ISSN 1018-5593; 1681-5084

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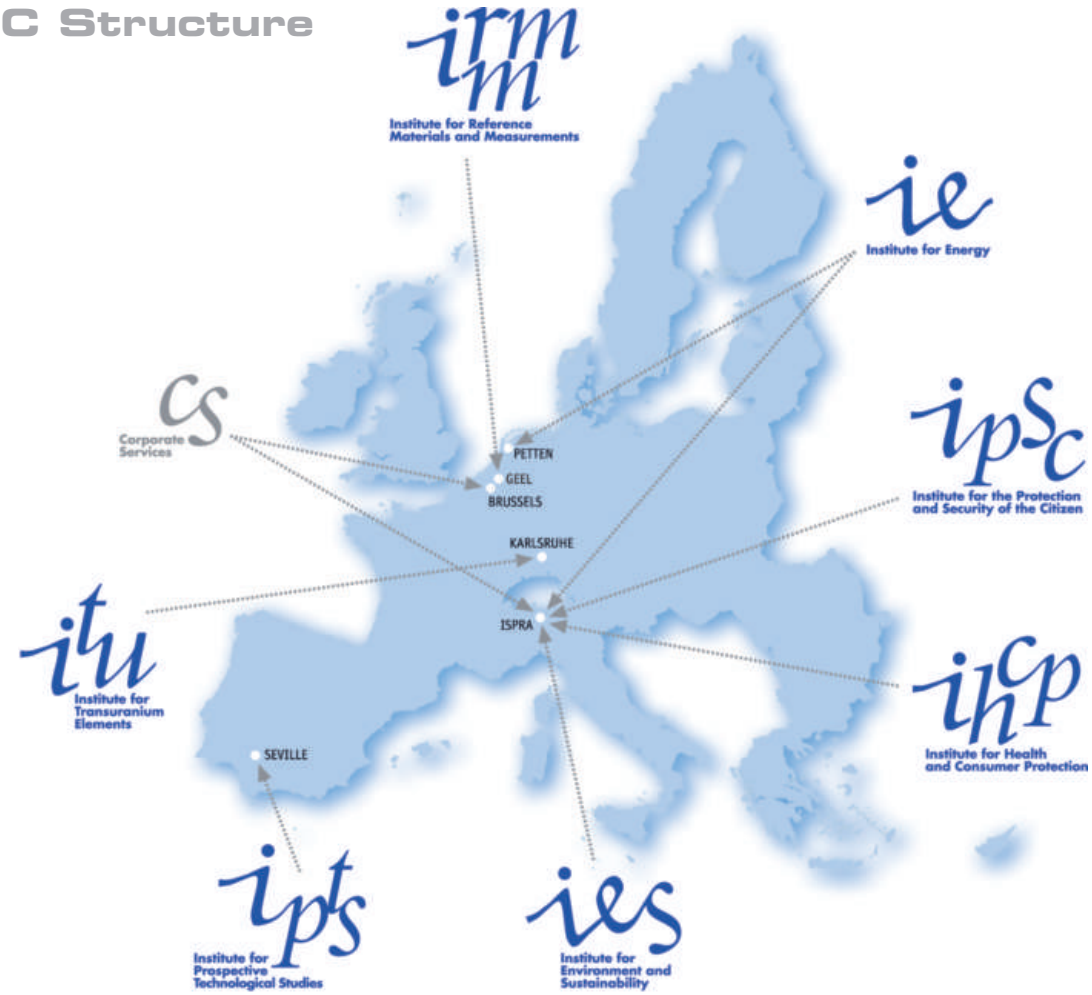
Institute for Reference Materials and Measurements

ANNUAL REPORT 2009

JRC
EUROPEAN COMMISSION
Report EUR 24246 EN

JRC Structure



The Institute for Reference Materials and Measurements

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IRMM – Confidence in measurements®

The vision for the JRC-IRMM is to be the European Commission reference, providing confidence in measurements in support of EU policies.

The mission of the JRC-IRMM is to promote a common and reliable European measurement system in support of EU policies.

JRC

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EUR 24246 EN
ISBN 978-92-79-15010-4
ISSN 1018-5593; 1681-5084
JRC59643
doi 10.2787/23251

Luxembourg: Publications Office of the European Union

Printed in Luxembourg


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in measurements®

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Message from the Director

I was extremely pleased to take up the Directorship of the JRC-IRMM on 1 November 2009, following the retirement of Alejandro Herrero Molina. I'd like to pay tribute to Alejandro's seven years at the helm, and I have inherited a well-managed institute with dedicated and exceptionally competent staff.

