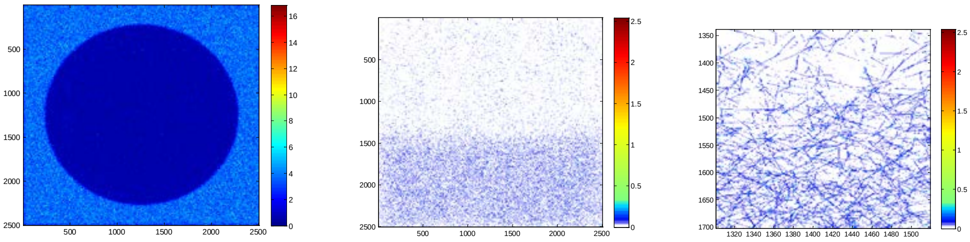

Fig. 2 Encoded image and its different zoom in

Synthesis of ZrO_2 nanoparticles for liquid scintillators for neutrinoless double beta decay detection

Susumu Takigawa [1)], Masanori Koshimizu [1)], Takio Noguchi [2)], Tsutomu Aida [2)], Seiichi Takami [3)], Tadafumi Adschiri [2,3,4)], Yutaka Fujimoto [1)], Keisuke Asai [1)]

[1)] Department of Applied Chemistry, Graduate School of Engineering, Tohoku University, 6-6-07 Aoba, Aramaki, Aoba-ku, Sendai 980-8579, Japan
[2)] New Industry Creation Hatchery Center, Tohoku University, 6-6-10 Aoba, Aramaki, Aoba-ku, Sendai 980-8579, Japan
[3)] Institute of Multidisciplinary Research for Advanced Materials, Tohoku University, 2-1-1 Katahira, Aoba-ku, Sendai 980-8577, Japan
[4)] Advanced Institute for Materials Research, Tohoku University, 2-1-1 Katahira, Aoba-ku, Sendai 980-8577, Japan

Corresponding author's e-mail: koshi@qpc.che.tohoku.ac.jp

The confirmation of the presence (or absence) of neutrinoless double beta decay is an important issue in current elementary particle physics. One of the experimental approaches is the use of liquid scintillators loaded with candidate isotopes for neutrinoless double beta decay. Conventionally, such isotopes have been loaded into liquid scintillators mainly as organometallic complexes to achieve a high solubility in toluene or xylene. However, the solubility is limited, and the loaded organometallic complexes often quench the scintillation.

In this study, we loaded nanoparticles into the liquid scintillator. This can be an alternative approach for the development of liquid scintillators for neutrinoless double beta decay detection. The nanoparticles can be well dispersed in organic solvents by modifying their surface with organic molecules. The use of small nanoparticles suppresses light scattering within the scintillator. To synthesize small nanoparticles with surfaces modified with organic molecules, we employed supercritical hydrothermal synthesis. In this study, we chose ZrO_2 nanoparticles, because ^{96}Zr is a candidate isotope for neutrinoless double beta decay.

In the hydrothermal synthesis, $Zr(OH)_4$, the precursor for ZrO_2, was dissolved in water, and a surface modifying agent, phenylhexanoic acid, was added. The hydrothermal synthesis was performed at 573 K and 30 MPa for 10 min. The reaction products were collected with toluene, and the dispersion was left undisturbed for one night. Subsequently, the precipitates were removed, and the organic fluorescent molecules PPO and POPOP were added to the solution to obtain the ZrO_2-nanoparticle-loaded liquid scintillator.

Figure 1 shows a photograph of the dispersion. The dispersion was irradiated with green laser light using a laser pointer to observe Tyndall scattering. The scattering indicated that the nanoparticles were dispersed. The Zr concentration in this dispersion was 1.4 wt%, as confirmed using inductively coupled plasma–atomic emission spectroscopy (ICP-AES). The size of the nanoparticles was less than 10 nm, as confirmed by transmission electron microscopy (TEM).

Figure 2 (a) shows the absorption spectrum of the ZrO_2 nanoparticle dispersion in toluene. The steep increase in intensity at wavelengths below 330 nm is attributed to the scattering of the incident light by the nanoparticles. Figure 2 (b) shows the X-ray-induced emission spectrum of the ZrO_2-nanoparticle-loaded liquid scintillator. A clear peak is observed at ~440 nm, which is attributed to

POPOP emission. The optical density of the ZrO_2 nanoparticle dispersion at the scintillation wavelength is negligible. Hence, these results indicate that the nanoparticles-loaded liquid scintillator is applicable in neutrinoless double beta decay detection.

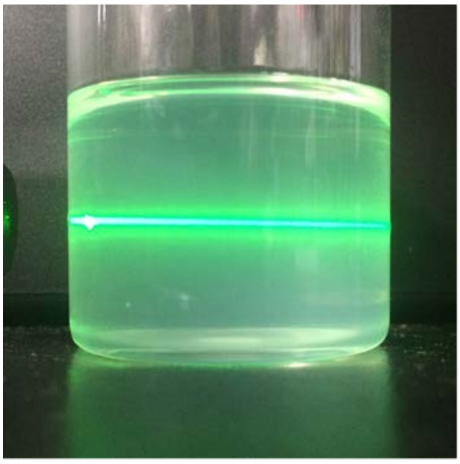

Fig. 1 Photograph of the ZrO_2 nanoparticle dispersion in toluene.

The dispersion was irradiated with a green laser light to observe Tyndall scattering.

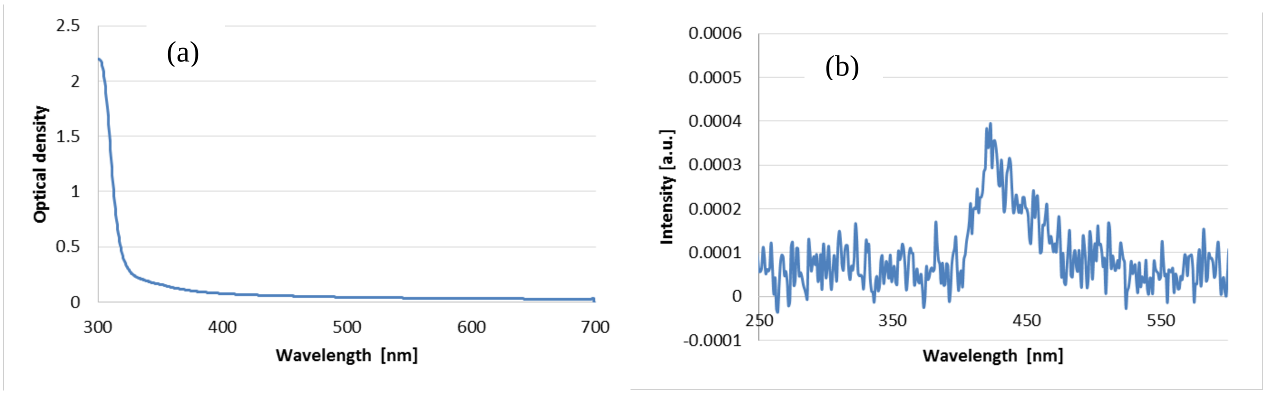

Fig. 2 (a) Absorption spectrum of the ZrO_2 nanoparticle dispersion in toluene. (b) X-ray induced emission spectrum of the ZrO_2-nanoparticle-loaded liquid scintillator.

Radionuclides determination by LSC during routine operation of nuclear power plants and its decommissioning.

RAJEC Pavol 1), DULANSKÁ Silvia 1), MÁTEL Ľubomír 1), ROSSKOPFOVÁ Olga 1)

1) Comenius University, Department of Nuclear Chemistry, Mlynská Dolina, Ilkovičova 6, 842 15 Bratislava, Slovakia

Corresponding author's e-mail: rajec@fns.uniba.sk

The government of the Slovak Republic decided on September 14, 1999 to realize the final shutdown of NPP V1 - units 1 and 2 in 2006 and 2008 respectively (SR Government decision No. 801/99). In decommissioning a nuclear facility, the radioactivity of various radionuclides has to be measured for estimation of the total inventory of radioactivity. Low and intermediate level radioactive wastes are disposed in a near-surface repository at Mochovce site. The repository, which was commissioned in September 2001is for disposal of low and intermediate level wastes (LILW) and construction of very low level wastes (VLLW) repository. It is a vault-type concrete structure repository with reinforced concrete containers as the final waste packages. The Mochovce repository is designed to receive acceptable radioactive wastes from decommissioned A-1 power plant at Jaslovské Bohunice, operational waste from NPPs V-1 and V-2 at Jaslovské Bohunice site and NPP Mochovce, as well as institutional radioactive wastes. The acceptance of radioactive waste in Mochovce repository is one of the many elements that directly contribute to the safety of the disposal system. Acceptance criteria for radionuclide inventory in the location of National Radioactive Waste Repository (NRR) Mochovce was added and all radioactive of wastes have to be check to meet the criteria valid for repository. The radioactivity in the radioactive of wastes might come from many radionuclides such as ^{60}Co, ^{55}Fe, ^{63}Ni, ^{137}Cs and other alpha or beta radionuclides. Gamma emitters such as ^{60}Co, ^{137}Cs can be easily determined by gamma spectrometry. However, the beta, X - ray and alpha emitters (difficult to measure radionuclides (DMR)) have to be separated individually from the matrix and other radionuclides before measurement. Declaration of radionuclides in radioactive waste for Regional Repository Mochovce is shown in Table 1. Many of the radionuclides in the table can be determined by LSC methods after previous separation technique. Nowadays, for separation of difficult to measure mainly ion exchange chromatographic and extraction chromatography are used. Some of the separation techniques used for determination of DMR and measured using LSC for radioactive wastes and routine operation of NPP samples at the Department of nuclear chemistry, Comenius University will be presented.

Tab. 1 Declaration of radionuclide in radioactive waste for Regional Repository Mochovce

Radionuclide	**Decay**	**Measurement method**	**Radionuclide**	**Decay**	**Measurement method**
C-14	β	LSC	Pd-107	β	LSC
Ca-41	EC	LSC, Low-Energy X Ray Detector,TIMS	Sn-126	β,γ	LSC, γ-spectrometry
Ni-63	β	LSC, Low-Energy X Ray Detector	I-129	β	LSC, Low-Energy X Ray Detector, ICP-MS
Ni-59	EC	LSC, Low-Energy X Ray Detector, TIMS	Sm-151	β	LSC, Low-Energy X Ray Detector
Se-79	β	LSC	Co-60	β,γ	γ-spectrometry
Sr-90	β	LSC, β-counting	Cs-137	β,γ	γ-spectrometry
Mo-93	EC	LSC, Low-Energy X Ray Detector, AMS	Cs-135	β	ICP-MS
Zr-93	β	LSC	Pu-238	α	α-spectrometry
Nb-94	β,γ	LSC, γ-spectrometry	Pu-239	α	α-spectrometry
Tc-99	β	LSC, ICP-MS	Am-241	α,γ	α- γ-spectrometry

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IN SITU MEASUREMENT OF 90SR ACTIVITY USING DIRECT LIQUID SCINTILLATION COUNTING (ISMAD LSC): APPLICATION TO DECOMMISSIONING PROJECTS

Vincent GOUDEAU [1)]

[1)] CEA, Direction of Fundamental Research, Unities for Protection Health and Security, Environment and Radiation Protection Service, Remediation section, 92265, Fontenay-aux-Roses, France.

Corresponding author's e-mail: vincent.goudeau@cea.fr

During the decommissioning of a nuclear site the operator must assure the protection of the workers and the environment. It must furthermore identify and classify the various wastes, while optimizing the costs. At all stages of the decommissioning radiological measurements are performed to determine the initial situation, to monitor the demolition and clean-up, and to verify the final situation.

To address these various operations, direct measurement methods can be used (onsite gamma or X spectrometry, autoradiography). For clean surfaces, contamination detectors can be used (COMO, LB, etc.). But for soils, especially damp ones, direct alpha or beta measurement is hard to performed, preparation is almost always needed.

Radiochemical analysis is crucial for the radiological evaluation of soils contaminated by alpha and beta emitting radionuclides such as plutonium and strontium. These analyses are expensive and time consuming, due to the many chemical preparations steps needed to purify the radionuclide to be measured. A sampling plan is needed to get an accurate characterization of the pollution. In this way, alpha liquid scintillation counting can be a precious tool.

This paper describes a study performed to highlight the capacity of the alpha LSC to detect abnormal counting rates in soils. Standard counting rates has been measured in clean soils then compared to soils containing plutonium and/or strontium. Moreover, comparison was made between strontium normalized method (ISO 18589-5 and experimental method (ISMAD).This protocol, including short preparation and high efficiency detection reveal itself useful and time-saving. This study was performed in the SMART mobile laboratories of the expertise platform in Fontenay-aux-Roses

Is it easy to improve radiochemical methods in respect of REACH regulation?

C. Gautier, C. Colin, J.P. Degros, P. Perret, C. Cruchet, M. Giuliani, E. Laporte and C. Mougel

Den - Service d'Etudes Analytiques et de Réactivité des Surfaces (SEARS), CEA, Université Paris-Saclay, F-91191, Gif-sur-Yvette, France

Corresponding author's e-mail: christele.colin@cea.fr

For more than twenty years, the LASE (Operator Support Analysis Laboratory) has been developing and implementing radiochemical methods devoted to the determination of Difficult To Measure radionuclides in radwaste. In particular, its expertise concerns the characterization of long-lived beta emitters such as ^{55}Fe, ^{63}Ni and ^{99}Tc in various matrices. The purification procedures applied for a long time are highly selective towards those radionuclides but mainly based on liquid-liquid extraction steps including the use of chloroform which is restricted through REACH regulation [1,2]. The elimination of this solvent is a real challenge for radioanalytical laboratories. Furthermore, the management of short-lived radioactive tracers such as ^{99m}Tc is becoming difficult. Consequently, alternative radiochemical methods have to be investigated.

The presentation will first focus on the results of speciation calculations (performed with Chess® software) which are of prime interest to better understand the involved chemical processes and to optimize the alternative separation methods before LSC measurements [2]. The second part of the work will deal with the improvements made for the analysis of ^{55}Fe, ^{63}Ni and ^{99}Tc. Extraction chromatography was introduced to maintain high decontamination factors towards the interfering elements [3,4]. TRU®, Ni® and TEVA® resins were respectively tested in the purification procedures dedicated to ^{55}Fe, ^{63}Ni and ^{99}Tc measurements by LSC. In order to increase the responsiveness of the laboratory, the implementation of rhenium instead of ^{99m}Tc as tracer was also investigated [5]. The results will be compared in terms of separation yield, selectivity and accuracy for real radioactive waste and effluent samples. In particular, the influence of the medium obtained after radiochemistry was investigated towards the quenching effect and the counting efficiency for LSC measurements. In the future, this approach can be extended to other radionuclides such as ^{93}Zr.

Table 1 Comparison of two radiochemical procedures applied to measure ^{55}Fe by LSC in various radioactive waste samples

Sample	Recovery yield (%) based on liquid-liquid extraction with cupferron	Recovery yield (%) based on TRU® resin	^{55}Fe (Bq/g) based on liquid-liquid extraction with cupferron	^{55}Fe (Bq/g) based on TRU® resin
Aluminum	72	82	3.70×10^5 ± 8 %	3.62×10^5 ± 8 %
Steel	23	80	3.80×10^3 ± 7 %	3.73×10^3 ± 7 %
Ion-exchange resin	74	86	2.62×10^3 ± 5 %	2.50×10^3 ± 5 %

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Pre-concentration method using oxalate co-precipitation with MnO_2 for determination of ^{90}Sr in seawater

Yong-gi Yun, Yong-Jae Kim, Ju-Yong Yun, Jung-Seok Chae

Korea Institute of Nuclear Safety

Corresponding author's e-mail: jschae@kins.re.kr

Strontium-90 is one of the most hazardous anthropogenic radionuclides originated from nuclear fission processes. Introduced ^{90}Sr into the environment can be incorporated into the organism through the calcium cycle. Eventually, it is accumulated in bones due to similar chemical characteristics with calcium and can give radiological risk to human health. Thus, the monitoring of ^{90}Sr in the environment is important for radiation protection. Especially since a considerable number of nuclear power plants are located on the cost, the ^{90}Sr survey in seawater is very important.

For determination of ^{90}Sr in seawater, various analytical methods have been used. Although many pre-concentration and separation methods using carbonate or oxalate precipitation have been applied, many steps must be taken to achieve high chemical recovery. However, since the activity concentration of ^{90}Sr in seawater is very low and decreasing with time, more than a dozen liters of seawater must be pretreated for ^{90}Sr analysis. It is very difficult and takes a great deal of labor to carry out the various steps of pretreatment of large amounts of seawater sample. Therefore, in this study, we investigated the pretreatment method which can obtain relatively high chemical recovery.

In order to simplify the pretreatment step, carbonate precipitation was omitted and a pre-concentration method using MnO_2 simultaneously with oxalate co-precipitation was examined. After addition of ^{85}Sr for determination of chemical recovery, oxalate co-precipitation was performed and then MnO_2 was added into the sample. The recoveries for only oxalate coprecipitation method was 65%, and the recoveries for the addition of MnO_2 with oxalate coprecipitation method was 75%. The detail experimental results of method validation will be discussed during presentation.

Evaluation of ^{14}C analysis using Pyrolyser system for decommissioned materials

Daeji Kim [1)], Phil E Warwick [2)], Ian W Croudace [2)]

[1)] Korea Institute of Nuclear Safety, 62 Gwahak-ro, Yuseong, Daejeon, 34142, South Korea
[2)] GAU-Radioanalytical, University of Southampton, Eropean Way, Southampton, SO14 3ZH, UK

Corresponding author's e-mail: kimdj@kins.re.kr

The requirement for ^{14}C and ^{3}H measurement in a wide range of materials has increased in recent years mainly as a result of accelerated decommissioning programmes with its associated waste sentencing requirements. However, as ^{14}C and ^{3}H is a pure low-energy beta emitter, any analytical procedure usually involves collection of representative samples, isolation of ^{14}C and ^{3}H from the sample and measurement of the ^{14}C and ^{3}H activity using liquid scintillation counting (LSC).

Complete oxidation of diverse sample types is always a critical issue of any combustion method because it will directly affect the extraction of or ^{14}C or ^{3}H from specific samples and ultimately the accurate measurement. The factors affecting the oxidation rate include maximum temperature, oxidant gas (i.e. oxygen or air) and total combustion time.

The recovery of ^{14}C in carbonate depend on the temperature and speciation of materials. To achieve complete oxidation of ^{14}C in carbonate using combustion furnace method, heating sample up to 900°C is required.

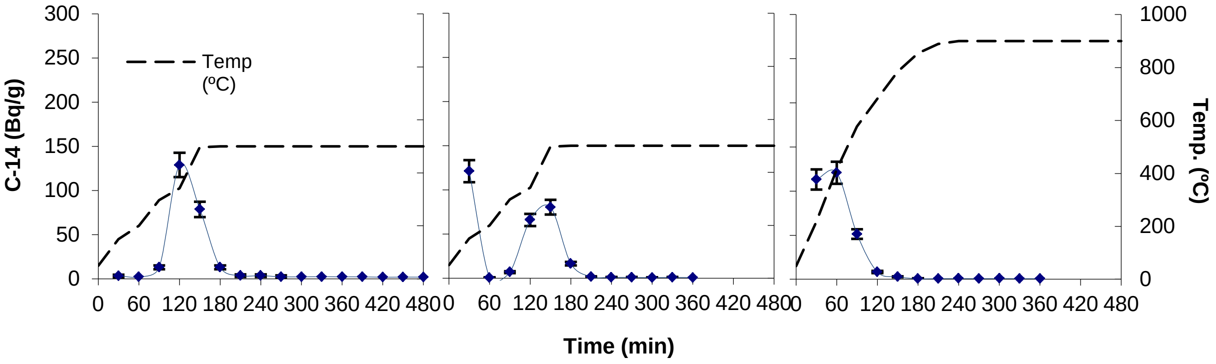

Fig.1 Evolution profiles of ^{14}C in thymidine in water spiked on (A) paper, (B)& (c) sand

In this study, the temperature dependence of ^{14}C release from a standard sources (^{14}C in thymidine) was investigaed. These can be applied to a range of materials commonly encountered during decommissioning and environmental surveys. Evolution profils provide valuable information which can provide insights into the association and form of carbon-14. These studies can help to develop robust analytical procedures for a range of waste types.

ON THE DEVELOPMENT OF AN EXTRACTION CHROMATOGRAPHIC RESIN FOR THE SEPARATION AND DETERMINATION OF SELENIUM

Dirks, C.[1], Happel, S.[1], Bombard, A.[1]

[1] TrisKem International, 3 rue des Champs Geons, 35170 Bruz, France

Corresponding author's e-mail: shappel@triskem.fr

Measurement of Selenium is of importance regarding public health as the ratio between beneficial daily intake and toxicity is rather low [1], [2]. Also from the radioprotection perspective, Se-79 as a long-lived fission nuclide ($T_{1/2}$=3.56(40) 10^5 a, β^- =150,9(17) keV [3]) with high mobility in environment, is of concern regarding waste management and decommissioning [4], [5]. As a long-lived pure beta emitter it is considered to be one of the difficult to measure (DTM) radionuclides, requiring special attention with respect to sample preparation before measurement, for LSC as well as for ICP-MS.

Selenium has due to the existence of different oxidation states a complex chemistry which makes its separation challenging, amongst other a careful control of the oxidation state is required. The aim of this work is the development of fast and easy separation methods allowing for the selective separation of Selenium from a variety of sample matrices.

Several extraction chromatographic resin prototypes have been tested and characterized for Se and numerous other elements. Based on obtained data most suitable resins were chosen and further characterized, including elution studies, allowing the development of a promising method for the separation of Se based on the best suitable resin prototype.

Further first tests were performed in order to evaluate the suitability of some of the tested resins for use in DGT (Diffusive Gradients in Thin films) devices.

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Quantitative determination of Ni-63 wipe tests by liquid scintillation analysis (LSA)

S. Wolf

Helmholtz-Zentrum Berlin für Materialien und Energie GmbH, Germany

In 2015 there were refurbishing works of a former radiochemical laboratory of the BER I research reactor. In the course of contamination monitoring and free release a Ni-63 contamination had to be considered. The investigations of contamination of Ni-63 was done with wipe tests and this was the reason for our group to create a method of quantitative determination.

Ni-63 ($t_{1/2}$ = 100 y) is a product of neutron activation formed by neutron capture from Ni-62, a stable isotope of nickel [1]. In general, at the central collecting depot of radioactive waste of the state of Berlin there are Ni-63 samples from electron capture detectors (ECD) or activated steels from neutron sources.

Methods for determining Ni-63 have been reported for many sample matrices especially related to interferences with Fe-55 [1-4]. The investigations in our group were done with the Hidex 300 SL, the first commercial liquid scintillation analyzer equipped with three Photomultipliertubes (PMT) to facilitate the triple to double coincidence ratio (TDCR) counting [5]. Comparable measure-ments were done with the Tri-Carb 2200 CA from Packard.

Before the investigation with the wipe tests a reference solution was used for an investigation through a variation of scintillation cocktails and under different quench conditions. 3 methods were investigated for the Hidex 300 SL. These were the external standard method, the TDCR method and the correction by a TDCR quench. Especially the TDCR method (deviation mostly around 5%) has delivered satisfactory results and requires no production of its own quenched standard set.

Glass fiber filters and paper filters were contaminated with the liquid reference and analyzed. Furthermore glass plates were contaminated and examined with dry or wet wipe tests.

Excellent agreements was observed for a wide range of standard activity with glass fiber discs. They are not as robust as paper filters, but they are transparent through the scintillation cocktail and can better absorb the cocktail.

It has also been found that it is possible to determine Ni-63 using the TDCR method when the glass fiber filter is added to the inner wall of a 20 ml vial [6]. This approach to measurement is less time-consuming and it requires less scintillation cocktail.

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The Radiological Characterization of Concrete and Soil from Decommissioning Process of Research Reactor

Jong Myoung Lim, Hyuncheol Kim, Yoon Hee Jung, Young Yong Ji, Chang Jong Kim, Kun Ho Chung, Mun Ja Kang

Environmental Radioactivity Assessment Team, Korea Atomic Energy Research Institute, Korea, 111 Daedeok-daero 989 beon-gil, Yuseong,gu, Daejeon, 34057, Korea

Corresponding author's e-mail: jmlim@kaeri.re.kr

The decommissioning, the final phase in the life-cycle of nuclear facility, is a complex process involving activities such as decontamination, dismantling ad demolition of equipment and structures and management of resulting wastes [1]. The radiological characterization for providing reliable information on quantity and type of radionuclides is very important for the determination activities to 1) decontamination processes, 2) dismantling procedures, 3) radiological protection of worker and environment, 4) waste classification and 5) their resulting costs [1]. Nuclear decommissioning wastes (e.g., biological shield, soil, structure materials of reactor, metals, etc.) have been consistently generated from facilities on decommissioning process. For the proper handling options of such nuclear wastes, a rapid and accurate analytical method should be developed for evaluating radionuclides concentrations.

In this study, the analytical procedures for radiological characterization of the radionuclides including gamma emitters, H-3, C-14, I-129, Fe-55, Ni-59, Ni-63, Sr-90, Tc-99, and gross alpha were developed for the sample of concrete and soil matrix. The process of sample preparation and measurement is composed of four main process: direct measurement, pyrolysis, alkali fusion, and acid leaching for specific target nuclides. The validated process were applied to analysis of the concrete and soil samples from decommissioned TRIGA MARK III research reactor of KAERI.

According to the validation results, we could conclude that the methods of pyrolysis for H-3, C-14, and I-129, alkali fusion for gross alpha, Fe-55, Ni-59, and Ni-63, and acid leaching for Tc-99 and Sr-90 could be used for rapidly and fully recovering the specific target nuclides from the samples with a high degree of accuracy and precision. Finally, the activity of H-3, C-14, and Fe-55 was relatively high level in the concrete samples. As shown Fig. 1, the relationship was obviously observed between the Fe-55 and gamma emitters in the concrete samples.

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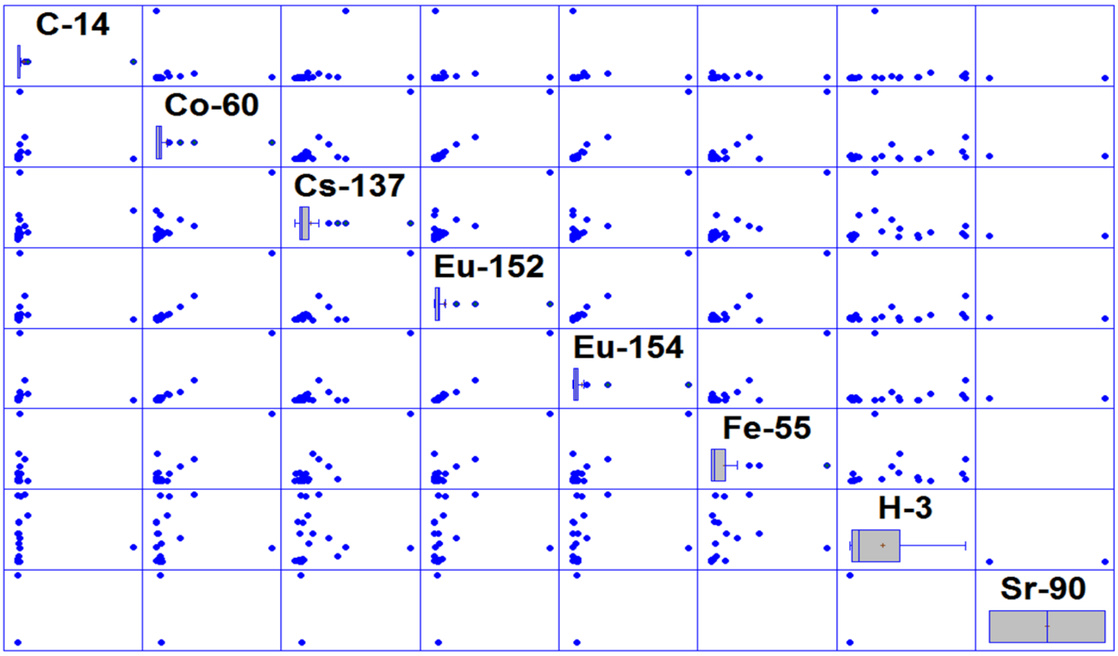

Fig. 1. The relationship between analyzed nuclides.

Laboratory infrastructure and utilization plan for radiochemical analysis

Kahee Jeong [1)], Kyu-min Song [1)], Eun Kyung Jung [1)], Seung Chul Park [1)]

[1)] KHNP Central Research Institute, 70-1312 Gil, Yusong-daero, Yusong-gu, Daejeon, 34101

Corresponding author's e-mail: kaheejeong@khnp.co.kr

The radiochemical analysis is an essential necessity for the management and disposal of radioactive wastes. The demand of radiochemical analysis is expected to increase rapidly as the decommissioning of NPP K-1 unit in Korea, which will be starting in the near future. KHNP-CRI has been building the infrastructure for the contribution of the smooth progress of decommissioning like the radiochemical analysis laboratory and analysis instruments.

In this study, the design of laboratory and selection of measuring instruments are introduced. Also, the application plan of this laboratory and LSC system is as well proposed. Through the use of pyrolyser as sample in pre-treatment and LSC as a form of measurement, beta emitter nuclides (^{3}H, ^{14}C, ^{90}Sr, etc.) can be tested in the radioactive wastes generated during decommissioning. The separation and recovery tests are performed using simulated samples with the standard sources of ^{3}H and ^{14}C. Therefore, was seen good recovery rate of more than 90 %.

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A plastic scintillation counter for tritiated water measurement as vapor

Etsuko Furuta

Ochanomizu University, 2-1-1, Otsuka, Bunkyo-ku, Tokyo 112-8610, Japan

Corresponding author’s e-mail: furuta.etsuko@ocha.ac.jp

Introduction Using plastic scintillators, a liquid scintillation counter (LSC) is able to measure low-energy beta emitters with high counting efficiency without organic waste generation. Especially, plastic scintillator sheets (PS-sheets) are useful for non-volatile compounds measurement [1] and plastic scintillator pellets (PS-pellets) are useful for volatile compounds measurement [2]. However, 20 mL vials for a general LSC have limits for available volumes to both methods. So, the detection limits described with the unit of per mL were not so high. For improvement of the detection limit and for future development of PS use, I made a prototype device for the PS-sheets [3]. It was success to get a higher detection limit. Then, I challenged to develop another new prototype device for the PS-pellets and studied to measure tritiated water as vapor.

Experimental The outside of the detector box of a new prototype device (PSC-va) was same as that of PSC-1 [3]; however, a pair of photomultiplier tubes (PMTs) was installed with a 36.3 mm gap. Figure 1 shows photographs of the inserted equipment with an acrylic vial and a 20 mL low-potassium glass vial which were filled with the PS-pellets. Sample assemblages preparation method using PS-pellets for the PSC-va was described in Fig.2 which was the same way to describe for an LSC vial [2]. Two new vials made by quartz and acryle were prepared for the PSC-va, and both of new vials and a 20 mL vial for LSC were used in this study.

Tritiated water (HTO; Moravek Biochemicals, USA) was used as the radioactive sample and a dilution series was prepared using distilled water, with concentration ranging from 1.3 Bq/mL to 500 Bq/mL. A liquid scintillation cocktail (LS; ACS-2, GE Healthcare UK Ltd., UK) was used for determining the activity (dps=Bq) of sample solutions, which were measured using a Tri-Carb

Fig. 1 Photos of the insert part of the plastic scintillator device of PSC-va with PS-pellets.

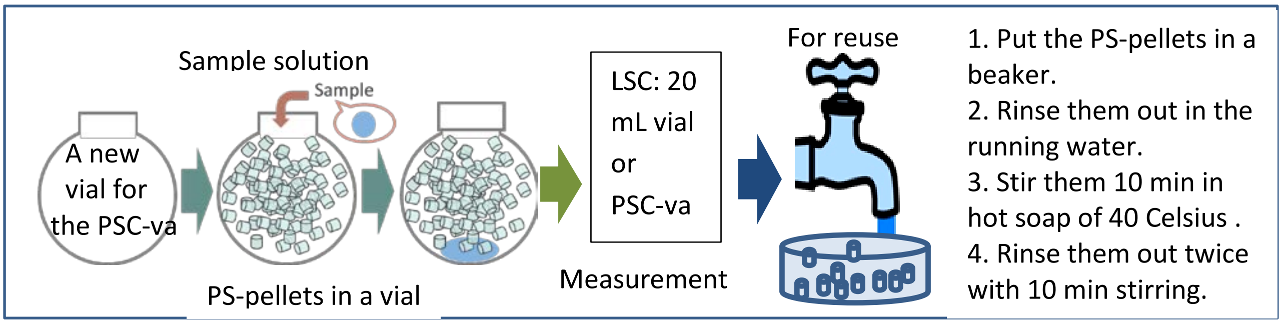

Fig.2 Preparation and reuse method of PS-pellets before and after measurement

3110TR (PerkinElmer, USA) liquid scintillation counter (LSC). Three samples of identical activity.were prepared in each investigation.

Results and discussion The counting efficiency, which was defined as cpm / dpm ×100, was stable up to 1 h after the start of the measurement. The measurement time was much shorter than that of LSC, which was required for 4 h measure, because the position of the vial was nearer to the PMTs and the vial was heated by the PMTs. PS-pellets measurement using PSC-va was very suitable for HTO measurement.

Two types of new vials did not work well because of leakage of HTO. One vial was made of quartz by blow molding; the other was made from acrylic sheets using laminating molding and had a screw-mouth. The quartz vial could not attach a ditch for screwing the inner gum cap. Therefore, when a silicon cap was used, a leakage clearly occurred approximately 2%, as observed from the comparison of the maximum stable counting efficiency of the first day and that of the second days. The acrylic vial also showed comparable leakage to the quartz vial on the second day. Using the inner gum cap for the LSC vials can stop leakage of HTO for more than 20 days [2]. Thus, although the technique of HTO vapor measurement with the PS-pellets is useful, a new type of perfectly sealed vial is necessary for further development of the PSC-va. The new vials should enable the application of a greater volume of samples compared to the 20 mL vials.

Conclusion The new type prototype device for PS-pellets was developed and the elapsed time for getting stable activity was 4 times shorter compared with that of an LSC use. It was merit of Plastic Scintillation Counter for vapor measurement. However, new type vials of quartz and acrylic never worked well because of leakage. I will choose more good materials for a new design vial to get higher counting efficiency and it is necessary to achieve lower detection limits.

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^{90}Sr and ^{99}Tc determination in Radioactive Ion Exchange Resins coming from Nuclear Wastes

Barrera, J.[1]; Bagán, H.[1], Monroy F[2]., Tarancón, A.[1,3]; García, J.F[1].

1) Department of Chemical Engineering and Analytical Chemistry, University of Barcelona, Marti i Franqués, 1-11, ES-08028, Barcelona, Spain

2) Laboratorio de Desechos Radioactivos, Instituto Nacional de Investigaciones Nucleares, Carretera México-Toluca s/n. 52750 Mexico.

3) Serra-Húnter Program, Generalitat de Catalunya, Barcelona Spain.

Corresponding author's e-mail: jfgarcia@ub.edu

The decommissioning of nuclear facilities and fuel reprocessing are two of the main activities involved on the generation of materials contaminated in a higher or lesser extent with radionuclides. Decisions regarding to how these materials have to be disposed depends on their radioactivity levels and composition. Ion Exchange Resins are quite common materials among the nuclear wastes. Their activity is related to the separation processes in which they are involved. At any case, once they are used some amount of radioactivity may remain in and it is necessary to perform their characterization. This task is of special difficulty in the case of pure beta emitters in which direct, fast and reliable measurement are not available. In such scenario, for accurate determination the sample has to be taken, solved, treated to remove interfering radionuclides and measured by liquid scintillation or proportional counting. Analytical procedures for pure beta emitters generated in Nuclear Power plants like ^{99}Tc and ^{90}Sr have been developed for several types of samples although there are complex samples like Ion Exchange Resins in which few methods are described.

In this study, a procedure for ^{99}Tc and ^{90}Sr activities determination by using Plastic Scintillation Resins (PSresin) in Ion Exchange Resins used in nuclear waste purification process is presented.

The procedure includes the dissolution of the Ion Exchange resin using nitric acid and oxygen peroxide in a close vessel, the separation of ^{99}Tc and ^{90}Sr regarding interfering radionuclides by using an specific PSresins in a SPE cartridge, and the further measuring of the corresponding PSresin in the SPE cartridge format in a scintillation detector. For ^{99}Tc, PSresin used is based on quaternary amine extractant and separation is achieved at HCl 0.1M media; whereas for ^{90}Sr determination, PSresin is based on the use of a crown ether extractant and the separation conditions are HNO_3 6M.

The chemical recoveries of the separation with PSresin are very high in both cases, higher than 99% for ^{99}Tc and around 93 % for ^{90}Sr with variability of 3 % in this last case. Regarding the detection efficiencies, values are of 91 ± 2 % for ^{99}Tc and 94 ± 5 % for ^{90}Sr.

The procedure proposed can be considered a useful alternative to the already existing procedures because the use of PSresin reduces the reagents, wastes and time required to perform the analysis.

X-ray detection capabilities of plastic scintillators incorporated with hafnium oxide nanoparticles surface-modified with phenyl propionic acid

F. Hiyama1), T. Noguchi2), M. Koshimizu1),
S. Kishimoto3), R. Haruki3), F. Nishikido4), T. Yanagida5), Y. Fujimoto1), T. Aida2),
S. Takami6), T. Asdchiri2,6,7), K. Asai1)

1) Department of Applied Chemistry, Graduate School of Engineering, Tohoku University, 6-6-07 Aoba, Aramaki, Aoba-ku, Sendai 980-8579, Japan
2) New Industry Creation Hatchery Center, Tohoku University, 6-6-10 Aoba, Aramaki, Aoba-ku, Sendai 980-8579, Japan
3) High-Energy Accelerator Research Organization (KEK), 1-1 Oho, Tsukuba, 305-0801, Japan
4) National Institute of Radiological Science, National Institutes for Quantum and Ragiological Science and Technology, 4-9-1 Anagawa, Inage-ku, Chiba 263-8555, Japan
5) Nara Institute of Science and Technology, 8916-5 Takayama, 630-0192, Japan
6) Institute of Multidisciplinary Research for Advanced Materials, Tohoku University, 2-1-1 Katahira, Aoba-ku, Sendai 980-8577, Japan
7) Advanced Institute for Materials Research, Tohoku University, 2-1-1 Katahira, Aoba-ku, Sendai 980-8577, Japan

fumiyuki.hiyama.t2@dc.tohoku.ac.jp

[Introduction] The demand for fast detection of X-ray synchrotron radiation has increased due to the widespread use of intense beams and time resolved measurements. For proper detection of the X-ray synchrotron radiation, the detectors must show high detection efficiency and high time resolution. One of the potential candidates for fulfilling this demand is a detectors equipped with a plastic scintillator. However, the detection efficiency is low because the plastic scintillator comprises low atomic number elements. One solution to this problem is the use of heavy-metal-doped plastic scintillators. Our aim is to enhance the detection efficiency by doping HfO_2 nanoparticles surface-modified with phenyl propionic acid (PPA). The oxide has high transparency and high density. Thus, the incorporation of HfO_2 nanoparticles resolves the issues of low density and low effective atomic number in the case of plastic scintillators. The purpose of modifying the nanoparticle surface with PPA is to improve the dispersibility of the nanoparticles into the plastic. In this study, we fabricated plastic scintillators incorporated with HfO_2 nanoparticles surface-modified with PPA and characterized the X-ray detection capability.

[Experimental methods] HfO_2 nanoparticles were prepared using the supercritical hydrothermal synthesis method with PPA as the surface modifier. Subsequently, the nanoparticles were mixed with polystyrene and (2-(4-tert-butylphenyl)-5-(4-phenylphenyl)-1,3,4-oxadiazole fluorophore and were dissolved in tetrahydrofuran (THF). The nanoparticles were added in quantities of 0, 5, 10, 15 and 20 wt% to polystyrene. Finally, the samples were obtained after evaporating THF at room temperature for a day. The thickness of the samples was 1.0 mm. Pulse-height spectra and time resolution curves were recorded with 67.4 keV X-rays.

[Result and Discussion] Fig. 1 shows the pulse-height spectra of the scintillators exposed to a 67.4 keV

X-ray beam. The photoelectric peak position shifts to lower channels as the nanoparticle content increases, indicating that the light yield is lower at higher HfO_2 concentrations. It is thought that the nanoparticles act as quenchers, because the light yield is lower at higher HfO_2 concentrations. However, the total counts are higher for the plastic scintillators with added nanoparticles. The time resolution curves of the detectors equipped with the nanoparticle-added plastic scintillators are shown in Fig. 2. As Expected, no long tails were observed in the time-resolution curves, even at high nanoparticle concentrations. Table 1 summarizes the detection efficiency of X-ray photons and the time resolution for the different nanoparticle concentrations. The detection efficiency was calculated with comparison with NaI(Tl). The detection efficiency improved up to 3.3 times due to the addition of nanoparticles. However, there was a possibility of an optimum nanoparticle concentration because the detection efficiency decreased for the 20 wt% concentration. Nanosecond time resolution was successfully achieved for all the scintillators.

[Conclusion] The detection efficiency for high-energy X-rays was successfully enhanced with the incorporation of HfO_2 nanoparticles into plastic scintillators. Nanoparticles are prospective candidates for the fabrication of superior materials for plastic scintillators without deterioration of the emission intensity of scintillation.

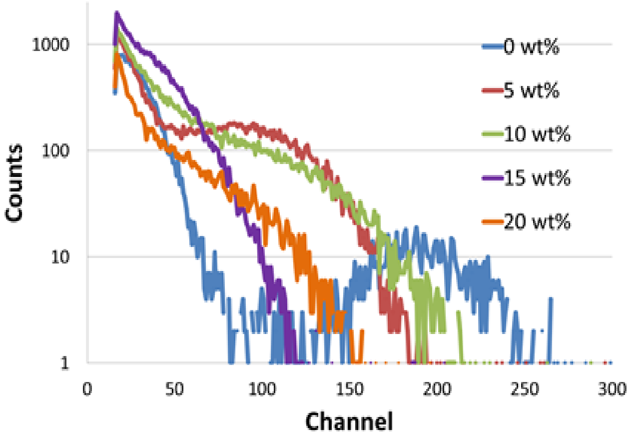


Fig. 1 Pulse-height spectra.

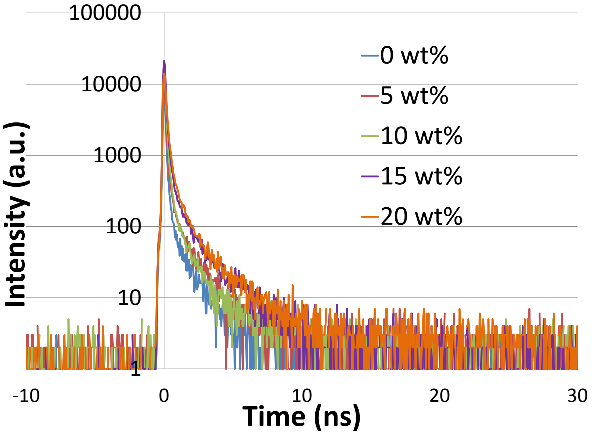


Fig. 2 Time resolution curves.

Table 1. Nanoparticle contents and detection efficiencies of 67.4 keV X-ray photons.