Prior to becoming Director of JRC-IRMM, I was the JRC Director of Programmes and Stakeholder Relations since February 2007. Before joining the European Commission, I was the Director of an institute related to material sciences at the Wroclaw University of Technology, in my native Poland.

In 2009, the JRC took on the challenge of reinvigorating its organisation by the creation of a new vision and strategy. The JRC's new vision is to be a trusted provider of science-based policy options to EU policy makers to address key challenges facing our society, underpinned by internationally recognised research. Reflection on the JRC's strategy continued throughout 2009, and the strategy will be adopted and followed by an implementation plan in 2010.

The thematic area of reference materials and measurements will be one of the seven priorities of the strategy, and the institute will consolidate its role in promoting a common and reliable European measurement system in support of EU policies. Construction work continued in 2009 on a new building designed principally for the production of reference materials. It will be the largest building on the JRC-IRMM site. The building also comprises laboratories for elemental and protein analysis, a class-three biosafety laboratory, offices and new meeting rooms, and is on schedule to open in the autumn of 2010.

The refurbished linear electron accelerator facility (known as GELINA) was officially inaugurated by JRC Director-General, Roland Schenkel, in October 2009 – thus marking the end of a series of improvements made over several years. GELINA is now equipped with modern state-of-the-art systems adapted to the demands of present-day neutron data experiments, offering significant operational advantages such as increased throughput of neutron data measurements and greater precision.

Also in 2009, a new collaboration agreement was signed by JRC-IRMM (representing the European Atomic Energy Community) and the Institut National de Physique Nucléaire et de Physique des Particules in France, covering common interests such as the improved understanding of neutron emission reactions and the fission process, developments in experimentation and sample fabrication and characterisation.

A new three-year project was launched in 2009 under the stewardship of JRC-IRMM which will bring together European and Turkish experts in measurement science. The project is funded by the European Union under the instrument for pre-accession assistance (IPA). Through the project, JRC-IRMM will provide consultancy, training and support to metrological institutes in Turkey. JRC-IRMM will also provide training in the field of chemical and ionising radiation metrology to laboratories and universities, in order for them to optimise the use of services they obtain from the national metrology institute, and to help them incorporate metrology-related subjects into educational curricula.

I'd like to thank my staff at the institute. It is only thanks to their scientific, technical and administrative competence that the JRC-IRMM is able to drive forward with its important mission – providing confidence in measurements in support of EU policies.

Krzysztof Maruszewski
Director JRC-IRMM

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About the institute

The Institute for Reference Materials and Measurements (IRMM) is one of the seven Institutes of the Joint Research Centre (JRC), a Directorate-General of the European Commission, providing independent scientific and technical support to Community policy-making. The JRC-IRMM was founded in 1957 under the Treaties of Rome and started operation in 1960 under the name of the Central Bureau for Nuclear Measurements (CBNM). Today, JRC-IRMM is one of the world's leading reference material producers, expert adviser in food safety and quality and bioanalysis as well as a valued provider of reference measurement data. Its management system is certified according to ISO 9001, ISO 14001 and OHSAS 18001 and its scientific units hold several accreditations.

JRC-IRMM is one of the world's leading reference material producers, and 21 new reference materials were released in 2009.

Mission and tasks

JRC-IRMM promotes a common and reliable European measurement system in support of EU policies. The primary task of JRC-IRMM is to build confidence in the comparability of measurement results by the development, production and dissemination of internationally accepted quality assurance tools, namely validated measurement methods, reference materials, proficiency testing and reference measurements.

JRC-IRMM participates in the activities of the international metrology organisations such as the International Committee for Weights and Measures (CIPM) and the network of European metrology institutes (EURAMET). Through an agreement with the European Co-operation for Accreditation (EA), JRC-IRMM helps to improve the measurement capabilities of hundreds of laboratories in all Member States. JRC-IRMM staff also contribute actively to the work of standardisation bodies like the European Committee for Standardisation (CEN) and the International Organization for Standardisation (ISO). The JRC-IRMM operates four European Union reference laboratories (EU-RLs), formerly called Community reference laboratories.

Core competencies

The core competencies of JRC-IRMM are development, production and distribution of reference materials, development and validation of methods for food and feed analysis, bioanalysis, isotopic measurements, neutron physics and radionuclide metrology. These competencies are applied in a variety of research fields: food and feed safety and quality, biotechnology, sustainable agriculture, environment, health, nanotechnology and nuclear safety and security. The scientific knowledge base of JRC-IRMM is acquired and maintained by both fundamental and applied research in the respective areas.