Nanoparticle content (wt%)	Thickness (mm)	Detection efficiency (1.0 mm of conversion)	Time resolution (ns)
0	1.00	0.30 (0.34)	0.97
5	0.90	0.87 (0.97)	0.94
10	0.95	1.02 (1.07)	0.93
15	1.0	1.12 (1.12)	0.99
20	1.0	0.34 (0.34)	1.15

Plastic scintillation foils for the measurement of the alpha and beta emitting radionuclides.

Merín, R.[1)], Tarancón, A.[1,2)], Bagán, H.[1)]; García J.F.[1)]

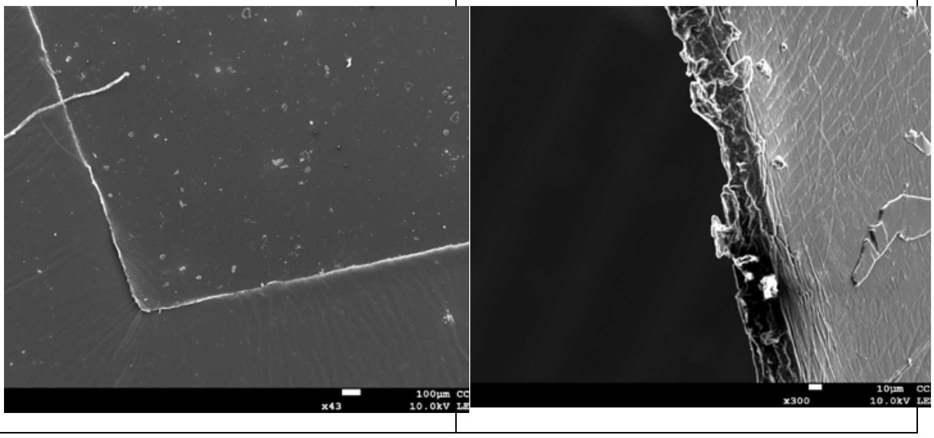
[1)] Department of Chemical Engineering and Analytical Chemistry, University of Barcelona, Marti i Franqués, 1-11, ES-08028, Barcelona, Spain.
[2)] Serra-Húnter Program, Generalitat de Catalunya, Barcelona Spain.

Corresponding author's e-mail: alex.tarancon@ub.edu

Plastic scintillators can be produced in different shapes and sizes. This capability is a great advantage on the design of innovative radioactivity measurements procedures. Thin film is a shape with few references and that can be an excellent platform for developing strips for radioactive contamination control or for fast and selective screening radioactivity analysis.

In this work, we present the preliminary results obtained with plastic scintillation foils prepared using the solvent evaporation method. Using this methodology, polystyrene based scintillating foils (PSfoils) of thickness between 45 and 125 micrometers have been produced by changing the amount of solution to be evaporated (from 30 to 60 g.). Changes on the thickness (from 40 to 200 micrometers) can also be achieved by modifying the polymer concentration in the solution (from 1.5 to 6%). Figure 1 shows the images of a foil made of polystyrene and 85 micrometers of thickness.

Fig. 1 PSfoils made of polystyrene



Through this methodology PSfoils obtained presented a reproducible thickness. As an example the mean value of thickness (measured at five different points) of 9 PSfoils produced by evaporation of 40 g of polymeric solution was of 62 ±10 micrometers.

PSfoils were characterized for the measurement of a high-energy beta emitter (^{36}Cl) and an alpha emitter (^{241}Am) in two counters (Quantulus and Triathler) in two configurations: by deposition of the radioactive solution over one PSfoil or by placing the radionuclide sticked between two PSfoils.

Results obtained in each configuration for polystyrene PSfoils of 82 ± 7 micrometers of thickness are shown in table 1

Table 1: Values of detection efficiency obtained for ^{36}Cl and ^{241}Am in two measuring configurations in the Quantulus detector.

	^{36}Cl	^{241}Am
One foil	63 ± 5	90 ± 5
Two foils sticked	97 ± 2	112 ± 13

In the case of ^{36}Cl, the energy of the beta particles is too high and in some cases it is not deposited enough energy on the foils to produce a detectable scintillating signal. Moreover spectrum is located at medium energies since not all energy is deposited in the scintillator. In the case of ^{241}Am the situation is improved since the probability of interaction due to the low penetration power of the alpha particles is higher and therefore the efficiency values are very high and spectrum is in form of a sharp peak located at high energies.

From the results obtained it can be concluded that PSfoils can be produced in a controlled and reproducible way. Moreover, they can be used for the measurement of beta and alpha radionuclides and represent a viable way to be use as a screening technique analysis, especially in nuclear decommissioning samples, or as a surface stick control detector for beta and alpha particles.

Fabrication and Characterization of 3D Printed Plastic Scintillator

Sunghwan Kim[1)], Youl-Hun Seoung [1)]

[1)] Department of Radiological Science in Cheongju University, Cheongju, 28503, Korea

Corresponding author's e-mail: kimsh@cju.ac.kr

Plastic scintillator is widely used for radiation detection in medical, science, industry applications. It possesses good properties such as tissue equivalence, short decay time, cheap, chemical stability and so on. In general, plastic scintillator is made by thermal polymerization method, which take long time and difficult to produce in complex shape. In this study, we fabricated and characterized a UV-curable plastic scintillator by 3D printing technology. The resin for 3D printing is prepared by ourselves. It used co-polymers TMPTA (trimethlylolpropane triacrylate, SigmaAldrich. Co.), DHPA (dipentaerythritol hexaacrylate, SigmaAldrich. Co.), and Ultima GoldTM organic scintillator (PerkinElmer). Darocur MBF (SigmaAldrich. Co.) is used as photo-initiator. Using DLP 3D printer (http://attosystem.co.kr) and the prepared resin, a cylindrical plastic scintillator is printed. X-ray induced luminescence spectrum of the 3D printed scintillator is measured with an X-ray tube from a DRGEM having a W-anode. For the emission spectrum, QE65000 fiber optic spectrometer (Ocean Optics) is used. Figure 1 shows emission spectrum obtained between 380~520 nm peaking at 423 nm. The observed emission spectrum match well with the quantum efficiency curve of the morden photomultiflier tubes. Decay time spectrum under alpha particles excitation is measured by using 400 MHz flash analog to digital converter. Signals from the PMT are fed directly into a 400 MHz FADC which is located in a VME (versa module eurocard) crate and read out by the Linux-operating personal computer through the VME-USB2 (universal serial bus) interface with a maximum data transfer rate of 10 Mbytes/s. The DAQ (data acquisition) system and the analysis program are written in the framework of the ROOT package. Decay time of plastic scintillation is found to be ~12 ns.

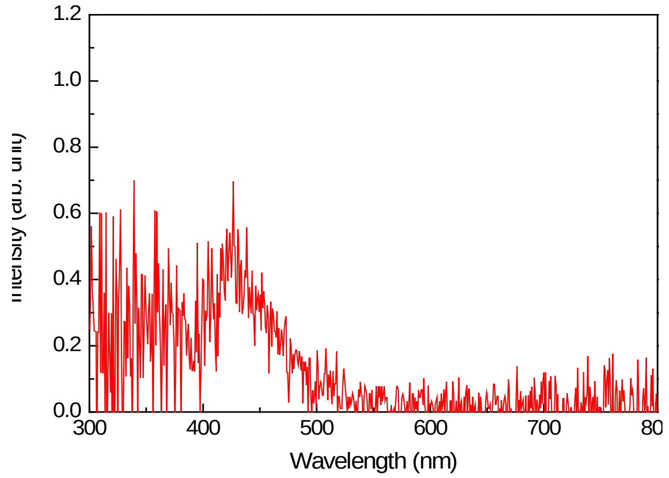


Fig. 1 Emission spectrum of 3D printed plastic scintillator excited by X-ray

We have successfully fabricated scintillation resin for 3D printing technique and characterized the prepared scintillator using our resin. Result show that the scintillator provides the possibility of combining 3D printing technology and it can find its applications in the human dosimetry etc.

Acknowledgment

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Rapid separation and easy measurement using plastic scintillator for radiostrontium in seawater

Haruka Minowa [1], Yuka Kato [2], Yoshimune Ogata [3]

[1] Radioisotope Research Facility, Jikei Univ., 3-25-8 Nishi-shimbashi, Minato-ku Tokyo 105-8461, Japan
[2] Measuring Systems Engineering Dept., Hitachi, Ltd., 6-22-1 Mure Mitaka-shi Tokyo, 181-8622, Japan
[3] Radioisotope Research Center, Medical Branch Nagoya University, 65 Tsurumai-cho, Showa-ku Nagoya-city 466-8550, Japan

Corresponding author's e-mail: minowa@jikei.ac.jp

The need for rapid analysis for emergency environmental samples has increased after the nuclear accident at Fukushima Nuclear Power Plant in March, 2011. The conventional method to analyze radiostrontium (Ref. 1) is time-consuming complicated procedures with deleterious substances. A simple, quick and safety analysis of radiostrontium in environmental sample is desired. The purpose of this work is to develop a new method to analyze and to measure radiostrontium.

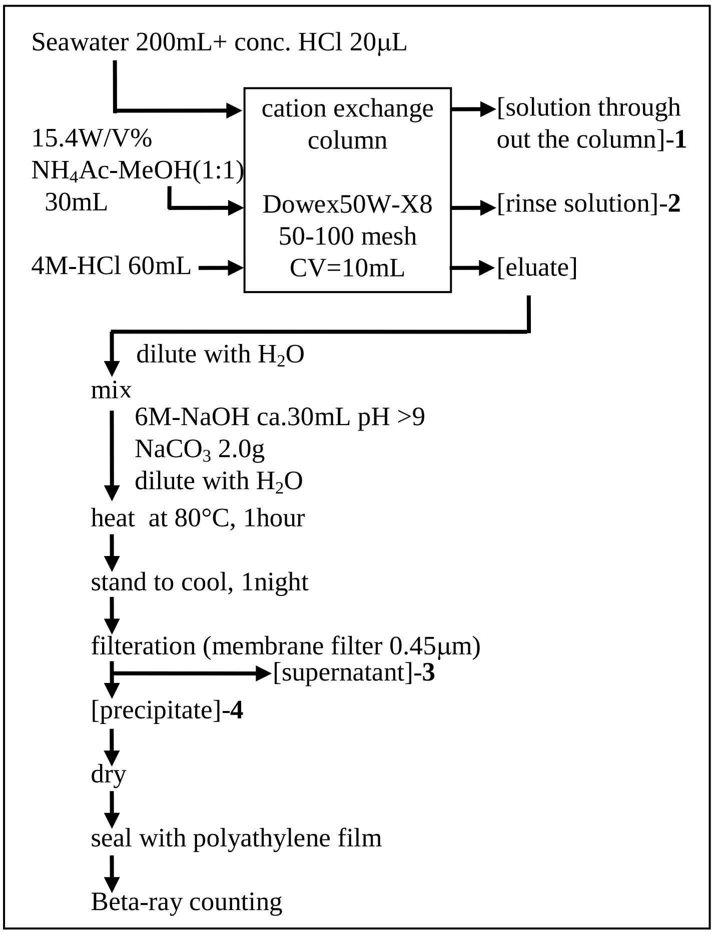


Fig. 1 Chemical procedure for strontium in seawater

The chemical procedure for separating strontium from seawater is shown in Fig. 1. A column separation with cation exchange was executed. Artificial seawater (Daigo's Artificial seawater SP, Nihon Pharmaceutical Co., Ltd.) or natural seawater, each volume was 200 mL, were loaded to a column with cation exchange resin (Dowex^{TM} 50W-X8, 10 mL of resin volume). After loading the sample solution through on the column to absorb cations, the column was rinsed with 30 mL of 15.4W/V% ammonium acetate-methanol (1:1) solution to elute the untargeted elements such as Ca, Mg and Pb. Continuously, Sr was eluted from the column with 60 mL of 4M-HCl. Two grams of powdered-calcium carbonate was added and the solution was heated for one hour at 80°C under alkali condition (pH>9). Then, carbonate was precipitated by cooling for one night. Finally, the precipitate was collected on a membrane filter (MF Cellulose membrane filter, 0.45μm, 45mmϕ, Millipore Corp.), and sealed with polyethylene films after dried at room temperature. The chemical yields of strontium in each fraction from artificial seawater measured by spiked ^{90}Sr or ^{85}Sr. In the case of ^{85}Sr, the activity was determined using a well-type NaI gamma counter (AccuFLEX γ7001, Hitachi, Ltd.).

Table 1 Yield of strontium for each fraction

Fraction	Artificial seawater [1)]
1. solution through out the column	0.4 %
2. rinse solution	3.8 %
3. supernatant	1.1 %
4. precipitate	94.8 %

[1)] spiked with ^{85}Sr

Figure 2 shows the elution profile of this procedure applied to artificial seawater. Natural Ca, Mg, Pb and Bi were separated from Sr and do not interfere for radioactivity measurement of Sr. The total chemical yield of Sr on the filter was 95% in this method as shown in Table 1. For the determination of activity of 200 mL of the seawater containing 8 mg/L of stable Sr as usual surface seawater, 10 mL of cation exchange resin was appropriate. The time required in this chemical separation procedure was 24 hours, although it took about two weeks in the conventional manner.

A new method to measure ^{90}Sr on the filter was developed using a plastic scintillator bottle with a commercial liquid scintillation counter. The sample was measured with a low-background liquid scintillation analyzer, (AccuFLEX LSC-LB7, Hitachi, Ltd.). The detail of the method is described in another study of ours. The minimum detectable concentration (MDC) of this method with cation exchange coupled with plastic scintillation counting is estimated as 0.1 BqL^{-1} for 50 minutes' measurement.

Simple method of the analysis for radiostrontium in seawater became possible by this procedure. This method is applicable to the rapid screening of seawater samples.

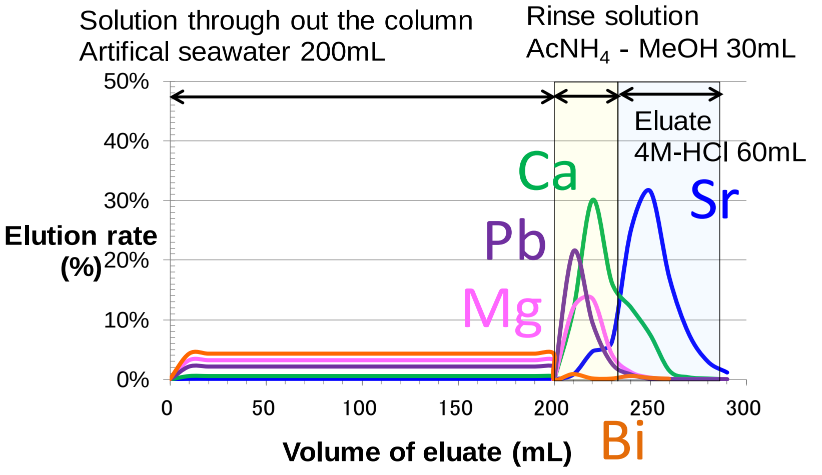


Fig. 2 The elution profile for cation exchange column

Reference:

1 . Ministry of Education, Culture, Sports, Science and Technology JAPAN, "Analysis Method for Radioactive Strontium" (in Japanese) Radiation Measurement Method Series 2, 2003

A Laboratory Study of Scintillation Counting of Polycarbonates

Krasimir K. Mitev (1)

(1) Faculty of Physics, Sofia University “St. Kliment Ohridski”, 1164 Sofia, Bulgaria
Corresponding author’s e-mail: kmitev@phys.uni-sofia.bg

The polycarbonates have high ^{222}Rn absorption ability [1] and this property have been largely used for development of ^{222}Rn measurement techniques (see [2,3] and the references therein). Recently it was observed that when ^{222}Rn is absorbed in the polycarbonate the alpha- and beta- particles emitted by it and its short-lived decay products create light in the polycarbonate material. The origin of this light is currently not accurately known - it may be Cherenkov radiation caused by high energy beta- particles of ^{214}Pb and ^{214}Bi [4] or scintillation light emitted by the polycarbonate material [5]. Despite that the exact origin of this light is not precisely known, it has already been used for radon measurement by liquid scintillation (LS) analyzer [4] and the method is shown to be applicable to radon in soil-gas measurements[6]. The future development and application of this approach requires more laboratory studies of the scintillation counting of polycarbonates exposed to ^{222}Rn.

The objective of this work is to perform a methodological laboratory study of the technique for measurement of ^{222}Rn absorbed in polycarbonate material by LS analyzers. For this purpose Makrofol DE polycarbonate pellets have been exposed to reference ^{222}Rn concentrations using the exposure facility described in [7]. After the exposure the pellets were transferred to 20 ml plastic LS vials and the ^{222}Rn activity in the vials was measured by a high purity germanium detector. Afterwards the vials were measured on a RackBeta 1219 LS analyzer (Fig. 1) and the counting efficiency for counting ^{222}Rn in equilibrium with its short-lived decay products is determined (Table 1). The experiment was repeated with ten samples and the mean counting efficiency of counting ^{222}Rn absorbed in 15.0 g polycarbonate pellets in equilibrium with its progeny in plastic LS vials with RackBeta1219 LS spectrometer was found to be $\varepsilon_c=1.18\pm0.12$.

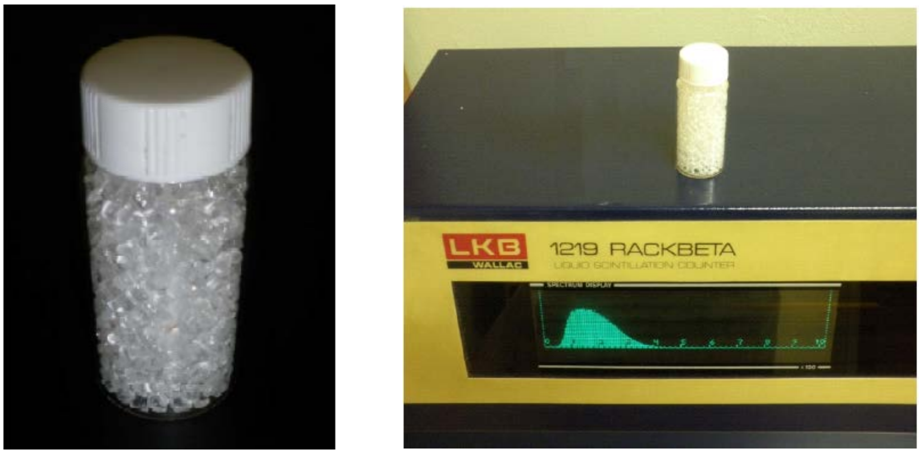
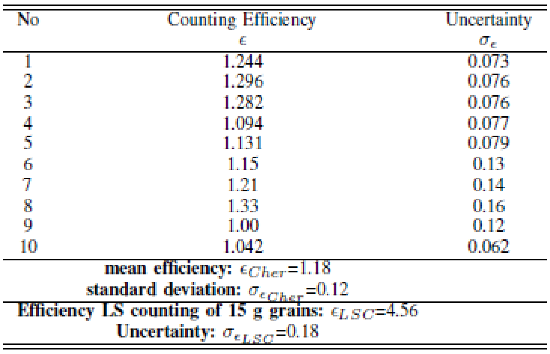


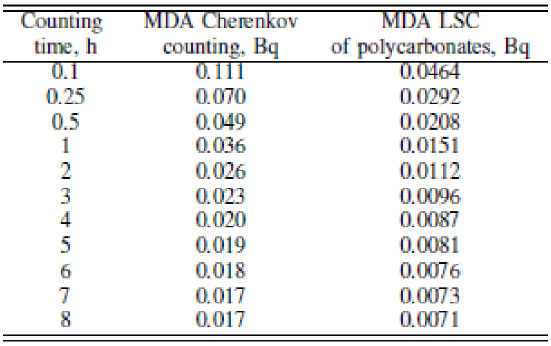
Figure 1. A 20 ml high performance glass vial fully filled with 15g polycarbonate pellets (left). The vial and the RackBeta LS analyzer in the course of measurement of a plastic PVE vial filled with 15g of polycarbonate pellets exposed to ^{222}Rn (right).

The above counting efficiency is lower than the counting efficiency of LS counting of polycarbonate pellets with liquid scintillator filled in the vials (ε_C=4.56±0.18, see [8] for detals), but is high enough to allow practical application. Note that there is an advantage of the direct measurement of polycarbonates compared to the measurement with liquid scintillation cocktail as there is no waste created during the sample preparation and few months after the measurement the pellets can be used again. In order to assess the sensitivity of the technique the approach of Curie [8] was used to estimate the minimal detectable activities (MDAs) when counting 15 g of polycarbonate pellets (referred to as "Cherenkov counting" in Table 2) and when counting 15 g polycarbonate pellets in a LS vial with added Ultima Gold LLT LS cocktail (referred to as "LSC counting of polycarbonates" in Table 2). The estimated MDAs of the scintillation counting of polycarbonate pellets are higher than those of the counting with LS cocktail, but are sufficiently low to allow practical application of the method such as those described in [6].



No	Counting Efficiency ϵ	Uncertainty σ_ϵ
1	1.244	0.073
2	1.296	0.076
3	1.282	0.076
4	1.094	0.077
5	1.131	0.079
6	1.15	0.13
7	1.21	0.14
8	1.33	0.16
9	1.00	0.12
10	1.042	0.062
mean efficiency: ϵ_{Cher}=1.18		
standard deviation: $\sigma_{\epsilon_{Cher}}$=0.12		
Efficiency LS counting of 15 g grains: ϵ_{LSC}=4.56		
Uncertainty: $\sigma_{\epsilon_{LSC}}$=0.18		

Table 1. Counting efficiencies of counting of 15 g of polycarbonate pellets in plastic LS vials.



Counting time, h	MDA Cherenkov counting, Bq	MDA LSC of polycarbonates, Bq
0.1	0.111	0.0464
0.25	0.070	0.0292
0.5	0.049	0.0208
1	0.036	0.0151
2	0.026	0.0112
3	0.023	0.0096
4	0.020	0.0087
5	0.019	0.0081
6	0.018	0.0076
7	0.017	0.0073
8	0.017	0.0071

Table 2. Minimal detectable activities of counting 15 g of polycarbonate pellets in plastic LS vials and in plastic LS vials filled with LS cocktail.

In another experiment we studied the relation between the net LS counting rate and the mass of the grains in the vials. A very good linear relation is observed (Fig. 2, left). The linearity experiment is performed also in various times after the closing of the vials and it was found that after 5 h after closing the vials (the time needed to attain secular equilibrium between the absorbed ^{222}Rn and its short-lived progeny in the vials) the dependence of the net LS counting rate vs. the mass of the pellets in the vials remain the same (Fig. 2, right). The results of a 350 min follow-up of the net counting rates of the samples are shown in Fig. 3 and compared to the expected ^{222}Rn decay curve. These results show that when a LS cocktail is added to the pellets inside the vials the net LS counting rate decreases approximately with the half-live of ^{222}Rn. The signal decrease of polycarbonate pellets without LS cocktail is faster (see Fig. 3) and we attribute this to desorption of ^{222}Rn from the pellets and its absorption in the walls of the plastic LS vials. Another experiment to study the transparency of the polycarbonate pellets to their light was also performed and show that the pellets are largely transparent to their own light (results not shown here, will be presented on the conference).

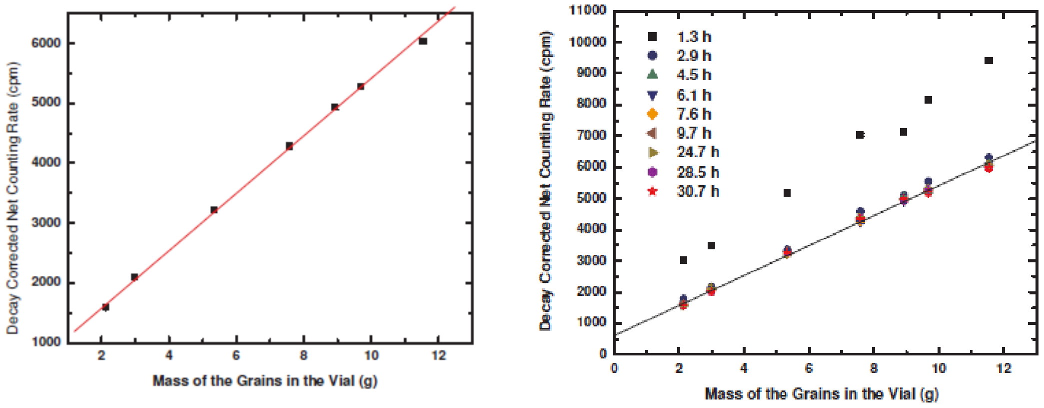


Figure 2. Decay corrected net LS counting rate vs mass of the grains in the plastic vials. Left: measurement 5 hours after closing the vials. Right: Follow-up to 30.7 h after closing the vials.

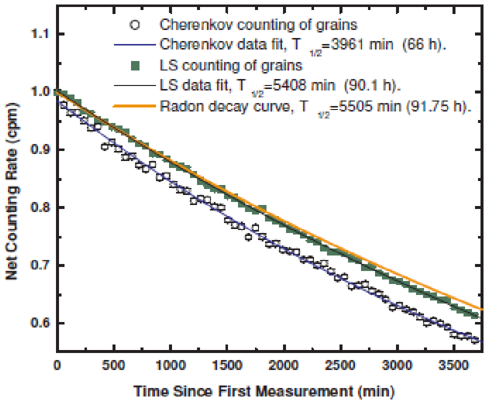


Figure 3. Follow-up of the net LS counting rate of 15 g polycarbonate pellets (Cherenkov counting) and 15 g polycarbonate pellets in plastic vial filled with liquid scintillator (LS counting).

This study presents methodological results for counting ^{222}Rn absorbed in polycarbonate pellets with LS analyzers. The counting efficiency is determined and MDAs of the counting of PC pellets are estimated. The LS counter response is linear with respect to the amount of the pellets in the vials and the follow-up of the net LS counting rate of the vials shows that it decreases faster than the ^{222}Rn half-life. The latter is attributed to desorption of the radon from the polycarbonates and absorption in the walls of the plastic vials. More results on the transparency of the polycarbonate pellets to their light and on the dependence of the radon absorption in the pellets will be presented on the conference. The results of this study will support the future development of techniques for radon in air and radon in soil-gas measurements based on scintillation counting of polycarbonates.

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Wednesday

3 May 2017

1. **Session: Application in natural radionuclides measurements and other topics**
2. **Session: Radionuclides metrology using LSC, standardization and quality assurance-2**

Monitoring of radioactivity of water supplies in Poland – first year of the implementation of the Euratom/51/2013 Directive

Izabela Chmielewska and Stanislaw Chalupnik

Silesian Centre for Environmental Radioactivity, Central Mining Institute, Katowice, Poland

Corresponding author's e-mail: ichmielewska@gig.eu

In November 2015 the Ordinance of the Ministry of Health related to the monitoring of radioactivity of drinking water supplies has been issued. It is an implementation of the European Commission Euratom/51/2013. Actually the year 2016 is the first year of the implementation of the Ordinance into practice. Very important for the introduction of the Ordinance is application of LS spectrometry methods for the monitoring of the radionuclides, present in water. Polish Ordinance requires monitoring of tritium, radon and radium isotopes in water. In the Silesian Centre for Environmental Radioactivity, Central Mining Institute, measurement methods for all mentioned above radionuclides are based on LSC.

In the paper results of the first year of the monitoring of radioactivity in drinking water supplies are presented. Almost 2000 water samples have been analysed within 2016, in most of the samples results of monitoring were below the detection limits of particular methods. No elevated concentrations of tritium have been found, only in several cases the elevated concentration of radon were found, exceeding 100 Bq/l. In case of radium isotopes, their activities are used to estimate the total indicative dose. For the several water supplies, the estimated annual dose for users exceeded reference value 0,1 mSv/year.

The preliminary investigations will continue till the end of the year 2017. At that time it will be possible to have to final pattern of distribution of radioactivity in drinking water supplies in Poland.

Determination of Lead isotope ratios for Nuclear Forensic signatures from uranium mine products in South Africa

Manny Mathuthu and Ntokozo Khumalo

North-West University (Mafikeng), Cnr University Drive and Albert Luthuli Road, Center for Applied Radiation Science and Technology (CARST), 2735, Mmabatho, South Africa

Corresponding author's e-mail: Manny.Mathuthu@nwu.ac.za

Abstract

Nuclear terrorism has led to newer investigative methods aimed at building national nuclear forensic libraries. The objective of this work was to resolve nuclear forensic signatures by applying the Liquid Scintillation Mass spectrometry for geo-chronology compared with Inductively Coupled Plasma Mass Spectrometry Isotopic Ratio technique on a uranium containing sample. The results show that uranium ore signatures are significantly different from processed uranium signatures. Uranium in the ore deposit exhibits geochemical signatures of the radiogenic or anthropogenic formations of the ore, while that from the processed tailing wastes exhibits geographical signatures of the mine location in South Africa. In this preliminary research, we also applied the Isoplot program to check the results from the ICP-MS ratio technique following the procedure adopted by Schoene [1].

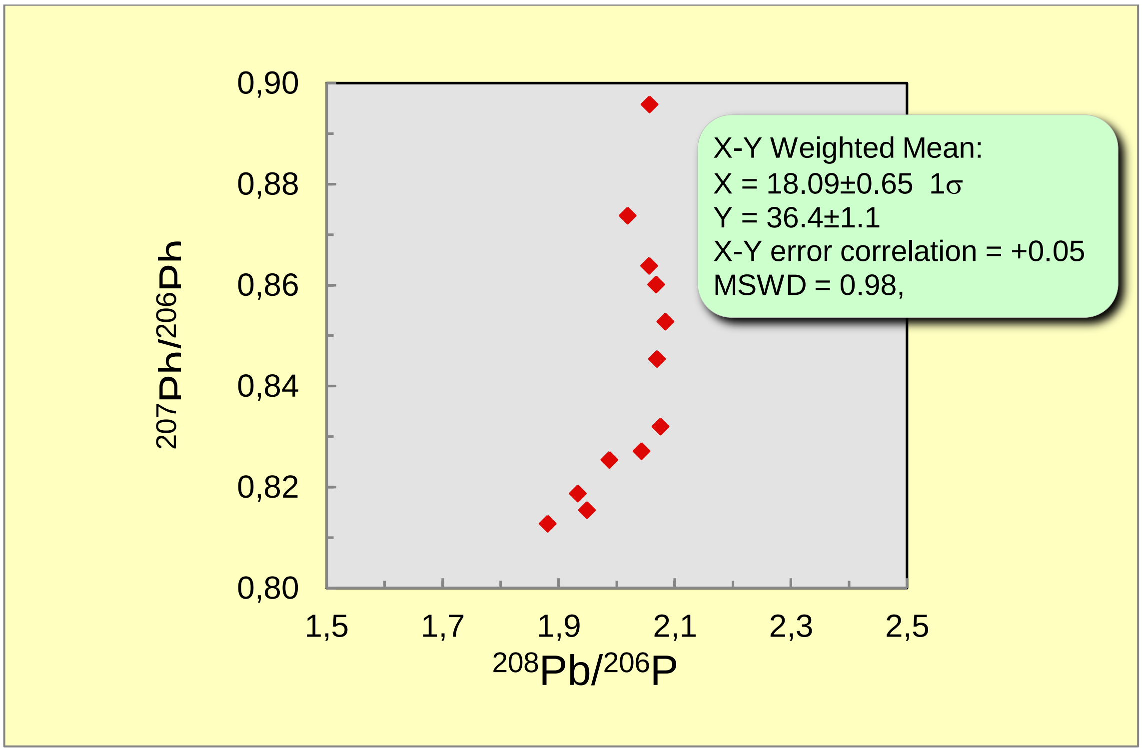
Table 1 Lead isotopic ratios for water samples before and after **^{208}Pb/^{206}Pb** normalization for mass balance correction using ICP-MS Isotopic ratio method [2]

Sample ID	**Measured**	**^{208}Pb/^{206}Pb - normalized**	**Measured**	**^{208}Pb/^{206}Pb normalized**	**Measured**	**^{208}Pb/^{206}Pb normalized**
	^{207}Pb/^{206}Pb	**^{207}Pb/^{206}Pb**	**^{208}Pb/^{206}Pb**	**^{208}Pb/^{206}Pb**	**^{204}Pb/^{206}Pb**	**^{204}Pb/^{206}Pb**
CW4	0.7715 ± 0.0065	0.8254 ± 0.0075	1.8575 ± 0.0095	1.9872 ± 0.097	0.0541 ± 0.0045	0.0578 ± 0.0062
WV14	0.8268 ±	0.8738 ±	1.9102 ±	2.0187 ±	0.0544 ±	0.0574 ±

	0.0071	0.0067	0.0093	0.0084	0.0045	0.0058
DAM31/3	0.7506 ± 0.0068	0.8154 ± 0.0064	1.7938 ± 0.0089	1.9487 ± 0.0096	0.0540 ± 0.0043	0.0586 ± 0.0049
WV13	0.7899 ± 0.0076	0.8271 ± 0.0067	1.9506 ± 0.0099	2.0426 ± 0.0097	0.0595 ± 0.0039	0.0623 ± 0.0057
DSW9/14	0.7276 ± 0.0067	0.8128 ± 0.0075	1.6839 ± 0.0089	1.8810 ± 0.0096	0.0368 ± 0.0029	0.0411 ±0.0033
DSW21/11	0.7488 ± 0.0065	0.8187 ± 0.0077	1.7678 ± 0.00 98	1.9329 ± 0.0088	0.0451 ± 0.0027	0.0493 ± 0.0036
DSW199	0.8155 ± 0.0059	0.8454 ± 0.0073	1.9961 ± 0.0096	2.0693 ± 0.0079	0.0581 ± 0.0039	0.0602 ± 0.0041
DSW7/12	0.8599 ± 0.0065	0.8958 ± 0.0068	1.9741 ± 0.0097	2.0564 ± 0.0089	0.0558 ± 0.0039	0.0581 ± 0.0037
DSW43/19	0.8290 ± 0.0059	0.8638 ± 0.0057	1.9730 ± 0.0096	2.0558 ± 0.0089	0.0514 ± 0.0043	0.0536 ± 0.0045
DSW39/17	0.8044 ± 0.0077	0.8320 ± 0.0076	2.0064 ± 0.0087	2.0753 ± 0.0095	0.0515 ± 0.0047	0.0532 ± 0.0046
DSW18/3	0.8270 ± 0.0069	0.8528 ± 0.0077	2.0208 ± 0.0089	2.0837 ± 0.0089	0.0559 ± 0.0045	0.0577 ± 0.0051
DSW4/5	0.8292 ± 0.0081	0.8601 ± 0.0079	1.9935 ± 0.0097	2.0678 ± 0.0099	0.0569 ± 0.0043	0.0590 ± 0.0038
AVER	0.7984 ±	0.8436 ±	1.9107 ±	2.0183 ±	0.0528 ±	0.0557 ±

	0.0069	0.0072	0.0088	0.0098	0.0044	0.0048
SDEV	**0.0407**	**0.0261**	**0.1101**	**0.0660**	**0.0063**	**0.0058**
%RSD	**5.0929**	**3.0962**	**5.7614**	**3.2722**	**11.8801**	**10.3423**
NIST SRM 981		0.91464 ± 0.00033		2.1681 ± 0.0008		0.059042 ± 0.000037

The Isoplot Code provides tools for the manipulation and plotting of radiogenic-isotope ratios, especially U-Pb, [2-8] [5, 9, 10]. The research chose this Isoplot program to check the results from the ICP-MS ratio technique following the procedure adopted by Schoene [1] as presented in Figure 1 below. The graph helps to analyze the ages of the samples from the mine.



X-Y Weighted Mean:
X = 18.09±0.65 1σ
Y = 36.4±1.1
X-Y error correlation = +0.05
MSWD = 0.98,

Fig. 1 ^{208}Pb/^{206}Pb Isochron

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Evaluation of Methods for the Determination of ^{210}Pb and ^{210}Po in Ca-rich Ash Samples

Banu Ozden[1], Taavi Vaasma[2], Madis Kiisk[2], Alan Henry Tkaczyk[2]

[1] Institute of Nuclear Sciences, Ege University, 35100, Bornova, İzmir, Turkey
[2] Institute of Physics, University of Tartu, W. Ostwaldi 1, 50411, Tartu, Estonia

Corresponding author's e-mail: ozdenbanu@yahoo.com

The measurement of natural radioactivity in the environment from naturally occurring radioactive material (NORM) is of significant importance for evaluating the radiological impact of non-nuclear industrial activities. Pb-210 and its daughter ^{210}Po, with half-lives of 22.3 years and 138 days respectively, are naturally occurring radionuclides widely used as tracers in environmental studies. These radionuclides originate from the radioactive decay of the naturally occurring ^{238}U series present in the earth's crust. The activity concentrations of these radionuclides can be enhanced due to the operation of phosphate, oil and gas industries, combustion of fossil fuels and other forms of energy production. Many different radiochemical methods are used globally to determine the activity concentration of naturally occurring radionuclides. Therefore, it is very important to choose suitable, precise and fit for purpose techniques and radiochemical methods.

The focus of this study is therefore on the measurement of ^{210}Po and ^{210}Pb in Ca-rich ash samples which are collected from oil shale-fired power plants in Estonia. Several parameters have been investigated such as digestion methods, separation of radionuclides, chemical efficiency, minimum detectable activity (MDA), and accuracy of the method. The most important factor in the success of the recommended method is the dissolution of the Ca-rich ash samples. For this reason; sample mass, type of acid, volume of reagents, temperature and time of heating have been promoted to understand the digestion process. We first demonstrated the most suitable microwave digestion method from a series of analysis for Ca-rich samples containing silicates. According to the analysis duration, the amount of acid used, the repeatability of analysis, the most suitable and available digestion method was selected for ash samples. The purpose of this study was to elucidate the applicability of the proposed methods for determination of the activity concentrations of ^{210}Pb and ^{210}Po in Ca-rich ash samples by LSC.

The samples were counted by an ultra-low level liquid scintillation spectrometer Quantulus 1220 (Perkin Elmer). The method was tested using IAEA (International Atomic Energy Agency) RGU-1 and IAEA-444 reference materials. Spectral calibration/peak identification which included the optimization of α/β discrimination system (pulse shape analyser - PSA), and recovery have been made by ^{209}Po and ^{210}Pb standard solutions.

This study demonstrates the applicability of the proposed method for determination of the activity concentrations of ^{210}Pb and ^{210}Po in Ca-rich ash samples which are collected from oil shale-fired power plants in Estonia. The ^{210}Po results obtained for RGU-1 and IAEA-444 reference materials indicate that the dissolution and radiochemical separation method is applicable for the samples with higher activity. Chi-square goodness-of-fit test was used to compare the results for the low and high levels of activity measurements. There is a significant difference in the measurement of low-level parallel samples (x^2=11.154, P<0.05). However, Chi-square goodness-of-fit test indicated that the difference in the measurements of high-level of parallel samples is not significant (x^2=2.219, P>0.05). The results indicated that the method is suitable in terms of repeatability for high-level of parallel samples. The differences observed in efficiency for low-level of parallel samples could be linked to the inappropriate LSC method for ^{210}Po measurements.

Studies of anthropogenic ^{129}I using accelerator mass spectrometry from corals and oceanwater samples.

Ching-Chih Chang1,2), George S Burr1,3,4), A J Timothy Jull1.2), Joellen Russell2), Yue-Gau Chen3), Chuan-Chou Shen3), Dana Biddulph1), Lara White1), Nancy G Prouty5) and Weijian Zhou4)

1) NSF-Arizona AMS Laboratory, University of Arizona, Tucson, AZ, USA
2) Department of Geosciences, University of Arizona, Tucson, AZ, USA
3) Department of Geosciences, National Taiwan University, Taipei, Taiwan
4) Shaanxi Province Key Laboratory of Accelerator Mass Spectrometry Technology and Application, Institute of Earth Environment, CAS, Xi’an 710043, China
5) US Geological Survey Pacific Coastal & Marine Science Center, Santa Cruz, CA, USA

Corresponding author’s e-mail: jull@email.arizona.edu

The long-lived radionuclide ^{129}I (15.7 × 10^6 yr) is well-known as a useful environmental tracer. It is easily measured using accelerator mass spectrometry (AMS). At present, the global ^{129}I in surface water is about 1-2 orders of magnitude higher than pre-1990 levels. Since the 1990s, anthropogenic ^{129}I produced from nuclear reprocessing plants has been the primary source of ^{129}I in marine surface waters of the Atlantic and around the globe. Prior to that time, nuclear weapons testing was the primary source and peaked in the 1950s-1960s. Corals that produce annual growth layers offer an excellent archive to study ^{129}I and have been used in a few cases to reconstruct time series that reach back to pre-nuclear times. We present ^{129}I time series records from two sites in the South China Sea; Rabaul, Papua New Guinea, in the South Pacific; and Guam in the North Pacific to complement earlier South Pacific records (specifically Solomon Islands and Easter Island). Taken together, these provide a broad picture of the distribution and spread of ^{129}I in the Pacific over the past 60 years due to atmospheric nuclear testing. We also report on studies to trace the ^{129}I from the Fukushima nuclear incident in 2011 and measurements from the US West Coast and elsewhere. Although samples close to Japan show an increase in ^{129}I, this has not so far been observed (up to Dec 2016) above the signal background on the West Coast nor in Taiwan.

Comprehensive development of radiochemical methods at DTU Nutech

Jixin Qiao

The Center for Nuclear Technologies, Technical University of Denmark, DTU Risø Campus, Roskilde, Denmark

Corresponding author's e-mail: jiqi@dtu.dk

The Center for Nuclear Technologies, Technical University of Denmark (DTU Nutech) has long-term experience on development of radiochemical methods for various hard-to-measure radionuclides. These methods have been successfully applied to analysis environmental samples for Danish environmental monitoring program operated at DTU Nutech, tracer and radioecological studies, as well as characterization of various waste materials from nuclear decommissioning activities.

This paper aims to give an overview of analytical methods developed over the past six decades at DTU Nutech (Fig. 1), with focuses on technical features of high effective radiochemical methods employed for determination of typical beta emitters (e.g., ^{3}H, ^{14}C, ^{55}Fe, ^{63}Ni) with the use of LSC. Analytical performance of these radioanalytical methods are discussed in detail. Examples are also given to illustrate their application in the work related to the decommissioning of Danish nuclear reactors, at Risø, Roskilde, Denmark.

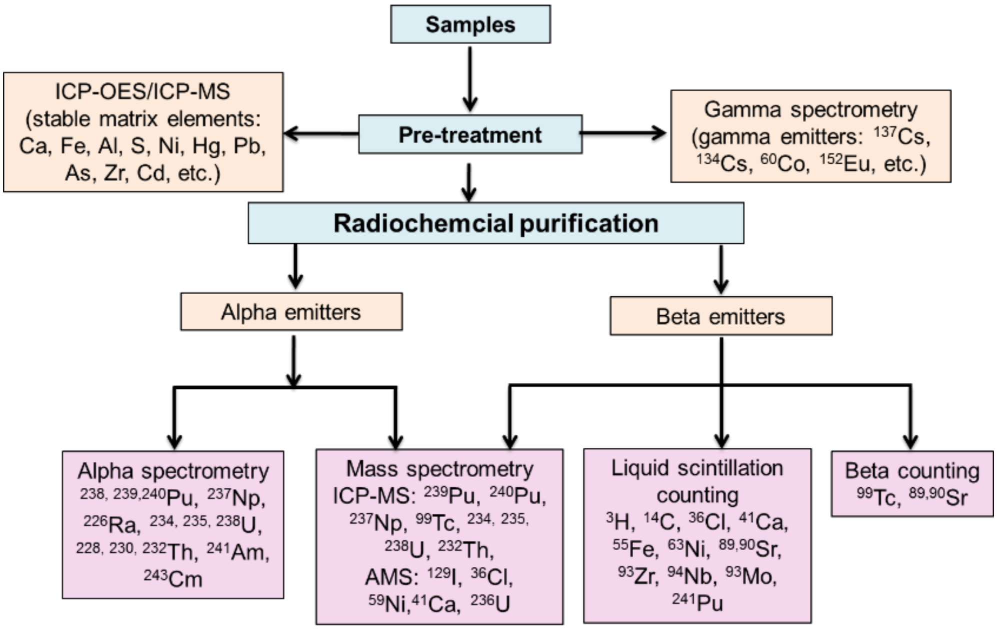

Fig. 1. Measurement techniques established at DTU Nutech for comprehensive determination of radionuclides.