JRC-IRMM staff are members of numerous committees, their working groups and scientific boards of international organisations. JRC-IRMM's work in the field of metrology and standardisation is widely recognised. For instance, various technical committees of ISO use expert advice of JRC-IRMM on reference materials for their specific application fields, and JRC-IRMM experts participate actively in the work of AOAC International, VAMAS, IFCC, etc. Many testing methods validated by JRC-IRMM together with its collaborators have been approved as standards of CEN and ISO.

Glove-box with an overpressure of argon, being used to prepare polyimide foils.

Special infrastructure

The research facilities include multi-functional and flexible laboratories for the development and production of reference materials and advanced analytical laboratories. The JRC-IRMM laboratories are well equipped for carrying out demanding tasks whether to solve a food related or an isotope measurement problem. The dedicated facilities for reference material production are able to handle large amounts of various types of materials, even those hazardous for health. Controlled storage conditions for all materials are available. The radionuclide metrology laboratory houses instrumentation for extremely accurate radioactivity measurements and small amounts of radioactive substances can be studied in the underground laboratory of JRC-IRMM located at the Belgian Nuclear Research Centre (SCK•CEN) in Mol, Belgium.

Checking the reference points before operating a milling machine in the workshop.

JRC-IRMM operates a 150 MeV linear electron accelerator (GELINA) and a 7 MV light-ion Van de Graaff accelerator. The two accelerators of JRC-IRMM, used for neutron production, are complementary in their experimental conditions and among the best such installations in the world. In the recent review of the JRC - chaired by Sir David King - the GELINA accelerator of JRC-IRMM was cited as one of the "efficient facilities absolutely necessary for the European nuclear research programme". The two accelerators can accommodate external users via a project on access to large scale facilities (EUFRAT).

Site developments in 2009

Construction work continued in 2009 on a new building designed principally for the production of reference materials. It will be the largest building on the JRC-IRMM site. The building also comprises laboratories for elemental and protein analysis, a class-three biosafety laboratory, offices and new meeting rooms, and is on schedule to open in the autumn of 2010.

In Belgium, all public forests and private forests larger than 5 hectares must develop a "forest management plan". The JRC-IRMM grounds are approximately 38 hectares, of which 25 hectares is wooded, so an extended forest management plan was approved and launched in 2009. A key target of the plan – which runs for 20 years – is to achieve at least 20% of domestic tree species by the end of planning period.

In 2009, good progress was made with the construction of the new building at JRC-IRMM. The reference materials production building will be the largest on site, and it is on schedule to be opened in the autumn of 2010.

The JRC-IRMM grounds are approximately 38 hectares, of which 25 is wooded area. In 2009, a forest management plan was approved. A key goal of the plan – which runs for 20 years – is to achieve at least 20% of domestic tree species by the end of planning period.

3 Management team

Director
Krzysztof Maruszewski

Reference materials
Hendrik Emons

Neutron physics
Peter Rullhusen

Food safety and quality
Franz Ulberth

Isotope measurements
Philip Taylor

Management support
Doris Florian

Infrastructure and site management
Marc Wellens

Informatics and electronics
Bartel Meersman

Safety, Health, Environment & Security
Pierre Kockerols

(Situation December 2009)

Facts & Figures

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Staff in 2009:

	male	female	total
officials	136	78	214
PhD and post-doctoral fellows	16	29	45
fixed-term employees	13	11	24
seconded national experts	2	0	2
trainees	0	1	1
Total	**167**	**119**	**286**

Budget in 2009:

JRC-IRMM is funded by the JRC budget from the EU Framework Programmes for Research and Technological Development, both of the European Community and the European Atomic Energy Community (EURATOM). The institutional budget is supplemented with competitive income.

Competitive income (k€) in 2009	
Contracts with other DGs	1 987
Participation in indirect actions	921
Work for third parties	724
Reference material distribution	2 177
Total	**5 809**

Publications in 2009:

A large part of the research done at JRC-IRMM is reported in scientific publications and is publicly available. In addition to articles published in refereed scientific journals and conference proceedings, valuable information can be found in the Scientific and Technical Research series. A bibliographic list of selected scientific and technical articles and reports is given at the end of this report.

Publications 2009	
Monographs and articles	81
JRC Scientific and Technical Reports	45
Contributions published in conference proceedings	24
PhD theses	3
Total	**153**


ERM-BF428a
Sample No: 0000
GHB119 Cotton
ERM-BF427c
Sample No: 0000
9140 Maize
ERM-BF427a
Sample No: 0000
ERM-BF413g
Sample No: 0000
MON 810 Maize
ERM-BF428c
Sample No: 0000
GHB119 Cotton
ERM-BF413
Sample No: 0000
MON 810 Maize
ERM-BF428b
Sample No: 0000
GHB119 Cotton
ERM-BF427b
Sample No: 0000
Maize
ERM-BF427d
Sample No: 0000
Maize
ERM-BF413a
Sample No: 0000
MON 810 Maize

5 Reference materials

Introduction

The reliability and comparability of analytical and testing data across Europe – and globally – greatly depend on quality assurance tools, in particular, certified reference materials. Reference materials are needed for developing, calibrating, validating and controlling methods of analysis. Today, reference materials are needed to fulfil the requirements of standards for the accreditation of testing and calibration laboratories. JRC-IRMM is one of the leading producers of certified reference materials in the world, particularly in the clinical, food, GMO and nuclear application areas.

JRC-IRMM released 21 new certified reference materials in 2009, covering fields of application such as clinical chemistry, microbiology, genetically modified organisms and nuclear safeguards.

In September 2009, the institute organised and hosted the first global encounter of various institutes producing chemical reference materials. Participants came from the USA, South Korea, Canada, Australia, China, South Africa, Japan, Germany and the United Kingdom. The representatives discussed common challenges in the field, and agreed to forge a network for exchanging early-stage information on their development programmes.

Hendrik Emons, Head of the Reference Materials Unit, received the Reference Material Achievement Award from AOAC INTERNATIONAL. His nomination cited JRC-IRMM's work on the development, production and certification of certified reference materials (CRMs) for a broad range of application areas.

Hendrik Emons receives the Reference Material Achievement Award from Darryl M. Sullivan, President of AOAC INTERNATIONAL (September 2009).

5.1 First ever higher-order reference materials for genetic testing

In 2009, three reference materials produced by JRC-IRMM for genetic testing became the first ever reference materials to be accepted and nominated as higher-order reference materials in the area of genetic testing.

The reference materials (IRMM-490/IFCC, IRMM-491/IFCC and IRMM-492/IFCC) were developed and produced by JRC-IRMM in cooperation with the International Federation of Clinical Chemistry and Laboratory Medicine (IFCC). They are plasmid-based materials, suitable for the quality control of the identification of a human genetic mutation. This single point mutation (G20210A) is located in the gene coding for the human prothrombin (Factor II) and was identified as one of the risk factors in venous thrombosis events.

At international level, the Joint Committee for Traceability in Laboratory Medicine (JCTLM) reviews whether reference materials used in the clinical field can be nominated to be higher-order reference materials. This committee was established through the cooperation of three organisations: the Bureau International des Poids et Mesures (BIPM), the International Federation of Clinical Chemistry and Laboratory Medicine (IFCC) and the International Laboratory Accreditation Cooperation (ILAC).

A higher-order reference material for in vitro diagnostics needs to fulfil the requirements laid down in ISO 15194 and ISO 15193. The reference materials of JRC-IRMM mentioned above are now listed in the JCTLM database for higher-order reference materials, and are the first CRMs for nominal properties in this database.

Digital polymerase chain reaction (PCR) for the quantification of a DNA copy number ratio.

5

5.2 New microbiological reference materials

JRC-IRMM has extended the range of microbiological reference materials based on the BioBall® approach by releasing two new certified reference materials. They are intended to help analytical laboratories develop and validate testing methods for *Candida albicans* (the most common, opportunistic fungal pathogen in humans) and *Enterococcus faecalis* (also an opportunistic bacterial pathogen).

Under normal circumstances, *Candida albicans* lives in the mouth and gastrointestinal tract of 80% of the human population with no harmful effects. However, it becomes an infectious agent when there is some change in the body's environment that allows it to grow out of control. The resulting condition is known as candidiasis moniliasis, or a "yeast" infection.

Reference material for analysing Candida albicans, *which lives in the mouth and gastrointestinal tract of 80% of the human population.*

Enterococcus faecalis inhabits the gastrointestinal tracts predominantly of humans, but also of many animals. Because of their ability to acquire high level resistance to antimicrobial agents, enterococci have emerged as nosocomial pathogens worldwide. Nosocomial infections are infections which are acquired in a hospital or a healthcare service unit, but which are not secondary to the patient's original condition. *Enterococcus faecalis* causes 80-90% of human enterococcal infections.

Measurement issues at low levels of colony forming unit (CFU) and practical limitations in the use of previous certified reference materials for microbiology prompted the search for alternative approaches. JRC-IRMM cooperated with BTF in Australia, who prepared materials with a precise number of bacterial cells into a single BioBall® using a modified flow cytometer. The BioBall® format can be conveniently used with common microbiological plating methods without having to perform additional sample preparation steps.