Simulation of neutron encode imaging with liquid scintillator filled capillary array

Leifeng Cao 1),2), Jian Teng 2),

1) Laser Fusion Research Center, China Academy of Engineering Physics, P. O. Box 919-986-6, 621900, China
2) Science And Technology on Plasma Laboratory, P. O. Box 919-986-6, 621900, China

Corresponding author’s e-mail: leifeng.cao@caep.cn

Liquid scintillator filled capillary array has been suggested to be a detector for neutron encode imaging in laser driven inertial confinement fusion research. The aim is to get better spatial resolution than former used method. Primary simulations were performed with Monte Carlo method and it provided meaningful information for judging the feasibility. Further possible efforts for such an issue were also discussed.

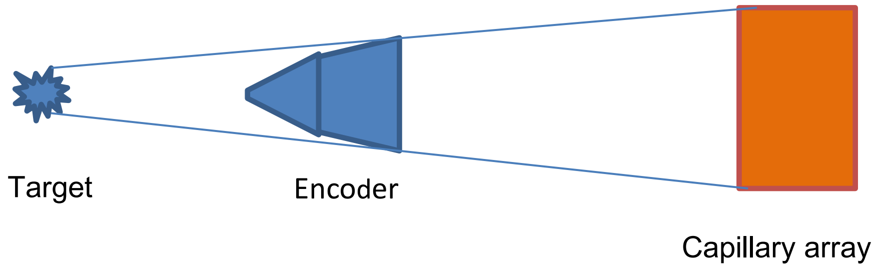

Fig. 1 scheme of the system

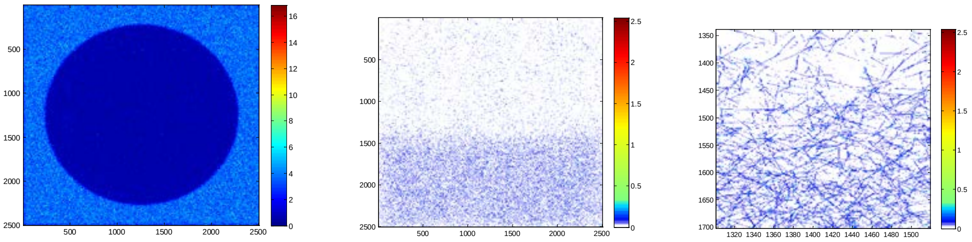

Fig. 2 encoded image and its different zoom in

Estimation of α-emitting polonium radionuclides in proton irradiated lead bismuth targets by LSC-TDCR method

Dibyasree Choudhury, Susanta Lahiri*
Saha Institute of Nuclear Physics, 1/AF Bidhannagar, Kolkata-700064, India
*E-mail: susanta.lahiri@saha.ac.in

Lead bismuth eutectic (LBE) is now projected as converter targets in various upcoming facilities like EURISOL projects. The incoming high intensity, high energy proton beam will produce intense flux of neutrons which will ultimately help to produce secondary energetic beams upon reaction with fissile material like UC_x. Besides its use in production of secondary beam, the LBE target may itself act as an intense source of clinically important and exotic radionuclides produced through various nuclear reactions like spallation, fission etc.

Recently, an experiment was carried out at CERN-ISOLDE to make a total inventory of radionuclides produced in 1.4 GeV irradiated thick LBE target (1). In this experiment 111 radionuclides including ^{206,207}Po were quantitatively identified in a cylindrical LBE target of 8mm length and 6mm diameter. However, α-emitting ^{208,209,210} Po could not be identified in the LBE targets for understandable reason, though their production is highly probable through ^{209}Bi (p,xn) or (p,g) reactions. These α-emitting radionuclides are of concern especially from the radioprotection point of view.

In this paper for the first time, an attempt has been made to quantify the total activity of alpha emitting polonium radionuclides in proton irradiated LBE targets. The liquid scintillation counting technique in conjunction with triple to double coincidence ratio has been applied for quantification of polonium radionuclides

The entire experiment was carried out in a low energy accelerator which is an experimental simulation of the earlier experiment carried out with 1.4GeV proton beam. The LBE targets of 4.8mg/cm^2 thickness were irradiated with proton beam of varying energies i.e. 9-18MeV at Variable Energy Cyclotron Centre (VECC), Kolkata, India. For measurement by LSC, the irradiated targets were dissolved separately in measured amount of 0.1M nitric acid. A fraction of these solutions were mixed with aqualite ultra low level cocktail solution. The samples were subjected to liquid scintillation counting by TDCR technique in Hidex 300SL LSC counter. The alpha triple spectrum (figure1) clearly indicated the presence of two different polonium radionuclides in the matrix. The alpha counts are being taken at different time intervals to confirm the half life of the produced polonium radionuclide. The total amount of polonium produced at each energy has been quantified. Polonium production has been found to increase with increase in proton energy (figure 2).

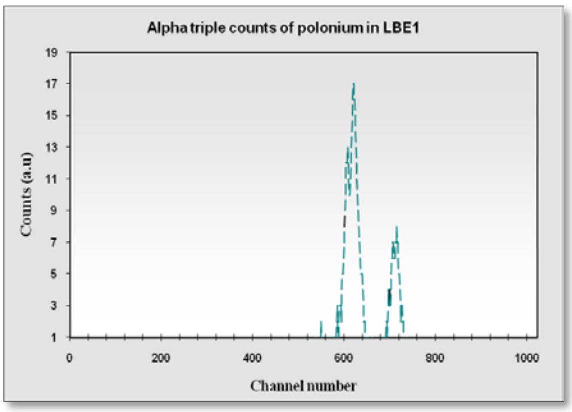

Fig. 1: Alpha triple spectrum of proton irradiated LBE target

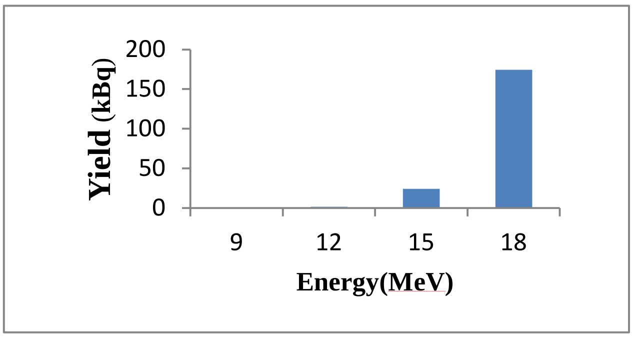

Fig 2: Diagram of yield of polonium at different proton energies

Estimation of polonium radionuclides turns out to be extremely significant. Alpha emitting polonium radionuclides has been quantified in a complex matrix such as LBE target for the first time. It is difficult to obtain proper standard for such type of complex systems. Hence the TDCR technique is only way to find out the absolute activity. However, more rigorous experiment is needed in future to validate the results.

The work has been carried out as part of the SINP-DAE-12 five year plan project "Trace Ultratrace Analysis and Isotope Production (TULIP)"

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Uncertainty Assessment in Liquid Scintillation Counting

B. E. Zimmerman [1]

[1] Physical Measurement Laboratory, National Institute of Standards and Technology, 100 Bureau Dr., Gaithersburg, MD, 20899-8462 USA

Corresponding author's e-mail: bez@nist.gov

The identification and quantification of uncertainty components is a critical part of any measurement; indeed, no result should be considered complete unless it is accompanied by a comprehensive uncertainty budget. The philosophies and methods for carrying out uncertainty analyses vary between laboratories and even between individuals, but the general process is defined by the Guide to Uncertainty in Measurement [1] and its supplements. For many counting techniques, the measurement model is simple, with a small number of variables that are linked by a linear mathematical relationship. This allows the error propagation equation to be easily defined. For radionuclide metrology applications of liquid scintillation (LS) counting, the situation is more complicated. This is due not only to the relatively large number of possible variables, many of which are highly correlated, that can go into the determination of an activity value, but also because of the need to apply calculational models to determine the detection efficiency. This prevents the measurement equation from being written in a simple, closed form. The fact that every radionuclide, because of its unique chemical and decay properties, presents its own set of measurement challenges also suggests that a "cookbook" approach to assessing uncertainties is impractical. This means that a measurement model and analysis approach needs to be defined for every case.

This talk is intended to be pedagogical in nature and will first provide an overview of the GUM approach to uncertainty assessment in LS-based metrology applications. Using examples from the CIEMAT/NIST efficiency tracing and Triple-to-Double Coincidence Ratio methods, the development of measurement models for these techniques will then be explored. Special emphasis will be placed on the use of sensitivity analysis and Monte Carlo techniques for estimating the magnitude of certain components. Finally, a method to deal with certain types of correlated LS counting data will be presented.

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With a little bit of effort, manufacturers could make better LS counters, even suitable for radionuclide metrology!

Philippe Cassette

CEA, LIST, Laboratoire National Henri Becquerel (LNE-LNHB), Bât. 602 PC111, CEA-Saclay 91191 Gif-sur-Yvette Cedex, France

Corresponding author's e-mail: philippe.cassette@cea.fr

In the field of radionuclide metrology, two main LSC measurement methods are used, both based on the free parameter model [1]: the Triple to Double Coincidence Ratio method (TDCR) requiring a specific 3-PMT counter and the CIEMAT/NIST efficiency tracing method (CNET) which can be implemented on a standard commercial LS counter. There is a consensus to consider that these methods are complementary and that the combination of the two approaches allow the determination of the optimal parameters to use for the calculation of the detection efficiency. This concerns the value of the kB parameter governing the model of the non-linearity of the scintillator, but this approach is also useful to determine the optimal shape factor of the beta spectrum to be used. During a recent specialists' meeting on LS techniques in radionuclide standardization, the attendee faced the sad conclusion that, nowadays, there is no longer a commercial LS counter available on the market which could fulfill the requirements of a metrological instrument for the application of the CNET method. There are two main reasons for this situation; firstly several companies making good instruments disappeared during the last twenty years and secondly most of the users of LS counters, who deal with environmental measurements, are not always aware of the drawbacks of the instruments they use. In this context we think that commercial instruments can and must be upgraded to meet the needs of the metrological community, and we present in this paper the mandatory requirements for decent LS instruments which would benefit all users.

The key points for improvement, ta achieve metrological quality concern the following main points: accuracy and resolution of the measurement date and time reported by the counter, accuracy of the live-time determination, linearity of the counter, determination and control of the threshold of the PMT's discriminator levels, optimization of the dead-time unit, definition of a robust quenching level indicator, traceability of the reference clock used to define the measurement live-time, possibility to disconnect the active guard (or any background reduction system), and possibility to record and save the spectrum obtained with the external source to determine the quenching index. All these points will be detailed in this paper and it will be shown that most of the proposed improvements do not require significant modifications of the actual counters, and thus can be realized with only a modest investment.

Even if the LS metrology community is not so large, instrument manufacturers must not underestimate the influence of the National Metrology Institutes on all the laboratories using LS techniques, especially when these laboratories are accredited. We strongly think that the future of LSC instruments is in the improvement of their metrological properties and not in the addition of gadgets developed for marketing purposes.

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Implementation of ISO/IEC 17025 in a low level liquid scintillation tritium laboratory

Ll.Pujol, M.E. Perez-Zabaleta

Centro de Estudios y Experimentación de Obras Públicas (CEDEX), Alfonso XII, 3, 28014 Madrid, Spain

Corresponding author's e-mail: luis.pujol@cedex.es

The practical experience on the implementation of ISO/IEC 17025 compliant quality system in a low level liquid scintillation tritium laboratory of CEDEX (Madrid, Spain) is described. This paper summarizes the need for a quality system and accreditation, the process of a quality system implementation, the quality system structures, and the formal accreditation of our laboratory by the Spanish Accreditation Body (ENAC). Also, the improvements in the management, technical and service quality which resulted from implementation of this system are briefly reported.

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Bilateral comparison of ^{14}C activity measurements at the NCBJ RC POLATOM and the ENEA-INMRI

T. Ziemek [1], M. Capogni [2], R. Broda [1], P. De Felice[2], T. Dziel [1], A. Listkowska [1]

[1] National Centre for Nuclear Research Radioisotope Centre POLATOM, Andrzeja Sołtana 7, 05-400 Otwock - Świerk, Poland

[2] ENEA-Italian National Institute of Ionizing Radiation Metrology (ENEA-INMRI), Via Anguillarese, 301, 00123 Santa Maria di Galeria (Roma), Italy

Corresponding author's e-mail: tomasz.ziemek@polatom.pl; marco.capogni@enea.it

A bilateral comparison between the Italian National Institute of Ionizing Radiation Metrology, belonging to ENEA (Italy) and named ENEA-INMRI, and the National Centre for Nuclear Research Radioisotope Centre POLATOM (Poland) on activity measurements of a solution of ^{14}C was organized in the year 2015 and piloted by POLATOM.

The aim of the comparison was to implement in both Institutes a new national ^{14}C activity standard and check the performances of the TDCR counters available both at POLATOM and ENEA by measuring a medium-energy long-lived (half-life of 5700 y) pure-beta emitter ^{14}C ($E_{e,max}$ = 156.48 keV), which is of particular importance in many applications. Particularly it was interesting to compare results of on-line in the FPGA digital board and off-line in the CAEN Desktop digitizer analyses in two TDCR systems. ^{14}C standards are used, in fact, in metrology for self-normalizing and calibration of LSC counters. ^{14}C is also used in radiometric dating. Furthermore, waste containing ^{14}C are continuously generated by the nuclear industry in nuclear reactor operations, spent fuel reprocessing, radioisotope production and medical research [1]. The discharge limits for ^{14}C in most countries are essentially based on the recommendation of the ICRP [2]. Assessing the impact of ^{14}C releases in the environment means setting-up and implementing technical and organizational procedures for ensuring public health. In fact, measurements of ^{14}C in coolant circuit of BWR and PWR reactors are required by U.S. Nuclear Regulatory Commission. New standards of this radionuclide could aid plant (and other facility at which fission reactions or spent fuel processing take place) in making accurate and precise measurements.

To perform the comparison, a set of six sources in 20 mL high-performance PerkinElmer glass vials filled-in with 10 mL of Ultima Gold liquid scintillator, containing radioactive master ^{14}C solution, was prepared. The above sources were measured at POLATOM in the period of two days. Three of the ready-to-measure sources in liquid scintillator have been sent to the ENEA-INMRI.
The solution was measured at POLATOM by the TDCR method in two various counters. The first one was a "classical" TDCR system with three PMTs working in coincidence with a liquid scintillator. The second one with an additional NaI(Tl) detector in gamma channel was equipped with the FPGA XILINX Spartan-3AN digital board [3]. The sources were measured also by the CIEMAT/NIST method in the Tri-Carb 2910 TR and the Wallac 1411 scintillation counters [4]. The measurements at ENEA-INMRI were performed using the TDCR method in two different counters: the Hidex 300SL 'Metro' version counter [5] and a portable TDCR counter equipped with

the CAEN Desktop digitizer DT5720 [6]. The new electronics, FPGA based and implemented on the home-made TDCR counters at both Institutes, were also tested.

All results obtained by both laboratories were in excellent agreement. The degree of equivalence between two laboratories was $D_{ij} = A_i - A_j$ = -0.08 kBq/g with its expanded uncertainty U_{ij} = 0.38 kBq/g (k = 2) [7].

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Long-term performance of liquid scintillation laboratory of LRM, SCK•CEN

Vasile M[1], Verrezen F.[1], Loots H.[1], Verstrepen D.[1], Bruggeman M.[1]

[1] SCK•CEN, Boeretang 200, 2400, Mol, Belgium

Corresponding author's e-mail: fverreze@sckcen.be

The Low-level Radioactivity Measurements (LRM) Expert Group is part of the Environmental, Health and Safety (EHS) Institute of the Belgian Nuclear Research Center (SCK•CEN). LRM is performing many different types of radionuclear measurements for internal and external clients in many types of environmental and biological samples. In order to guarantee the quality of our results to our clients, LRM is accredited according to ISO 17025 and certified according to ISO 14001 [1, 2]. The organization of LRM is subdivided in several laboratories, such as a Sampling and Sample Preparation group, the Alpha-particle spectrometry, Gross Alpha/Beta counting and Gamma-ray spectrometry laboratories, the laboratories for the measurement of Strontium/Iodine and Radium-Radon and also the Liquid Scintillation Counting laboratory. One of the requirements of the ISO 17025 is to demonstrate the quality of our methods and the reliability of our results by participating in proficiency tests.

The Liquid Scintillation Laboratory of LRM has a long history of proving its capability and quality of the reported results. The laboratory is equipped with three TriCarb (Perkin Elmer) and two Quantulus 1220 (Perkin Elmer) counters. LRM has been participating since 1990 in different intercomparison exercises organized by renowned organizations such as PROCORAD, IAEA, BfS and NPL, focusing mainly on ^{3}H and ^{14}C measurements in urine or environmental samples.

For the evaluation of the reported results the zeta-score is used and the figures containing the obtained values by LRM along the years for ^{3}H and ^{14}C measurements will be presented and discussed in the paper.

Recently a method for evaluating the proficiency test results and for the confirmation of the calculated uncertainty of the results, based on the external quality assessment of the data was reported [3, 4]. The method was first described in detail by Meijer at al., 2002 and presented on the 2016 PROCORAD conference by C. Guichet [3, 4]. LRM applied this method using the data from the last 25 years to compare our reported uncertainty with the long-term uncertainty obtained applying the method described by Meijer at al., 2002 [3]. Discussion and comparison of the results will be described in detail in the paper.

The long-term analysis revealed that the results of the Liquid Scintillation laboratory of LRM are consistently in good agreement with the target values, that the calculated uncertainty is very well covering all sources of uncertainty and that the applied measurement methods are not subjected to any absolute or relative method bias.

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LSC calibration stability over long timescales

Pawel Gaca, Phillip E. Warwick, Ian W Croudace

GAU-Radioanalytical Laboratories, University of Southampton, National Oceanography Centre, European Way, Southampton SO14 3ZH, United Kingdom

Liquid scintillation spectrometers are widely used in nuclear and radiopharmaceutical industries for the quantification of alpha and beta emitting radionuclides. Robust calibration of liquid scintillation (LS) spectrometers is fundamental to accurate liquid scintillation measurement. However, the detection efficiency will vary depending on a range of factors including sample composition, scintillant type etc and this must be accurately corrected for during measurement. In addition, the detection efficiency will vary with time due to deterioration of instrument optics. The most commonly used approach is to calibrate the LS spectrometers using a set of standards of known activity and a varying amount of quench.

Full calibration of a liquid scintillation counter is time consuming, particularly if a large range of radionuclides are analysed by the laboratory. The frequency at which full calibrations are performed will depend on the laboratory and is based on a range of practical and operational factors. However, to inform this choice, it is useful to understand the long term stability of liquid scintillation counters and the rate of degradation of the system. In addition, knowledge of the long term stability of the LS calibration can be used in assessing method uncertainties associated with calibration. This work presents the summary of the Quantulus LSC calibrations performed annually using various radionuclides: ^{63}Ni, ^{55}Fe, ^{36}Cl, ^{45}Ca, ^{147}Pm, ^{241}Pu, ^{99}Tc for a period of 10 years and assesses the long term stability of the instruments.

Counting efficiency calibrations for all the radionuclides were prepared using certified standard solutions. The different quench level for any given set of calibration standards was achieved by the addition of varying amounts of a quenching agent (e.g. nitromethane) or by using different sample solution to scintillation cocktail ratios. The deviations in counting efficiencies were checked for selected SQPE values representing varying degree of quench; from heavily quenched samples (SQPE 1), through a mid-level (SQPE 2) to relatively weakly affected ones (SQPE 3). Counting efficiency data for ^{99}Tc (βmax= 293.6 keV) and ^{3}H (βmax= 18.571 keV) are presented in Fig 1 and Fig. 2.

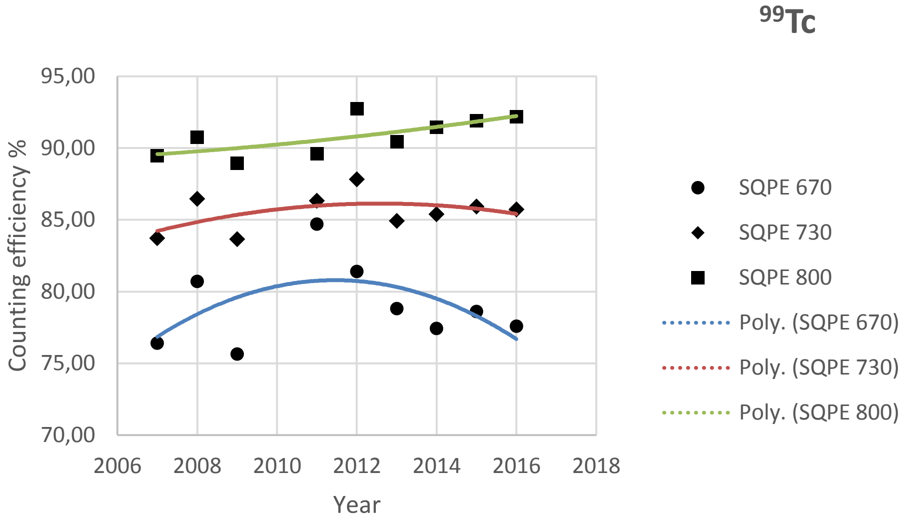

Fig. 1 Counting efficiency change for selected SQPE values for ^{99}Tc.

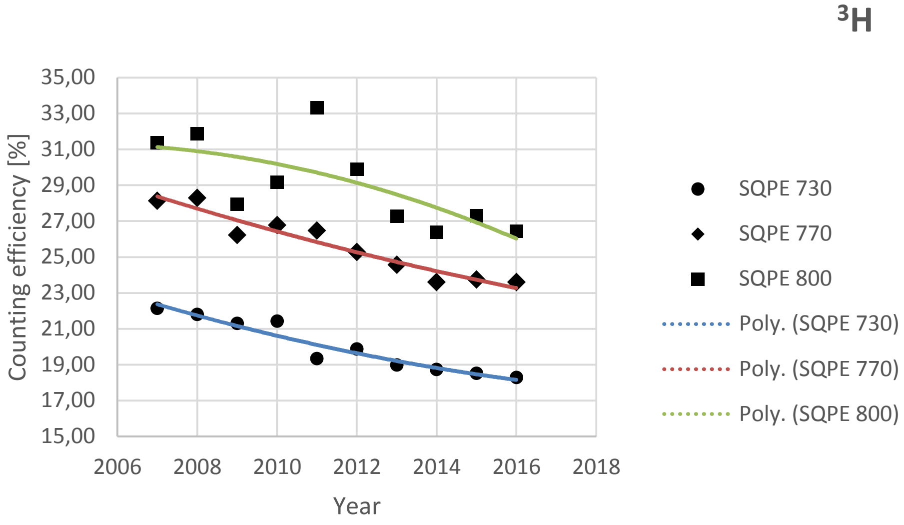

Fig. 2 Counting efficiency change for selected SQPE values for ^{3}H.

The observed deviations suggest much higher counting efficiency change for radionuclides measured in a low-energy part of the LSC spectrum than for the high-energy beta emitters. For ^{3}H, the counting efficiency shows slow and regular decreasing trend for all the tested SQPE values – on average the counting efficiency drop is approx. 0.5 % per annum. The results for ^{99}Tc, although exhibiting a higher degree of scatter, show no clear decreasing trend – the counting efficiency for all the tested SQPE values seem to be stable over time scale examined. Such result suggest the possibility of extending the calibration periods beyond the typical annual regime to longer periods for high-energy beta emitters and the necessity to maintain tighter calibration frequency for low-energy beta emitters.

Thursday

4 May 2017

1. **Session: Plastic scintillator and application**
2. **Session: Application in environmental radioactivity-2**
3. **Session: Application in medicine and other topics**
4. **Poster session-2**

Plastic scintillators and related analytical proposals for radionuclide analysis.

García J.F.1), Tarancón, A.1,2), Bagán, H.1);

1) Department of Chemical Engineering and Analytical Chemistry, University of Barcelona, Marti i Franqués, 1-11, ES-08028, Barcelona, Spain.
2) Serra-Húnter Program, Generalitat de Catalunya, Barcelona Spain.

Corresponding author's e-mail: jfgarcia@ub.edu

Liquid and Plastic scintillation started together on the decade of the 1960's. In spite of their similar potential capabilities, plastic evolved to be used in large area detectors whereas liquid scintillation focused its use on radionuclide activity determination. This was the situation until the end of 1990's when an increasing interest appeared to explore the capabilities of Plastic Scintillators for radionuclide analysis.

The detection mechanism of both liquid and plastic scintillation for alpha and beta particles detection was described as similar by J. Birks but pointing out some differences related to how the particle energy is transferred in both cases to generate the photon emission. These differences are responsible of the variations on the quenching processes and of the different temporal distribution of the photon avalanches produced by alpha and beta particles.

Liquid Scintillation (LS) is a well known technique widely implemented with success on routine determinations of radionuclides on materials coming from different fields as nuclear industry, medicine, biochemistry, geology, archaeology, environmental protection. However two main limitations can be pointed out: the production of mixed waste, difficult to be disposed; and its lack of selectivity in alpha and beta emitters determination. For alpha's, this lack of selectivity is due to the signal broadening along the process of signal production; and for beta's, as consequence of their intrinsic energy distribution.

The interest for Plastic Scintillation (PS) comes from their contribution to overcome some of the above mentioned limitations.

Capabilities of Plastic Scintillators in form of microparticles for direct activity determinations on solution samples have been widely studied for several alpha and beta emitters. For alpha's, the results obtained show high detection efficiency and a spectra distribution at reasonable high energies in good concordance with LS observations. For beta emitters, detection efficiencies and spectra position are also similar to those obtained by LS except for low energy emitters which detection efficiency decreases drastically. The results improve when solution samples are evaporated to dryness onto the Plastic scintillator or when microparticles of low diameter are used.

This behaviour evidences a particularity of PS with regard LS for alpha and beta determinations: the lost of energy on their way from the emission point in the solution until the particle reach the scintillating material (particle quenching). The interactions along this path reduce the energy of the emitted particle in a more important relative proportion as lower is the energy of the emission. This phenomenon is the origin of the main drawback of the PS measurements which is the low detection efficiency of low beta emitters as ^{3}H.

Another difference between PS and LS, already pointed out by J. Birks, is the different temporal distribution of the photon avalanches when they are produced by beta and alpha disintegrations. In the case of PS, both distributions are more similar and the discrimination between pulses produced by both types of particles is more difficult and requires specific secondary solvents to improve the resolution.

However the main contribution of the Plastic Scintillators to the analysis of alpha and beta emitters is based on their solid nature. This characteristic appears as a property that opens the possibilities both for on line continuous analysis and for selective scintillating determinations.

Selective Scintillation analysis can be achieved by using the Plastic Scintillating Resins (PSresin) that joint separation and detection steps contributing to overcome the lack of selectivity already pointed out. PSresins are based on a Plastic scintillator supports coated by a extractant selective to the radionuclide to be determined. This approach has been studied for different radionuclides and types of samples and it shows a clear shortcut of time and resources required for samples processing. Recently, different procedures of support production and extractant addition are under study with promising results and potential application to routine determinations.

Thus, PS and PSresins in particle form can be the base for new discontinuous, continuous or automatic determinations procedures that can be applied to environmental and decommissioning related problems.

Measurement of tritium with plastic scintillators in large vials of a low background LSC -an organic waste-less method-

Etsuko Furuta [1], Yuka Kato [2], Shinji Fujisawa [2]

[1] Ochanomizu University, 2-1-1 Otsuka Bunkyo-ku Tokyo, 112-8610, Japan
[2] Hitachi Ltd., 6-22-1 Mure Mitaka-shi Tokyo, 181-8622, Japan

Corresponding author's e-mail: furuta.etsuko@ocha.ac.jp

Introduction For trace radioactive nuclides in our environment, measurement methods are required to check of low level radioactivities. So, some low background LSCs have been developed into a 3 Photomultiplier Tubes' coincidence mechanism and one of them is possible to measure large vials. At the same time, usage of large vials means generation of huge amounts of organic wastes after measurement using general liquid scintillator. It becomes an environmental load. We have developed an organic waste-less measurement method by using plastic scintillators (PS) [1-3]. By our method, non-volatile compounds could be measured using small size PS-sheets [1], which had a limit of applying sample volume to maximum 25 μL. Additionally, volatile compounds could be measured using PS-pellets, and the counting efficiency was higher than those of liquid scintillator when the sample volume was only 5 μL [2]. Unfortunately, the counting efficiency of the PS-pellets decreased when the loading sample volume increased. So, we needed an instrument which was able to apply to more large volume. Then, we studied measurement of tritium compounds with PS-sheets and pellets using 100 mL wide mouth Teflon vials and/or 145 mL small mouth polyethylene vials. For both of PS-sheets and PS-pellets, counting efficiency depending on the radioactivities was studied and detection limits were calculated using PS's background.

Experimental Tritium compounds (Moravek Biochemicals Inc., USA) were tritiated water as a volatile compound and ^{3}H-methionine as a non-volatile compound. The PS-sheet was 69 mm in height, 20mm in width and 0.5 mm in thickness (BC-400: Saint-Gobain, USA), which was able to put 1 mL sample on one sheet and it was spread out for the entire surface with plasma treatment.

Fig.1 Photos of PS-sheets and pellets in large mouth Teflon vials.

The PS-sheets surfaces were treated with a plasma device for hydrophilicity (PR-101, Izumi Co., Japan), which is able to treat 10 sheets within 1 min with one time. The plasma source is air and the plasma box is vacuumed approximately 200 Pa. The contact angles of water solution were approximately improved from 97° with non-treatment to 21° with 1 min treatment. And the PS-pellets were 3 mm both in diameter and length (EJ-200: G-tech, Japan). The vials were 100 mL Teflon (Sanplatec Co., Japan) and 145 mL polyethylene (ZINSSER ANALYTIC, Germany) which cap was uGV2-CAP (Meridian, UK). The PSs with vials are shown in Fig.1. The low background LSC used was AccuFLEX LSC-LB7 (Hitachi Ltd., Japan) which is able to apply 20 vials at one time measurement. The sample treatment methods for the PS-sheets and pellets were same as the references [1, 2]. The detection limit A_D [Bq/mL] was calculated by the following equations:

$$A_D = x_D \times 100/\varepsilon \times 1/60/v \quad x_D = k^2/2\ [1/t_S + (1/t_S^2 + (4x_B)/k^2\ (1/t_S + 1/t_B\ )\ )^{1/2}]$$

where ε [%] is counting efficiency, v [mL] is sample volume, k is a coverage factor corresponding to a confidence interval of 95% when k equals 2, x_B was the count rate of the background and t_S and t_B are measurement time of the sample and background, respectively.

Results and discussions With the PS-sheets for a non-volatile compound; the 64 PS-sheets were able to put in one Teflon vial. So, maximally 64 mL sample solution was applicable as one sample. The back ground of the 64 sheets was 8.60 ± 2.37 cpm. So, the detection limit of 64 sheets with 25.0% counting efficiency at 60 min, which was the case of 1 mL sample solution on 1 PS-sheet, was 2.30 $\times 10^{-3}$ Bq/mL when the background was measured 60 min.

With the PS-pellets for a volatile compound; the net weights of the PS-pellets in the 100 mL Teflon vial and the 145 mL polyethylene vial with full filled were 82.8 ± 0.9g and 96.9 ± 0.6 g, respectively. The background of the Teflon vial was lower than that of polyethylene vial. So, the detection limits of the Teflon and the polyethylene vials were almost same. However, the relationships between the net count rate and radioactivity with the Teflon vials and the polyethylene vials were deferent to each other as shown in Fig.2. The details were shown in the Conference.

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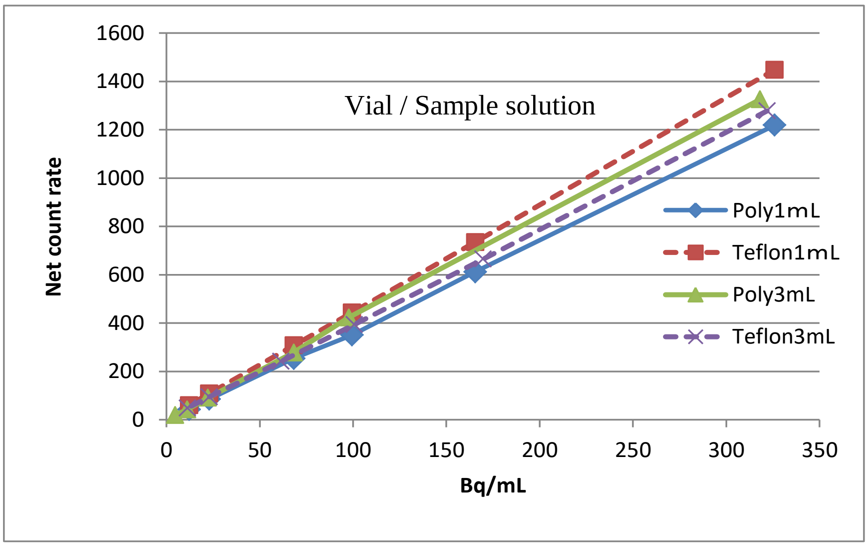


Fig.2 HTO measurement with 2 kinds of large vials by LSC LB7.

Synthesis and characterisation of scintillating microspheres made of polystyrene/polycarbonate for ^{222}Rn measurements

Pelay, E.[1]; Tarancón, A.[1,3]; Mitev, K.[2]; Dutsov, Ch.[2]; Georgiev, S.[2]; Boshkova, T.[2]; García, J.F[1].

1) Department of Chemical Engineering and Analytical Chemistry, University of Barcelona, Marti i Franqués, 1-11, ES-08028, Barcelona, Spain
2) Faculty of Physics, Sofia University “St. Kliment Ohridski”, 1164 Sofia, Bulgaria
3) Serra-Húnter Program, Generalitat de Catalunya, Barcelona Spain.

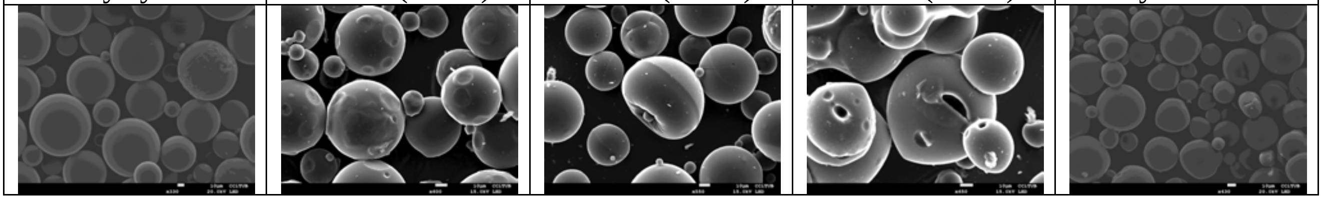
Corresponding author’s e-mail: kmitev@phys.uni-sofia.bg

^{222}Rn represents one of the most relevant radioactive threats to the population since its contribution to the total dose received by humans is estimated to be around 40% and this value can be higher in those areas with high concentration of natural radionuclides[1-2]. ^{222}Rn concentration in water and air is considered in several regulations and for this purpose several measuring methods have been developed. ^{222}Rn quantification in water samples can be done by liquid scintillation (LS) counting or high-resolution gamma spectrometry (HRGS) if concentrations are high enough[3-4]. In ^{222}Rn –in-air studies methods used include techniques based on absorption in polymers[5], Lucas cells, ionization chambers or charcoal absorption among others. Nevertheless, the fast and reliable measurement of ^{222}Rn is still an issue in some applications, like in the soil-gas measurements and ^{222}Rn in air monitoring, where improved methods are needed.

A new approach to deal with this kind of measurements is the use of scintillating polymers. It has been demonstrated that polycarbonates are highly efficient for the absorption of ^{222}Rn[6]. On the other hand, scintillation microspheres (PSm) made of polystyrene have been demonstrated to be a promising material for the measurement of the alpha and beta radionuclides[7], as well as ^{222}Rn[8]. The motivation of this work is to produce PSm from mixture of polystyrene/polycarbonate or pure polycarbonate and thus to combine the high ^{222}Rn absorption ability of polycarbonates with the favorable counting properties of the PSm. The objective of the work is to present the development of the PSm made of polystyrene and polycarbonate and the results of their characterization from the point of view of morphology, capacities to measure radioactivity and ^{222}Rn absorption behavior. Five different PSm composed of polystyrene (100%), mixtures of polystyrene and polycarbonate Makrofol DE (75/25, 50/50, 25/75 in percentage) and polycarbonate (100%) have been synthesized using the extraction/evaporation method[9]. From the morphological point of view (Figure 1) only PSm made of polystyrene are perfectly spherical. PSm composed of mixtures of polymers present holes or two different parts clearly differentiated suggesting that polycarbonate and polystyrene are not miscible when solved in the organic solvent. Finally PSm made of polycarbonate present a non-spherical form but similar size to those of polystyrene.

Figure 1: PSm synthetized

Polystyrene | PS/PC (75:25) | PS/PC (50:50) | PS/PC (25:75) | Polycarbonate



Radiometric capabilities have been evaluated by measuring active solutions of ^{3}H, ^{36}Cl and ^{241}Am with the different PSm in a Quantulus detector (Table 1). The results obtained show that PSm made of polystyrene present the better values from the point of view of the detection efficiency and the quenching parameter. The addition of polycarbonate causes a decrease of the quenching parameter, which can be attributed to chemical quenching effect. This quenching effect is relevant in the measurement of low-energy beta emitters but not in the case of high-energy beta emitters or alpha emitters. In the last two cases, the most influence is caused by the increase of the particle size observed in PS/PC 75:25 and 25:75 due to the agglomeration of particles produced in the synthesis process.

Table 1: Detection efficiencies and SQP(E) of the polystyrene; polystyrene/polycarbonate and polycarbonate PSm

	PS	PS/PC 75:25	PS/PC 50:50	PS/PC 25:75	PC_MAKD
^{3}H det. eff. (%)	0.9 ± 0.1	0.30 ± 0.03	0.66 ± 0.03	0.47 ± 0.03	0.53 ±0.02
^{36}Cl det. eff. (%)	97 ± 1	84 ± 3	96 ± 1	91 ± 2	93 ± 1
^{241}Am det. eff. (%)	81 ± 1	53 ± 4	85 ± 2	74 ± 1	83 ± 1
SPQ(E)	806 ± 3	799 ± 4	765 ± 4	745 ± 5	712 ± 3

In order to study the ^{222}Rn absorption properties of the synthesized PSms, they were exposed to ^{222}Rn containing air in the exposure facility described elsewhere[10]. The ^{222}Rn activity concentration during the exposure (A_V) was monitored with an AlphaGuard RnTnPro radon monitor. The temperature during the exposure was 21 ^{O}C and the exposure time was 72 h. After the end of the exposure the PSms are transferred to 20 ml high performance LS glass vials (fully filled with PSm) and the ^{222}Rn activity absorbed in the PSm (A_{PSm}) is determined by high purity germanium detector measurements. The ^{222}Rn absorption capabilities of the PSm are characterised by the sampling efficiency[8]: $\varepsilon_S = \frac{A_{PSm}}{A_{air}}$, where A_{air} is the ^{222}Rn activity contained in the air with volume, equal to that of the PSm (i.e. $A_{air}=A_V V$, where V is the volume of the PSm). The results are given in Table 2.

Table 2: Sampling efficiencies of the polystyrene; polystyrene/polycarbonate and polycarbonate PSm

PSm type	ε_S
PS	6.0 ± 1.1
PS/PC 75:25	6.6 ± 1.7
PS/PC 50:50	6.1 ± 1.1
PS/PC 25:75	5.5 ± 1.4
PC_MAKD	5.03 ±0.89

The results in Table 2 do not show systematic increase of the ^{222}Rn absorption properties with the increase of the polycarbonate content in the PSm. This is an unexpected result since the Makrofol D polycarbonate has remarkably high radon absorption ability[10] compared to other polymer materials. According to the theoretical model which describes the ^{222}Rn absorption in PSm[8] and using the ^{222}Rn solubility and diffusion length in Makrofol DE polycarbonates from[12], the ^{222}Rn sampling efficiency of PSm made from polycarbonate is expected be 4 times higher than the sampling efficiency of polystyrene made PSm. The fact that we do not see increased sampling efficiency of Makrofol DE PSm compared to the polystyrene PSm (see Table 2) suggests that the ^{222}Rn absorption properties of the polymers is not only a function of their material content, but also on the way of their production. This hypothesis will be tested experimentally by measurement of the ^{222}Rn absorption properties of the particular raw materials from which the PSm are made and evaluation of the differences in the ^{222}Rn absorption properties between the raw materials and the PSm synthesized from them. More results will be presented at the conference.

In this work we demonstrate that it is possible to produce plastic scintillation microspheres from polycarbonate and mixtures of polycarbonate/polystyrene in different proportions. The study of the radiometric capabilities of the newly synthesized PSms shows that the addition of polycarbonate causes slight decrease in the high-energy beta- particles and alpha- particles detection efficiency and slightly increases the quenching of the PSm. The study of the ^{222}Rn absorption capabilities of the newly synthesized PSm shows that the theoretical expectation of increased ^{222}Rn absorption capabilities of the PSms made of polycarbonate is not justified. This result shows new insights in the understanding of the radon absorption properties of the polymers and implies that the ^{222}Rn absorption is not dependent only on the material composition of the polymer, but also on the way of its production. Future studies of this hypothesis are scheduled and more results will be presented on the conference.

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Development of stable extractive scintillating materials for quantification of radiostrontium in aqueous solutions

A. F. Seliman, V.N. Bliznyuk, T. A. DeVol

Environmental Engineering and Earth Sciences Department, Clemson University,
Clemson, SC 29634-0919, USA.