The resulting reference materials, IRMM-354 and IRMM-355, contain *Candida albicans* and *Enterococcus faecalis*, respectively, at a level of approximately 900 colony forming units per material sphere. The certified values are based on interlaboratory characterisation studies.

5.3 Supporting legislation on banned flame retardants

In 2009, JRC-IRMM released two new certified reference materials (CRMs) to help analytical laboratories better detect two classes of flame retardants banned under the RoHS Directive[1]. Polybrominated flame retardants are used in a wide variety of products to inhibit or resist the spread of fire, for example, paper, furniture upholstery and electrical and electronic devices.

These chemicals can bio-accumulate in blood, breast milk, and fat tissues, and they are reported to impair the development of the nervous system and cause hormonal imbalances. For this reason, the European Union decided to ban the use of two classes of flame retardants, namely polybrominated diphenyl ethers (PBDEs) and polybrominated biphenyls (PBBs) in electric and electronic devices.

This ban was formalised in the RoHS Directive, and an upper limit of 1 g/kg for the sum of PBBs and PBDEs was set. Several intercomparisons at international level showed that the current quality of measurement results was not sufficient to reliably enforce the 1 g/kg limit. JRC-IRMM therefore produced two certified reference materials (CRMs) for the determination of PBDEs and PBBs in polymers. The two reference materials (ERM-EC590 and ERM-EC591) were custom made to contain all relevant PBDEs and PBBs at levels close to the legal limit. Certified values were assigned for 10 PBDEs and PBBs, and additional information for another 18 substances is provided. In addition, the total bromine content was certified, as the measurement of total bromine is often used as a screening method for brominated flame retardants.

1 RoHS Directive (2002/95/EC): Restriction of the use of certain hazardous substances in electrical and electronic equipment.

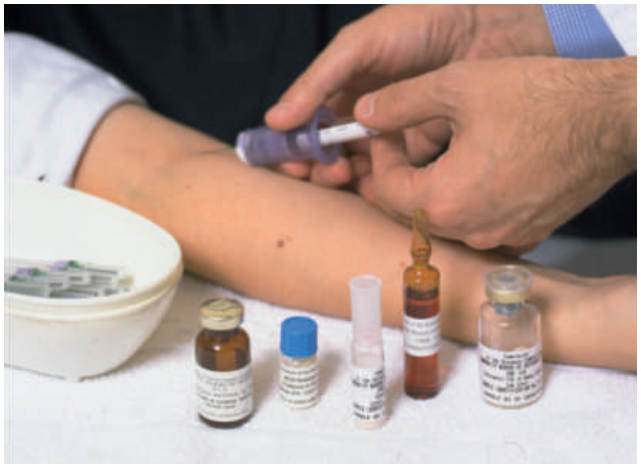
Certified reference materials from JRC-IRMM, intended to help analytical laboratories better detect two classes of flame retardants banned under the RoHS Directive.

The two materials are intended as quality control tools for analytical laboratories. Data obtained from measuring these CRMs can identify laboratories' shortcomings and generally lead to increased competence in measuring these flame retardants. Additionally, laboratories can use these materials to demonstrate the accuracy of their measurements, thus increasing the confidence in the results and contributing to an effective implementation of the RoHS Directive.

5.4 Reference materials for clinical chemistry

JRC-IRMM released a new reference material certified for C-reactive protein (CRP), which is a very sensitive marker of inflammation and tissue damage. CRP is frequently used to diagnose bacterial and viral infections, to assess disease activity in inflammatory conditions like rheumatoid arthritis, and to determine the long-term risk of cardiovasular disease and heart attacks, making it one of the most important analytes in clinical chemistry.

The reference material, ERM-DA472/IFCC, is the successor of the ERM-DA470 – which was used worldwide as the standard for CRP. After the release of ERM-DA470, manufacturers of in vitro medical devices (IVD) began referencing their calibrants and controls to that material, and the measurement discrepancy between laboratories became substantially lower. The new material will ensure continuity in the standardisation of CRP, which is crucial in clinical chemistry.



The new reference material for C-reactive protein is intended to be used to assign values to calibrators that are an integral part of in vitro *diagnostic medical devices.*

The reference material is intended to be used to assign values to calibrators that are an integral part of in vitro diagnostic medical devices (IVD). The EU Directive on In Vitro Diagnostic Medical Devices (IVD-MD) (Directive 98/79/EC) requires traceability of calibrants and control materials to reference measurement procedures and/or reference materials of higher order. The International Federation of Clinical Chemistry (IFCC), which actively supports standardisation in clinical chemistry, has collaborated with JRC-IRMM on the development of ERM-DA472/IFCC.

5

5.5 Uranium and plutonium spikes to control irradiated nuclear fuel

Large-sized dried spikes (LSD) have become a fundamental part of the fissile material control of irradiated nuclear fuel. They are provided to the European Commission Directorate-General for Energy as a fundamental part of the fissile material control of irradiated nuclear fuel at the on-site laboratories in Sellafield (UK) and La Hague (France).

A new set of LSD spikes (IRMM-1027m) for the determination of uranium and plutonium by isotope dilution mass spectrometry in solutions of spent fuel from reprocessing plants has been prepared and certified for uranium and plutonium isotopic contents.

The new batch of large-sized dried spikes contains circa 50 mg of uranium with an ^{235}U amount fraction of 19.5% and circa 1.8 mg of plutonium with a ^{239}Pu fraction of 97.8% in each individual vial, covered with a thin layer of organic material (cellulose acetate butyrate) as a stabilizer. The uranium and plutonium content was certified based on values from mass metrology of a validated automated system. Verification of the contents of the spike was done by isotope dilution mass spectrometry at JRC-IRMM. The values measured for the dried covered spikes agreed well with those calculated from the weights of starting materials dissolved and the weights of the final solution.

In 2009, JRC-IRMM produced new large-sized dried spikes (IRMM-1027m) for nuclear safeguards verification.

5.6 Compatibility study of plutonium isotopic reference materials

Measuring amounts of plutonium is recognised as one of the most important tasks in fissile material control. Analysts have the difficult task of measuring plutonium over a range of levels, from large (multi-gram) amounts down to traces in the environment. JRC-IRMM is one of the few institutes worldwide that prepares and certifies plutonium reference materials. These materials are used by European and international safeguards authorities and nuclear laboratories for fissile material control.

Flame sealing of quartz ampoules for uranium isotope reference materials.

An interlaboratory calibration campaign using state-of-the art measurement procedures was carried out to demonstrate the suitability and performance of JRC-IRMM's isotopic plutonium reference materials for safeguards verification and environmental measurements. The traceability of the certified values of the plutonium isotopic contents to the International System

of Units (SI) was established and the new batch of uranium and plutonium large-sized dried spikes (IRMM-1027m) was confirmed as a primary reference material.

The outcome of the exercise underlines JRC-IRMM's leading role as a provider of isotopic plutonium reference materials.

Loading of a solution of certified plutonium isotope reference material for mass spectrometric analysis.

5.7 Developing documentary standards

In 2009, JRC-IRMM made significant contributions to the revisions of international standards, including ISO Guide 34, ISO 15193 and ISO 15194.

ISO Guide 34 describes the general requirements for reference material producers. Adherence to this guide ensures that reference materials, which include certified reference materials (CRMs) are produced according to the same principles worldwide. JRC-IRMM chaired the working group for the revision of ISO Guide 34. It clarified a number of issues, such as the necessary extent of method validation as well as metrological traceability of assigned values and of the measurement results upon which they are based.

ISO 15193 and ISO 15194 are mandated standards and related to Directive 98/79/EC on *in vitro* diagnostics devices (IVD). Both standards were due for revision according to the ISO scheme. JRC-IRMM acted as a liaison to the ISO Committee on Reference Materials (ISO/REMCO) and significantly contributed to the revision process. The revised ISO 15193 and ISO 15194 standards were released in 2009.

ISO 15193 concerns the requirements for the content and presentation of reference measurement procedures, which are key in achieving the traceability to higher order reference materials and methods as required by the IVD Directive. The standard was updated according to the newest related ISO references. Technical aspects, such as the relationship between a reference method and reference measurement procedure, have been clarified.

ISO 15194 originally addressed the description of reference materials, and underwent substantial changes in the new revision. The standard now focuses on the requirements for certified reference materials for *in vitro* diagnostics and the content of supporting documentation. It has been brought in line with the ISO Guide 30-35 series, which are the most relevant standards related to the production and certification of reference materials.

Nuclear physics

Introduction

Many issues related to nuclear safety and security require accurate calculations using nuclear data. Measurements of neutron-induced reactions and cross-section standards, and absolute measurements of radiation i.e. radionuclide metrology, have been key activities of the JRC-IRMM since it started operation in 1960. JRC-IRMM focuses on neutron data for standards, safety of operating reactors, handling of nuclear waste and waste transmutation and the design of new and different types of reactor systems and fuel cycles.

Measurements carried out at its linear electron accelerator facility (known as GELINA) and the Van de Graaff facility have played a significant role in establishing and improving the evaluated nuclear data file maintained at the databank of the Nuclear Energy Agency (NEA) of the Organisation for Economic Cooperation and Development (OECD).