Corresponding author's e-mail: ayman@clemson.edu

The research presented here is the development of sensor materials that are applicable for real-time *in situ* quantification of radiostrontium in aqueous solutions. Three different approaches were applied to prepare highly selective structures for simultaneous extraction and detection of radiostrontium using on-line detection methods. Flow-cells were created by packing ≈ 0.06 g of the resin/scintillator into polytetrafluoroethylene tubing to a resin bed length of 4.0 ± 0.1 cm and 0.16 cm inner diameter. The detection efficiencies were quantified with Flow Scintillation Analyzer (FSA) IN/US β-Ram model 5 (LabLogic Systems, Inc). The instrument digital output was connected to a laptop has Laura software package and the data was acquired as a multichannel scaling spectrum with a 30-s dwell time. The first approach at development of a sensor involved the simple mixing the commercially available SuperLig®620 resin with commercially available granulated scintillator. Three different granulated scintillator were investigated, but the SuperLig®620/CaF_2:Eu mixture (1:2) performed the best as an extractive scintillating sensor with detection efficiency of 54.3±1.3%. The second approach involved the incorporation of SuperLig®620 solid phase extraction particles into porous scintillating polyvinyltoluene (PVT) beads formulated with the organic fluor monomer 2-(1-naphthyl)-4-vinyl-5-phenyloxazole (vNPO). The new extractive scintillating composite showed good selectivity and detection efficiency of 53.6 ±2.4% for ^{90}Sr. The third approach involved growing scintillating polymer brushes on the surface of the silica particles of the SuperLig®620 in a two-step process. The first step involved modifying the surface with 3-Methacryloxypropyltrimethoxysilane (MPS) in ethanol or toluene to form vinyl-SuperLig®620. The second step involved polymerization of scintillating polymer brushes on the surface of the vinyl-Superlig® 620. The polymerization was conducted in a thin layer of dimethylformamide (DMF) on the surface of the Superlig®620 or in toluene solution as suspended particles which resulted in detection efficiency of 27.4±1.7% or 32.5±1.4%, respectively. The sensor demonstrated a high sensitivity and a chemical stability. The detection system has sufficient sensitivity for real-time measurement of ^{90}Sr at a wide range of radioactivity levels including the US maximum contaminant level (MCL) of 0.33 Bq/L for ^{90}Sr in drinking water.

Development of continuous inflow tritium in water measurement technology by using electrolysis and plastic scintillator

Jun Woo Bae, Ukjae Lee, Choon Wie Lee and Hee Reyoung Kim

Ulsan National Institute of Science and Technology, 100, Banyeon-ri, Eonyang-eup, Ulju-gun,
Ulsan, 44919, Republic of Korea

*Corresponding author's e-mail: skypia12@unist.ac.kr

In-situ tritium monitoring system in water based on plastic scintillation and electrolysis method was developed. It could detect the 250 kBq/L water tritium sample for 5-minute measurement. Tritium monitoring has been issued importantly in Korea in terms of radiological safety for the public since 4 Pressurized Heavy Water Reactor (PHWR) in Gyeongju has been operated where the oldest one had started its operation in April, 1983. People living around the nuclear power plants has worried about the safety of drinking water. Tritium concentration or radioactivity of a water sample is hard to analyze quickly. The analysis of the tritium has been implemented by using high temperature and liquid scintillation counting (LSC) method in the laboratory, which required long time for sample pretreatment and cause radioactive waste problem from the use of inorganic and organic solvent despite exactly measuring method for even extremely small quantity of sample.

In this study, a real-time in-flow tritium measurement system based on a plastic scintillator was developed. Fig. 1. shows a scheme of the tritium in water continuous inflow measurement system. The system consisted of electrolysis part and detection part. The electrolysis part included filters and an electrode. It electrolyzed the water sample and pure hydrogen gas was obtained. The measurement part consisted of plastic scintillator flow channel. The produced hydrogen gas entered the channel and light signal from interaction of tritium with scintillator was measured and analyzed by photo-multiplier tube and multi-channel analyzer.

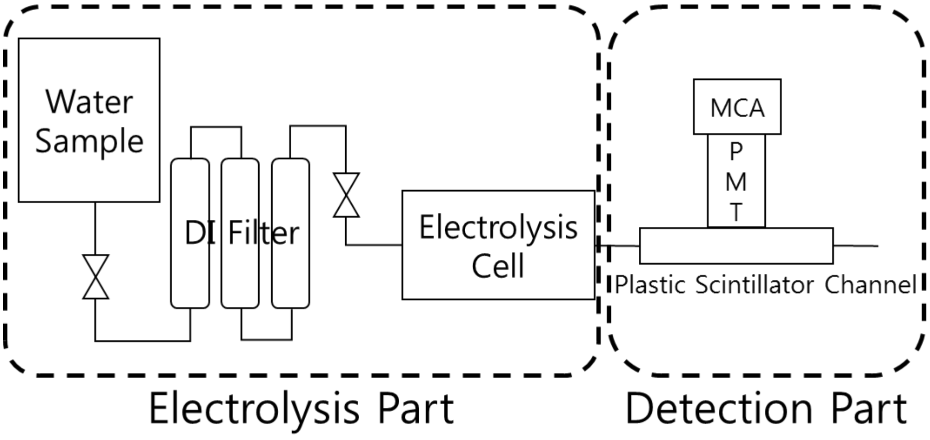

Fig. 1. Scheme of the measurement system

The DI water and a tritium contaminated water sample were used. The volume of tritium sample was 20 mL in water state and its volumetric radioactivity concentration was 250 kBq/L.

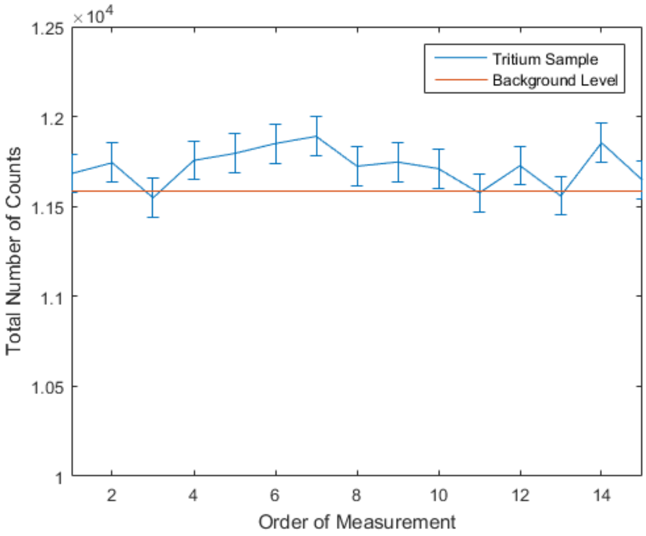

Fig. 2. Measured data of tritium sample with continuous measurement

Fig. 2 shows the number of counts measured every 5 minute. The measurement was conducted continuously. In Fig. 2, the average of total counts from tritium sample was 11720±101.86 and background was 11585±88.754 for 5 minutes. It was confirmed that the tritium monitoring system could continuously and quickly measure the tritium contamination in water quickly showing some errors where the estimation of MDA was to be included for the future work.

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$^{89}Sr/^{90}Sr$ determination in milk in emergency situations by using PSresins

Marina Sáez-Muñoz 1), Héctor Bagán 2), Alex Tarancón 2), José F. García 2), Josefina Ortiz 1), Sebastián Martorell 1)

1) Laboratorio de Radiactividad Ambiental, MEDASEGI Research Group, Universitat Politècnica de València, Camino de Vera s/n, 46022, Valencia, Spain

2) Departament de Química Analítica, Universitat de Barcelona, Diagonal 647, E-08028 Barcelona, Spain

Corresponding author’s e-mail: masaemuo@etsii.upv.es

In emergency situations, such as those caused by nuclear and radiological accidents, terrorist attacks using radiological dispersal devices (RDDs) or “dirty bombs”, and improvised nuclear devices (INDs), numerous short- and long-lived radionuclides will be present, requiring rapid identification and quantification. In these situations, radiostrontium is one of the most important radionuclides to determine. ^{89}Sr ($T_{1/2}$=50.56 days) and ^{90}Sr ($T_{1/2}$=28.79 years) can migrate through the food-chain into the milk and dairy products and represent an important risk specially for infants.

Pure beta emitters, such as ^{89}Sr and ^{90}Sr, require a radiochemical separation prior to measurement. In most cases, extraction chromatographic columns are employed [1-3]. However, the use of PS Resins for strontium, based on plastic scintillator microspheres coated by a selective extractant (DtBuCH18C6), unifies chemical separation and sample measurement preparation in a single step [4]. The main advantages are the reduction in time, effort and reagents required for analysis. Moreover, unlike Liquid Scintillation (LS), no mixed waste is generated.

This study describes a new and fast procedure for ^{89}Sr/^{90}Sr determination in milk based on the use of PS Resins for strontium separation. The pre-treatment of the sample is performed following the main steps proposed by Kabai et al. [2]. It consists of using trichloroacetic acid as acidifying agent for protein and fat precipitation and the separation of alkaline-earth elements by oxalates precipitation. The isolation of strontium isotopes from other alkaline-earth elements is achieved using PS Resin for strontium separation in a 2mL cartridge. After interferents elimination with different elution steps, the cartridge is directly measured in a LS plastic vial by QUANTULUS 1220. In case of both ^{89}Sr and ^{90}Sr presence, spectral deconvolution is used for activity calculation.

For strontium isolation and measurement, the PS Resin specifications have been previously characterized and the equipment has been calibrated for radiostrontium measurement in milk. Furthermore, different types of milk (different origin and fat content) have been analyzed during the study. Good separation yields were obtained, with a limit of detection for 100mL of milk sample below 1 Bq/L, and a reduction in the time of analysis.

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Development of low cost per unit area plastic scintillator materials for radiation detection and monitoring applications

Lee F. Thompson [1], John K. Clapham [2], Tom Deakin [1,2] and Chris Steer [3]

[1] The University of Sheffield, Department of Physics and Astronomy, Hicks Building, Hounsfield Road, Sheffield, S3 7RH. UK.
[2] LabLogic Systems Ltd., Paradigm House, 3 Melbourne Avenue, Broomhill, Sheffield, S10 2QJ. UK.
[3] AWE plc, Aldermaston, Reading, RG4 4PR, UK.

Corresponding author's e-mail: l.thompson@sheffield.ac.uk

In this work, we describe a recent research and development programme that has led to the scaled-up production of injection-moulded plastic scintillator tiles based on a polystyrene (PS) base material that can be arranged in a variety of array geometries for large-scale monitoring applications, such as in the use of muon tomography applications for the passive detection of special nuclear material [1], the imaging of nuclear reactors [2] or imaging volcanoes [3,4]. The resulting tiles are based on a size of 200 (w) x 200 (l) x 5 (d) mm and feature four longitudinal 1.5 x 1.5 mm cross-section grooves that permit the insertion of wavelength-shifting (WLS) or conventional optical fibres for the capture of scintillation light and hence efficient coupling to a photosensor (Figure 1).

Fig. 1. Illustration of an injection-moulded plastic scintillator tile.

Optimisation of the concentration of scintillation fluors (p-Terphenyl, pTP and 1,4-bis(5-phenyloxazol-2-yl) benzene, POPOP) has led to a light output peak wavelength at 420 nm to match the majority of commercial photosensors (Figure 2) and muon detection efficiencies in excess of 97% for a tiled thickness of 15 mm (three individual layers), >85% for two layers and in excess of 60% for a single tile thickness. The performance of a double thickness of a 2m x 2m array of these tiles, configured as a trigger for a demonstrator cargo container scanning system is given in Figure 3.

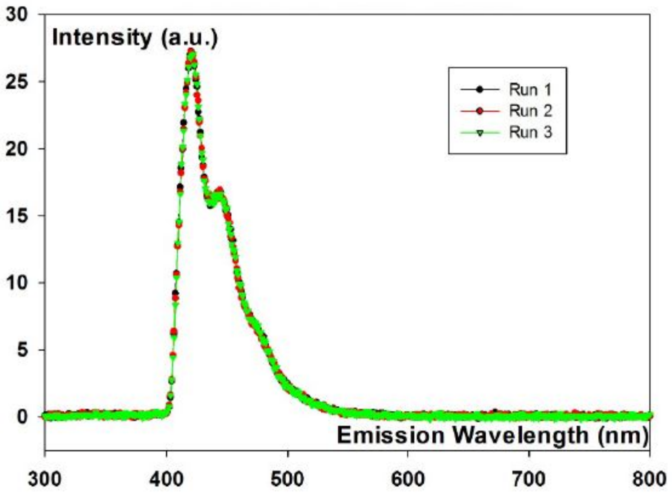

Fig. 2. Emission spectra of tile material showing peak emission at 420 nm.

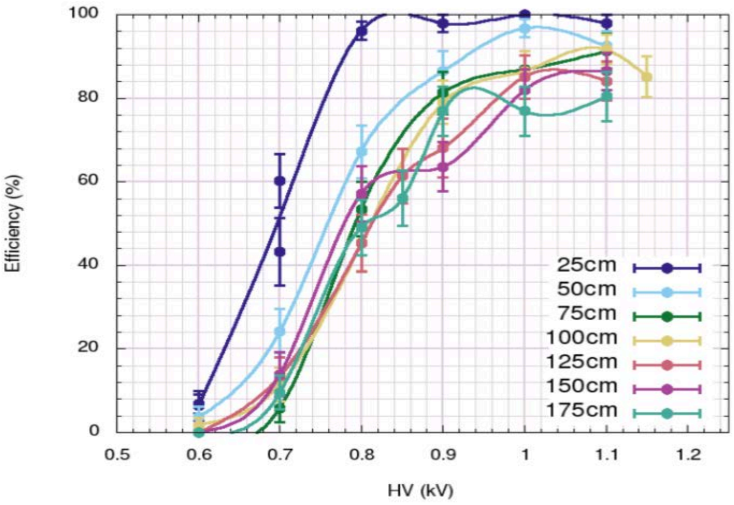

Fig. 3. Muon detection efficiency as a function of PMT high voltage for different distances from a single PMT in a 2m x 2m array of scintillating tiles.

More recently, we have focused on the addition of Gd-based dopants to improve neutron sensitivity and hence enable discrimination between neutrons and gammas. Results on the sensitivity of these tiles to neutron and gamma sources will be presented.

This work has demonstrated the feasibility of scaled-up production of low cost per unit area scintillation tiles using injection moulding. Future work will focus on building on the successes of this proof of concept to develop a range of PS-based scintillating materials in various physical configurations, such as blocks, larger area tiles and bars to form the basis of modular radiation detection systems that can be interfaced with various signal readout platforms based on existing (e.g. photomultiplier tube-based) and novel (e.g. Silicon photosensor-based) detectors. The primary objective will be on delivering plastic scintillator with enhanced sensitivity to gammas and neutrons (for detecting fissile material) in a wide range of formats that can be combined to satisfy numerous physical requirements whilst maintaining the low-cost nature of the material.

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Comparison of several methods for measuring ^{222}Rn in drinking water

Ll.Pujol, M.E. Perez-Zabaleta

Centro de Estudios y Experimentación de Obras Públicas (CEDEX), Alfonso XII, 3, 28014 Madrid, Spain

Corresponding author's e-mail: luis.pujol@cedex.es

The European Union published in 2001 a Recommendation concerning the necessity of performing ^{222}Rn measurements in drinking water (EC, 2001). As a consequence of this Recommendation, the CEDEX (Centro de Estudios y Experimentación de Obras Públicas) and the Douro River Basin Authority carried out a preliminary ^{222}Rn determination on drinking waters of the Douro Basin. Water samples for ^{222}Rn determination were collected in winter 2009 in nine sites. Three techniques were used for ^{222}Rn measurement in drinking water: degassing method followed by the counting in an ionization chamber (IC), liquid scintillation (LS) and gamma spectrometry (GS). Environmental samples were measured in the field using the IC and, the same samples were measured in the laboratory using LS and GS. The results obtained by the three techniques are compared and discussed according the new Euratom Drinking Water Directive (2013/51/Euratom), referred as E-DWD, that requires monitoring of radioactive substances such as radon.

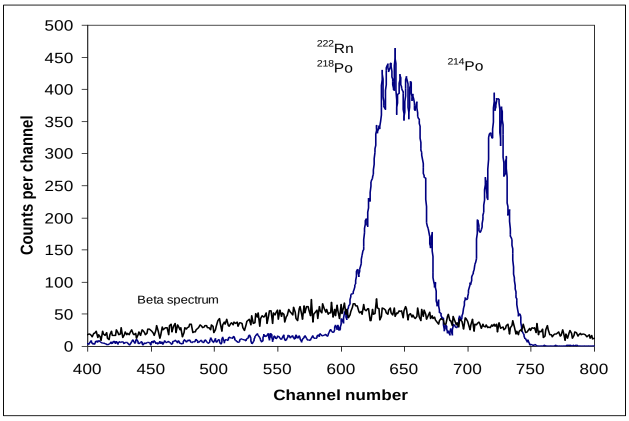

Fig. 1 Alpha and beta spectra from sample DU07 using LS.

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Application of LSC Techniques in Environmental Radiochemistry Research in China

Chunli LIU

Beijing National Laboratory for Molecular Sciences, Fundamental Science Laboratory on Radiochemistry & Radiation Chemistry, College of Chemistry & Molecular Engineering, Peking University, Beijing 100871, PR China

China's nuclear industry has been developed for more than 60 years, and some areas near nuclear facilities may be contaminated or polluted, which need to be remediated or rebuilt meanwhile, the operation of nuclear power plants may cause some release of radioactive materials to the surroundings of the NPP. To assess the pollution of an area need robust background information.

Liquid scintillation counting has long been used for measurements of environmental radionuclides such as ^{3}H, ^{90}Sr, ^{227}Ac, ^{99}Tc. For all of these measurements, accurate background measurement is absolutely important. In this paper, the LSC technique used in environmental radiochemistry research and possible future background measurements in China will be presented and discussed.

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Adsorptive Removal of Lead-210 Using Hydroxyapatite Nanopowders Prepared From Phosphogypsum Waste

Nazife ASLAN, Gülten ÖZÇAYAN

Turkish Atomic Energy Authority, Sarayköy Nuclear Research and Training Center,
Radiation Metrology Division, 06983 Saray, Kazan, Ankara-TURKEY

Corresponding author’s e-mail: nazife.aslan@taek.gov.tr

Recently, several organic and inorganic adsorbents are used for the separation and purification processes of metal ions and removal of radionuclides from radioactive and industrial wastes. In this study, calcium hydroxyapatite (HA) was prepared by phosphogypsum waste and its adsorption behavior towards Pb-210 was evaluated. The influence of the various experimental parameters such as pH, concentration of adsorbent, contact time and adsorbent quantitity on the adsorption process was also investigated and the adsorption capacity of HA was found to be 98% on the conditions, i.e. pH 6.0 – 6.5, adsorbent quantity 2g, contact time 1 h at room temperature. The results indicated that synthesized hydroxyapatite can be used as an efficient adsorbents for removing lead-210 from aqueous media.

Experience after 10 years of monitoring ^{14}C in the vicinity of the Nuclear Power Plant Krško, Slovenia

Ines Krajcar Bronić(1), Bogomil Obelić(1), Jadranka Barešić(1), Damir Borković(1), Nada Horvatinčić(1), Andreja Sironić(1), Aleš Volčanšek (2), Borut Breznik (2)

(1) Ruđer Bošković Institute, Bijenička c. 54, 10000 Zagreb, Croatia
(2) Nuclear Power Plant Krško (NEK), Vrbina, Slovenia

In this presentation we will summarize the 10-year-long experience in monitoring ^{14}C activity in the close vicinity of the Krško Nuclear Power Plant (NPP) in Slovenia. Atmospheric CO_2 samples have been taken in 2-month periods during the regular operation of the power plant, or in shorter periods during refuelling periods. Biological samples (apples, corn, wheat, grass, vegetables) have been samples twice a year (in summer and autumn) in two circles around the NPP, inner and outer, and at the control point 12 km from the plant. The aim of the monitoring was to estimating a possible influence of the NPP on environmental ^{14}C levels and on the effective dose of local population through food chain. Increase of ^{14}C activity in atmospheric CO_2 was observed during and immediately after the refuelling of the power plant, performed every 18 months. Good correlation between the total ^{14}C activity released in gaseous effluents and the ^{14}C activity of the atmospheric CO_2 has been observed. ^{14}C activity in plants collected close to the Krško NPP is always higher than the activities on the control point, and depends both on the distance from the exhaust of the plant ventilation system and on wind direction: it is higher on the location in the SW-NE direction that coincided with the most pronounced wind directions. Higher ^{14}C activities have been determined in plants collected in summer after the spring refuelling than in those collected during the following vegetation period after the autumn refuelling. This can be explained by the uptake of the CO_2 of higher ^{14}C activity for the process of photosynthesis after spring refuelling. To estimate the realistic effective dose due to ingestion to the population in the vicinity, a model of food consumption has been proposed. The calculated dose for the population at the NPP vicinity is not significantly different from the dose for the population at the control point.

Comparison study on low-level Sr-90 measurements for the Dutch food monitoring program

Vos van Avezathe, A.[1)], Tuinen, ir. S.T. van[1)]

[1)] RIKILT Wageningen University & Research, Akkermaalsbos 2, 6708 WB Wageningen, The Netherlands

Corresponding author's e-mail: annemieke.vosvanavezathe@wur.nl

Background

The RIKILT in Wageningen performs a national monitoring program on the presence of Sr-89 and Sr-90 in several food and feed products [1]. Regularly, these validated and accredited measurements are performed with a Quantulus measuring apparatus, with which a detection limit of about 1 Bq/kg fresh weight after four hours measuring time could be achieved. Last year, a new Hidex system was installed, aiming at an identical detection limit as the Quantulus apparatus. Since the Hidex system differs technically from the Quantulus in some fundamental measuring and analysing techniques, before the Hidex system could be used as official system for the monitoring program, a thorough validation study was performed.

Main methods

In the validation study the measuring results of the Quantulus and the Hidex were compared. In this, also the similarities and differences of the whole analysing process are considered.

Currently, all activities related to the Sr analyses on the Quantulus apparatus are described in standard operating procedures (SOPs), a primary condition for a validated and accredited methods [2,3,4]. So, this will also be requested fo the analyses performed with the Hidex.

The main difference between the two apparatuses is the number of PMT detectors: 2 for the Quantulus and 3 for the Hidex system [5,6]. Many comparison studies on H-3 and C-14 measurements have been performed providing a lot of information about the technical performance of these systems [7,8,9]. However, not many research has been performed on the intercomparison of Sr-90 measurements.

Compared to the Quantulus system, a higher background is expected for the Hidex system. Our research focuses on obtaining a MDA of 0.1 Sr-90 Bq per vial, corresponding to the requested MDA for the accredited measurement of 1 Bq/kg fresh food weight. For milk, the requested MDA for the non-accrediated measurement is even 5 times lower, corresponding with 0.02 Bq per vial and 0.2 Bq/kg fresh milk. According to earlier experiments performed with the Hidex the validated

TDCR algorithm should for reaching the requested MDA's not result in very much longer measuring times than the current measuring times with the Quantulus (4 hours).

The measurement strategy consisted of the following steps:

- Preparation and measuring of several samples from standard solutions with Sr-90 only;
- Interpretation of the measuring results by making calibration curves;
- Adapting the measurement and equipment settings (e.g. measurment order, measuring time, counting, ionized and measurement chamber delay);
- Preparation and measuring of several samples from standard solutions, with varying Sr-89/Sr-90 ratio;

This measurement cycle was repeated as often as necessary until the requested MDA were obtained.

Results and interpretation

Amongst other, the following samples were measured:

Table 1. Ratio's Sr-89/Sr-90 samples

Sample nr	Sr-89	Sr-90	Sr-89/Sr-90 ratio
	(Bq/vial)	(Bq/vial)	
1 (blanco)	-	-	N/A
2	1.11	-	N/A
3	-	0.7	N/A
4	5.82	0.7	8.3
5	5.82	0.35	17
6	11.64	0.35	33
7	0.58	0.07	8.3
8	1.11	3.68	0.30
9	0.54	3.51	0.15
10	0.53	7.12	0.07
11	0.11	0.35	0.31

Table 2. Results: Figure of Merit (values) comparison Hidex – Quantulus

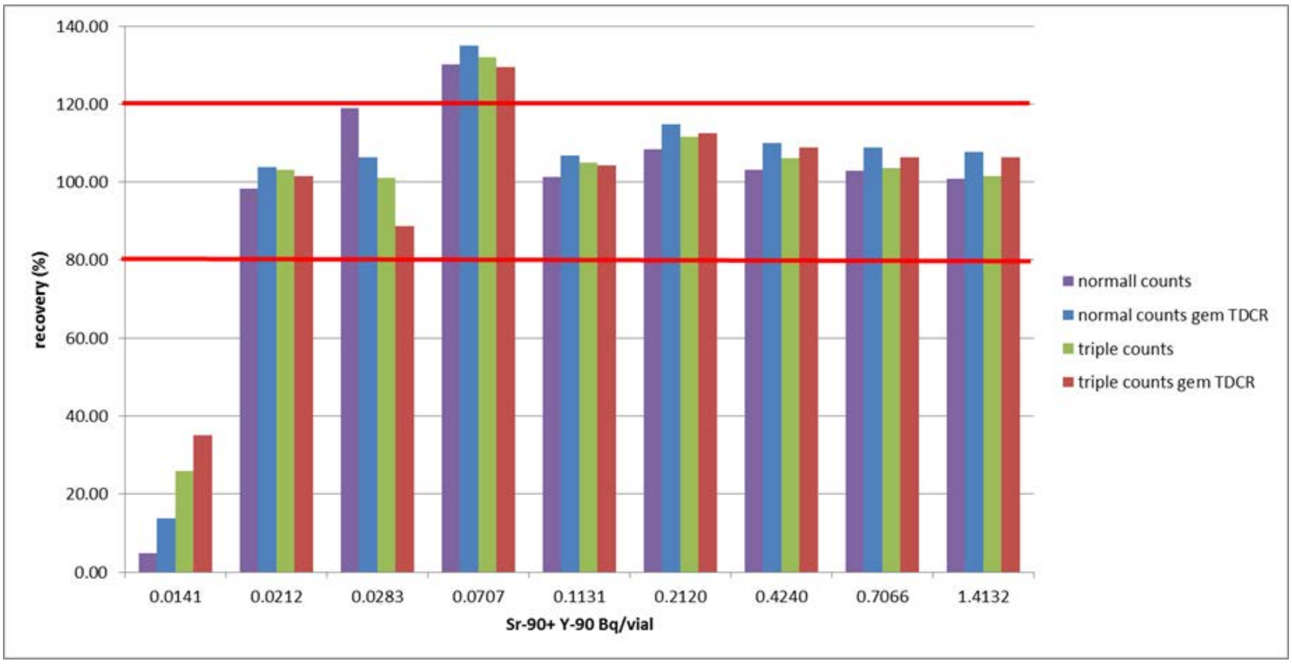
Instrument	ROI	Mode	Efficiency	BKG	FOM
	(channel)		(%)	(cpm)	(Eff^2/BKG)
Hidex	5-850	normal	92.5	24.67	347
	5-850	triple	92.5	18.15	471
	450-850	normal	77	7.96	745
	450-850	triple	77	7.88	752
Quantulus	5-950	N/A	82.4	3.83	1773
	300-674	N/A	58	2.06	1633

During the validation study the following observations were made:

- The Hidex system, designed for low level measurements, and equipped with a cooling system, shows a significantly higher background than the Quantulus apparatus. Consequently, this higher background is challenging the demands of measuring in the lower activity range and meeting our request considering the low MDA's;
- Even within one measuring cycle with the Hidex, the TDCR-factor showed remarkable fluctuations. However, as a result of the extensive and specific sample treatment it is expected that the quench for all samples is identical. This is confirmed by observations on the samples before and after the measurements: no changes in the samples during the measurements were observed. Therefore, we assumed the TDCR factor to be constant during one measuring cycle. This improved our results considerably.
- The effects of luminescence as a result of other processes than radioactivity were as much as possible eliminated by:
    - Delaying the start of the analyses by the Hidex up to 30 minutes after having closed the Hidex unit and starting the measurement cycle (eliminating spontaneous luminescence);
    - Setting the ionized delay on 10 seconds (eliminating possible electrostatic effects);
    - Setting the measurement chamber delay on an additional waiting time of 120 seconds (eliminating possible luminescence as a result of movements of the vial within the system).
- The measuring results of the blanco samples for normal counts varied between 24 and 30 cpm.

The following measurement results were obtained:

- We found quite some differences in the Figure of Merit (FOM) values [10]. The FOM found for the Quantulus were 2-4 times better than for the Hidex system, depending on the Region of Interest taken and the amount and the TDCR calculation algorithm.
- Despite these large differences in FOM, we were able to reach the MDA's requested for the Hidex. Figure 1 shows the correlation between the activity being present in the samples and the activity measured. The blue and red lines represent the recoveries with an averaged TDCR of the measuring series. The green and purple lines represent the recoveries where for every single measurement the TDCR factor is determined and reported in the analysis software.



From Figure 1 it can be concluded that using an averaged TDCR leads to better results, i.e. leads to recovery values closer to the 100%.

Conclusions

Assuming a constant TDCR factor for one measurements series with a measuring time of 6 hours we were able to reach the requested MDA of 0.1 Bq per vial for Sr-90 in fresh food.

Possible perspectives

Although the current results are showing that the requested MDA of about 0.1 Bq/vial – corresponding with 1 Bq/kg in food - is almost achieved, there are still some issues to be solved.

For instance, it is still not clear why the variations in the TDCR during the measurements occur, and why we are not able to reach the theoretically calculated MDA of the Hidex systems. In this, special attention will be given to the fluctuations in the blanco samples.

Secondly, for achieving the current MDA some assumptions have been made, which will be tested and subsequently must be confirmed.

Finally, we found during some test measurements a large difference in the number chemcial and luminiscence counts in. Possibly, there is a temperature and humidity dependency of this factor. The possible variations have to be investigated more in detail. Having performed this tests we will be able to decide whether we are going to use the logged TDCR-values during the measurements or an average determined TDCR-value for the whole series.

If all uncertainties have been cleared there will be some final tests determining the MDA for Sr-90 in milk, where we aim at a 5 times lower MDA (0.02 Bq/vial) than for food materials.

After this additional validation process it is expected that the Hidex system can get the formal status as measuring apparatus in our food monitoring program.

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Determination of ^{90}Sr and ^{89}Sr in water and urine samples by chemical separation and Cherenkov counting with LSC system

S. Ridone, D. Arginelli, M.C. Botta

ENEA - Radiation Protection Institute - Integrated Laboratory of Radioactivity Measurement and Monitoring, Strada per Crescentino, 41, I-13040 Saluggia (VC), ITALY

Strontium-90 is generated by uranium fission reaction and it can be present in nuclear reactor liquid wastes or spent fuel. Strontium compounds are generally water-insoluble, but they may be transformed in water-soluble compounds by chemical reactions. So they represent a source of internal contamination through ingestion, entering in human diet, in addition to inhalation, both for workers and for people in general. For this reason it is important to develop accurate and practical methods to monitor this radionuclide, both in drinking water for public health purposes and in urinary excretion, especially for acute and chronic exposure in workplaces.

At the Integrated Laboratory of Radioactivity Measurement and Monitoring, a radioanalytical method to determine ^{90}Sr with a satisfactory and constant chemical yield and a low level of Minimum Detectable Activity (MDA) has been optimized, in both environmental and biological samples.

According to different type of matrix, every sample was adequately mineralized with concentrated HNO_3 and successively dissolved into 8M HNO_3. Strontium-90 was extracted by chromatography using a 1M 4,4'(5')-di-t-butylcyclohexano 18-crown-6 in 1-octanol resin, commercialized, as prepackaged columns, by Eichrom^{TM} (Sr Resin^{TM}), in order to remove interfering radionuclides, like ^{40}K, ^{140}Ba and ^{137}Cs.

Sample activity has not been measured directly on ^{90}Sr radiation, but by using a liquid scintillation spectrometer, able to detect Cherenkov photons, generated by daughter radionuclide ^{90}Y, while reaching secular equilibrium with the mother radionuclide. In case of suspected presence of short-live fission product ^{89}Sr, which emits Cherenkov photons, too, it is possible to elute, by Eichrom^{TM} (Sr Resin^{TM}), ^{90}Y and subtract its contribution to total Cherenkov counts, so to determine separately ^{89}Sr and ^{90}Sr (via ^{90}Y) activity.

Convenient sample measurements during 15 days were fitted by an exponential equation type: Y=A*(1-EXP(-□*X))+B, that allowed to exclude possible presence of other β-emitter radionuclides (including negligible ^{90}Sr Cherenkov emission, corresponding to 2.4% of ^{90}Sr/^{90}Y Cherenkov counting) and, eventually, calculate ^{90}Sr activity even before secular equilibrium achievement.

Chemical yield of ^{90}Sr was calculated by an external standard, where the same matrix of the sample to be analyzed has been spiked with a known higher activity of ^{90}Sr. The constant chemical yield allows to use this technique for routine analyses, also without using an internal standard of stable strontium salt, which would require supplementary techniques, like atomic absorption spectrometry.

This method has been successfully validated through the participation to the ALMERA Proficiency Test for ^{90}Sr and ^{89}Sr determination in drinking water and to PROCORAD intercomparison exercises for ^{90}Sr determination in urine samples (Table 1 and 2).

Table 1 Comparison between IAEA-TEL-2016-04 ALMERA PT target value, laboratory reported value and relative bias. Sample 1 and Sample 2 are drinking water

Sample	**^{90}Sr**			**^{89}Sr**		
	Target	**Rep. value**	**Rel. bias**	**Target**	**Rep. value**	**Rel. bias**
2016	**[Bq·L^{-1}]**	**[Bq·L^{-1}]**	**%**	**[Bq·L^{-1}]**	**[Bq·L^{-1}]**	**%**
1	14.07±0.5	14.51±0.65	-1.29	-	-	-
2	20.5±0.5	19.02±0.83	-7.22	373±15	394±20	+5.63

Table 2 Comparison between PROCORAD assigned value, laboratory reported value and relative bias, for the years 2013, 2015 and 2016. All the samples are urine.

Sample	**^{90}Sr**		
	Ass. value	**Rep. value**	**Rel. bias**
2013	**[Bq·L^{-1}]**	**[Bq·L^{-1}]**	**%**
A	5.08±0.16	4.97±0.36	-2
C	1.09±0.05	1.12±0.11	+3
2015	**[Bq·L^{-1}]**	**[Bq·L^{-1}]**	**%**
A	2.38±0.12	2.04±0.36	-14
C	5.38±0.27	5.05±0.79	-6
2016	**[Bq·L^{-1}]**	**[Bq·L^{-1}]**	**%**
A	2.70±0.14	2.71±0.30	0
C	5.14±0.70	5.24±0.54	+2

Liquid Scintillation Counting in Quality Control of PET radiopharmaceuticals

Mikael Jensen [1)], Thomas Jørgensen [1)]

[1)] The Hevesy Laboratory, Frederiksborgvej 399 - Building 202, DK-4000 Roskilde, Denmark

Corresponding author's e-mail: kmje@dtu.dk

The widespread use of short lived radiopharmaceuticals for PET (Positron Emission Tomography) has forced a new honing of the standard tools for producing, handling and measuring activity. Especially the use of ^{18}F-FDG in oncological PET-CT has forced a qualitative and quantitative revolution in Nuclear Medicine. Other ^{18}F labelled compounds are being introduced every year, and the entire field of PET radiopharmaceuticals is growing at a tremendous pace that is pushing both available methods and the regulatory framework to the limit.

A simple, but very important set of quality specifications for these new life-saving radiopharmaceuticals relates to the radiations emitted. By nature, the best and most suited isotopes for PET only emit positrons in their decay, making them indistinguishable from each other by conventional gamma spectroscopy. The half-life then becomes the best way to tell both radionuclide identity and radionuclidic purity (RNP) of a given radiopharmaceutical preparation.

While the isotope identity can easily be established by two consecutive gamma spectrum measurements 5-10 minutes apart , the RNP is a difficult thing to measure. Especially so because modern regulatory guidelines (The European Pharmacopeia monographs for ^{18}F-FDG etc.) call for very high purity ; > 99.9% , understood as purity maintained throughout the shelf-life of the product, typically 4-5 half-lives of ^{18}F . At time of analysis this may require ability to detect impurites at the $3*10^{-5}$ activity level.

If one takes this requirement seriously, it is necessary to consider all possible interfering radioactive isotopes as possible contaminants. The very nature of the current production methods do introduce possibility of coproduction of longer lived isotopes with little or no gamma emission, making their detection dependent on either x-ray or beta-particle detection. A typical cyclotron target will inevitably also make ^{3}H (from ^{18}O(p,t)^{16}O) and ^{55}Fe (from nuclear reactions in the target foil). As the production facilities often have multiple uses of their cyclotron and often handle several different isotopes, the risk of the gamma-silent isotope sneaking into the product increases. Equally important is the fact that conventional gamma spectroscopy, even based on modern high-purity germanium detectors, have difficulties detecting and quantifying gamma-impurities on top of the normal high-count rate annihilation radiation spectrum, especially if the gamma lines in question lie below 511 keV. Even detection of gammas at the 1% level will require long counting periods and 0.1%-0.01% detection almost impossible.

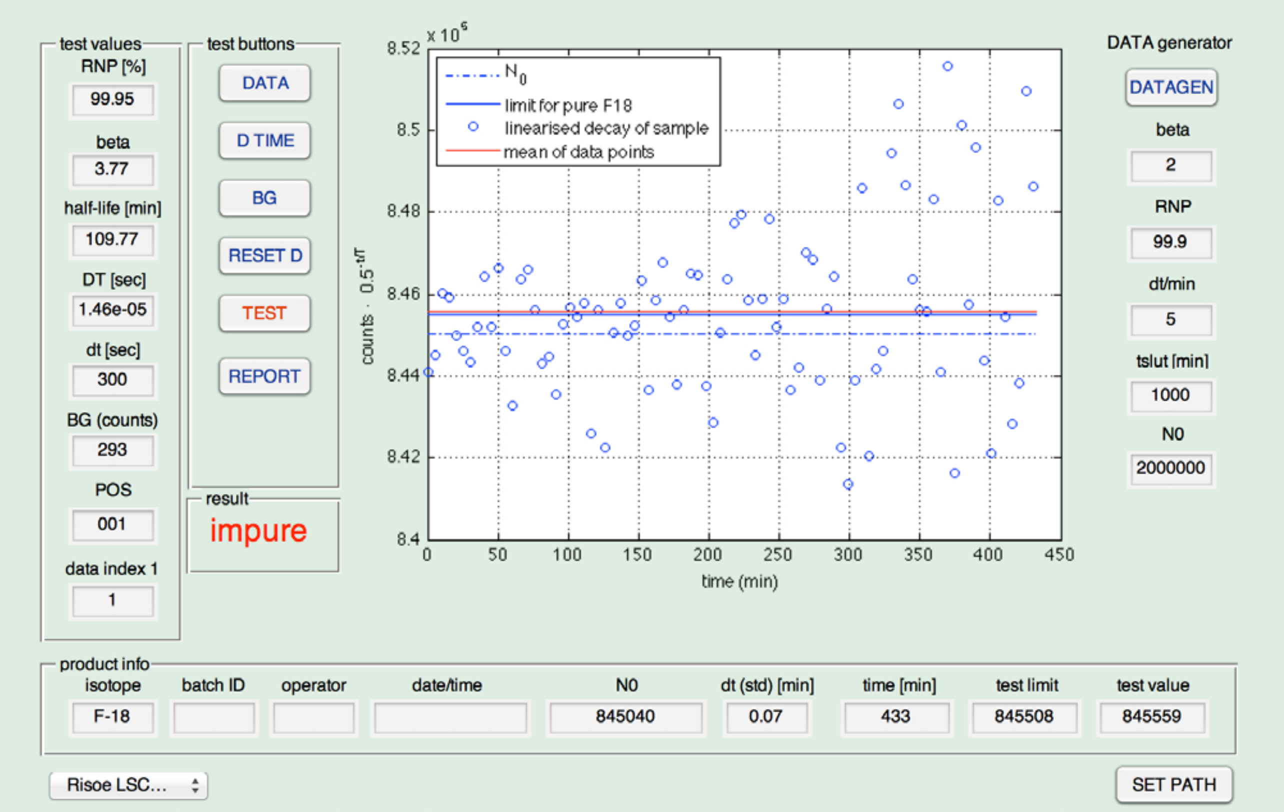
Here the liquid scintillation counting comes in as very useful tool. It may have been forgotten or abandoned by most new radiopharmacists, but it lends itself excellent to this use. It does detect all radioisotopes, no matter of their emissions, and it will, because of the inherent high detection efficiency, the short dead-time and the low background offer a good way of getting the half-life (half-lives) measured with high precision.

Using both old and new LSC counters (LKB Wallac Rackbeta 1214 or HIDEX SL-300) we have developed and tested a universal way of measuring the decay rates and doing a statistically stringent analysis of the RNP,- in the shortest possible time period. The statistical details depend in a predictable manner on important “facts of life”: the ratio between half-lives of the constituent isotope versus the impurity(ies), the ratio between detection sensitivity in the LSC, and the background of LSC instrument. We have previously shown (1,2) that this parameter space typiccaly allow for a robust RNP method even with simple LSC instrumentation.

We have also made this method available as a program (Matlab) that can do on-line or off-line purity tests (ref.2.). The graphical user interface is shown in figure 1.

In a typical validation experiment, we can, in less than 300 minutes, experimentally detect 0.05% of ^{64}Cu (T½=12.7 h) in an otherwise pure ^{18}F sample. To do this we only use a few microliters of the product and any old or new LSC instrument. To allow for the broadest possible detection of impurities, the energy window of the LSC is set wide open, just above the electronic noise level. Nuclear betas (even from tritium), X- and gamma ray secondary electrons, conversion electrons, Auger electrons, Cerenkov, even alphas are captured with large detection efficiencies.

No spectroscopy is used, no calibration necessary. Just measure the half-life!



RNP [%] 99.95
beta 3.77
half-life [min] 109.77
DT [sec] 1.46e-05
dt [sec] 300
BG (counts) 293
POS 001
data index 1 1

result: impure

beta 2
RNP 99.9
dt/min 5
tslut [min] 1000
N0 2000000

isotope	batch ID	operator	date/time	N0	dt (std) [min]	time [min]	test limit	test value
F-18				845040	0.07	433	845508	845559

Figure 1: Graphical User Interface for the T½ analysis using LSC

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LSC methods for analysis of radionuclide impurity of ^{99m}Tc eluate in quality control of ^{99}Tc-^{99m}Tc generators

Xiaolin Hou

Technical University of Denmark, Center for Nuclear Technologies, Risø Campus, DK-4000, Denmark

Corresponding author's e-mail:xiho@dtu.dk

The radionuclidic impurities in ^{99m}Tc eluate (sodium pertechnetate injection) used in the medical diagnosis and therapy, especially the long-lived radionuclides is a major concern in the application of this product, because these impurities might interfere the efficiency of diagnosis and therapy, meanwhile the injection of radionuclide impurities will also cause an extra radiation to the patients. Therefore, it is required by the medical authorities that ^{99m}Tc eluate from the ^{99m}Tc-^{99}Mo generator has to contain limited amount of radionuclides other than ^{99m}Tc. According to Ph.Eu Monograph [1] , the major concerning radionuclides in the sodium pertechnetate injection (^{99m}Tc eluate) from a ^{99m}Tc-^{99}Mo generator produced by fission reaction of uranium are ^{99}Mo, ^{131}I, ^{103}Ru, ^{89}Sr, ^{90}Sr, alpha emitting impurities and other gamma (beta) emitting impurities. As a quality control of the ^{99m}Tc-^{99}Mo generator, the ^{99m}Tc eluate from each batch of generator has to be analyzed for the above mentioned radionuclidic impurities. Among all these radionuclides, the gamma emitters such as ^{99}Mo, ^{131}I and ^{103}Ru can be directly measured using gamma spectrometry after 3-4 days cooling of the eluate to remove most of ^{99m}Tc (6.5×10^4 by 4 days through radioactive decay). This work presents a method of chromatographic separation combined with liquid scintillation counting (LSC) measurement for measure beta emitters (^{89}Sr, ^{90}Sr) and total alpha and beta activities.

1. Removal of ^{99m}Tc and ^{99}Mo for determination of total alpha and beta activities

Due to the very high radioactive concentration of ^{99m}Tc in the eluate (1-10 GBq), and might also high ^{99}Mo due to the leakage. An anion exchange chromatographic method was applied to remove ^{99m}Tc and ^{99}Mo in order to measure total alpha and beta activities. The eluate prepared in 0.1M HCl – 0.9%NaCl solution was loaded to a 2 ml anion exchange column (AG1-×4, 50-100 mesh) [2], and the column was washed with 2 ml of 0.1 m HCl solution, the effluent and wash are combined for measurement of total alpha and beta activity and used for further radioactive strontium separation. It was found that the decontamination factor of this procedure to ^{99m}Tc and ^{99}Mo is higher than 10^4, while the most of other possible alpha emitters such as uranium and plutonium isotopes (from the irradiated enriched uranium) are remain in the solution, the recovery of Pu and U are higher than 98%. Meanwhile the most possible beta emitters including ^{89}Sr, ^{90}Sr, ^{134}Cs and ^{137}Cs are not absorbed on the column, it was observed that >98% strontium and caesium remain in the effluent and washes. However, it should be mentioned that ^{131}I, and part of ^{103}Ru, ^{106}Ru as anions are absorbed on the column. Therefore the measured total beta activity in the eluate of 99mTc does not include ^{131}I, ^{99}Mo and ^{103}Ru, which are measured directly by gamma spectrometry.

2. Separation of strontium for determination of ^{89}Sr and ^{90}Sr

Radioactive strontium in the effluent and wash from the anion exchange chromatographic separation was separated using Sr-column (Triskem). Half of the solution (effluent + wash) was taken and prepared in 8.0 HNO_3 by addition of 65% HNO_3, 2 mg Sr and 2 mg Y carrier/hold-back carrier are added. The solution is loaded to a 2 ml Sr column, and the column is washed with 8.0 M HNO_3. Strontium on the column is eluated using 10 ml of 0.05M HNO_3 solution. This eluate is directly used for measurement of ^{89}Sr and ^{90}Sr by LSC. The tracer experiment has shown that the recovery of Sr in this procedure is more than 98%, and the decontamination factor for most of radionuclides including ^{99m}Tc and ^{99}Mo are higher than 10^4. Due to quite quantitative separation of strontium in the two steps above, it is not necessary to measure the chemical yield of strontium.

3. Measurement of total alpha and total beta by LSC using discrimination function

Based on the α/β discrimination function in the LSC instrument, total alpha and beta activities in the separation sample from ^{99m}Tc eluate are measured. To 2 ml separated solution (0.1 M HCl-0.45% NaCl), 10 ml of Ultima Gold scintillation cocktail is added. A satisfied separation of alpha from beta was obtained at SPA of 100. The investigation using ^{90}Sr and ^{242}Pu as representative of alpha and beta radionuclides has shown that the cross-over of alpha to beta window is less than 3% and the beta to alpha window is less than 2%.

4. Measurement of ^{89}Sr and ^{90}Sr using Cherenkov radiation by LSC

The separated radioactive strontium in 0.05M HNO_3 solution was measured by LSC using Cherenkov counting. After Sr column separation, the solution is immediately measured (within 3 hours) to obtain the activity of ^{89}Sr before the ingrowth of ^{90}Y from ^{90}Sr. The solution is then kept for 5-7 days for ingrowth of ^{90}Y, and measured again for ^{90}Y. The Cherenkov counting efficacies for ^{89}Sr and ^{90}Y are measured using standard solution of 89Sr and 90Y to be 42% and 62%, respectively. The ^{90}Sr activity is calculated by the measurement results of two measurements and the ingrowth time from the separation to second measurement. Due to very low counting efficiency of ^{90}Sr by Cherenkov counting, the inference of ^{90}Sr to the measurement of ^{89}Sr is mainly come from the formed ^{90}Y. In the case of radioactive ratio of ^{90}Sr/^{89}Sr lower than 1, this interference is less than 4%. The interference of ^{90}Y to ^{89}Sr measurement is corrected based on the second measurement for ^{90}Y+^{89}Sr.

Summary

With the developed method combined with gamma spectrometry, all required radioactive measurement in the Ph. Eur. Including total alpha, total beta, ^{89}Sr and ^{90}Sr can be completed, the detection limits reached by this method can satisfied with the requirement in the Ph. Eur. 9.0 (6×10^{-7} for ^{89}Sr, 6×10^{-8} for ^{90}Sr and 10^{-9} for alpha emitting radionuclides relative to ^{99m}Tc in the eluate when injection). This method has been successfully used for the quality control of ^{99m}Tc-^{99}Mo generators.

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Functionalization of Polymers with Fluorescent and Neutron Sensitive Groups for Efficient Neutron and Gamma Det

Uwe Greife, Adam Mahl, Alan Sellinger, Henok Yemam

Colorado School of Mines, Golden, CO 80401, USA

We report here on the synthesis of new organic-based materials for use as dopants and/or matrices in scintillators which are to be used as low cost detector materials that provide both neutron and gamma detection while achieving high discrimination between gammas and neutrons so that they can be used for replacement of 3-He based detectors". Our approach intends to achieve its objectives through:

- Develop highly fluorescent groups that will be chemically incorporated into a polymer matrix, thus preventing aggregation of the fluorescent groups during processing and/or application that is known to reduce efficiency;
- Develop highly fluorescent boron (10-B) containing materials that can act as both neutron and alpha/gamma detection groups, and
- Develop "bulky" 3-D highly fluorescent groups that can be physically mixed into the polymer matrix, but will not migrate or aggregate due to its bulky 3-D structure.

The talk will focus on the performance (light output and pulse shape Discrimination (PSD)) of p-terphenyl, PPO and fluorene variants which were designed to be easily incorporated into a poly vinyl toluene (PVT) matrix and to, even had high doping levels, exhibit good mechanical properties.

P-terphenyl (PTP) is used widely in plastic scintillator production but is only soluble to below 3% in (PVT), our first approach was to synthesize a wide variety of PTP variants. Here we were able to correlate lower melting point with increased solubility and were able to manufacture the first terphenyl based overdoped plastic scintillator samples exhibiting PSD, good mechanical stability and light yield comparable to commercial and PPO based samples. In a second series of syntheses we worked on planar variants of fluorene which showed high solubility even when exhibiting high melting points. The best performance so far is shown by our newest approach on PPO variants where our emphasis is on improving the hardness of our samples compared to the overdoped PPO scintillator approach which is since a few years also commercially available.

Additionally, new pathways in the incorporation of 10-B for thermal neutron sensitivity and work on co-polymerization of PVT with functionalized groups will be presented.

A comparison of LSC and Isotopic Techniques in Resolving Nuclear Forensic Signatures in Uranium Mining and Processing

Ntokozo Khumalo and Manny Mathuthu

North-West University (Mafikeng), Cnr University Drive and Albert Luthuli Road, Center for Applied Radiation Science and Technology (CARST), 2735, Mmabatho, South Africa

Corresponding author's e-mail: ntokozohkhums@gmail.com

Abstract

Nuclear forensics was introduced as a division in science to resolve unanswered questions working together with law enforcement and intelligence. This is the branch of science that is associated with the analysis or characterization of seized nuclear or radioactive material based on chemical and isotopic composition, age dating and physical parameters in determining the origin of the interdicted radiological nuclear material. This parameters are called signatures and are stored in a nuclear forensic library so each country can easily have access to them for comparison in the case of seizure. The aim of this research was to compare the Liquid Scintillation Counter and ICP-MS Isotopic techniques (*1*) in resolving nuclear forensic signatures from South African Uranium Mining and Processing.

Samples were collected from a Gold mine in Carletonville, South Africa and they were analyzed using ICP-MS (Perkin Elmer NexIon 300Q) and Liquid Scintillation Counter (Perkin Elmer Quantulus Ultra Low Level). The results obtained from tailing dam 1 show an average of Al (567.11), Cr (4.42), Cu (0.47), Fe (1781.69) and Zn (0.46) mg/L. Based on this elemental concentration, Al, Cr, Cu and Zn are at trace level whereas Fe is at a minor level specifically for this site. This family of elements is indicative of a carry through from a precursor ore. La (0.046), Na (5.22), Sr (0.16) and Y (0.029) are low. As a result it shows that open cast mining is not done in this mine. The concentration of uranium is below 10 ppm for both tailing dam 1 and 2 (Table 3 and 4). This marks an absence of a blackshale deposit which generally occurs in carbonaceous marine shales. Four REE were found to be a fingerprint for this mine on both tailing dam 1 and 2. These include La, Ce, Pr and Nd. The REE concentrations for tailing dam 2 are slightly higher than that of tailing dam 1. All samples showed a strong positive Tb anomaly indicating no fractionation of HREE (Tb– Lu). Probably this is due to Geo Tectonic activity associated with the Witwatersrand area (Orkney earthquake of 2014). The Seismic instability might be causing hydrothermal enrichment of Tb. The signatures generated in this study will support and strengthen nuclear security towards combating theft of nuclear or radioactive material thereby promoting peaceful use of such material.

Table 1: Average REE concentration for tailing dam 1 (*1*), (*2*)

REE	average	Std dev	Std error	min	max
La	0.046	0.012	0.0035	0.030	0.061
Ce	0.093	0.024	0.0074	0.058	0.12
Pr	0.010	0.0026	0.00080	0.0060	0.014
Nd	0.032	0.0078	0.0024	0.021	0.045
Sm	0.0057	0.0016	0.00047	0.0040	0.0080
Eu	0.0013	0.00047	0.00014	0.0010	0.0020
Gd	0.0068	0.0018	0.00055	0.0050	0.010
Tb	0.0012	0.00041	0.00012	0.0010	0.0020
Dy	0.0046	0.0021	0.00064	0.0030	0.010
Ho	0.0010	0.00045	0.00014	0.00	0.0020
Er	0.0019	0.00094	0.00029	0.0010	0.004
Tm	0.00	0.00	0.00	0.00	0.00
Yb	0.0016	0.00067	0.00020	0.0010	0.0030
Lu	9.09E-05	0.00030	9.09E-05	0	0.0010

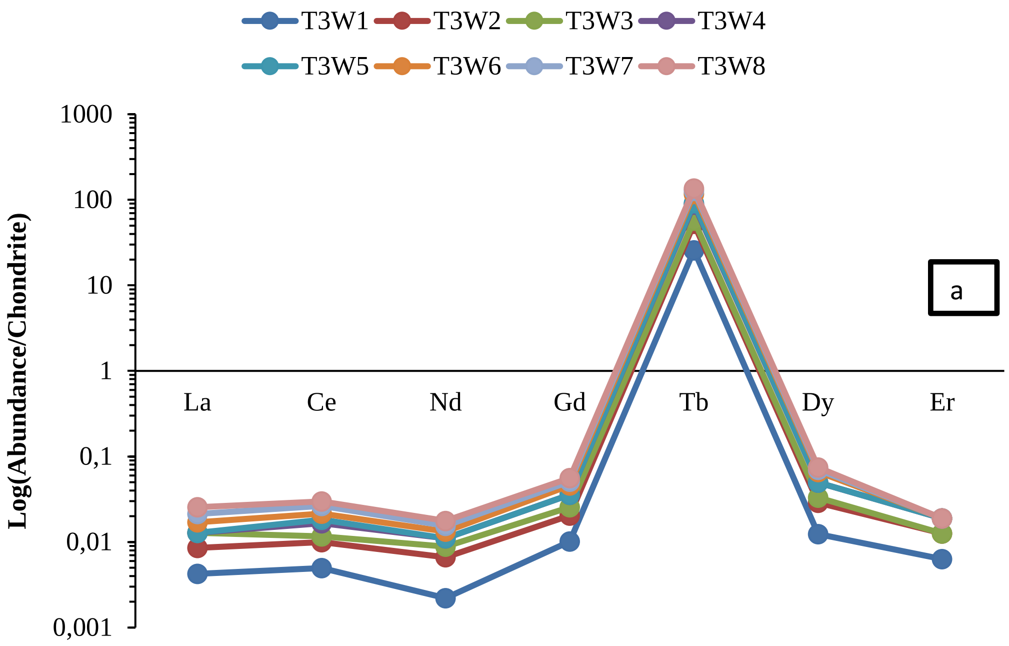

Figure **20 (a)**: Chondrite normalized Lanthanide (REE) patterns for tailing dam 3 (*3*, *4*).

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Luminescence free counting of H-3 facilitated by Hidex 300 SL/600 SL TDCR triple coincidence counters

Risto Juvonen, Hidex Oy, Mustionkatu 2, 27800 Turku, Finland,

risto.juvonen@hidex.com

Luminescence is one of the major interferences in scintillation counting causing increase of the background and variation in the results. Regardless of the origin, luminescence is low energetic and yields in emission of single photons. As such it's the lowest energetic true photon emitting phenomena in the samples and challenging to distinguish from multiphoton beta events.

As standard procedure, scientists are waiting for hour to several days for luminescence decay. One can also subtract Chemiluminescence CPM from the results but as the luminescence count rate is relatively low and variable this may yield in high uncertainty of the results. Very often samples are counted repeatedly and outliers are removed. One can also set a luminescence free energy window above luminescence region. With conventional counters this works relatively well with higher energy isotopes like 14C but yields in too low counting efficiency for 3H. With triple coincidence counters Hidex 300 SL and 600 SL, the counting efficiency is higher and instruments can record triple coincidence counts and double coincidence counts separately. These features allow luminescence free methods also for 3H in water. At Hidex Counters the feature is called Luminescence Free Counting Mode (LFCM). In LFCM the counter records only the coincidence counts that are free from luminescence. These counts are 1) all triple coincidence counts and 2) double coincidence counts at above luminescence ROI (above channel 110).

Conventional methods (decay of luminescence and removal of outliers) were compared to luminescence free counting method for measurement of 3H in water (Table 1).

Table 1. 3H performance comparison of 300 SL to Wallac Quantulus

- 8+12 ml, 4 h measurement time, 3 s uncertainty
- Samples were stabilized for 20 h before the start of the measurement

	Normal mode	Lum free mode	Quantulus
Bg	4.7 CPM	2.8 CPM	0.5 CPM
Eff	37 %	24 %	25 %
Ld	2.4 Bq/L	2.8 Bq/L	1.7 Bq/L
Outliers	High risk	Low risk	High risk

As conclusion the counting efficicncy is reduced but the detection limit is only slightly higher than that in normal count mode due to lower background count rate. As an advantage luminescence free mode allows more or less immediate start of the measurement after sample preparation and there is no need to detect and exclude outliers after the measurement. This yields in shorter measurement times in total and higher reliability of the results as the risk for outliers is reduced.

Optimization of a/B separation using 2D graphical tool

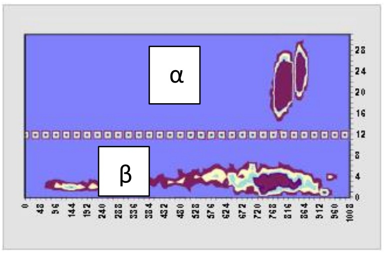
Ville Haaslahti, Hidex Oy, Mustionkatu 2, 27800 Turku, Finland,

ville.haaslahti@hidex.com

Liquid Scintillation counting provides a convenient and sensitive detection of alpha isotopes in presence of betas and gammas. Typical applications include Rn-222, Ra-226 and gross alpha/beta measurements. Alphas are separated from betas based on the pulse decay time. Selection of materials (cocktails and vials) and sample to cocktail ratio is essential for good separation efficiency and resolution.

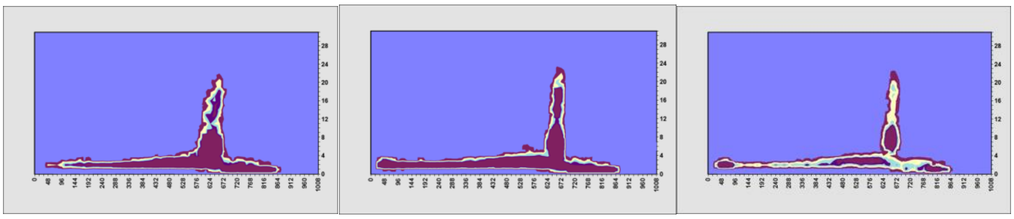
The alpha/beta discriminator must be calibrated for each type of application. This is done conventionally by doing so called misclassification. Pure beta and alpha standard samples are measured with different discriminator settings yielding in so called misclassification curve to record discriminator with minimum misclassification of betas as alphas and vice versa. The challenge for this method is that optimum value is different for different isotopes and for different degree of quenching. As consequence there is risk for high uncertainty of results if the isotopes or conditions at unknown samples are different than those at misclassification (e.g. gross alpha/beta).

The calibration and optimization of alpha/beta discriminator at Hidex counters is based on 2D graphical optimization too. Alphas and betas are printed in same 2D spectra and optimum discriminator can be defined visually.



Same 2D graph can be used to optimize the conditions (e.g. cocktail, sample to cocktail ratio and vial type) and to verify the quality of results of unknown samples. For calibration one can use even unknown sample with some alphas but also the same standards as used for conventional misclassification.

An example of optimization: Sr-90/Am-241 sample measured on different conditions



UltimaGold AB in Glass
-alpha and beta regions merged together

UltimaGold AB in Teflon
-better than previous but not optimum

AquaLight AB in Teflon
- zero misclassfication

2D graph can be used as a tool 1) for optimizing measurement conditions, and 2) for quality control of the results.

Most important factors to optimize the conditions are 1) selection of optimum vial and cocktail, 2) reduction of quenching by optimizing sample to cocktail ratio and 3) optimizing alpha/beta parameters of the instrument.

Measurement of Tritium, gross alpha/beta and ^{222}Rn with the new Quantulus GCT 6220 according to council directive 2013/51 Euratom

Ronald Edler

The name of the first institution, address, post code, country

The PerkinElmer, Schnackenburgallee 114, Hamburg 22525,Germany

Corresponding author's e-mail: Ronald.edler@perkinelmer.com

Since the publication of the council directive 2013/51 Euratom in October 2013 **[1]** the analysis of drinking water was in the focus again of many laboratories European wide. This directive lays down requirements for the protection of the health of the public with regard to radio isotopes in water intended for human consumption. In order to avoid time consuming sample preparation for individual isotopes, gross alpha/beta measurements became very attractive again. In addition, Tritium and Radon have to be determined in drinking water and measurements are very often performed with liquid scintillation counting. Meanwhile the new council regulation is part of many national regulations. The council directive is a kind of frame for the European countries and sets some important parameters such as sampling rates, maximum indicative dose resulting from isotopes in the water and required detection limits of the instrumentation as shown in the table below. According to the council directive the maximum allowed indicative dose is 0.1 mSv. In case the activity of the gross a/b measurement is below 0.1 and 1.0 Bq/L respectively, it can be assumed that the total indicative dose is below 0.1 mSv/year.

Table 1 Activity and detection limits for analysis of drinking water

Isotope	Max. activity allowed in water	Required detection limit
^{3}H	100 Bq/L	10 Bq/L
^{222}Rn	100 Bq/L	10 Bq/L
Gross α	0.1 Bq/L	0.04 Bq/L
Gross β	1.0 Bq/L	0.4 Bq/L

In addition to activity limits and required detection limits the council directive sets some numbers for sampling and analysis frequencies. This resulted in increasing number of samples and sensitive instruments are required to enable the laboratories to do the measurements in acceptable counting times. Some of the European countries such as Germany **[2]** have set even lower detection limits for some of the isotopes. This resulted in challenging detection limits especially for the gross a/b measurement. In most cases enrichment is necessary to reach the required detection limits. Especially waters with higher amounts of dissolved ions can limit the sensitivity of the liquid scintillation counter due to uptake limitations of the cocktail when using waters with high ionic strength. As a consequence of the low required detection limits sensitive liquid scintillation counters are necessary to obtain reliable data with acceptable measurement times. Our investigations demonstrated that the new Quantulus GCT 6220 has very low and stable background for $\alpha-$ and $\beta-$ measurements without compromising the counting efficiency. Spill from $\alpha-$ into the $\beta-$channel and vice versa was very low even at elevated quench levels. In total this resulted in very good sensitivities. The gross alpha/beta measurements were performed according to ISO 11704 **[3]** and ASTM D7283-13 [**4**]. Tritium measurements were performed according to ISO 9698 **[5]** and ^{222}Rn measurements were performed according to ISO 13164-4 **[6]**. All results are reported according to ISO 11929 **[7]**. The sensitivity of the Quantulus GCT 6220 instrument results in very short counting times for ^{3}H, ^{222}Rn and gross $\alpha-/\beta-$measurements thus allowing higher throughput of samples required by many laboratories involved in drinking water analysis.

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Thursday

4 May 2017

Poster session-2

Assessment of ^{90}Sr by liquid scintillation counting (LSC) in marine fish species consumed in the city of São Paulo

C.H.R. Saueia[1], B. Castilho[1], M. B. Nisti[1], B. P. Mazzilli[1]

[1] IPEN – CNEN/SP, Av. Professor Lineu Prestes 2242, 05508-000, Brazil

Corresponding author's e-mail: chsaueia@ipen.br, bcastilho@ipen.br, mbnisti@ipen.br, mazzilli@ipen.br

The study of artificial radionuclides dispersion into the environment is very important to control the nuclear waste discharges, nuclear accidents and nuclear weapons testing. The accident in Fukushima Daiichi Nuclear Power Plant on 11 March 2011, released several radionuclides in the marine environment by aerial deposition and liquid discharge, with various level of radioactivity. The ^{90}Sr was one of the elements released into the environment, it is estimated that its atmospheric releases ranged from $3.3.10^{-3}$ to 0.14 PBq. The ^{90}Sr is produced by nuclear fission with a physical half-life of 28.79 years with decay energy of 0.546 MeV. The ^{90}Sr is chemically similar to calcium and has a biological half-life of 49.3 years, it can bio accumulated in fish muscle and bone. Due to its radio toxicity, the ^{90}Sr has been extensively studied in the marine environment.

The aim of this study is to quantify the concentration of the ^{90}Sr in the muscles of marine fish species most consumed in São Paulo city. The species and scientific names of the fishes analyzed were bluefish (pomatomus saltatrix), croaker (micropogonias furnieri), hake (merluccius merluccius), Smooth-hounds nei (Mustelus spp.), sardine (sardinella brasiliensis) and mullet (Mugil brasiliensis). The determination of ^{90}Sr by liquid scintillation counting (LSC) measurement was performed using a 1220 Quantulus™ Ultra Low Level Liquid Scintillation Spectrometer.

The results obtained in this study can be used to ensure the quality of the fish consumed in the São Paulo city.

Carbon-14 concentration in aqueous samples from southeast of China

BAO Li

China Inst. for Radiation Protection

E-mail: blinda008@163.com

There is an increasing concern with the radiological impact associated with the discharge of long-lived radionuclides. Carbon-14 is one of the most significant radionuclides in the group because of its long half-life (5730yr) and the ease with which it assimilates into biological system.

It is more difficult in monitoring radiocarbon in seawater than in fresh water because there is a lot of SO_4^{2-} and other impurity. We investigated a simple, reliable radiocarbon measurement procedure for all types of water samples using the bubbling and direct absorption method, followed by low-level liquid scintillation spectrometry. A bubbling line was established for environmental aqueous samples which can deal with 50 L sample by one time. Carbo-sorb E was used for absorb CO_2 and the LS spectrometer was Tri-carb 3170. The recovery of sample pretreatment (bubbling line) can up to 99% and the full recovery is 90%, with the LLD of 0.20mBq/L.

This method was designed for routine analysis of water samples, and it is proposed for use in environmental radiocarbon monitoring programs. Several aqueous samples from southeast of China were determined using this process. For sixteen seawater samples from Fujian Province, the DIC concentration was 16.7~25.8 mg/L and radiocarbon concentration was 4.32~6.82mBq/L. The results of sixteen seawater samples from Hainan Province were 21.4~23.4 mg/L and 5.48~6.71mBq/L, four seawater samples from Guangxi Province were 12.4~17.3 mg/L and 2.37~3.29mBq/L.

Some surface water and underground water samples were also measured. Radiocarbon concentration of eight surface water samples from Hainan Province were 5.42~9.03mBq/L, and the results of eight surface water samples from Fujian Province were 4.46~8.38mBq/L. While the radiocarbon concentration of eight underground water samples from Hainan and Fujian Province were 7.13~12.70mBq/L and 7.71~10.62mBq/L respectively. The difference in different district was not obviously, but the radiocarbon concentration in underground water was a little higher than surface water.

Optimization of rapid ^{90}Sr/^{90}Y screening method in waters via Cherenkov radiation detection

Ivana Stojković (1), Jovana Nikolov (2), Nataša Todorović (2)

(1) University of Novi Sad, Faculty of Technical Sciences, Trg Dositeja Obradovića 6, 21000 Novi Sad, Serbia
(2) University of Novi Sad, Faculty of Sciences, Trg Dositeja Obradovića 3, Department of Physics, Novi Sad, Serbia

Corresponding author's e-mail: ivana_st@uns.ac.rs

Radiostrontium ^{90}Sr is present in spent nuclear fuel and radioactive waste from nuclear reactors as a product of nuclear fission, as well as in fallout from nuclear weapon tests and nuclear accidents. Its long physical and biological half-life ($T_{1/2}$ = 28.8 y) makes ^{90}Sr most biologically hazardous radionuclide of major concern in environmental contamination studies. Its determination in water by conventional methods is time-consuming and assumes expensive and sophisticated radiochemical separation techniques followed by gas proportional or liquid scintillation counting.

System configuration, optimization and validation of a simple method for ^{90}Sr screening in water via Cherenkov radiation detected by a low-level liquid scintillation counter Quantulus 1220^{TM} has been presented in this paper. Although most energetic electrons from ^{90}Sr beta spectrum ($E_{max} = 546\ \text{keV}$) exceed Cherenkov threshold in pure water ($263\ \text{keV}$), more realistic is Cherenkov counting of ^{90}Sr daughter, ^{90}Y, that generates beta spectrum with $E_{max} = 2283\ \text{keV}$. ^{90}Y has a half-life of 64.1 h, thus, this pair is in secular equilibrium in environmental samples.

Low diffusion polyethylene vials, high performance glass counting vials and low potassium borosilicate glass vials of 20 ml volume were used in order to examine their potential effect on counting process. The derived efficiency from calibration curves was 45.86(9) %, and a minimum detectable activity of 0.32 Bq l^{-1} in a 20 ml water sample in polyethylene vial has been achieved during 300 minutes of counting. However, environmental water samples might be colored which would induce color quenching, one of the most important problems that affect Cherenkov counting. The sample channel ratio (SCR) method has been applied to correct this effect.

Described analytical procedure and measurement technique was tested by participating in the IAEA world-wide proficiency tests on determination of ^{90}Sr in water samples. Presented method has some advantages over conventional ones, since it is ultra fast, simple, non-destructive and inexpensive test for efficient screening of water samples without any chemical pre-treatment. Its limitation is inability to correct possible interferences between ^{89}Sr and ^{90}Sr spectra that might occur in environmental water samples in case of nuclear accident.

Evaluation of different LSC methods for ^{222}Rn determination in waters

Ivana Stojković 1), Jovana Nikolov 2), Nataša Todorović 2)

1) University of Novi Sad, Faculty of Technical Sciences, Trg Dositeja Obradovića 6, 21000 Novi Sad, Serbia

2) University of Novi Sad, Faculty of Sciences, Trg Dositeja Obradovića 3, Department of Physics, Novi Sad, Serbia

Corresponding author's e-mail: ivana_st@uns.ac.rs

Monitoring of ^{222}Rn in drinking or surface waters, as well as in groundwater have been performed in connection with geological, hydrogeological and hydrological surveys and health hazard studies. Liquid scintillation counting is often preferred analytical method for ^{222}Rn measurements in waters that allows multiple-sample automatic analysis. It implies mixing of water samples with organic scintillation cocktail, which triggers radon diffusion from the aqueous into organic phase for which it has a much greater affinity, eliminating possibility of radon emanation in that manner.

Two direct LSC methods that assume different sample composition have been presented, optimized and evaluated in this study. One-phase method assumed direct mixing of 10 ml sample with 10 ml of emulsifying cocktail (OptiPhase HiSafe 3 or Ultima Gold AB). Two-phase method involved usage of water-immiscible cocktails (High Efficiency Mineral Oil Scintillator, Opti-Fluor O or Ultima Gold F). Calibration samples were prepared with aqueous ^{226}Ra standard in glass 20 ml vials and counted on ultra-low background spectrometer Quantulus 1220$^{\text{TM}}$ equipped with PSA (Pulse Shape Analysis) circuit which discriminates alpha/beta spectra. Since calibration procedure is carried out with ^{226}Ra standard, which has both alpha and beta progenies, it is clear that PSA discriminator has vital importance in order to provide reliable and precise spectra separation. Consequentially, calibration procedure was done through investigation of PSA discriminator level influence on ^{222}Rn efficiency detection, using ^{226}Ra calibration standard in wide range of activity concentrations.

Evaluation of presented methods was based on obtained efficiency detections and achieved Minimal Detectable Activity (MDA). Comparison of presented methods, accuracy and precision as well as different scintillation cocktail's performance was considered from results of measurements of ^{226}Ra spiked water samples with known activity and environmental samples.

Use of the IAEA rapid simultaneous determination of ^{89}Sr and ^{90}Sr in milk as a routine method for environmental purposes

R. Idoeta, M. Herranz, S. Rozas and F. Legarda

Dpto. de Ingeniería Nuclear y Mecánica de Fluidos, ETSI de Bilbao, Universidad del País Vasco UPV/EHU, Alda. Urquijo s/n, 48013 Bilbao, Spain

Corresponding author's e-mail: raquel.idoeta@ehu.eus

Rapid methods useful for nuclear accident or incident situations have to be implemented and validated in radioactivity measurement laboratories so as to be ready to give a quick answer to governments, regulatory organizations and people in the case of such situations. This implementation is done though the training of experimented technicians and the regular application of these methods in the laboratories so as to be ready to apply them efficiently in case of an atypical emergency situation.

Accordingly, the best way to do so is to use the same method for both routine and rapid or emergency situations. To do this, an analysis of the conditions under which a rapid method could be used as a routine one should be accomplished.

The aim of this work is to analyse the performance of the rapid method for the simultaneous determination of ^{89}Sr and ^{90}Sr in milk, published by the IAEA [1], based on the radiochemical separation of strontium using cation exchange and extraction chromatography followed by the measurement of the sample using Cherenkov counting and afterwards liquid scintillation counting, to be used as a routine environmental procedure.

There are different international regulations, like the Codex Alimentarius [2], with Guidelines levels for certain radionuclides to be applied to commodities moving in international trade, or the Council Regulation (Euratom) 2016/52 [3], laying down maximum permitted levels of radioactive contamination of food and feed following a nuclear accident or any other case of radiological emergency, which ask for methods able to provide rapidly ^{89}Sr and ^{90}Sr activity concentrations in milk but without any requirement in detection limits, that can be relatively high.

However, from an environmental point of view, the lowest detection limits for ^{89}Sr and ^{90}Sr in milk are requested as, for example, in the Spanish regulation on the detection limits that laboratories have to obtain in the framework of the Radiological Monitoring Environmental Plans [4]. So, if the IAEA rapid method is to be considered as a routine method for environmental purposes, it should be able to provide those lowest values for detection limits for ^{89}Sr and ^{90}Sr in milk.

So, a study on which values of the measurement parameters would help to meet this requirement is performed. That is, the sample volumes, counting times, time lapse between the two measurements and background counting time are varied in order to obtain the optimal values of these parameters to be able to use the IAEA method as an environmental one. A numerical simulation of the IAEA method is applied to a set of hypothetical samples with different ^{89}Sr and ^{90}Sr activities that could be expected in the regulations and also considering different ^{89}Sr /^{90}Sr ratios.

Proposal for optimal selection of measurement parameters in case it is used for routine environmental purposed is finally presented.

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Ra-226 determination by LSC and alpha-spectrometry in by-products from oil industry

R. Idoeta, M. Herranz, K. Olondo, S. Rozas and F. Legarda

Dpto. de Ingeniería Nuclear y Mecánica de Fluidos, ETSI de Bilbao, Universidad del País Vasco UPV/EHU, Alda. Urquijo s/n, 48013 Bilbao, Spain

Corresponding author's e-mail: raquel.idoeta@ehu.eus

Assessment of ^{226}Ra contents in different by-products generated during oil extraction and processing is a very important task of the Radiological Protection of workers. Different products like water, scales, and sludge have very different ^{226}Ra activity concentrations. These by-products are also very different from the physical and chemical point of view and should be treated as very complex matrices. They have different composition and densities, organic matter, elements like barium and rare-earth, and some of them can also be flammable.

If direct gamma-ray spectrometry is carried out for their determination, larger detection limits are obtained, so, other methods which include the radium chemical separation process should be chosen to assess the activities of those matrices.

This paper focuses on the determination of radium contents by two different methods: co-precipitation followed by liquid scintillation counting (LSC), via assessment of ^{222}Rn, and by ion-exchange and alpha spectrometry (AS). The advantages and disadvantages of the two procedures as methods for achieving low detection limits in by-products from oil industry are evaluated. Three different matrices have been tested for this analysis: scales, sludge and oil.

Liquid scintillation measurement by means of radon emanation requires about 25 day span from the preparation of matrices to the measurement in order to allow equilibrium to be achieved. On the other hand, alpha spectrometry can be carried out about one week after sample preparation and despite the fact that the counting time is much longer than that usual for scintillation, it can be considered a quicker method.

Detection limits obtained are around 1E-04 Bq for both AS and LSC but with different counting times, 120 and 45 hours, respectively. Uncertainties obtained are also somewhat lower in liquid scintillation measurements.

Problems arising in alpha spectrometry when ^{225}Ra is used as a yield tracer, like electrodeposition yield, lack of control on ^{225}Ac behaviour and impact on the results of the different times involved in all the steps of the separation process are analysed. Similarly, other problems arising if liquid scintillation counting is chosen, using Ba as a yield tracer, like stability of the samples and the possible interference of the barium content of the samples in the radiochemical yield are also analysed.

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Pilot Studies on ^{90}Sr in Meat, Milk and Dairy Products from the Polish Market

Wiatr K.[1)], Rachubik J.[1)], Piskorska-Pliszczynska J.[1)]

[1)] National Veterinary Research Institute, Partyzantow 57, 24-100 Pulawy, Poland

^{90}Sr and its daughter ^{90}Y are highly radiotoxic isotopes. Because of its affinity to bone tissue, ^{90}Sr can cause bone cancer or leukemia. Presence of ^{90}Sr in the environement is caused by atmospheric nuclear tests and nuclear power plants catastrophes (Chernobyl 1986, Fukushima 2011). Due to limitted information on ^{90}Sr activity levels in foodstuffs new studies are desired. In the first phase of our work we are focused on meat (poultry, pork, beef, fish), milk and dairy products (cows, goats, sheep). A method used for analyses is based on liquid-liquid extraction of ^{90}Y and LSC measurement. Obtained data show that activity of ^{90}Sr in all meat matrices is below MDA. In cow milk, 5 of 12 samples have activity slightly above MDA. Activity of ^{90}Sr in goat and sheep milk ranges from 0.02 to 0.08 Bq L^{-1}. It suggests that goat and sheep milk is a better indicator of pollution with ^{90}Sr . Studies require continuation because of a small number of analysed samples.

Biolubricant measurements by direct LSC method

Romana Krištof, Jasmina Kožar Logar

Jožef Stefan Institute, Jamova 39, SI-1000, Slovenia

Corresponding author: romana.kristof@ijs.si

The direct LSC method is known as one of the methods providing fast and repeatable results of bio-component quantification. Recently, accuracy and trueness has been demonstrated by various intercomparison exercises as well as its incorporation into DIN 51637 standard. In last years, development of so called bio-lubricants, lubricants produced fully or partly from renewable sources, has been made and introduction to the market has occurred. The tendency of the society to use more environmentally sustainable sources has arisen a need for the analytical methods which can quantify bio-part of the resource.

In-house method was used for the initial test measurements of the various liquid lubricants. We will demonstrate that no changes to the sample preparation and measurement need to be done in order to conduct bio-lubricants quantification. LS sample are prepared by simple mixing of Ultima Gold F™ scintillator and lubricant sample. Results of calibration, initial measurements of lubricants on the market as well as some of validation parameters will be presented. We will demonstrate that the direct LSC method can easily be adopted for lubricant measurements and used on routine basis if such need will be expressed by the governmental authorities.

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Optimisation of Tri-Carb Liquid Scintillation Counter for Cherenkov Counting of ^{90}Sr/^{90}Y

Ivana Coha, Gorana Kranović, Željko Grahek

Ruđer Bošković Institute,Bijenička cesta, Zagreb, 10000, Croatia
Corresponding author's e-mail: *ivana.coha@irb.hr*

Strontium radioisotopes, especially ^{90}Sr, attract attention for many decades by radiochemical laboratories worldwide. Since it is pure beta emitter, its separation from matrix is desirable but most of the time complex and time consuming. The researches all over the world are publishing new, more rapid methods for its isolation and detection. The most recent method developed in our laboratory is its isolation from large sample volumes on highly selective extraction chromatographic resins, and measurement directly on column [1]. The procedure includes separation of strontium from matrix on Super Lig 620 column followed by off line on column detection via Cherenkov counting on commercially available instrument. The goal is further development of the method and determination of low level activity of ^{90}Sr present in the environment by Cherenkov detection.

Generally, the advantage of Cherenkov counting technique is simplicity of sample preparation. The sample is not diluted with any scintillation cocktail and therefore it remains unchanged and it can be recovered for further analysis. The drawback of the method is lower efficiency of the detection in comparison with liquid scintillation counting (LSC). Therefore, the goal of this research is to optimise detection efficiency on commercially available instrument, Tri-Carb 3180 TR/SL. The characteristic of this instrument is patented time-resolved technology, primarily developed for LSC of low level activity samples. This feature has also influence on Cherenkov detection [2]. By using different media it is possible to increase detection efficiency. For example, if ^{90}Sr/^{90}Y is counted in different acid solutions (Figure 1), detection efficiency in low level counting mode (LLCM) compared with normal counting mode (NCM) is decreased, whereas in nitric acid solution is similar.

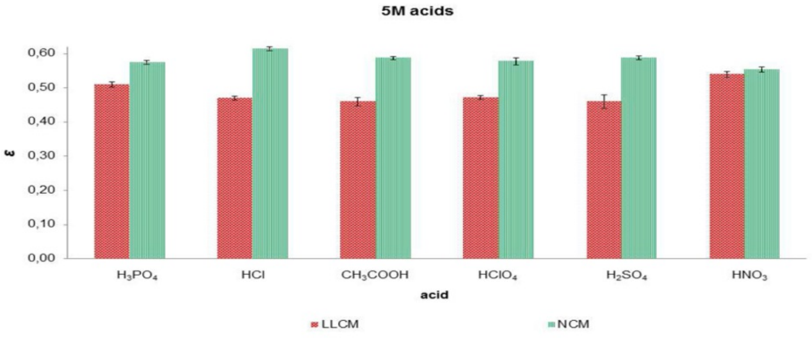

Fig. 1 Cherenkov counting efficiency on Tri-Carb for different 5 M acids in low level and normal counting mode

Within this research, it will be investigated more in detail (by using different media, column type, etc.) as well as other parameters wich were set up for LSC (coincidence time, delay before burst etc.).

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Sample pre-treatement for HTO measurements in biota and seawater

Nikolaus Hermanspahn 1), Michael Lechermann 1)

1) Institute of Environmental Science and Research Ltd, 27 Creyke Rd, Christchurch 8041, New Zealand

Corresponding author’s e-mail: Nikolaus.hermanspahn@esr.cri.nz

The Institute of Environmental Science and Research operates environmental radioactivity monitoring programmes for the New Zealand government and commercial clients. The focus of the monitoring programmes is on human health impacts from anthropogenic radionuclides.

Low level applications require tritium enrichment to reach the required sensitivity. Biota and seawater samples need pretreatment before tritium enrichment process can be started. Pre-treatment for biota samples is commonly performed by freeze drying and subsequent processing of the water (HTO) condensate.

We will present layout, operational characteristics and method development results for sample pre-treatement for tritrium analysis using a Genevac miVac-Duo evaporator system fitted with a SpeedTrap collection system.

Results will include extraction efficiency and recovery and overall measurement accuracy as obtained in proficiency tests.

Determination of Sr-90 in soil and sediment samples with double energetic window Method by LSC

Funda Barlas Şimşek

Turkish Atomic Energy Autority, Çekmece Nuclear Research and Training Center, 34303, Küçükçekmece, , İstanbul-Turkey

Corresponding author's e-mail: simsekbarlas@gmail.com

^{90}Sr is one of the more hazardous fission products that have reached the natural environment in larger quantities after atmospheric nuclear weapon tests and large-scale nuclear accidents like Chernobyl and Fukushima Daiichi. ^{90}Sr is a fission product with E_{max} of 546 keV and half-life of 28.5 y. In general it is in equilibrium with its daughter,^{90}Y, E_{max} of 2282 keV and half-life of 2.67 days (L'Annunziata, 1998). With a long half life it exhibits a biochemical behavior similar to calcium and after entering the human body, most often by ingestion of contaminated foodstuffs or water, can be mainly deposited in bones and bone marrow, as well as in blood and soft tissues (Lopes et al., 2010).

Various analytical methods have been developed for the determination of ^{90}Sr in environmental and nuclear samples using different techniques such as beta counting and liquid scintillation spectrometry (Juznic and Fedsina 1986; Randolph 1971).

In this work, a methodology for the determination of ^{90}Sr in soil and sediment samples is presented. The method consists of 3 basic steps: oxalate precipitation to remove potassium, chromatographic separation of strontium from interferences utilizing Sr Resin (Eichrom Technologies LLC), and oxalate precipitation of strontium to evaluate the chemical yield. . Following the chemical procedure an aliquot of the dissolved sample (1 mL) is mixed with 14 mL of scintillation cocktail (Insta-Gel Plus, Perkin Elmer) in a 20-mL container vial and the sample activity is determined using a liquid scintillation spectrometer, Perkin Elmer Quantulus 1220. The chemical recovery is determined by gravimetry. Counting efficiency is evaluated with a known activity of ^{90}Sr standard solution after the chemical procedure above is applied to this solution which is in secular equilibrium with the progeny ^{90}Y. Determination of the activity concentration of ^{90}Sr was performed using the double-energetic windows method which relies on the direct determination of ^{90}Sr (Vajda et al., 1992). For the successful determination of this radionuclide two counting windows were optimized to eliminate the overlapping of the beta spectra of ^{90}Sr and ^{90}Y.

The validity of results was tested by performing the same procedure on IAEA- 375 standard reference material and QC samples. The combination of chromatographic separation of Sr from acidic media followed by liquid scintillation counting gives reliable results for ^{90}Sr determination in soil and sediment samples. Also the method is relatively fast since the digestion, separation and counting procedures require about 15 hours, except the time required for the samples to become stable before the counting process. Moreover, the procedure achieved a good chemical yield, in the range 60–90% and counting efficiency about 95%.

Finally, the method was also tested by participation in IAEA intercomparison program, with soil samples. The results showed a good correlation between the measured value and the reported value.