In 2009, the refurbished GELINA facility was officially inaugurated - marking the end of a series of improvements made over several years. Numerous major systems were upgraded, including the control room, the injector, the electrical power distribution system and the magnet power supplies. Due to the specialised nature of the work, most of the design, development and installation work was done in house by JRC-IRMM staff. GELINA is now equipped with state-of-the-art systems adapted to the demands of present-day neutron data experiments, offering significant operational advantages such as increased throughput of neutron data measurements and greater precision.

Checking the electronics settings in GELINA' s injector pulsing system. During operation, these electronics are working on a hot deck at 80,000 volts above earth potential.

JRC Director-General, Roland Schenkel, inspects a control panel of the refurbished linear accelerator during its inauguration (12 October 2009).

The Euratom transnational access project, 'European facility for innovative reactor and transmutation neutron data' (EUFRAT), enables outside users to access JRC-IRMM's accelerator facilities. In 2009, two calls for proposals were launched, which resulted in 17 approved projects.

Also in 2009, a new collaboration agreement was signed by JRC-IRMM (representing the European Atomic Energy Community) and the Institut National de Physique Nucléaire et de Physique des Particules in France, covering common interests such as the improved understanding of neutron emission reactions and the fission process, developments in experimentation and sample fabrication and characterisation.

Thin uniform deposits are needed as targets for JRC-IRMM's neutron data measurements, and the target preparation activity has seen a revival in recent years at JRC-IRMM. Following the production of active targets (^{235}U) last year, thin deposited layers of ^{6}LiF, Au and ^{10}B were prepared and characterised by physical vacuum deposition in 2009.

Preparation and characterisation of thin film nuclear targets by vacuum evaporation.

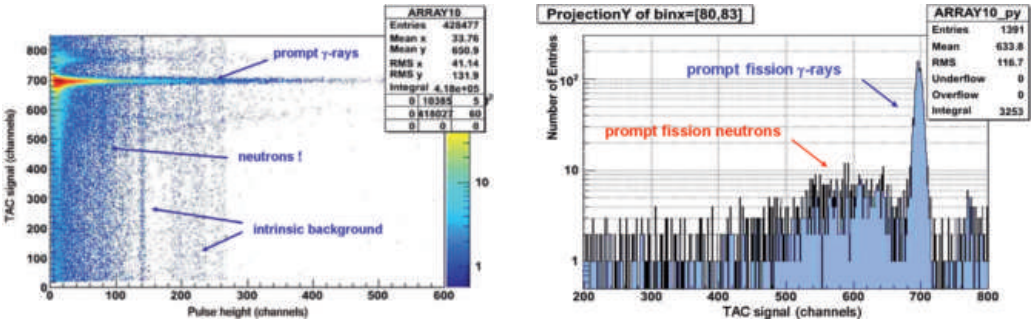
6

6.1 Lanthanum halide detectors for unprecedented accuracy of fission gamma-ray measurements

Inside a nuclear reactor, the nuclear fission process is accompanied by the emission of prompt neutrons and γ-rays. The latter quantity is extremely important for the development of next generation nuclear reactors, in particular for making accurate heat calculations and detailed risk analyses. A specific challenge for the modelling of new generation reactor neutron kinetics is the calculation of the γ-heat deposition, e.g. in steel and ceramic reflectors without uranium oxide blankets, which must be known with an accuracy as low as 7.5%.

A promising approach is the use of recently developed cerium-doped lanthanum halide crystal scintillation detectors in conjunction with an ultra-fast fission event trigger based on artificial diamonds.

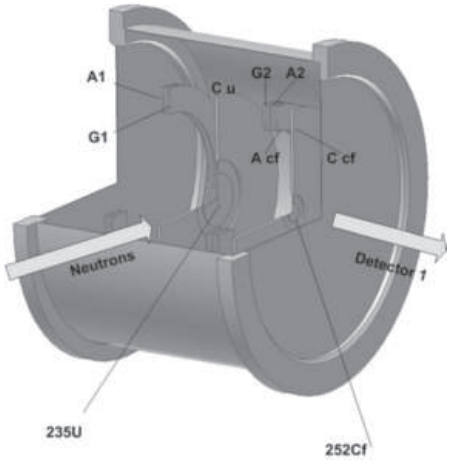
At the JRC-IRMM Van de Graaff facility, state-of-the-art lanthanum-halide detectors were successfully introduced and characterized with the goal of measuring prompt fission γ-ray data with unprecedented accuracy. In conjunction with artificial diamond detectors as fission event triggers, coincidence timing resolution better than 1ns was achieved, providing excellent neutron/γ-ray separation by means of time-of-flight. The very good energy resolution, which is better than 4% at 662 keV, enables the investigation of the impact of isomeric γ-decay from fission fragments on the measured prompt emission spectra.