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Determination of Pu in environmental water samples using TEVA microextraction chromatography separation and liquid scintillation counter measurement

Bo Liu, Ze-Yi Yan, Tong-Huan Liu, Keliang Shi, Wangsuo Wu

Radiochemistry Lab, School of Nuclear Science and Technology, Lanzhou University, Lanzhou 730000, China
Corresponding author's e-mail: yanzeyi@lzu.edu.cn

The most common isotopes of plutonium in the environment were ^{238}Pu, ^{239}Pu, ^{240}Pu and ^{241}Pu. Except β emitting radionuclide ^{241}Pu, all the others give rise to α emission and have long half-lives (^{238}Pu: 4.3×10^{10} year, ^{239}Pu:5.5×10^{15} years, ^{240}Pu: 1.22×10^{11} year), which lead to long-term persistence in the environment. Due to the nuclear activities by the people, it has been reported that more than 10^{16} Bq of $^{239+240}$Pu have been already released into the environment. Plutonium can migrate into the water and soil by the action of monsoon and ocean current, and enter the body of human through the food chain. As the consequence of so much harm to human, the accurate quantification of Pu in diverse environmental samples, especially in environmental water samples, becomes an important work to the environmental regulatory authorities. The present work mainly aims at the rapid determination of trace level Pu in water samples by a vacuum box system equipped with micro-TEVA extraction columns followed by detecting α decay of $^{239+240}$Pu using low background liquid scintillation counter (LSC).

Table 1 Comparison of the determined concentrations of Pu with conventional values

Sample	Concentration by conventional method (mBq L^{-1})	Concentration by present method (mBq L -1)	Yield, %
Artificial sample 1	9.7 ± 0.3	9.8 ± 0.4	98.0
Artificial sample 2	48.8 ± 2.8	49.2 ± 4.1	98.4
Artificial sample 3	96.5 ± 4.5	97.4 ± 3.6	97.4
Water sample 1	8.2 ± 0.3	8.5 ± 0.5	83.5
Water sample 2	42.3 ± 3.6	43.3 ± 5.2	85.7
Water sample 3	8.5 ± 0.5	8.6 ± 0.3	88.3

[a] Value were the average of six replicates (± standard deviation).

[b] Values were obtained using the China GB11225-89.

[c] The detection limit was 6×10^{-3} Bq L^{-1}.

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Optimization of liquid scintillation counting technique for a European interlaboratory comparison in gross alpha/beta activity concentrations in water

Viktor Jobbágy[1)], Andrej Rožkov[1,2)], Mikael Hult[1)], and Gulten Özçayan[3)]

[1)] European Commission, Joint Research Centre (EC-JRC), Retieseweg 111, 2440 Geel, Belgium
[2)] Now at European Commission, Research Executive Agency, Rogier 16, 1210 Brussels, Belgium
[3)] Turkish Atomic Energy Authority, Sarayköy Nuclear Research and Training Center, Saray Mah, Atom Cad, No:27, 06890 Kazan/Ankara, Turkey

Corresponding author's e-mail: viktor.jobbagy@ec.europa.eu (European interlaboratory comparison) and andrej.rozkov@ec.europa.eu (method elaboration)

An interlaboratory comparison (ILC) in gross alpha/beta activity concentrations in water was organized among 71 environmental radioactivity monitoring laboratories in Europe by the Institute for Reference Materials and Measurements (from 1 July 2016 JRC-Geel) (Jobbágy et al., 2015).

The main goals of the comparison was to obtain more information on the measurement methods used by the EU Member States to report the results on water radioactivity in the environment under the Articles 35, 36 of the EURATOM Treaty and to compare the performance of the participating laboratories with regard to employed methods (Jobbágy et al., 2013 and 2015).

For the determination of the reference values of the three different interlaboratory water samples, four independent standard methods were considered including a liquid scintillation counting (LSC) method. Our main intention was to contribute to the reference values of comparison samples used in this interlaboratory comparison with accurate LSC results. For this purpose, the optimization of an LSC method based on the ISO 11704 (ISO, 2010) international standard was performed.

The ISO 11704 standard provides sometimes general recommendations on sample preparation and measurement procedures of gross alpha/beta using LSC in drinking water. However, the general recommendations in the standard could be elaborated further in view of this special case (three known ILC samples) and for the sake of establishing a reference value when more where more labour-intensive work can be accepted in comparison to routine measurements. An important aspect was to optimize the standard method for the measurement of drinking water samples with relatively low gross alpha and beta activity concentrations with accuracy adequate for this ILC.

First, the procedure of the radionuclide pre-concentration was adjusted for each of the three different types of ILC water samples with regard to their mineralization. During the sample preparation the main aims were to minimize the adsorption of the radionuclides on the glassware and to avoid cross-contamination of the prepared samples. The pre-concentration procedure was verified by spiking water samples with known activities of ^{241}Am and ^{90}Sr/^{90}Y standard solutions.

The chemical yield was evaluated by spiking the samples using a KCl solution with known (natural) ^{40}K activity.

Next, sample preparation and then the sample measurement conditions with the low-background LS counter were optimized paying special attention to the following parameters:

1. Sample preparation: Selection of scintillator cocktail, volumetric ratio between water sample and cocktail, effect of mineralization and pH on sample quench, material of the LSC vial.

2. Measurement: Measurement time, spectra window, counting efficiency, spillover correction coefficients and pulse shape analysis setting.

The settings of the alpha/beta discriminator pulse shape analysis (PSA) value were determined for each type of water samples. The alpha and beta counting efficiency was determined using radionuclide calibration solutions. The optimized LS counting method was verified using samples spiked with ^{241}Am and ^{90}Sr/^{90}Y standard solutions. The minimum detectable activity concentrations obtained with the elaborated method were 5 mBq·L^{-1} and 20 Bq·L^{-1} for the gross alpha and gross beta radioactivity, respectively.

As a part of the work, analysis of the most common errors in the determination of the gross radioactivity was conducted. These errors include underestimation of the background fluctuations during the sample measurement, variation in PSA value settings, loss of radionuclides during the pre-concentration from water samples of relatively large mineralization and build-up of radon decay products in samples.

The reference value was established by four expert laboratories using different LSC methods. After analysing the interlaboratory water samples the results of the optimized LSC method were compared to the other methods from the reference value determination campaign as an example shown for one of the ILC water samples in Figure 1.

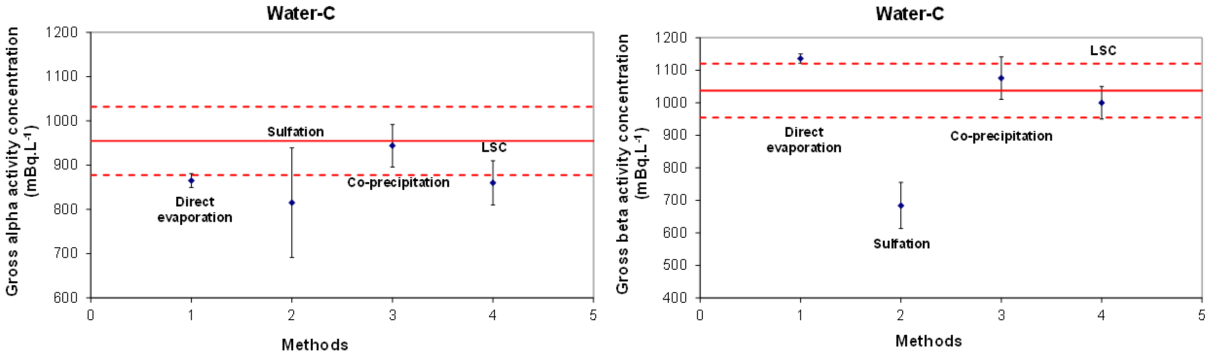

Fig 1. Comparison of independent gross alpha/beta methods for water C spiked sample. Activity concentration values are given with their combined uncertainties (k=1). Red solid line shows the mean activity concentration and red dashed line shows the upper and lower values of combined measurement uncertainty of mean activity concentration (k=1).

In general it can be concluded that the LSC gross alpha/beta activity concentration measurement results were in agreement with the mean values within their limits of uncertainty. The performance of the LSC method described here proved to be adequate for gross alpha/beta activity measurements in the drinking waters used in this ILC. This LSC method is straightforward and capable of providing relatively rapid quantitative and semi-qualitative measurement information about gross activity levels in drinking water samples.

The optimized LSC method is suitable to provide results of the gross radioactivity in the drinking water with the level of the trueness and precision similar to that obtained using other radioactivity screening techniques.

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Radionuclide extraction from aqueous solutions by Ionic Liquids

Gabriele Wallner, Raphlin Leyma, Sonja Platzer, Marzieh Habibi, Wolfgang Kandioller and Regina Krachler

Institut für Anorganische Chemie, Universität Wien, Währingerstr. 42, A-1090 Wien, Austria

Corresponding author’s e-mail: gabriele.wallner@univie.ac.at

Ionic liquids (ILs) are salts with a low melting point (below 100 °C) and they are composed of completely dissociated large ions. ILs have many advantages and they are of growing interest in the field of green chemistry. Some of the unique properties of the most widely studied ILs include the large liquid temperature range, high thermal stability, electrical conductivity, and tunable physical properties.[1] Especially the extraction of uranium by using ILs is of enduring interest in the literature as it may also be relevant to spent fuel reprocessing.[5,6]

The presented work is part of a project dealing with purification processes for drinking water. The aim of our work was the extraction of Uranium, Pb-210 and Po-210 from aqueous solutions with different ILs. Furthermore, a successful back-extraction of the radionuclides from the IL is desirable in order to be able to re-use the IL for several times. Investigations in our laboratory demonstrated that extraction of uranium from water is possible with [A336][TS][2,3], tricaprylmethylammonium thiosalicylate, as well as from maltol-based ILs, namely [A336][Mal] (tricaprylmethylammonium maltolate) and [C101][Mal] (tetradecyltrihexylphosphonium maltolate).[4] The ILs under investigation now are [A336][HNBA] and [A336][Ant] (trioctylmethylammonium 2-hydroxy-5-nitrobenzoate and anthranilate, respectively), as well as [PR4][HNBA] and [PR4][Ant] (trihexyltetradecylphosphonium 2-hydroxy-5-nitrobenzoate and anthranilate, respectively).

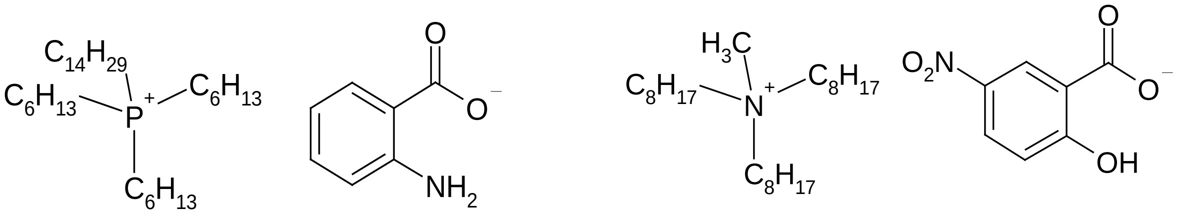

Figure 1: some of the ILs investigated: [PR4][Ant] and [A336][HNBA]

Investigations were performed with artificial water samples: uranyl nitrate solution with known uranium concentration was added to about 10 mL of distilled water, then NaOH was added in order to set a specific pH-value. The resulting uranium content is 27.7 µg $U_{nat.}$ (sum activity 683 mBq or 41 dpm for U-238 and U-234) in 10 ml corresponding to a uranium concentration of 2.8 mg/L. This solution is then mixed with the respective ILs. After centrifugation the phases were separated and an aliquot of the aqueous phase was analysed by liquid scintillation counting (LSC) to ensure that the uranium was removed. For Pb-210 and Po-210 the added activities to the aqueous phase was about 500 mBq, respectively. Afterwards 0.05M HNO_3 was used for back extraction of the radionuclides from the IL phase.

Generally, all investigated ILs showed a high extraction yield for uranium and for Po-210, while Pb-210 was only extracted in very low percentage rates. From the adsorption isotherms we learned that at least one of the investigated ILs was able to take up a rather high quantity of uranium.

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Applicability of the 3M Empore™ RAD disks for rapid determination of ^{90}Sr and ^{99}Tc by liquid scintillation counting

Vasile M[1], Loots H.[1], Verstrepen D.[1], Avci H.[1], Verrezen F.[1], Bruggeman M.[1]

[1] SCK•CEN, Boeretang 200, 2400, Mol, Belgium

Corresponding author's e-mail: mvasile@sckcen.be

In the framework of the 'Drinking Water Directive' that specifies maximum allowed concentrations of radioactivity in drinking water, the determination of certain artificial radionuclides may also be required. The quantification of these radionuclides should be made with an appropriate sensitivity. The sensitivity, expressed by the detection limits of the methods, are also specified by the EU Directive 2013/51/EURATOM. One of the anthropogenic radionuclides which has to be quantified in drinking water is ^{90}Sr, which is a beta emitter commonly analysed by liquid scintillation counting or with a gas-flow proportional counter after its radiochemical separation. Strontium-90 and ^{89}Sr, a beta-particle emitter as well, will be present in the environment due to nuclear accidents [1, 2].

We investigated the applicability of the commercially available 3M Empore™ Strontium RAD disk for ^{90}Sr separation and pre-concentration from drinking water as an alternative for the commonly used conventional separation procedures. After filtering of the water sample through the 47 cm 3M membrane disk, this is placed in a polyethylene scintillation vial with 20 mL Optiphase HiSafe III cocktail being ready for liquid scintillation counting. The measurements were done using a Quantulus™ 1220 liquid scintillation counter. The procedure used is described by 3M Empore™ in Test Method SR-95 [3]. For the total efficiency calibration, certified reference solutions traceable to the SI units were used. In the experiments one liter water samples spiked with ^{90}Sr were measured. Also the limitations of the procedure using 3M Empore Strontium RAD disk were studied by analysing different types of water: drinking water, waste waters, but sea water as well.

Based on the experience obtained from the study of Fons-Castells et al., 2016, the required impregnation time of the disk with cocktail was determined to be ~ 5.5 hours [4]. A total efficiency of (95.8± 4.1) % for ^{90}Sr was obtained when the molarity of the samples was 2M using HNO_3.

Samples acidified to pH < 2 were also tested and a small decrease in the total efficiency of the ^{90}Sr was noticed; a total efficiency of (92.7 ± 4.0) % was obtained.

Several studies revealed that along with strontium isotopes, radium and lead isotopes were also retained [5, 6, 7]. When only ^{210}Pb and ^{228}Ra (both are low energy beta emitters) are present in the sample than their interference can be eliminated by setting-up a measurement window for ^{90}Sr (beta-particle energy of 546 keV). The presence of ^{226}Ra (an alpha-particle emitter), is also a possible source of interference, especially when no alpha-beta discrimination is used, like in our case. The peaks of ^{226}Ra and its decay products will overlap with ^{90}Sr peak making imposible its quantification.

By applying the above described procedure for spiked, drinking waters and waste waters free of radium isotopes or ^{210}Pb, good results were obtained. The results of spiked sea water samples showed large deviations from the expected values due to the interference of the high concentrations of salts present in sea water.

In the paper the results obtained using the 3M Empore^{TM} Strontium and Technetium RAD disks are compared with the results obtained using Sr-spec and TEVA resins for ^{90}Sr and ^{99}Tc activity concentrations, respectively.

The advantages of the use of the 3M Empore^{TM} Strontium disk are that it is much faster than the routine procedures based on extraction chromatography columns, which take several days, it offers a simple sample preparation and produces less dangerous wastes. The disadvantages are the interferences from radium isotopes (which are present in natural waters in higher amounts than ^{90}Sr [5]) and the limited capacity for strontium uptake in case of waters with high amounts of salts.

We plan to investigate the possibility of using the 3M Empore^{TM} Strontium disk for solid environmental samples and also for rapid determination of ^{89}Sr and ^{90}Sr by liquid scintillation counting.

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The Belgian St-Adele fountain as a natural sample source for an interlaboratory comparison exercise on radon measurement in water

Doumont C.1), Di Pasquale S.1), Braekers D.1)

1) Institute for radioelements IRE Elit, 1 avenue de l'espérance, B-6220 Fleurus, Belgium

Corresponding author's e-mail: damien.braekers@ire-elit.eu

^{222}Rn is naturally present in underground and surface waters. It's a descendant of the natural ^{238}U and a key indicator of the potential present of radiotoxic isotopes like ^{210}Po or ^{210}Pb. Furthermore, the new European directive Euratom 2013/51 1aying down requirements for the protection of the health of the general public with regard to radioactive substances in water intended for human consumption set up a parametric reference level on the activity concentration of radon in water.

In this framework, the radon measurement in water becomes an important analysis method like the tritium and the gross alpha-beta measurements in a rapid screening strategy for monitoring of drinking water. The need of ^{222}Rn reference samples for the validation of the measurement technic by environmental laboratories is increasing.

A natural spring source located in the south of Belgium (St-Adele fountain) is used as a reference sample by our laboratory for 5 years as a quality control sample for the radon measurement in water. The method used is the liquid scintillation (LS) counting after extraction of radon and its daughter's products in the non-miscible water LS cocktail. The ^{222}Rn activity is determined by measuring the alpha activity of ^{222}Rn and of its daughter products (^{218}Po and ^{214}Po). The typical radon concentration of the source is fluctuating between 50 and 100 Bq/l depending both on air and water temperatures. Additionally, the complete analysis of the natural isotopes vector shows the presence of a small amount of natural uranium (~3µg/l). Since 2014 our laboratory is yearly organizing its own interlaboratory comparison exercise.

In 2016, six different laboratories participate in this exercise using their own sampling procedure and measurement method (liquid scintillation counting, gamma spectrometry and emanation Lucas method). The sampling was performed with a syringe in the case of LS technic and by filling different types of container for the other methods.

The results of participants are in good agreement. However an influence of the sampling methodology with two clear sets of results is evidenced.

The variation of radon activity concentration over the sampling period (~2h) has also been evaluated (see Fig. 1). The stability is acceptable and the fluctuation is below 3%.

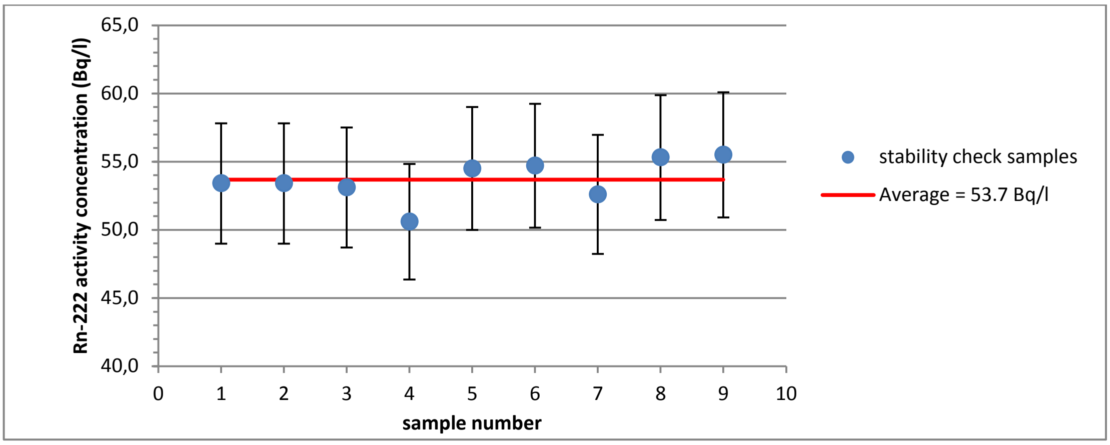

Figure 1: stability test of the radon activity concentration during the sampling

Comparison of two methods for C-14 analysis from essential oils using LSC

I. Vagner [1)], C. Varlam [1)], I. Faurescu [1)], D. Faurescu [1)], F. Bucura [1)]

[1)] National R&D Institute for Cryogenics and Isotopic Technologies – ICSI Rm. Valcea, 240050, 4 Uzinei, Romania

Corresponding author's e-mail: irina.vagner@icsi.ro

The paper describes the use of two preparation methods, direct method and CO_2 absorption method, to report ^{14}C results for two essential oils obtained from plants (basil and lavender).

Direct method presumed mixing of the samples directly with the scintillation cocktail in a ratio of 8:12 (g:ml). The CO_2 absorption method presumed the combustion of the samples and bubbling of the CO_2, after the proper purification, in a home-made scintillation cocktail.

In order to report and to compare the results obtained on ^{14}C analysis applying the two preparation method (Bq/ g of sample with Bq/ g of carbon) we determined the elemental composition of the samples to be analyzed. The obtained results showed differences of around 10% in ^{14}C activity expressed in Bq/g of carbon and around 3% to the obtained uncertainty. Problems that may occur on the conversion of the ^{14}C activity measured using the two methods are the elemental analysis of the samples and their uncertainties. Verification of these preparation methods to eliminate elemental analysis on conversion of the ^{14}C activity obtained was done using ethanol, knowing its chemical formula and implicitly its carbon content.

The two studied methods were used to investigate the uncertainty of the sample preparation procedure for the CO_2 absorption method.

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Measurement of radiocarbon and tritium in a single sample: simultaneous and radiochemistry approaches

Arūnas Gudelis, Lina Gaigalaitė, Rasa Gvozdaitė, Inga Gorina

Center for Physical Sciences and Technology (FTMC), Savanorių Ave. 231, Vilnius, LT-02300, Lithuania

Corresponding author's e-mail: arunas.gudelis@ftmc.lt

Aqueous samples from the nuclear installation (radioactive waste repository) were measured by the liquid scintillation counting method. Groundwater samples were gathered from the monitoring wells installed at different directions and distances from the vault [*Gudelis et al., 2009*]. Atmospheric humidity was collected with the aid of samplers designed for monitoring tritium and radiocarbon emissions from the nuclear power plants. Ultima Gold LLT cocktail was used for sample preparation at two sample-to-cocktail ratios: 4 mL of water to 16 mL of scintillator or 10 mL of water to 10 mL of scintillator. LSC spectra revealed presence of radiocarbon and tritium at various activities. In Fig. 1, one can see similar activities of both radionuclides or prevailing tritium concentration (red and green spectra, respectively). Double-label approach has been used to quantify ^{14}C and ^{3}H from simultaneous measurement [*L'Annunziata, 2004*].

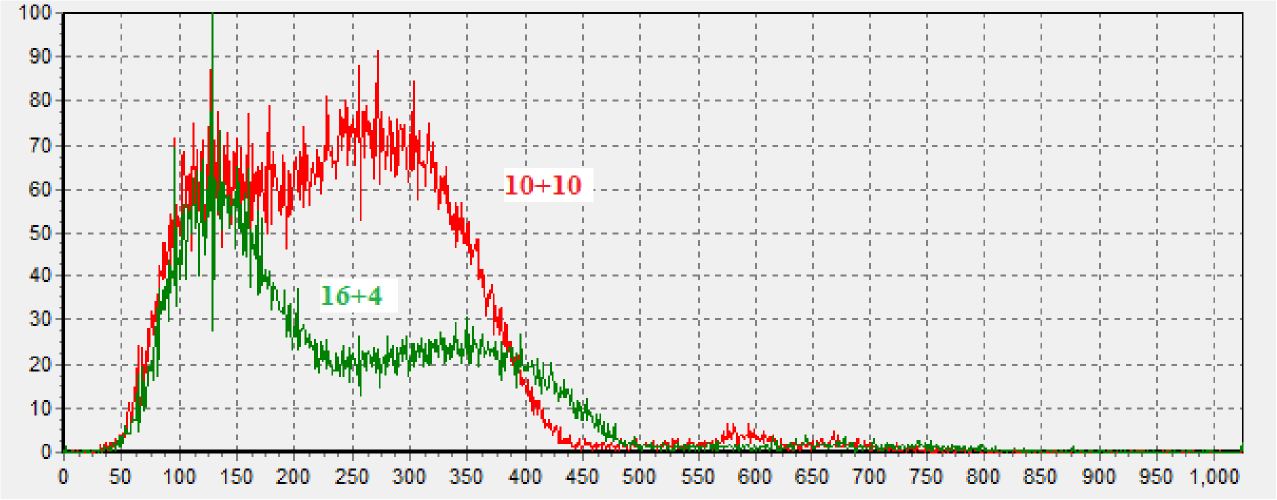

Fig. 1 Radiocarbon and tritium spectra in aqueous samples obtained at different sample-to-cocktail ratio

Reference materials traceable to the National metrology institute (FTMC) were used for calibration. Radiochemistry methods were also applied: wet acidifying procedure for the determination of ^{14}C at low activities of radiocarbon and Tritium colums (Eichrom Technologies) for the separation of tritium in the samples with a presence of ^{14}C at higher levels.

Results were used within the Project No. MIP-050/2011 with the Lithuanian Science Council for assessing radiocarbon and tritium transfer from the near-surface radioactive waste repository to the both groundwater and ground-level atmosphere.

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Determination of ^{226}Ra in natural water samples by liquid scintillation counting

Szabolcs Osváth [1] [2], Péter Rell [1], Júlia Kövendiné Kónyi [1], Gyula Szabó [1]

[1] National Public Health Center, National Research Directorate for Radiobiology and Radiohygiene, Budapest, Anna utca 5., 1221, Hungary
[2] Current workplace: Technical University of Denmark, Center for Nuclear Technologies (DTU Nutech), Roskilde, DTU Risø Campus, Frederiksborgvej 399, 4000, Denmark

Corresponding author's e-mail: osvath.szabolcs@osski.hu

Determination of ^{226}Ra in drinking waters, natural waters and other environmental samples is an important task due to its high radio-toxicity. A relatively fast, simple and reliable method has been developed for determination of ^{226}Ra from natural water samples, using radiochemical separation and liquid scintillation counting (LSC). This method is based on using ^{133}Ba as tracer, sorption on MnO_2 Resin® and precipitation of $BaSO_4$. $BaSO_4$ was converted into $BaCO_3$, solubilized, and then measured by LSC.

0.5-1.5 L sample was taken, filtered through a 0.45 µm filter and pH was adjusted to 4-7 using 1 M HNO_3 or 1 M NaOH. After that 4 Bq ^{133}Ba tracer and 1.25 g MnO_2 Resin® were added. The sample was stirred for at least 1 hour, and the resin was allowed to settle (preferably for a night). The supernatant was decanted, the resin was centrifuged and the supernatant was discarded. Ba and Ra were eluted from the resin using 12 mL 6 M HCl, 1 drop 30% H_2O_2 was added to make the solution colorless, then it was filtered through a 0.45 µm filter. 100 µL 1:1 acetic acid was added to make complex with Pb^{2+}. 19 mg $Ba(NO_3)_2$ and 5 mL 40% (saturated) Na_2SO_4-solution were added. The sample was slightly warmed and stirred for 30 minutes to form $BaSO_4$ precipitate, then it was centrifuged (3000 rpm, 5 min) and the supernatant was discarded. The precipitate was suspended in 2 mL saturated K_2CO_3 solution and it was evaporated with 3x2 mL distilled water to dryness to convert $BaSO_4$ to $BaCO_3$. 30-40 mL distilled water was added, and the slurry was centrifuged. This step was repeated until the pH of the supernatant reached 7 (typically 3 times). The $BaCO_3$ precipitate was dissolved in 1 mL 0.5 M HNO_3, 1 drop 30% H_2O_2 was added to make it colorless. The solution was transferred into a 20 mL plastic LSC vial, 16 mL Ultima Gold LLT cocktail was added and LSC measurement was performed using a Tri-Carb 2900TR (Packard, USA) equipment.

Focal point of this method is that the same LSC measurement was used for determination of activity of ^{133}Ba tracer as well as that of ^{226}Ra analyte (see Figure 1), as proposed by Tinker at al. [1]. As of pulse decay discriminator setting, 140 ns discrimination time was used to reduce the spillover of ^{133}Ba counts into the α-counting region. Activity and recovery of ^{133}Ba were calculated on the basis of the β-LSC spectrum. Recovery of ^{226}Ra was assumed to be the same as that of ^{133}Ba. Activity of ^{226}Ra was calculated using channels 90-500 of the α-LSC spectrum, after subtraction of spillover of ^{133}Ba counts into the α-counting region. In the region mentioned above, peaks of all α-emitter progenies of ^{226}Ra (namely ^{222}Rn, ^{218}Po and ^{214}Po) are present. Their in-growth was taken into account using a calibration curve based on the results and equations described by Fons et al. [2]:

$$A_{sample}^{\alpha}(t) = \left\{1 + 3 \cdot \left[1 - exp\left(-\frac{\ln(2) \cdot t}{91.68\ hours}\right)\right]\right\} \cdot A_{sample}(t = 0)$$

where $A_{sample}(t = 0)$ is the ^{226}Ra-activity of sample at separation, $A_{sample}^{\alpha}(t)$ is the α activity of sample at t hours after separation and 91.68 hours is half-life of ^{222}Rn.

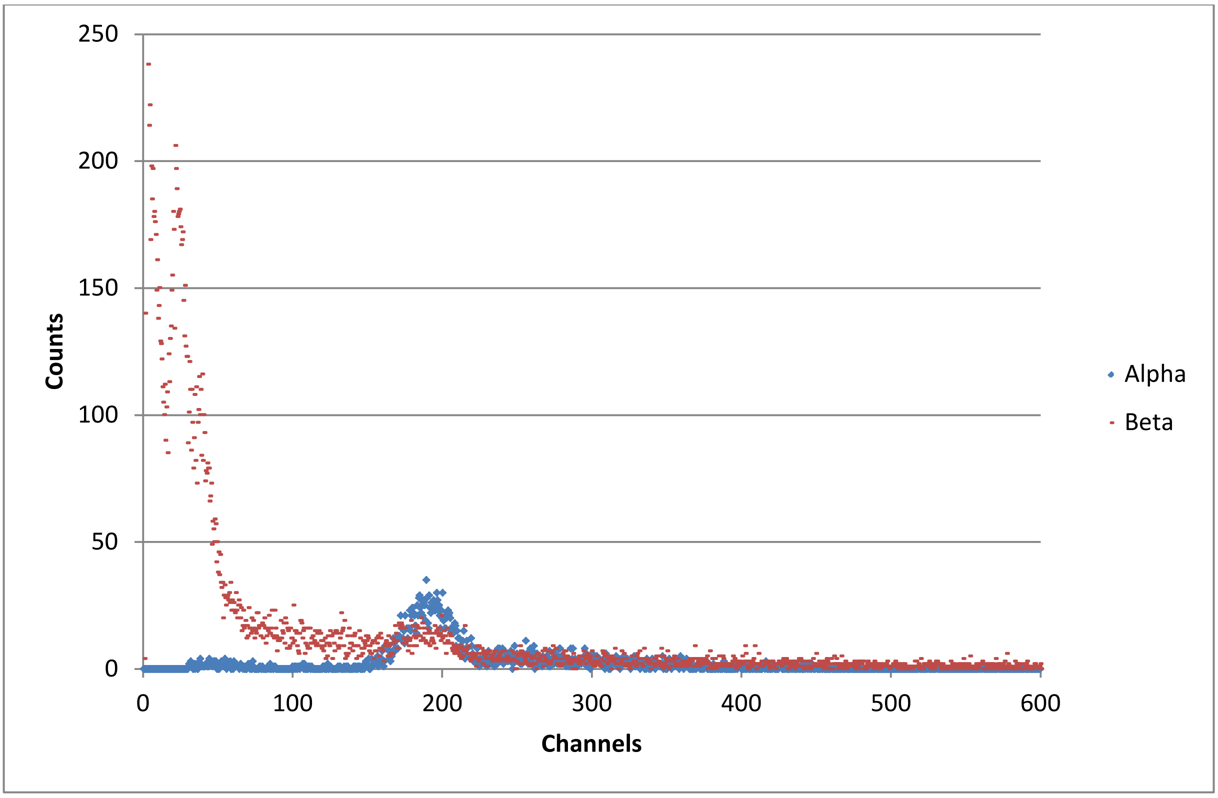

Figure 1 LSC spectrum of ^{226}Ra and ^{133}Ba. The peak in the α-spectrum corresponds to ^{226}Ra (4784 keV). The β-spectrum consists of counts from ^{133}Ba tracer and spillover of α counts of ^{226}Ra. As these spectra were acquired right after separation, progenies of ^{226}Ra are not present.

Some results of 38 analyzed water samples (bottled mineral waters, medicinal thermal waters and natural surface waters) can be seen in Table 1. Our experiments have showed that the average chemical recovery was 60(±15)%, and we could not find correlation between recovery and sample composition (alkaline earth content).

According to 2.2 cpm background between channels 90-500 of the α-LSC spectrum and applying 100 min detection time, typically 10 mBq of minimal detectable activity (MDA) was achieved. Therefore, analyzing a sample of 0.5-1.5 L, the limit of detection described in the Council Directive 2013/51/Euratom [3] (namely 40 mBq/L) is easily achievable. Typical relative uncertainty of results (in cases they were over MDA) was 8%, calculated using a coverage factor of 1 (k=1).

Table 1 Activity concentration of ^{226}Ra (mBq L^{-1}) in some popular Hungarian (and Rumanian) bottled mineral waters. The uncertainty is given using a coverage factor of 1 (*k*=1).

Brand name	Measured value
Balfi	< 10
Borsec [sparkling; Romania]	343±38
Borsec [still; Romania]	41±9
Fonyódi	< 10
Kereki Theodora	< 10
Kékkúti Theodora	< 10
Mizse	< 10
Natur Aqua	< 10
Óbudai Gyémánt	100±9
Szentkirályi	< 10
Visegrádi	159±14

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Radon-222 Radioactivity in Groundwater

Woosik Shin1), Sungwook Choung1), Byungwook Cho3), and Hyun Koo Kim4)

1) Korea Basic Science Institute (KBSI), Cheongju, 28119, Republic of Korea
2) Korea Institute of Geosciences and Mineral Resources (KIGAM), Daejeon, 34132, Republic of Korea
3) National Institute Environmental Research (NIER), Incheon, 22689, Republic of Korea

Corresponding author's e-mail: schoung@kbsi.re.kr

Radon (^{222}Rn) is an alpha-emitting radionuclide that is naturally derived from successive decay of uranium-238. The radon has been known to induce lung or stomach cancer due to internal radiation via inhalation or ingestion. Therefore, the International Agency for Research on Cancer (IARC) classified radon-222 as a carcinogen of Group 1 and many authorities of foreign countries such as US EPA, Denmark, Sweden, Norway and Finland regulate radon level between 2700 to 27000 pCi L^{-1} in drinking water for the decrease of radon exposure. In the case of Korea, there are no established standards for radon in drinking water. In particular, since the supply ratio of tap water in the rural area of Korea is relatively lower than the urban area, the dependence of groundwater as drinking water is quite high. This study was carried to provide the scientific background information for the establishment of radon standard for drinking water in Korea. The radon level in groundwater were investigated nationwide for 9 years (2006–2014). Samples were collected from 2143 public and private wells of the rural area, and analyzed by Liquid Scintillation Counter (LSC). The radon level in groundwater is up to 195000 pCi L^{-1} with an arithmetic mean of 2600 pCi L^{-1}. Compared to AMCL guideline (4,000 pCi L^{-1}) of U.S., 17 % of samples were exceeding the AMCL guideline for radon in drinking water, and the boreholes were distributed mainly into the western region of Korea. The estimated population who is potentially at risk of radon exposure through drinking groundwater was approximately 800000 resindents in 106 towns.

^{90}Sr in surface air of Rovaniemi (Finnish Lapland) in 1965-2011

Susanna Salminen-Paatero 1), Jussi Paatero 2)

1. Department of Chemistry, Radiochemistry, P.O. Box 55, FI-00014 University of Helsinki, Finland
2. Finnish Meteorological Institute, Observation Services, P.O. Box 503, FI-00101 Helsinki, Finland

Corresponding author's e-mail: susanna.salminen-paatero@helsinki.fi

A long-term time series (1965-2011) of air filters was studied, to observe the effect of different anthropogenic radionuclide contamination sources on Finnish Arctic environment. Weekly air filter samples collected in Rovaniemi (66°30′N, 25°44′E) were combined to contain collected aerosol particles from three months to five years, depending on the sampling year and assumed activity concentration level in the air filters.

First, the activity concentration of ^{137}Cs was determined from the sample sets by gamma spectrometry. Then the filters were ashed and wet-ashed with conc. HCl and HNO_3, with volume ratios of 1:3, respectively. From the digested samples, radionuclides ^{90}Sr, $^{238+239+240}$Pu, and ^{241}Am were separated with anion exchange and extraction chromatography, by TRU®, and Sr® resins (TrisKem International). The activity concentrations of Pu and Am isotopes were determined by alpha spectrometry.

After elution from Sr® resin column, the solutions of 0.05 M HNO_3 containing ^{90}Sr were set aside for 3 weeks. After that time, ^{90}Y/^{90}Sr was in radioactive equilibrium in the samples, and the activity concentration of ^{90}Sr in eluates was determined with Quantulus 1220 in the Cherenkov counting mode. The counting efficiency of Quantulus 1220 for ^{90}Sr was determined to be 65%. Radiochemical yield of ^{90}Sr was determined with ICP-OES, being 41-98%.

The activity concentration of ^{90}Sr in surface air of Rovaniemi varied from < 0.02 µBq/m^3 (in 2001-2005) to 145±30 µBq/m^3 (in 1965), being highest in 1965 (Fig. 1). Since then, the atmospheric concentration of ^{90}Sr has been constantly decreasing in Rovaniemi. The activity concentration values of Rovaniemi are in agreement with worldwide values since 1965 (UNSCEAR 1966, de Bortoli et al. 1966). Most of ^{90}Sr from atmospheric weapons testing performed before partial test ban treaty in 1963 has been scavenged from surface air after the deposition maximum in 1963, in other words, before the start of sampling at Rovaniemi in 1965. Therefore, the highest activity concentration values in 1963 are not comparable with values of later years. After summer 1986, the activity concentration of ^{90}Sr was below the detection limit in all the samples. It can be concluded from the decreasing trend of ^{90}Sr activity concentration in the surface air of Rovaniemi, that the origin of ^{90}Sr in Lapland has been mainly global fallout from nuclear weapons testing in 1950s and early 1960s. Only marginal effects of the Chernobyl accident or atmospheric nuclear tests between 1964 and 1980 can be seen.

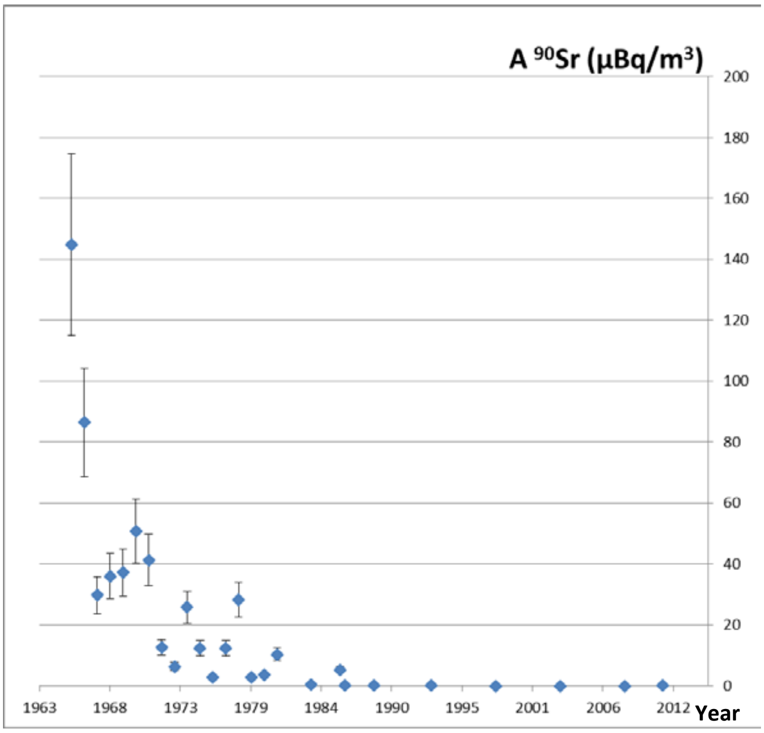

Fig. 1 The activity concentration of ^{90}Sr during 1965-2011 in surface air of Rovaniemi (µBq/m^3). The activity concentrations fell below MDA since summer 1986, and the value “half of MDA” has been used for those samples as activity concentration.

Acknowledgements:

This work was part of the project "Collaboration Network on EuroArctic Environmental Radiation Protection and Research (CEEPRA)". The project was financed by EU Kolarctic ENPI CBC 2007-2013 programme managed by the Regional Council of Lapland.

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The role of LSC methods in implementation of the Euratom Directive 51/2013 for the monitoring of radioactivity of water supplies in Poland

Stanislaw Chalupnik, Izabela Chmielewska

Silesian Centre for Environmental Radioactivity, Central Mining Institute, Katowice, Poland

E-mail: schalupnik@gig.eu

In October 2013, the European Commission issued the Directive Euratom/51/2013, related to the monitoring of radioactivity of drinking water supplies. In Poland this Directive has been introduced into the national law system in November 2015 as the Ordinance of the Ministry of Health. In the implementation of the Ordinance into practice the main role play methods based on LS spectrometry. This robust technique can be applied for monitoring of the majority of the radionuclides, required by the Ordinance, it means tritium, radon and concentration of radium isotopes in water.

In this paper the description of the Polish approach to the Euroatom Directive implementation is given as well as brief description of the LSC methods, used in the monitoring system of water supplies, provided by Silesian Centre for Environmental Radioactivity.

Automated dispersive liquid–liquid microextraction of ^{99}Tc from biological samples and hospital residues previous liquid scintillation counting

Marina Villar 1), Antoni Borràs 2), Fernando Vega 1), Víctor Cerdà 3), Laura Ferrer 2)

1) Radiopharmacy Service, Son Espases Hospital, Cra. Valldemossa 79, 07120 Palma de Mallorca, Spain.
2) Laboratory of Environmental Radioactivity-LaboRA, University of the Balearic Islands, Cra. Valldemossa km 7.5, 07122 Palma de Mallorca, Spain.
3) Department of Chemistry, Faculty of Science, University of the Balearic Islands, Cra. Valldemossa km 7.5, 07122 Palma de Mallorca, Spain

Corresponding author's e-mail: laura.ferrer@uib.es

An automated system exploiting in-syringe dispersive liquid–liquid microextraction (DLLME) for ^{99}Tc extraction and preconcentration from biological samples, i.e., urine and saliva, and hospital residues is presented. The detection was performed using a liquid scintillation counter Tricarb 2810 TR (Perkin Elmer). A window comprising 1–600 channels was used for counting. The counting time was 1800 s in normal mode, achieving a counting efficiency of 95.2% determined from a known ^{99}Tc activity. Given that the proposed method provides constant quenched samples, quenched curves were not required.

^{99}Tc is a beta emitter with a long half-life (2.111×10^5 years) and a high mobility in the different environmental compartments. One of the sources of this radionuclide is by its father ^{99m}Tc in medical diagnosis.

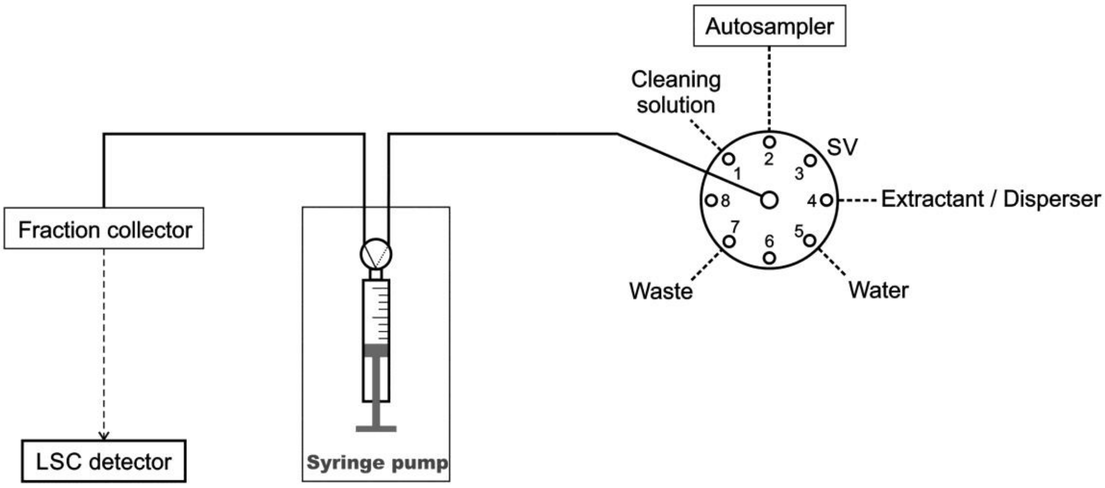

Fig. 1 Schematic depiction of the in-syringe dispersive liquid–liquid micro-extraction system for ^{99}Tc determination. HC: Holding Coil; LSC: Liquid Scintillation Counter; SV: Selection Valve.

A critical comparison between extractants and disperser solvents for ^{99}Tc DLLME is presented, e.g., tributyl phosphate (TBP), trioctylmethylammonium chloride (Aliquat®336), triisooctylamine (TiOA), as extractants in apolar solvents such as xylene and dodecane, and disperser solvents such as acetone, acetonitrile, ethanol, methanol, 1-propanol, and 2-propanol. The system was optimized by experimental design, and 22.5% of Aliquat®336 in acetone was selected as extractant and disperser, respectively. H_2O_2 was added to hospital residues to ensure that all Tc was totally oxidized to Tc(VII).

The present method has a ^{99}Tc minimum detectable activity (MDA) of 0.075 Bq with a high extraction/preconcentration frequency (8 h^{-1}). Moreover, volumes of organic solvents and sample are drastically reduced, and it has several benefits such as miniaturization, simplicity, low cost per analysis, high recovery, precision, and enrichment factor. Urine, saliva, and hospital residues were satisfactorily analyzed with recoveries of 82–119 %. Therefore, this system is proposed as a useful tool for monitoring the ^{99}Tc generated in hospitals from biological and residue samples.

Figures of merit	Value
Minimum Detectable Activity (Bq)	0.075
Intra-day precision (%) (n=10)	4.1
Inter-day precision (%) (n=5)	6.9
Extraction frequency (h^{-1})	8
Enrichment factor	5
Detection efficiency (%)	95.2
Extraction efficiency (%)	84

Table 1. Most relevant figures of merit of the developed system.

Radiobioassay method for the determination of isotopes from fuel particles

Raymond Ko (1), Dominique Audette (2), Chunsheng Li (3)

(1) Radiation Protection Bureau – Health Canada, 775 Brookfield Rd, K1A1C1, Canada
(2) University of Waterloo, 200 University Avenue West, N2L 3G1, Canada
(3) Radiation Protection Bureau – Health Canada, 775 Brookfield Rd, K1A1C1, Canada

Corresponding author's e-mail: raymond.ko@hc-sc.gc.ca

During a radiological-nuclear (RN) emergency, workers and civilians face dangers of contamination by radionuclides through inhalation, ingestion, or wounding. In the case of inhalation, aerosolized chelating agents can be used to accelerate the absorption of moderately soluble particles that have deposited in the lung, and increasing the overall rate of clearance from the body.

One of the research projects at the Radiation Health Assessment Division at Health Canada is the development of different inhalable chelating agents and evaluating their efficacy on rats which have been exposed to active fuel particles. To determine the effect of the chelating agents on each of the isotopes in the fuel particle (47% 137Cs, 30% 241Pu, 20% 90Sr, < 2% 154Eu, < 2% total alphas, < 2% other) in a high background of uranium, a sequential column based separation method based on extraction chromatography was developed and validated. The measurement of the beta emitters was done using liquid scintillation counting.

Homogeneity Assessment for Grass Items from Organically Bound Tritium Proficiency Test

Carmen Varlam 1), Cristina Bucur 2), Irina Vagner 1), Marius Constantinescu 1), Diana Bogdan 1), Ionut Faurescu 1)

1) Tritium Laboratory, Institute for Cryogenics and Isotopic Technologies ICSI, Rm. Valcea, Valcea, 240050, Romania

2) Health Physics Department, Cernavoda Nuclear Power Plant, Cernavoda, Constanta, 905200, Romania

Corresponding author's e-mail: Carmen.Varlam@icsi.ro

Starting with "1st workshop on Organically Bound Tritium (OBT) and its analysis" held in Balaruc les Bains, France in 2012, an international working group was established. Within the last 4 years, several inter-laboratory exercises were organized to improve our understanding of OBT behavior and the overall analytical performance.

The 4th OBT exercise organized by Environmental Laboratory of Nuclear Power Plant Cernavoda is in progress. The sample chosen for this proficiency test is grass. This was sampled, dried, and homogenised before to be dispatch by the organiser. Due to the different methods used by the participants to the exercise and, of course, due to ISO 13528* requirements, the homogeneity and stability of the proficiency test items are of primary interest for the quality and success of the exercise.

The paper presents homogeneity check for grass sample performed for hydrogen content and OBT analysis (Bq/l combustion water). 10 package of proficiency test items were chosen using a stratified random selection. 2 replicates from each package were analysed for Hydrogen content and OBT analysis. The homogeneity parameters were evaluated according to ISO 13 528. The two properties checked confirm that homogeneities is sufficient for the batch prepared for 4th OBT exercise.

References:

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Tritium in environment around NPP Krško

Romana Krištof[1)], Suzana Košenina[2)], Benjamin Zorko[1)], Jasmina Kožar Logar[1)]

[1)] Jožef Stefan Institute, Ljubljana, Slovenia
[2)] University of Ljubljana, Faculty of Health Studies, Ljubljana, Slovenia

Corresponding author: romana.kristof@ijs.si

Pressurized water reactor (PWR) is a heart of Krško Nuclear Power Plant (KNPP) positioned in south-eastern part of Slovenia. Environment of KNPP has been monitored for decades and tritium results show slightly elevated concentrations in regard to natural background in Sava River, precipitations and some bore hole water in the close vicinity of the nuclear facility. However, the measured values in all types of water are far below the permitted values of 100 Bq/L for drinking water in directives 98/83/ES and CD 013/51/Euratom. In very near future, the environment of KNPP will be changed because of the dam for hydroelectric power plant Brežice few kilometers downstream the Sava River. The water surface will increase and the river flow will be slowed down, so the change of tritium concentration in air, water, food and feed might be expected.

Analyses of tissue free water (TFW) as well as organically bounded tritium (OBT) are not the part of regular national monitoring program. The method for their determination in food, feed and soil has been introduced into the Laboratory for liquid scintillation spectrometry on Department for Low and Medium Energy Physics just recently. One of the main purposes was the documentation of possible changes in the environment of KNPP after changed hydrological circumstances. The presented values from the vicinity of KNPP and from Alpine region without any influence of nuclear facility are first measurement results of TFW in Slovenia. They represent the reference values for the natural background and background in the vicinity of KNPP. The TFW tritium concentrations in spring and fall samples will be evaluated also through correlations with tritium measurements in different types of water such as Sava River, water from gravel pits and precipitations.

Tritium content in tissue free water (TFWT) in African coconuts

Giscard Honoré Sonkwa Monthe[1)], Romana Krištof [2)], Jasmina Kožar Logar [2)]

[1)] University of Yaoundé I, Faculty of Science, P.O. Box 812, Yaoundé, Cameroon
[2)] Jožef Stefan Institute, Jamova 39, SI-1000, Slovenia

Corresponding author's e-mail: jasmina.logar©ijs.si

Coconut is important nutrition source for most people under the tropical and subtropical belt all over the World. The fruit becomes very popular also in so called western cultures because of its richness of vitamins, minerals and bioactive compounds that are essential for better health.

The paper of Narayan [1] attracted the attention with the thesis that the coconut fruit pre-concentrates the tritium in respect to tritium concentration in environmental water such as irrigation water and precipitation. Ripe coconuts from Cameroon, Ivory Coast and Equatorial Guinea were collected with the attempt to provide first tritium data also for African fruits. Cameroon's fruits were from the towns of Limbe, Mamfe and Mbonge. The nuts from Guinea were found at the market of Kribi while samples of Ivorian nuts were obtained at supermarkets in Giessen, Germany and Kranj, Slovenia.

Ripe coconut water was simply distilled and directly measured by Quantulus 1220 while sample preparation for Tissue Free Water Tritium (TFWT) determination in water from kernel milk was conducted on two different ways. Simple procedure was developed with the intention to be feasible also for majority of African laboratories. Grated coconut kernel was heated in sand bath. Evaporated water droplets were collected and distilled. The procedure was tested and validated by one of the usual methods in TFWT/OBT sample preparation, e.g. azeotrope distillation with toluene.

Preliminary results show consistent values for different coconuts from the same sampling points. Tritium values vary among different locations. Tritium content is always higher for kernel milk water than for ripe coconut water.

The comparison of sample preparation methods will be presented and commented. TFWT values for coconut water and milk from all mentioned locations will be evaluated and compared with results for Indian coconuts.

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Tritium internal dose estimation with liquid scintillators

A. Pántya[1], A. Andrasi[1], T. Pázmándi[1], T. Szarvas[1], G. Taba[2],

[1]Hungarian Academy of Sciences, Centre for Energy Research, H-1121, Budapest, 29-33 Konkoly Thege Miklós street, Hungary

[2]Semmelweis University, Department of Radiation Protection, H-1085, Budapest, 26 Üllői street, Hungary

The measurement of tritium in environmental and biological (urine) samples plays an important role in assessment of internal exposure. Tritium normally exists as tritiated water vapour (both in itself and adhered to aerosol particles) in the atmosphere of workplaces like the Budapest Research Reactor or the high level radioisotope laboratory of Isotope Institute Ltd. Budapest. This tritium can get into the body of workers by inhalation and also by absorption through skin. Since the inhaled tritium after the uptake is spreading over the body, the whole body is exposed to its radiation. For internal dose assessment it is necessary to consider in urine sampling the time after the intake, when the tritium concentration inside the body has already reached equilibrium. For obtaining the best performance comparison was carried out using two available liquid scintillation equipments (Packard 2200CA and Tri-Carb 4910) highlighting the uncertainty of measurements. The results were used for dose estimation with MONDAL-3 and IMBA codes. The uncertainty of the data acquisition system was implemented into the overall uncertainty analysis considering the following factors: time of intake, absorption type, AMAD (Activity Median Aerodynamic Diameter), etc. The final conclusions will be presented in our presentation.

Study on optimization of liquid scintillation counting during tritium measurements

Sung Jin Noh[1)], Hyojin Kim[1)], Hyun Kim[1)], Dong Hyeok Jeong[1)], Yeong-Rok Kang[1)*]

[1)] Research Center, Dongnam Inst. of Radiological & Medical Sciences, Busan 619-953, Republic of Korea

Corresponding author's e-mail: yeongrok@dirams.re.kr

The liquid scintillation counter (LSC) is widely used worldwide for the purpose of detecting tritium, which is a radionuclide emitting low-energy beta rays. There ara many studies that deal with the optimization of tritium analysis concerning the sample/coctail ratio, quenching, sample stability, and other parameters that can interfere with counting conditions [1-5]. The purpose of this work is the optimization of the counting performance for tritium determinations in water sample by direct counting, using the low background liquid scintillation system Quantulus 1220. To count the activity concentration in environmental samples using an LSC, the correlation between the quenching level and the counting efficiency must be analyzed and evaluated first. To determine the correlation between the activity concentration and the quenching, the external standard method was used in this study for quench correction. This method can be the most reliable when very accurate DPM values are required because users can control all aspects of the preparation of the quenched standards in order to represent the chemistry of their experimental samples most closely. A detailed description of the procedure for the preparation of quenched standards and a quench correction curve from the quenched standards is provided in [6]. For the standard source for quenching, glass-, plastic-, and teflon-vial-type series sets were prepared and used. For the glass vial type, the H-3 Ultima Gold Low Level Quenched Standard Set (assay value of 94,110 dpm/std ± 1.6%) supplied by PerkinElmer was used; and for the plastic and teflon vial types, 10 samples were prepared at a constant rate using a standard tritium sample (H-3 in MQ water: using 509.9 Bq/g 1.6%). For the three types of standard sources for quenching, the same liquid scintillation cocktail (Ultima Gold LLT) and 20 m*l* vial were used. In addition, the parameters for these three types of standard sources were set (Parameter Group 1: glass-vial-type standard source for quenching, Parameter Group 2: plastic-vial-type standard source for quenching, and Parameter Group 3: teflon-vial-type standard source for quenching) using the WinQ program. Then, counting was repeated 10 times for 5 min for each sample set to obtain the counts per minute, CPM, and SQP(E). The quenching correction curve was created on the basis of the counting and analysis results; then, the activity concentration calculated by the quenching correction curve and the known values were analyzed comparatively to determine the accuracy [Fig. 1]. To evaluate the properties according to the differences in the standard sources for quenching and to evaluate the tritium concentration for a rainwater sample in the external standard method, nine samples (three each for each vial type) were obtained through a pretreatment process, and these samples were counted 10 times (30 min each time) by applying the parameters for each type saved in the WinQ program. To identify the correlation between the quenching coefficient of the standard source for quenching and the counting efficiency for the purpose of performing the efficiency calibration of an LSC, a series of standard sources for quenching with different degrees of quenching prepared using tritium standard samples was counted.

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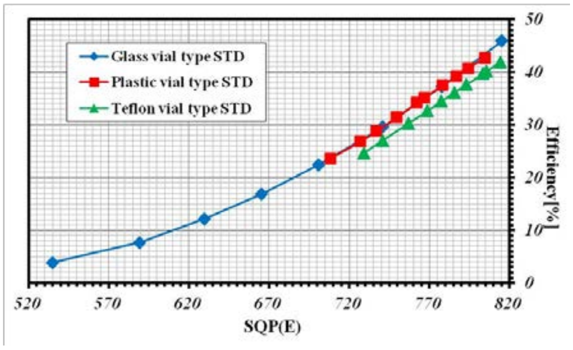

Fig. 1 set of quenching correction curves measured in different source for quenching.

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Determination of very low level tritium volume activities in surface water and precipitation in the Czech Republic

Diana Marešová, Eduard Hanslík

T. G. Masaryk Water Research Institute, p.r.i., Podbabská 2582/30, 160 00 Prague, Czech Republic

Corresponding author's e-mail: diana_maresova@vuv.cz

TGM WRI, p.r.i. has been involved in research of very low level tritium volume activities in water for a substantial amount of time. This contribution summarizes the results of tritium monitoring in surface water and precipitation in the Czech Republic since 1990. The samples have been pretreated by electrolytic enrichment from 2010.

Tritium volume activity was monitored in surface water and precipitation at sites not influenced by discharges from a nuclear plant. The frequency of sampling was once per month. Precipitation samples were accumulated monthly. The samples were distilled. The measurements were carried out with low background liquid scintillation spectrometer Quantulus 1220 according EN ISO 9698. The relative efficiency was about 26%. A mixture of 8 mL of the sample and 12 mL of ULTIMA GOLD LLT scintillator was measured for 800 min (for samples without electrolytic enrichment until 2010) and for 300 min (for electrolytically enriched samples). The minimum detectable activity at a significance level of 95% was 1.0 and 0.1 Bq/L respectively. Certified material (Czech Metrology Institute) was used for calibration. The initial sample volume was 500 mL for electrolytic enrichment. A sample with added of Na_2O_2 was electrolysed to a volume of 20 – 25 mL. Subsequently, the sample was neutralized with $PbCl_2$ and distilled. A set for electrolytic enrichment allows the simultaneous adjustment of 20 samples; the total elapsed flow is 1400 Ah.

The long-term development of tritium volume activity in surface water and precipitation was evaluated for 1990-2015 and for 1993-2016. The effective half-life time (T_{eff}) was determined to be 16.0 y (Fig. 1), which was longer than the physical half-life (12.32 y [1]). The reason for the longer half-life is the constant component (contribution by cosmic rays and contribution from nuclear facilities considered constant for the assessed period). The constant component contributes significantly to relatively low tritium volume activities. For these reasons, the annual average tritium volume activities were corrected for natural component generated by cosmic rays (c_{3HCR}) and for the estimated contribution from nuclear facilities worldwide via atmosphere (c_{3HNF}). T_{eff}=10.6 y was calculated for kinetics of tritium volume activity reduction, corrected for the natural component and the contribution from nuclear facilities (i.e. for residual contamination from atmospheric testing of nuclear weapons). The obtained T_{eff} was shorter than the one for development of uncorrected tritium volume activities. This component of tritium contamination will further decrease. Moreover, one can assume that the value of the effective half-life will increase. Similarly, the tritium volume activity was determined in precipitation. The obtained effective half-life of tritium was 25.9 y in precipitation for 1993-2015; 16.9 y after correction for the natural component and the component of distant transfer. The obtained half-life was longer that the physical half-life of tritium. It is obvious that the tritium component generated by natural processes begins to predominate over the component of tritium from nuclear bomb testing. Tritium volume activity was evaluated for 2010-2016 in detail (the electrolytically enriched samples). The characteristic seasonality of tritium in precipitation was observed. Consequently, the weighted averages of tritium were processed for all seasons (Fig. 2). The determined average values were maximal for spring and summer, and minimal for winter. This is a natural course of precipitation described in literature [2].

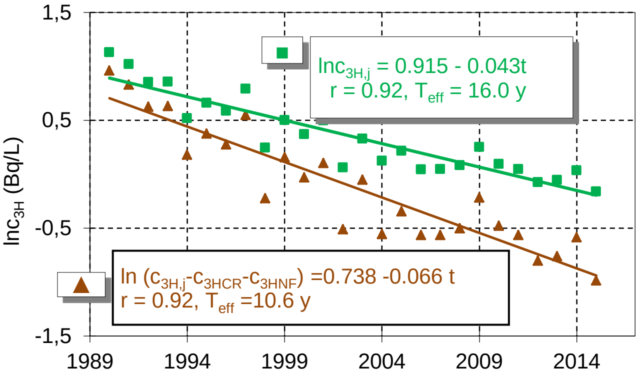

Fig. 1 The development of tritium volume activity in surface water (Elbe river basin) unaffected by discharge from nuclear power plants in the period 1990-2015.

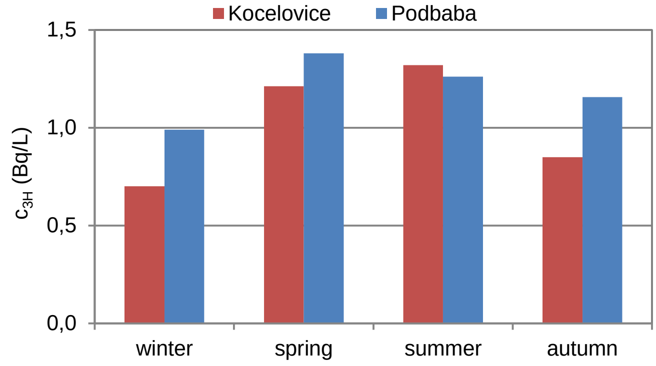

Fig. 2 Seasonality of tritium volume activity in precipitation at selected monitoring sites.

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Optimization of liquid scintillation counting conditions for tritium determination in sea water adjacent to Nuclear Power Plants

Feng Lin , Jianhua He , Tao Yu

Third Institute of Oceanography institution, 184# Daxue Road Xiamen city Fujian province, 361005, China

Corresponding author's e-mail: linfeng@tio.org.cn

In order to measure tritium activity in sea water near Nuclear Power Plants(NPP) with low-level liquid scintillation counter PE Quantulas 1220 spectrometer, we studied the measurement time, appropriate background of water, scintillation cocktail and sample/scintillation ratio. The minimal detectable activity of this method is 1.07Bq/L after optimizations. In order to test the reliability of the method, we participated in the comparison measurement of IAEA seawater samples. The result of our sample is 2.91±0.35 Bq/L which is only 3.9% to the relative deviation of IAEA reference value (2.80±0.06 Bq/L). It proves the feasibility of our method. Compare with the value reported, it is considered that the method is suitable for tritium measurement in waters adjacent to NPP.

Table 1 Effect of different background water and scintillation cocktails on the counting rate

Background water	Counting rate(cpm)	
	OptiphaseHisafe 3	Ultima Gold LLT
A	1.443	1.431
B	1.354	1.349
C	1.482	1.541
D	1.501	1.484
E	1.491	1.563
F	1.512	1.485

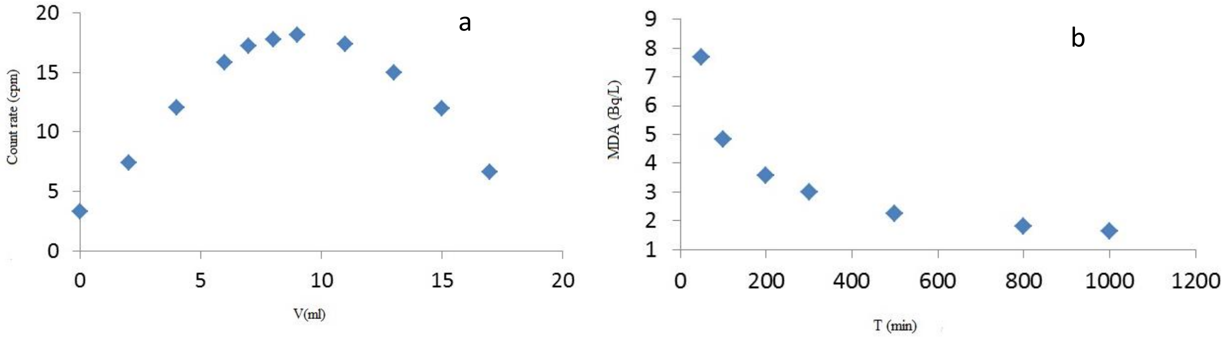

Fig1. a.Curves of counting rates measured with different volumes of samples

b. Minimal detectable activity as a function of time

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OBT measurement in annual ring of tree, Japan

Shinji Sugihara [1], Toshiya Tamari [2], Noriyuki Momoshima [2]

[1] Radioisotope Center, Kyushu University, 6-10-1 Hakozaki, Higashi-ku, Fukuoka 812-8581, Japan

[2] Kyushu Environmental Evaluation Association, 1-10-1 Matsukadai, Higashi-ku, Fukuoka 813-0004, Japan

Corresponding author’s e-mail: sugihara.shinji.248@m.kyushu-u.ac.jp

A large amount of radionuclides was released to the environment by the accident of Fukushima Dai-ichi Nuclear Power Plants (FDNPP) on March 2011. Tritium was also released from the FDNPP as water, and it would be transferred into vegetation. As for tritium, transfer speed in the environment is fast to exist as water. Therefore it is difficult to know the tritium concentration at the time of the accident. we can estimate tritium concentration when an annual ring was produced by measuring tritium (OBT: Organicaly Bound Tritium) in the annual ring of the tree.

In order to investigate the background of OBT in Japan and to estimate the tritium concentration at the time of the accident, annual ring OBT of the wood which collected in Fukushima and Gifu was measured.

Wood sample was cut into round slices, with the exception of the bark, and air-dried. We scraped out of the annual ring from the outside of the dried sample by one year. The scraped sample was soaked to the background water to except for the exchangeable OBT that was replaced with tritium situ during sample preparation. The water sample was obtained from the wood by the combustion method. Concentration of tritium in the water samples was measured by a liquid scintillation counter (LB-5, Hitachi Aloka Medical, Tokyo, Quantulus 1220, PerkinElmer) after mixing water and scintillation cocktail (Ultima GOLD LLT, Perkin Elmer) in a Teflon vial. Counting time was 1200 minutes per sample and an external standard channel ratio (ESCR) was measured at the same time for the correction of quenching.

The OBT concentration in an annual of 2011 was bigger than other annual rings reflecting the influence of the accidental release. It indicated that the tritium released by the accident was incorporated as OBT in plants and stored in the annual ring that was generated at the accident time 2011.

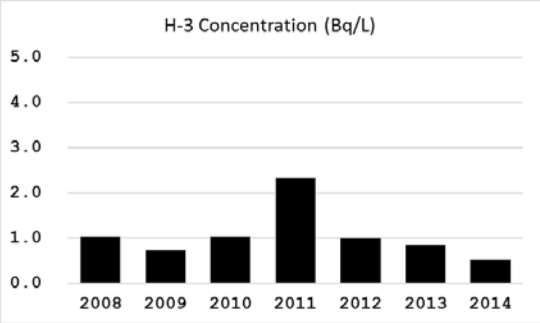

Fig. 1 Tritium concentration in annual ring

The decade tritium monitoring results in groundwater near a radioactive waste repository

Arūnas Gudelis, Inga Gorina, Lina Gaigalaitė

Center for Physical Sciences and Technology (FTMC), Savanorių Ave. 231, Vilnius, LT-02300, Lithuania

Corresponding author's e-mail: arunas.gudelis@ftmc.lt

In January 2005, two new boreholes were installed in close proximity of the vault of the near-surface radioactive waste repository. Since then, groundwater samples were taken from ten boreholes each month for monitoring radionuclide dispersion. Tritium content in the aquifer's upper and lower layers was measured by the liquid scintillation counting method involving an ultra low-level counter Quantulus-1220 [*Gudelis et al., 2006*]. The commercial cocktail Ultima Gold LLT was used for preparation of counting mixtures. For the quality assurance samples were prepared with different sample-to-cocktail ratios. The external standard technique was applied to correct for changes in counting efficiency (Fig. 1). Measurements were traceable to the National standard of activity which was based on the primary TDCR method and tested in the international comparison [*Cassette et al., 2016*].

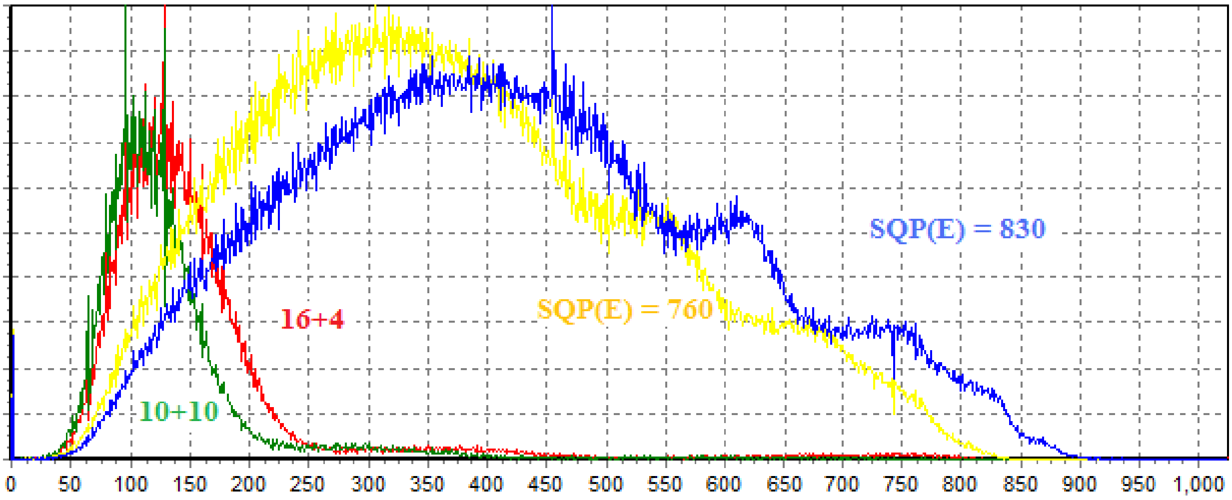

Fig. 1 Tritium spectra obtained at different sample-to-cocktail ratio and respective external standard spectra

The results of tritium determination in groundwater taken from boreholes #41 and #42 are presented in Fig. 2.

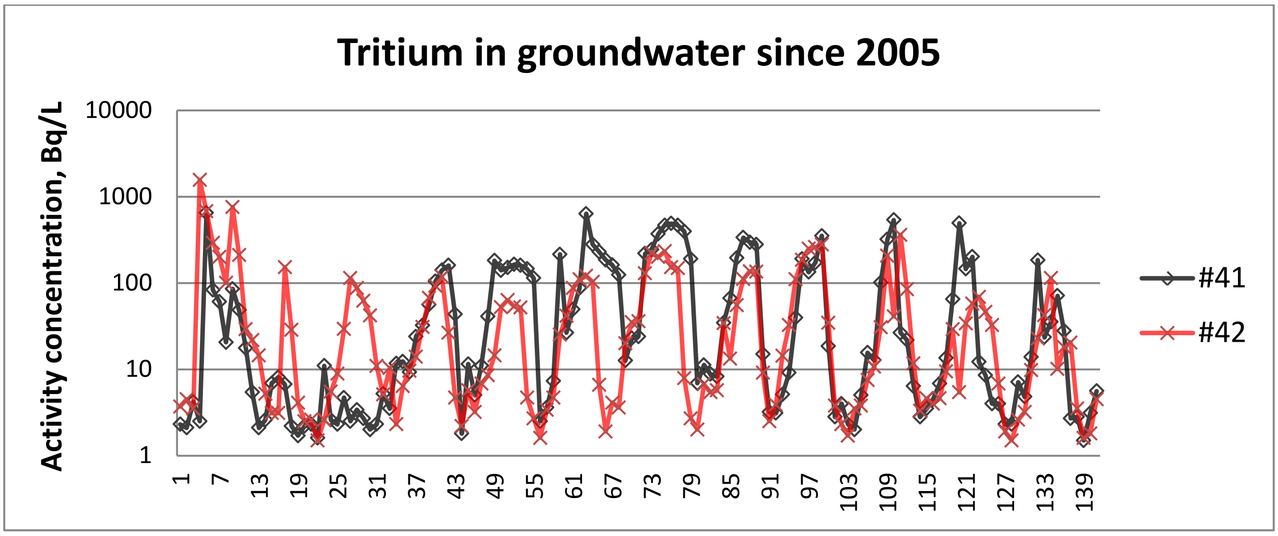

Fig. 2 Tritium in two boreholes installed close to the vault. The measurement number is shown on the x-axis

The results show seasonal variations of tritium activity concentration with maxima in the spring and minima in the late summer while the magnitudes are very similar. This is, probably, related to the changes in groundwater level and wash-out of tritium released before the installation of new physical barriers in 2006. No sharp increases indicate that there is no heavy leakage directly from the vault.

Accurate measurements are important around the nuclear installations to assess the effectiveness of physical barriers and impact to environment.

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Yeong-Rok Kang[1]*

[1] Research Center, Dongnam Inst. of Radiological & Medical Sciences, Busan 619-953, Republic of Korea

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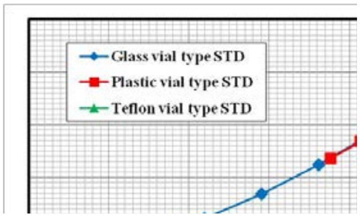

Fig. 1 set of quenching correction curves measured in different source for quenching.

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Simple LS Arrangement for Education and Measurement of Natural Radionuclides in Water Samples of Madagascar and Sri Lanka

S. Moebius 1), C-H. Graser 2), S. Rajaona 3), T. Moebius 1), L. Santiago 1), V. Waduge4)

1) German Society for Liquid Scintillation DGFS e.V., Spoecker Weg 54A, 76351 Linkenheim, Germany

2) Karlsruhe Institute of Technology (KIT), FTU, Hermann-von-Helmholtz-Platz 1, 76344 Eggenstein-Leopoldshafen, Germany

3) Université Antsiranana, Faculté des Sciences, BP 0, Antsiranana 201, Madagascar

4) Sri Lanka Atomic Energy Board, 60/460, Baseline Road, Orugodawatte, Wellampitiya, Sri Lanka.

info@dgfs-ev.eu

Liquid Scintillation (LS) is a universal and efficient, but nevertheless a simple measuring method. However, it lacks from the availability of low cost equipment with respect to the limited budget of educational institutions in developing countries. Our investigations aim at compiling a simple LS arrangement from widely available materials. It cannot substitute a more sensitive commercial equipment with automatic sample changer and α/β discrimination, but can promote LS in education to a broader group of users. The set up consists of a recycled photomultiplier tube (PMT) and a widely used nuclear analyzer (Berthold 2040) as central unit. The crucial step arises from the necessity of total light shielding of the PMT during manual sample changing. The instrumentation was adjusted for optimum counting conditions and calibrated for measurement. It turned out that the arrangement is suitable to quantify α, high and medium energetic β radiation down to 0.2 MeV maximum energy with efficiencies comparable to commercial instruments. However, the sensitivities without α/β discrimination are limited due to the high beta background. For ^{222}Rn in water directly and for ^{226}Ra via ^{222}Rn extraction after storage a detection limit of around 150Bq/L have been found. A customized mobile equipment was compiled at the University of Antsiranana in Madagascar and was successfully applied for LS measurement within a radioisotope training for graduate science students. Rn measurements in water and air samples in Madagascar in the Northern territories of Diego Suarez and its surroundings have been performed, as well as control measurements from the Black Forest and data are reported. The samples were remeasured by Triathler device in the more sensitive α counting mode and data were compared. No increased activity levels have been found for Diego which is in contrast to former measurements in the central areas around Antananarivo and Ansirabe. The use of the PMT for the analysis of Ra in drinking water, however, requires a sample preconcentration step e.g. by Radium RAD disks in order to satisfy their detection in areas of elevated natural radioactivity. Additional data have been compiled during a water sampling campaign in the monazite bearing mountain area of Sri Lanka. While only an occasionally slightly elevated ^{222}Rn activity concentration in granitic spring water could be found, a considerable increased beta level indicates the presence of Thoron daughter nuclides.

DGFS : 15 Years of Research, Education and Training in Liquid Scintillation – Heading for More

S. Moebius, J. Wendel, S. Erat

German Society for Liquid Scintillation DGFS e.V., Spoecker Weg 54A, 76351 Linkenheim, Germany

Juergen.wendel@kit.edu

The German Society for Liquid Scintillation DGFS as non-profit society was founded along the LSC2001 Conference in Karlsruhe with the aim of development and transfer of modern Liquid Scintillation (LS) to a broader community. Within the last fifteen years various analytical procedures with special emphasis on natural radionuclides like gross alpha/beta, Rn and Ra isotopes in air and water and NORM products have been developed or improved. They have been published recently in two handbooks on modern trends and measuring procedures for LS. Selected procedures of practical interest are surveyed in this paper.

Another key object of DGFS is the academical education and training in Liquid Scintillation: Various training courses on behalf of DGFS and IAEA in Germany, Brazil, Thailand and Australia are reviewed and discussed. At the moment the academical education at universities includes simple self made LS arrangements from widely available equipment in order to enable the method to be transfered as well to deveveloping countries with smaller budget (see as well separate paper). However, DGFS heads for more: Future programmes worldwide are presented in order to further spreading this modern, universal and future prospective methodology. You may help by active participation in our Society.

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Development of direct LSC method for biogenic fraction determination in fuels

Jovana Nikolov1), Ivana Stojković2), Milan Tomić3), Radoslav Mićić4), Nataša Todorović1)

1) University of Novi Sad, Faculty of Sciences, Department of Physics, 21000 Novi Sad, Serbia
2) University of Novi Sad, Faculty of Technical Sciences, 21000 Novi Sad, Serbia
3) University of Novi Sad, Faculty of Agriculture, 21000 Novi Sad, Serbia
4) Scientific-Technological Center, NIS-Naftagas, 21000 Novi Sad, Serbia

Corresponding author's e-mail: jovana.nikolov@df.uns.ac.rs

Depletion of oil and gas reserves, their uneven geographical distribution and constant pressure on the environment in the form of global warming, imposed commitment to use transportation fuels from alternative sources. These must be technically and environmentally acceptable, economically competitive and easily available. The current intention is to use biofuels as an alternative to fuels from fossil sources. Due to its ecological, economical and geopolitical significance, biodiesel is becoming an important part of most EU countries developing strategies. At least 10% of synthesized biodiesel is required in liquid fuels by the year 2020, which has stimulated various types of petrodiesel and bio-based component blends production, which triggered development of methods for exact, effective and reliable quantification of biodiesel content.

Method for biogenic component determination in liquid fuels by direct measurement of the ^{14}C activity concentration via liquid scintillation counting (LSC) technique has been developed and presented in this paper. Optimization of method assumed scintillation cocktail, optimal window and vial selection, fuel/cocktail ratio, PAC adjustment and best calibration procedure consideration of liquid scintillation counter. Biodiesel produced from two different feedstock materials was mixed with two commercial fossil matrices to produce fuel blends containing 1–100% FAME (Fatty Acid Methyl Esters), which were analyzed for calibration purposes. Analytical study of relation between the detected released energy and quench level led to establishment of calibration curves specific to the fuel types produced in this moment in Serbia. Minimal detectable biogenic content is assessed to be (0.47 - 1.1)% m for 300 min of measurement time, depending on analyzed fuel mixture. Measurements were performed on Ultra Low Level Liquid Scintillation Spectrometer 1220 Quantulus manufactured by Perkin Elmer.

Key words: Biodiesel, biogenic fraction, 14C, Liquid Scintillation Counting (LSC), optimization

Extraction and Characterization of Groundwater Colloids

Kaifeng Wang, Chunli Liu*

Beijing National Laboratory for Molecular Sciences, Fundamental Science Laboratory on Radiochemistry and Radiation Chemistry, College of Chemistry and Molecular Engineering, Peking University, Beijing, 100871, China

Corresponding author's e-mail: liucl@pku.edu.cn

Understanding the physical, chemical and biological properties of natural aquatic colloids in groundwater are of great importance for exploring the mobility behavior and speciation distribution of radionuclides in the near field of the repository[1-3]. Unfortunately, the knowledge about aquatic colloidal properties was quite incomprehensive due to their extremely low concentration. Though conventional centrifugation method can enrich aquatic colloids on membrane, it may change colloidal properties for the interactions with membrane surface[4].

In this paper, an ultrafiltration technique maintaining in situ hydraulic pressure was developed to enrich aquatic colloids effectively from groundwater without chemical disturbance, and the properties of aquatic colloids were characterized using multiple state-of-the-art analytical techniques: dynamic light scattering (DLS), scanning electron microscope (SEM), atomic force microscopy (AFM), X-ray diffracmeter (XRD), high resolution transmission electron microscopy (HRTEM), 3D excitation-emission matrix (EEM) fluorescence and inverted fluorescence microscopy (IFM).

The average equivalent circle diameter of colloids was 347 ± 188nm by SEM image (Figure 1). Two different morphological characteristics of colloids could be found in our AFM images, which seem to be described as erythrocyte and platelet structure (Figure 2). XRD patterns indicated the presence of quartz (SiO_2), gypsum ($CaSO_4 \cdot 2H_2O$), microcline maximum ($KAlSi_3O_8$), dolomite ($CaMg(CO_3)$) and lepidocrocite ($FeO(OH)$). The colloidal organic phase was characterized by EEM, as shown in Figure 3, in which four fluorophores can be observed, including humic-like (A), fulvic-like (C), tyrosine-like (B) and tryptophan-like (T). Bacteria was observed under 488 nm laser excitation by IFM, and 16S rDNA sequencing revealed that Thermomonas was the most likely genus the bacteria may belong to.

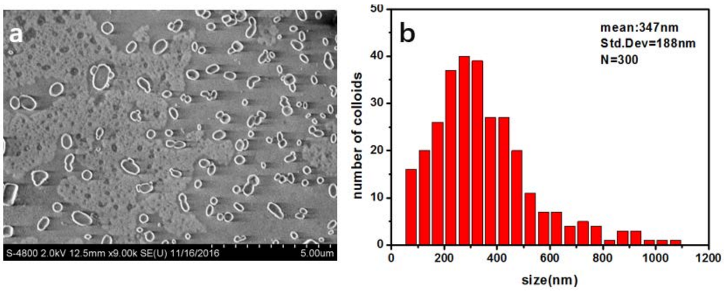

Fig. 1 (a) SEM image of colloids, (b) Size distribution histograms from SEM

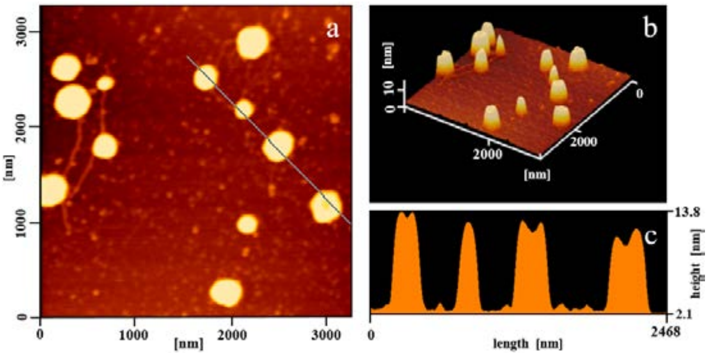

Fig. 2 (a) TM-AFM image of colloids absorbed on silicon slice, (b) section analysis (according to the blue line in part a), (c) 3D topographic image.

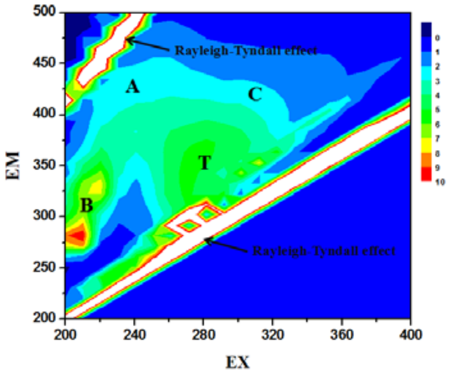

Fig. 3 EEM fluorescence for colloidal organic phase

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Evaluation of the LSC counting time for determination gross alpha and gross beta activity, ^{3}H and ^{90}Sr/^{90}Y

M. B. Nisti[1)], M. F. Máduar[1)], C. H. R. Saueia[1)], B. P. Mazzilli[1)]

[1)] IPEN – CNEN/SP, Av. Professor Lineu Prestes 2242, 05508-000, Brazil

Corresponding author's e-mail: mbnisti@ipen.br, mmaduar@ipen.br, chsaueia@ipen.br, mazzilli@ipen.br

Counting time can be defined taking into account the uncertainty of the measurement or the minimum detectable activity (MDA) required.

This paper aims to evaluate the liquid scintillation counting (LSC) time that provides a reduction in the uncertainty of the concentration measurement to acceptable levels.

The equipment used for the measurement was 1220 Quantulus™ Ultra Low Level Liquid Scintillation Spectrometer. The background and the standard solutions were measured for the determination of gross alpha and gross beta activity, ^{3}H and ^{90}Sr/^{90}Y, using different counting times: 15, 30, 60, 120, 180, 240 and 300 minutes.

For gross alpha and gross beta activity measurement, an initial step was added to the methodology, which consisted of heating the standard solution on a hot plate, at a temperature of 80 °C, for reduction of the volume. An aliquot of 5 mL of the final solution was mixed with 15 mL of the scintillation solution.

For ^{3}H determination, an aliquot of 50 mL of the standard solution was transferred with a volumetric pipette to a distillation apparatus, and was distilled at a controlled temperature from 96 to 97 °C with a heating controller. An aliquot of 5 mL of the distilled solution was mixed with 15 mL of the scintillation solution.

For ^{90}Sr/^{90}Y, an initial step of pre-concentration was added to the methodology, which consisted of heating on a hot plate, at a temperature of 80 °C, for reduction of the volume. An aliquot of 5 mL of the final solution was mixed with 15 mL of the scintillation solution.

All samples were counted in the appropriate vial with 20 mL capacity. The measurement of the background was carried out following the same procedure described for each radionuclide and using deionized water.

The performance of the LSC method for the measurement of gross alpha and gross beta activity, ^{3}H and ^{90}Sr/^{90}Y in water was evaluated by participating in Proficiency Tests (PT) organized by Instituto de Radioproteção e Dosimetria IRD/CNEN.

Secondary electron spectra measured in modified LSC vial

Katarzyna Szufa [1], Tomasz Mróz [2], Jerzy W. Mietelski [1]

[1] The Henryk Niewodniczański Institute of Nuclear Physics Polish Academy of Sciences,
ul. Radzikowskiego 152, 31-342 Kraków, Poland
[2] The Pedagogical University of Cracow,
ul. Podchorążych 2, 30-084 Kraków, Poland

Corresponding author's e-mail: jerzy.mietelski@ifj.edu.pl

In the recent times, nanoparticles of gold (GNP's) and high Z-number elements are widely applied in therapy as a tumor-selective radiation dose enhancement and Monte Carlo method is common approach for simulating doses from secondary electrons [1,2,3,4]. In our study, a standard 20 ml LSC plastic vial was modified in aim to determine spectra produced by secondary electrons induced by gamma-rays. Different, point gamma-ray sources (^{241}Am, ^{137}Cs, ^{60}Co) were mounted inside the top screwed cup of vial. In case of Cs and Co isotopes directly below it an appropriate height plastic cylinder was situated to filtrate direct beta radiation coming from the source. It was wrapped with black tape to eliminate Czerenkov radiation or scintillations generated inside it. The top of the vial was also wrapped out by black tape from the same reason. Suspensions of GNP's of different diameters were placed in such prepared vials, mixed with scintillation cocktail. Spectra were collected using Wallac 1414 Guardian counter. Results obtained are discussed.

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Gross Alpha – Beta Measurements of Water Samples from Wonderfonteinspruit Catchment Area in the Gauteng Province South Africa using LSC

Mashaba, M.[1], Kotze, D.[2], Tshivhase, V.M.[1], Faanhof, A.[1, 2]

[1] Northwest University, Centre of Applied Radiation Science & Technology (CARST), Mafikeng, South Africa.

[2] The South African Nuclear Energy Corporation SOC Limited, Pretoria, South Africa.

Corresponding Author's email: mashabamac@gmail.com

Liquid scintillation counting (LSC) offers an attractive method for fast screening of water samples contaminated with NORMs. The determination of gross α-β emitters with the LSC method requires an accurate PSA setting under various quenching conditions. The appearance of quenching in the sample affects not only counting efficiency but the α-β discrimination as well. The aim of the research is to evaluate the potential of a low-background liquid scintillation system with advanced spectrometry capabilities, the Quantulus 1220^{TM}, to be used directly for the determination of the gross α- and β-activities in environmental water collected from the Wonderfontein catchment area (WCA) without extensive sample preparation. The results were verified by element specific separations (U, Ra, Pb/Po and Th) followed by α-spectrometry at the radionalytical laboratories of the South African Nuclear Energy Corporation (Necsa). An acceptable correlation was obtained between LSC and α-spectrometry. The method has demonstrated that LSC is able to produce reliable result for measuring α/β-activity in water samples. The method developed offers an attractive and cost-effective alternative.

KEYWORDS

LSC, PSA, Quench, α/β-activity, WCA, NORMs.

Suitability of gross alpha region of interest for the determination of gross alpha and gross beta activities in water by liquid scintillation counting

João Abrantes [1)], Irene Lopes [1,2)], Anabela Mourato [1)], Maria José Madruga [1,2)], Mário Reis [1,2)]

[1)] Laboratório de Proteção e Segurança Radiológica, Instituto Superior Técnico, Universidade de Lisboa; Estrada Nacional 10, ao km 139.7, 2695-066, Portugal

[2)] Centro de Ciências e Tecnologias Nucleares, Instituto Superior Técnico, Universidade de Lisboa, Estrada Nacional 10, ao km 139.7, 2695-066, Portugal

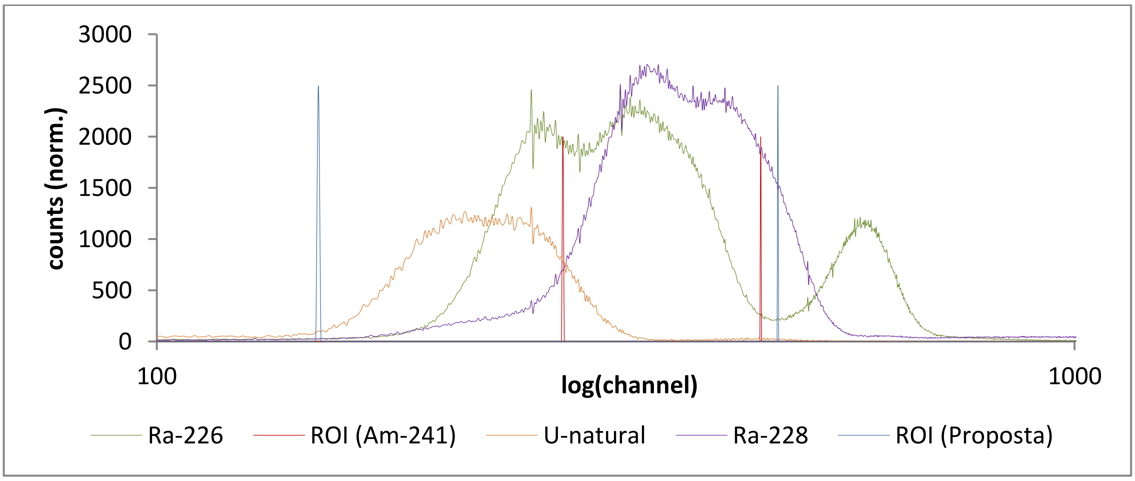
Corresponding author's e-mail: abrantes@ctn.tecnico.ulisboa.pt

Gross alpha and gross beta activity determination by liquid scintillation counters in waters intended for human consumption are nowadays routine methods in many laboratories worldwide.

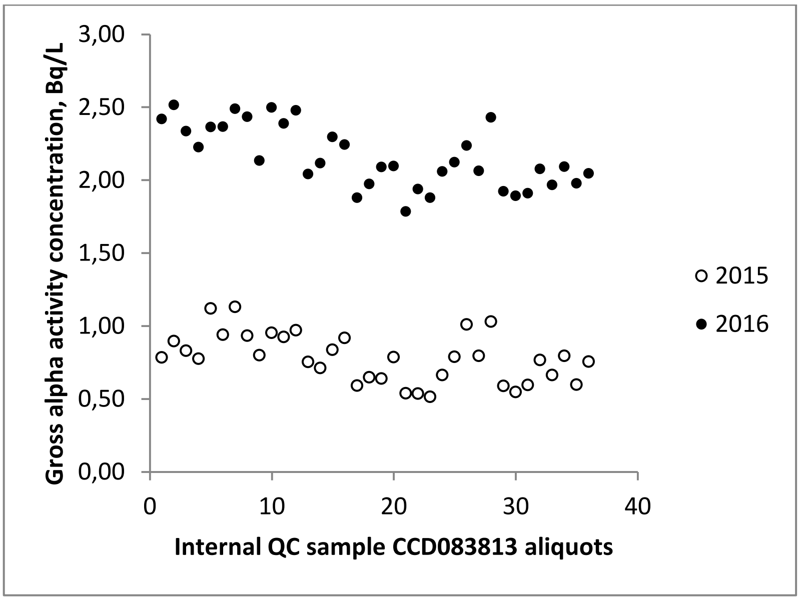
Radionuclide selection for calibration will depend on intended purpose of measurement. Consequently, different set-ups can be found for the same measurement instrument: region of interest, pulse discriminator, background, efficiency and spillover for both measurement windows.

Typically, in order to optimize measurement conditions, the setting of a proper region of interest (ROI) for both alpha and beta windows is achieved maximizing the figure of merit (FOM). Although the FOM algorithm is useful at constant quench conditions, the procedure must be adapted in order to take into account the dislocation of spectra to smaller energies in the presence of higher quench. Even so, in the presence of natural alpha emitters, the region of interest can exclude some of this emissions and must be extended.

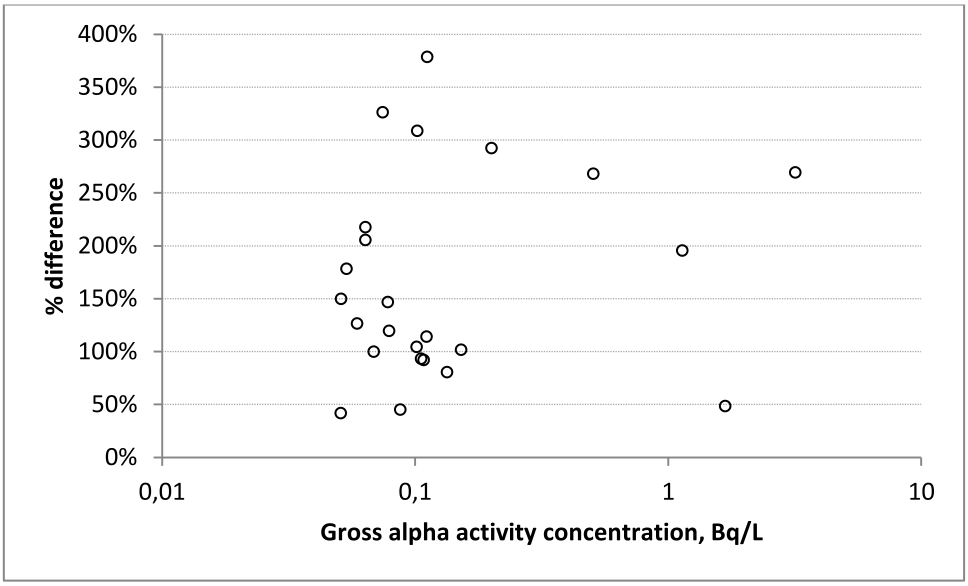
In the present work it is evaluated the suitability of this method in the presence of natural alpha emitters and results will be presented and compared for internal QC and intercomparison samples. Implications on calibration curves, detection limits and routine measurements is also discussed.



Normalized alpha spectra for different emitters and ROIs: in use, obtained with FOM algorithm with Am-241 (red) and proposed in this work (blue).



Gross alpha activity concentration for an internal QC sample calculated for different ROIs: in use, obtained with FOM algorithm with Am-241 (2015) and proposed in this work (2016).



Gross alpha activity concentration difference (%) for routine samples calculated for the two ROIs as a function of gross alpha activity concentration.

Limitations of the LSC technique in alpha spectrometry of transuranium elements

L. Toro, F. Baciu - Obrocea, A. Musat, G. Teodorov, A. Lefterovoci

Matefin Ltd, Bucharest, Romania, 87-89 Vulturilor street

toro.laszlo@matefin.com

The purpose of this work is to assess the possibilities of the use of LSC in the measurement of transuranic alpha emitting element (U, Pu and Am) concentrations. The experiments were performed to prepare an alternative method for the measurement of the concentrations of these elements if “classical” alpha spectrometry equippement (with PIPS semiconductor dectectors) is failed.

Normally the dosage of a given alpha emitting nuclide, a corresponding racer is added to the sample followed by a radichemical separation of the radioactive element (Am, Pu and U) and at the end an alpha spectrometrical measurement is performed using semiconductor detectors.

To see if in the final measurement the LSC spectrometry should replace the PIPPS detector spectrometry the following steps were performed:

- A given amount (1.5Bq) of transuranic tracer nuclide (^{243}Am, ^{242}Pu and ^{232}U) was added separatelly to 15ml scintillation cocktail (Ultima Gold and Aqua Light) obtainig two sets of sample
- Each sample was measured with a Hidex HL300 liquid scintilation counter using TDCR technique
- For each sample the energy resolution was calculated

Analyising the experimental results:

- The spectra obtained with Ultima Gold liquid scintilation cokctail is shifted with about 40keV toward the higher energies
- The shifting is more pronounced in the rising region of the absorbtion peak
- FWHM of the peaks resulted from ^{243}Am and ^{242}Pu are between 385-322keV, much higher related to the semiconductor spectrometry peaks (between 40 and 60 keV)
- The peaks obtained with Aqua Light scintillation cocktail are broader related to those obtained with Ultima Gold. Thew differences are in the measuring uncertainity range but are systematic
- In ther case of ^{232}U due to the presence of the daughters of the parent nuclides resulted a series of peaks (in fact two) wich contain peaks resulted from several alpha emitting nuclides.

According to the low resolution LSC technique is not suitable to use tracer nuclides in the measurement of alpha emmiting nuclides. The tracer will mask the the impulses obtained from the unknown nuclides.

^{222}Rn concentration in groundwater of geological rock types in South Korea using LSC

S.Y Cho, K.Y. Lee, Y.Y.Yoon, B.W. Cho, W. Yoon, H.I. Kwon, K.C.Ha

Geologic Environment Division, Korea Institute of Geoscience and Mineral Resources(KIGAM)

30 Gajeong-dong, Yuseong-gu, Daejeon, 305-350, Korea

Corresponding author’s e-mail: sycho@kigam.re.kr

The determination of ^{222}Rn concentration in groundwater is very important to understand the groundwater and surface-water interactions because it has been used frequently as a tracer form any geohydrological processes. ^{222}Rn contents were determined in a total of 5,200 groundwater samples collected from geological rock types in Korea. The liquid scintillation counting technique was employed for th measurement using Quantulus 1220. Their concentration levels were used to construct detailed distribution maps. The relationship between chemical characteristics and groundwater sampling depth was investigated in terms of EC, pH, and temperature. The concentration of ^{222}Rn groundwater ranges from 0.1 to 2,390 Bq/l with an average of 84.8 Bq/L. The average Rn contents of the rock types increase in the following order: Granite> Metamorphic rock > Sedimentary rock > Volcanic rock. The ^{222}Rn contents and their physicochemical components however, reveal no relationship. The Rn contents in groundwater were determined as 170 Bq/L, 210 Bq/L, 260 Bq/L with the depth of <50 m, 50-100 m, >100 m, respectively. When viewed from the average value, ^{222}Rn in deep ground was relatively higher. AS a result, ^{222}Rn contents of groundwater in Korea dependent on the the rock types and groundwater sampling depth.

Establishment of rapid LSC method for direct alpha/beta measurements in waters

Nataša Todorović [1]*, Ivana Stojković [2], Jovana Nikolov [1]

[1] University of Novi Sad, Faculty of Sciences, Department of Physics, Novi Sad, Serbia
[2] University of Novi Sad, Faculty of Technical Sciences, Novi Sad, Serbia

Corresponding author contact: natasa.todorovic@df.uns.ac.rs

Abstract. Gross alpha/beta measurements in drinking waters enable radiochemical composition analysis in environmental studies providing efficient screening method that indicates whether water sample contains elevated levels of any radionuclide. Routine gross alpha/beta activity monitoring in drinking waters has been carried out in laboratory for low-level radioactivity measurements in Novi Sad for a few years according to ASTM D 7283-06 method on liquid scintillation counter Quantulus 1220^{TM}.

Development of rapid and efficient screening method for gross alpha/beta measurements has been presented. This method is modification of conventional ASTM method and assumes direct mixing of water samples with liquid scintillation cocktail Ultima Gold AB, without any sample preparation. Optimization of the method involved determination of optimal sample-to-scintillation cocktail ratio based on the achieved detection limit and quench level of sample. Optimal value of Pulse Shape Analysis (PSA) discriminator was investigated for different sample:cocktail ratios to ensure accurate and reliable alpha/beta spectra separation. Calibration experiments have been carried out with ^{241}Am, and ^{90}Sr/^{90}Y aqueous standards for different sample:cocktail ratios, which included efficiency, MDA (Minimal Detectable Activity) and optimal PSA parameter determination.

Method's accuracy and validity has been tested on few water samples and spiked samples with various radionuclides and obtained activities have been compared to results obtained with ASTM D 7283-06 method. Presented method for direct alpha/beta measurements offers some advantages over conventional ASTM method, since it is ultra fast, simple and inexpensive test for efficient screening of water samples.

Key words: alpha/beta spectroscopy, Quantulus 1220^{TM}, optimization

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Friday

5 May 2017

1. **Session: Application in tritium studies**
2. **Session: Application in environmental radioactivity-3**

Tritium analysis strategy regarding activity concentration levels in monitoring situations

N. Baglana 1), C. Cossonnetb 2)

1) CEA/DAM/DIF - F91297 Arpajon - France.
2) IRSN/PRP-ENV/STEME/LMRE - Bât 501 - Bois des Rames, 91400 Orsay - France.

Tritium can be found in many types of samples originating from the environment, from nuclear plants (effluents, waste…), from facilities handling tritium (heavy water production, radiopharmaceutical, luminous paints…). Thus, activity concentrations levels to assess vary over a wide range starting from one Bq.L^{-1} to thousands or even millions of Bq.L^{-1}. Therefore, analytical strategy should be adapted regarding either technical and instrumental limitations but also taking into account safety issues. In addition, investigation on tritium forms and or tritium species are frequently asked to get a better insight on tritium behaviour, mainly to estimate the part of tritium which is tightly bound to the matrix. Hence, aiming at obtaining enough water to fulfil measurement objectives (accuracy, precision); the preparation process will be a function of sample types (gaz, liquid, solid).

Focusing on the measurement itself, Liquid Scintillation Counting (LSC) is widely used for tritium (and other □ emitters) activity concentration determination with counters designed by the manufacturers for different applications range. After a short overview of the activity concentration to assess regarding monitoring objectives, the different counters class (low level, standard, high level) will be investigated to assess their best performance field, their drawbacks. In addition, some information on an alternative technique (mass spec.) will be discussed aiming at providing its best applications range and to demonstrate its complementarity with LSC.

Apparatus for measurement of tritium in expiration with plastic scintillator

Etsuko Furuta [1), Tuyoshi Ito [2)

[1) Ochanomizu University, 2-1-1 Otsuka Bunkyo-ku Tokyo, 112-8610, Japan
[2) KAKEN Co. LTD., 1044 Hori Mito Ibaraki, 310-0903, Japan

Corresponding author’s e-mail: furuta.etsuko@ocha.ac.jp

Introduction After the Fukushima Daiichi nuclear power plant accident of Japan, many skilled laborers for radioactive materials treatment have been working at the site. They always wear masks not to inhale radioactive materials spread in the air. It is considered that their masks protect them from radioactivities attaching to dust, perfectly. On the other hand, tritiated water in the air was considered difficult to shut out with the filter of the masks. So, they always worry about their health. Also, the situations were same at the advanced thermal reactor “Fugen” of Japan and in the case of exchange of the neutron source at J-PARC: Japan Proton Accelerator Research Complex, where radioactive gases were spread from duct of mercury source. Actually, some workers were exposed to tritiated water significantly at the latter case. Once, there was an apparatus for measurement of tritium in expiration in Japan. However, the apparatus is not produced now, and even if a facility holds the old one, it needs 10 L breath for one measure and the result is gotten some days after the check. So, it is not easy to know the concentration of tritium in the expiration, now. Additionally, the old apparatus generated much amounts of radioactive organic wastes after the measurement because of use of liquid scintillator for LSC measurement. We designed a new type of apparatus which measure tritiated water in expiration using plastic scintillator pellets and a low background LSC of AccuFLEX LSC-LB7 (Hitachi Ltd., Japan). The outline of the apparatus and the measurement method will be reported.

Experimental A photo of the prototype apparatus is shown in Fig.1. A bag to collect the breath

Fig.1 A photo of apparatus: the left side is a control box and the right side shows the inside main part of apparatus

Homogeneity Assessment for Grass Items from Organically Bound Tritium Proficiency Test

Carmen Varlam [1)], Cristina Bucur [2)], Irina Vagner [1)], Marius Constantinescu [1)], Diana Bogdan [1)], Ionut Faurescu [1)]

1) Tritium Laboratory, Institute for Cryogenics and Isotopic Technologies ICSI, Rm. Valcea, Valcea, 240050, Romania

2) Health Physics Department, Cernavoda Nuclear Power Plant, Cernavoda, Constanta, 905200, Romania

Corresponding author’s e-mail: Carmen.Varlam@icsi.ro

Starting with “1st workshop on Organically Bound Tritium (OBT) and its analysis” held in Balaruc les Bains, France in 2012, an international working group was established. Within the last 4 years, several inter-laboratory exercises were organized to improve our understanding of OBT behavior and the overall analytical performance.

The 4th OBT exercise organized by Environmental Laboratory of Nuclear Power Plant Cernavoda is in progress. The sample chosen for this proficiency test is grass. This was sampled, dried, and homogenised before to be dispatch by the organiser. Due to the different methods used by the participants to the exercise and, of course, due to ISO 13528* requirements, the homogeneity and stability of the proficiency test items are of primary interest for the quality and success of the exercise.

The paper presents homogeneity check for grass sample performed for hydrogen content and OBT analysis (Bq/l combustion water). 10 package of proficiency test items were chosen using a stratified random selection. 2 replicates from each package were analysed for Hydrogen content and OBT analysis. The homogeneity parameters were evaluated according to ISO 13 528. The two properties checked confirm that homogeneities is sufficient for the batch prepared for 4th OBT exercise.

References:

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Determination of tritium in water using isotopic enrichment. Methodology improvements

Ana Rita Gomes (1), João Abrantes (1), Albertina Libânio (1), Maria José Madruga (1,2), Mário Reis (1,2)

(1) Laboratório de Proteção e Segurança Radiológica, Instituto Superior Técnico, Universidade de Lisboa, Estrada Nacional 10, ao km 139.7, 2695-066, Portugal

(2) Centro de Ciências e Tecnologias Nucleares, Instituto Superior Técnico, Universidade de Lisboa, Estrada Nacional 10, ao km 139.7, 2695-066, Portugal

Corresponding author's e-mail: argomes@ctn.tecnico.ulisboa.pt

Tritium (^{3}H) is present in the environment as a result of natural and anthropogenic sources. It is a pure beta emitter (Emax = 18.6 keV) with a half-life of 12.31 years and is produced in the atmosphere by nuclear reactions between fast neutrons that result from cosmic radiation and nitrogen atoms. Tritium levels in environmental samples increased between 1945 and 1963 due to nuclear testing. Since 1963 the activity in ^{3}H has been decreasing and currently levels are close to minimum detectable activity concentrations.

One of the methodologies used for ^{3}H determination in water samples is based on the electrolytic concentration of the sample. Following the water purification by distillation, the isotopic enrichment of the sample is carried out by electrolysis in direct current, followed by another distillation with $PbCl_2$. An aliquot of the distillate (≈ 8 g) is mixed with 12 g of scintillation cocktail Ultima Gold LLT® and the sample is measured by LSC (Tri-Carb 3170 TR / SL, Packard) in low-level mode with 300 min counting time . Under these conditions, a detection limit (LD) of about 0.40 Bq L^{-1} is reached.

The quality control of the method was improved considering more parameters in the uncertainty calculation. Beyond gross sample count rate, background count rate, counting efficiency, ^{3}H half-life, other parameters are now taking into account, such as weighings, direct current, electrolytic enrichment factor (Z) and enrichment parameter (P). Besides, P, Z and blank samples are evaluated in each batch to check if the electrolysis system is working properly.

The outcomes for the intercomparison participations in 2014 (IARMA ETRIT-PT-2014 - IARMA Proficiency Test on the Determination of Tritium in Water at Environmental Levels) and in 2015 (CSN - Intercomparación analítica entre laboratorios de radiactividad ambiental – agua) were Acceptable and Satisfactory, respectively. The IARMA results allowed validating the accuracy of the method.

A new efficiency measuring way for the liquid scintillation counting method

Qinghua Xu, Zhonghua Xiong, Shicheng Li

Institute of Materials, China Academy of Engineering Physics. P. O. BOX: 9071-14, NO. 9 Huafengxincun, Jiangyou, Mianyang 621907, Sichuan, PRC.

Corresponding author’s e-mail: 2371087183@qq.com

Liquid scintillation counting method is the most commonly used for measuring tritium concentration in the surface water. To optimize the tritium analysis, the calibration of background, combination of scintillant/vial, measurement of efficiency and optimization of the way to deal with sample and data were performed.

Table1 Background of ^{3}H counting mode

Entry	Vial type	Cocktails
1	-	-
2	glass	-
3	polyethylene	-
4	polyethylene	14
5	glass	5
6	glass	10
7	glass	14
8	glass	20

Quantulus 1220 ultra-low background liquid scintillation spectrometer was used in this work. The quantulus 1220 has two multichannel analyzers (MCA), MAC1 and MAC2. MCA 2 is used for measuring the spectrum of the guard and MAC1 is used for spectra record. Those two MCAs are divided in two halves, Half1 and half2 respectively. Half 1 measured pulses in the beta detector, half2 measured all the other or anti-coincident pulses.

A series of samples were analyzed to confirm the background (table 1) of the methods for measuring tritium concentration. The background spectra of SP12 were shown in Fig.1. The characteristic emission peaks for the glass vail with different volume cocktail had similar shape and position (Fig.1 left 5, 6, 7, and 8). The spectra of the empty glass vail and vail with cocktail had the analogy formation in the range of channels 50 to 400 (Fig.1 right). The channels range of measuring

Quench correction in the analysis of Organic Bonded Tritium (OBT) in biota samples

CHEN Qianyuan, LIU Changjun, ZHANG Rongsuo, XIANG Yuanyi, GONG Chuande

The *Radiation Monitoring Technical Center, Ministry of Environmental Protection, PRC*

Biota samples in the ambient of an operated Nuclear Power Plant were collected, after being cleaned up and dried to eternal weight under the temperature of around 85 centigrade, the dried samples were smashed into powders, place approximately 8 grams of sample powder evenly into each silica combustion boat, the totally six boats were inserted separately into the silica combustion tubes of the Pyrolyser-6 Tritium&Carbon-14 extraction apparatus, after the preset combustion program was run over , OBT contained in the biota sample was oxidized as tritiated water(HTO) and trapped inside of the Coolsafe cold trap under -110 centigrade.

10 grams of melted HTO was weighted into a polyethylene scintillation vial and mixed with 10 mL of UtimaGold LLT cocktail after fierce shaking. The sample was finally measured in the Quantulus 1220 Liquid Scintillation Counter.

Due to the impurity of the collected HTO, Quench index which shown as SQP(E) is tend to be differ from one sample to another, which means that a quench correction was required. First of all, several kinds of reagents, such as CCl_4, CH_3NO_2 and HNO_3,were chosen to be the tested quenching indicator. Then, different weights of reagents were added into the HTO reference& background sample and measured, so as to make a quench correction curve [SQP(E)-Efficiency]. Finally, quench curves which covers the SQP(E) range of the OBT samples of our lab best was chosen, therefore, CH_3NO_2 was chosen to be the quenching indicator in the measurement of OBT in biota samples.

When the measuring of sample was completed, SOP(E) value could be checked out in the data output file of Easyview sofware, therefore, the correspondent counting efficiency value of the counted sample could be checked out in the quench curve [SQP(E)-Efficiency], which was to be applied to calculate the activity concentration of OBT in biota samples.

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Tritium content in tissue free water (TFWT) in African coconuts

Giscard Honoré Sonkwa Monthe(1), Romana Krištof (2), Jasmina Kožar Logar (2)

(1) University of Yaoundé I, Faculty of Science, P.O. Box 812, Yaoundé, Cameroon
(2) Jožef Stefan Institute, Jamova 39, SI-1000, Slovenia

Corresponding author's e-mail: jasmina.logar©ijs.si

Coconut is important nutrition source for most people under the tropical and subtropical belt all over the World. The fruit becomes very popular also in so called western cultures because of its richness of vitamins, minerals and bioactive compounds that are essential for better health.

The paper of Narayan [1] attracted the attention with the thesis that the coconut fruit pre-concentrates the tritium in respect to tritium concentration in environmental water such as irrigation water and precipitation. Ripe coconuts from Cameroon, Ivory Coast and Equatorial Guinea were collected with the attempt to provide first tritium data also for African fruits. Cameroon's fruits were from the towns of Limbe, Mamfe and Mbonge. The nuts from Guinea were found at the market of Kribi while samples of Ivorian nuts were obtained at supermarkets in Giessen, Germany and Kranj, Slovenia.

Ripe coconut water was simply distilled and directly measured by Quantulus 1220 while sample preparation for Tissue Free Water Tritium (TFWT) determination in water from kernel milk was conducted on two different ways. Simple procedure was developed with the intention to be feasible also for majority of African laboratories. Grated coconut kernel was heated in sand bath. Evaporated water droplets were collected and distilled. The procedure was tested and validated by one of the usual methods in TFWT/OBT sample preparation, e.g. azeotrope distillation with toluene.

Preliminary results show consistent values for different coconuts from the same sampling points. Tritium values vary among different locations. Tritium content is always higher for kernel milk water than for ripe coconut water.

The comparison of sample preparation methods will be presented and commented. TFWT values for coconut water and milk from all mentioned locations will be evaluated and compared with results for Indian coconuts.

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Automation of the radiochemical procedures for the sequential separation of mixed radionuclides

Kun Ho Chung, Hyuncheol Kim, Jong Myoung Lim, Mun Ja Kang

Environmental Radioactivity Assessment Team, Korea Atomic Energy Research Institute, 989-111 Daedeok-daero, Yuseong-gu, Daejeon 34057, Republic of Korea

Corresponding author's e-mail: chungkh@kaeri.re.kr

To effectively determine the radioactivity of alpha and beta nuclides by LSC, Alpha Spectrometer or ICP-MS, it is necessary to remove all the interfering radionuclides. Interfering nuclides are typically removed by extraction chromatographic methods under gravitational flow or using a vacuum box. Many separation methods are manual and time-consuming. The automated radionuclide separators with a single column arrangement have been designed and used for the measurement of ^{99}Tc in groundwater [1] and $^{89/90}$Sr in milk [2].

An automated sequential radionuclide separator (ASRS) has been developed for the automation of the radiochemical procedures, which is to sequentially separate mixed radionuclides in environmental and waste samples. The ASRS with a tandem column arrangement can simultaneously, rapidly, selectively and automatically separate mixed radionuclides in a sample. The ASRS consists of a data acquisition device, peristaltic pumps, solenoid valves and columns. Three types of solenoid valve are used for ASRS with a tandem column arrangement. A 6-inlet port flow selection valve is used to select different chemical reagents. The 2-inlet port flow selection valve is used to select a sample or a chemical reagent. The 4-way flow distribution valve is used to collect eluated solution and connect two columns as a tandem arrangement. Fig. 1 shows the system configuration of ASRS for the sequential separation of mixed radionuclides.

Control software for the individual control of all instruments components is developed in Labview programming language. The control software consists of three windows called the front panel, which are virtual instruments for interfacing the software and ASRS. The first and second windows are used for calibrating the flow rate of peristaltic pumps. The final window is used to execute an automated radiochemical procedure for separating mixed radionuclides. The input parameters in the virtual instruments are the volumes, flow rates and selection of reagents for executing an automated radiochemical procedure.

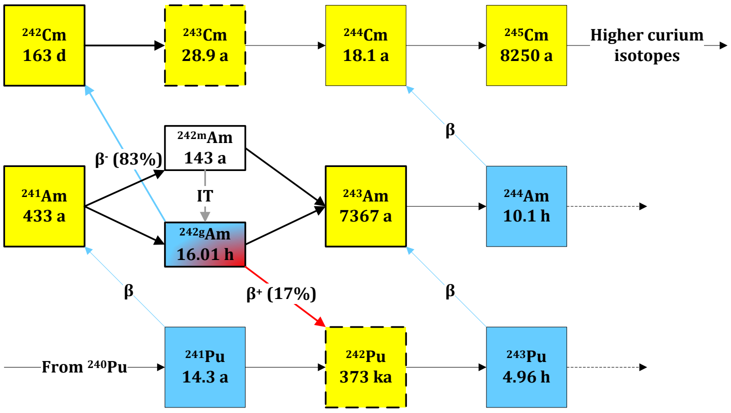
The ASRS, designed and built in our laboratory, was successfully applied to the sequential separation of mixed radionuclides in a sample. Compared to conventional radionuclide separation methods carried out manually, ASRS is faster, less labor intensive and expected to be widely used as a powerful and convenient tool for the automated chemical separation and purification of radionuclides at trace levels found in environmental samples.

Determination of ^{242m}Am by Extraction Chromatography and Liquid Scintillation Counting

Simon Jerome, Andy Pearce and Peter Ivanov

National Physical Laboratory, Teddington TW11 0LW, United Kingdom

Americium isotopes are generated in the nuclear fuel cycle, particularly where mixed oxide fuels are employed. Americium, mainly as ^{241}Am, occurs in the environment either through direct discharge of ^{241}Am from fuel reprocessing facilities or by β^- decay of ^{241}Pu that has been similarly released. The production of americium and curium proceeds as partially shown here:



Both ^{242m}Am and ^{242g}Am are key intermediaries in the formation of higher actinides, and that the relatively long half-life of ^{242m}Am (143(2) years) indicates that this nuclide may be found in the environment with ^{241}Am. Furthermore, the relative levels of ^{241}Am, ^{242m}Am (and ^{243}Am) may provide some information on the irradiation history of material containing americium. Thus, taken with other measurements, americium ratios may be employed to identify nuclear materials for forensic investigations of the source and age of such material. As the fission and activation cross sections of ^{242m}Am are relatively large (~6 500 barns and ~1 100 barns respectively), equilibrium between ^{241}Am and ^{242m}Am is reached fairly quickly, since for neutron fluxes greater than 10^{12} n cm^{-2} s^{-1}:

$$\frac{\lambda_{242m} + \sigma_{242m(n,\gamma)}\cdot\varphi_{th} + \sigma_{242m(n,f)}\cdot\varphi_{th}}{\lambda_{241} + \sigma_{241(n,\gamma)}\cdot\varphi_{th} + \sigma_{241(n,f)}\cdot\varphi_{th}} > 37$$

At equilibrium, the activity ratio of ^{242m}Am:^{241}Am approaches 6×10^{-2}, although measured values are wholly dependent on the irradiation and decay history of the material being measured.

In this paper, currently available techniques for separation and purification of americium by extraction chromatography are employed. A novel approach to the simultaneous determination of ^{241}Am, ^{242m}Am and ^{243}Am by liquid scintillation counting will be described; the technique is based on the recovery of americium isotopes by washing the active component of extraction

A comparison of methodologies in analysing ^{210}Pb-^{210}Bi-^{210}Po

Per Roos, Nikola Markovic

The DTU Nutech, Center for Nuclear Technologies

Corresponding author's e-mail: roos@dtu.dk

Gamma spectrometry, liquid scintillation counting, Geiger-Muller counting and alpha spectrometry are various ways by which ^{210}Pb, ^{210}Bi and ^{210}Po may be determined directly or indirectly. The choice of tracers and the ways they are determined as well as the choice of radiochemical separation methods opens up for a wide selection of possible ways to quantify the isotopes of interest. The presentation gives a summary of the methods used at DTU Nutech to isolate and measure these radioisotopes in various type of samples ranging from biological samples to solid lead.

The comparison of two liquid scintillation instruments for analysis of highly quenched samples.

Sarah Mullins [1,2], Megan Cook [1]

[1] Radiation and Nuclear Science, Department of Health, 39 Kessels Road, Coopers Plains QLD, 4108, Australia

Corresponding author's e-mail: Sarah.Mullins@health.qld.gov.au

Leachate derived from toxicity characteristic leaching procedure (TCLP) often contains soil particles which quench liquid scintillation spectra.

In this research, we compare results obtained from two instruments: the 1220 Quantulus Ultra Low Level Liquid Scintillation Spectrometer and the Aloka AccuFLEX LSC-LB7.

The method was performed on a solid sample with leaching fluids of pH 2.9, 5, 7 and 9.3. The solid sample and the leaching fluid were added to a 2L polyethylene extraction container and rotated in an end-over-end fashion at 30±2 revolutions per minute for 16-18hrs. Leachate solution was filtered through a glass fibre filter with an effective pore size of 0.5µm. The leachate solution underwent evaporation and reconstitution before addition of liquid scintillation cocktail and LSC analysis.

When final results have been obtained they will be used for establishing best practice in-house methods for application to government legislation and public health.

Subsequent determination of ^{90}Sr and ^{210}Pb in goat bones samples from Island of Mljet, Croatia

NODILO Marijana [1)], DULANSKÁ Silvia [2)], MÁTEL Ľubomír [2)], GRAHEK Željko [1)]

1) Ruđer Bošković Institute, Laboratory for Radioecology, Bijenička c. 54, 10 000 Zagreb, Croatia
2) Comenius University, Department of Nuclear Chemistry, Mlynská Dolina, Ilkovičova 6, 842 15 Bratislava, Slovakia

Corresponding author's e-mail: marijana.nodilo@irb.hr

The activity concentrations of ^{90}Sr and ^{210}Pb were determined in the bones of the goats from the island of Mljet, Croatia. The goats have been a generous food supply for centuries on this Croatian island, which is scarce of any abundant food source.

Strontium is a calcium chemical homology. Lead can easily and irreversible depot in bones. Consequently, they readily deposit in the bones during a life of an animal. Such bones can be used as a part of an environmental monitoring.

For every analysis, 10g of bone ash was used. Sequential separation of strontium and lead in the samples of the goat bones employing Eichrom´s extraction chromatography product Sr Resin, and solutions of nitric and hydrochloric acids, allows the determination of anthropogenic radionuclide ^{90}Sr and naturally occurring radionuclide ^{210}Pb with good chemical yields and detection limits. After separation ^{90}Sr was detected by Cherenkov radiation and ^{210}Pb by classical LSC detection. Determinated activities were from MDA (2.6 Bq/kg) to 60.5 Bq/kg for ^{90}Sr and from 8.4 to 95.1 Bq/kg (MDA 1.9 Bq/kg) for ^{210}Pb.

The use and the effectiveness of Sr Resin were successfully verified by analysing a certified reference material and reliable results have been obtained.

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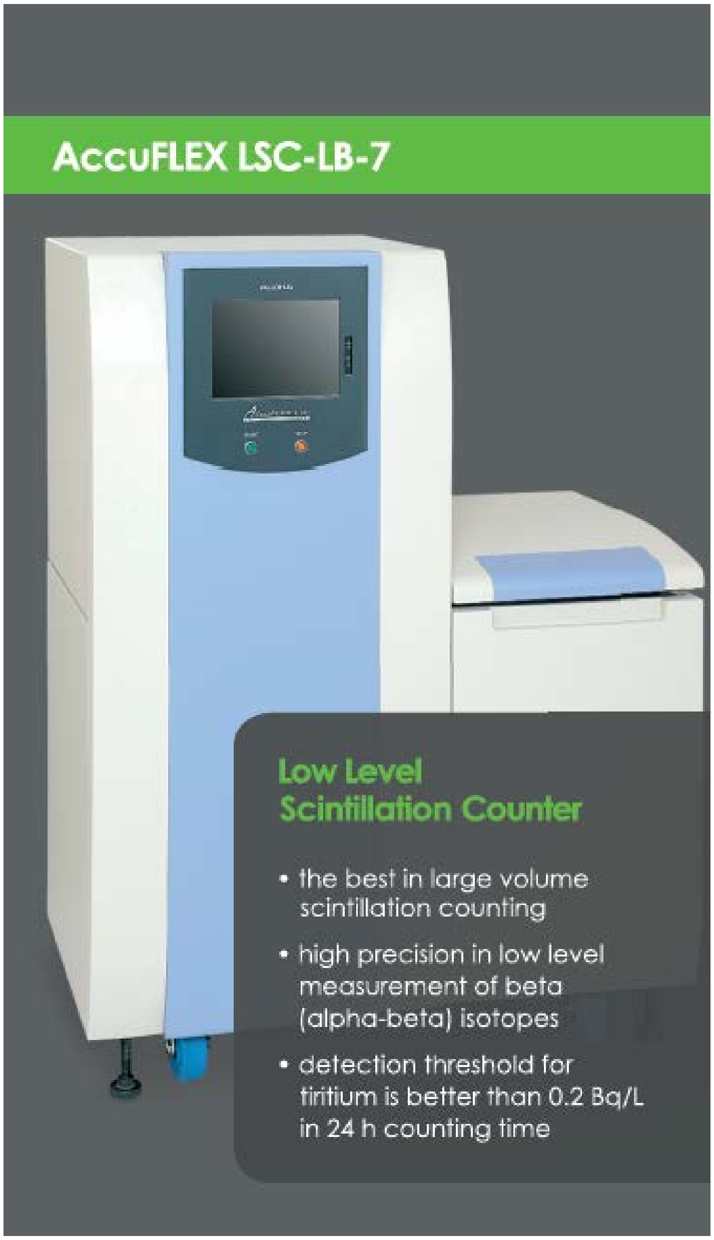


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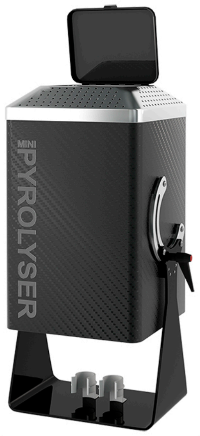

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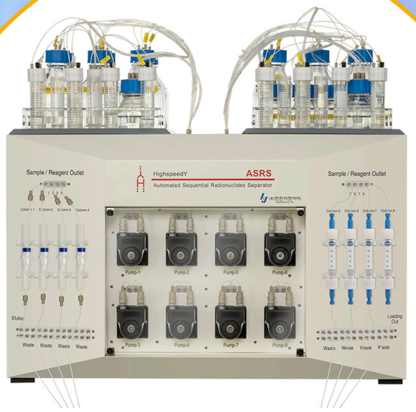

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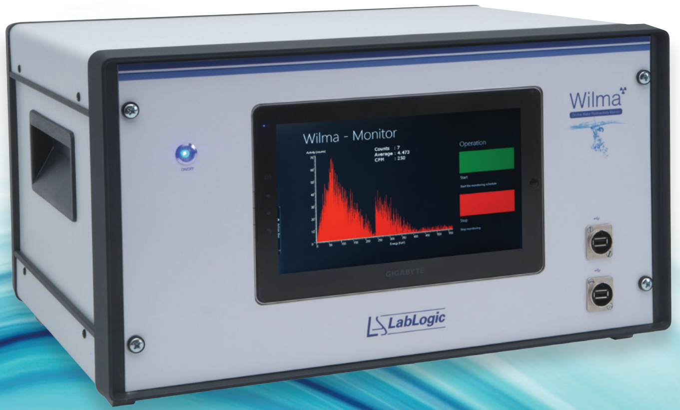

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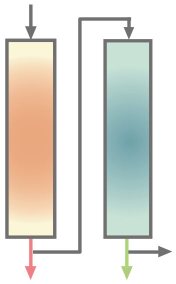

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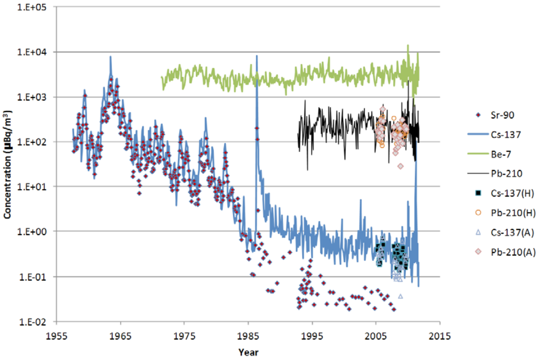

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