Prompt fission neutron and γ-ray time-of-flight versus detector pulse height (left); the excellent separation of prompt fission γ-rays from prompt neutrons is achieved by means of both time-of-flight (right) and corresponding pulse height.

6.2 Prompt fission neutron spectrum of uranium-235 at thermal energies

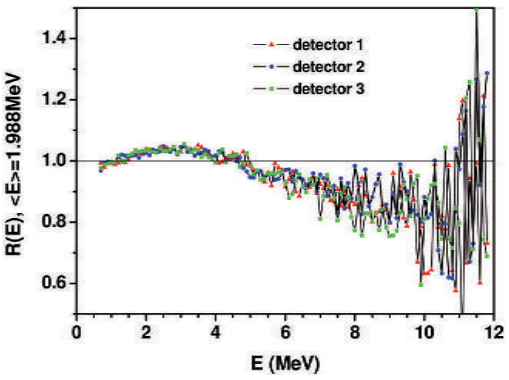
Since ^{235}U is the most important isotope for nuclear energy production, it is imperative to understand its prompt fission neutron spectrum. The prompt fission neutron spectrum from the ^{235}U(n,f) reaction has been investigated in many experiments at different incident neutron energies from thermal to the fast region. These efforts were motivated by an unresolved contradiction between microscopic and macroscopic data (integral average cross section and Keff experiments) which still exists today.



Schematic of the fission ionisation chamber showing the cathodes with U and Cf neutron sources (C_u, C_{cf}), the anodes and grids for the U section of the ionisation chamber (A_1, A_2, G_1,G_2) and the anode for the Cf section (A_{cf}).

The prompt fission neutron spectrum was recently measured using the time-of-flight technique at the cold-neutron prompt-gamma activation analysis (PGAA) facility at the Budapest Nuclear Research Reactor at 100 K incident neutron energy. This investigation was done to verify JRC-IRMM's results at the Van de Graaff facility at 0.5 MeV incident neutron energy, and also literature data measured over the past 20 years. The literature data contradicts the theoretical description of the spectrum, as well as integral and benchmark experiments.

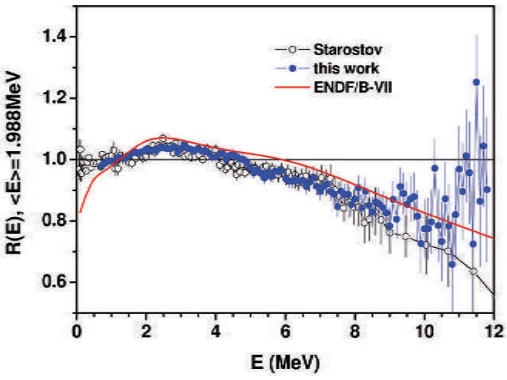
Three detectors at different angles, carefully shielded against scattered neutrons, were used in the Budapest experiment. A thin ^{235}U sample inside an ionisation chamber was used together with a ^{252}Cf sample as a reference standard. The results showed no difference in spectrum shape between the three different neutron detectors, thus the angular anisotropy of the prompt fission neutron spectrum observed at the Van de Graaff experiment could not be reproduced.



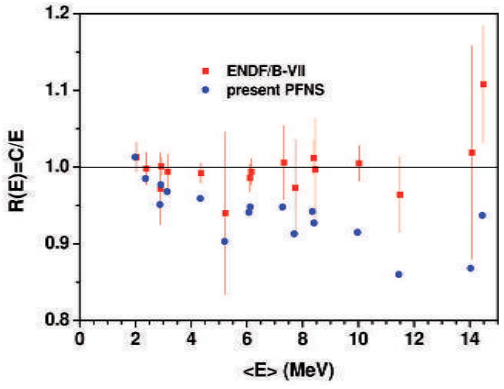
The prompt fission neutron energy spectra of ^{235}U measured with the three different detectors at the Budapest research reactor and compared to a Maxwellian distribution with mean energy of 1.988 MeV.

A comparison of the results with literature values was plotted. The measured average spectrum agrees (within the error bars) with data from Starostov *et al.* in the energy range 0.7-11 MeV. However, the present results – as well as the results in literature – contradict what's known as the the ENDF/B-VII evaluation (full line). The theoretical model – which is based on assumption of neutron emission from fully accelerated fragments – does not produce the newly measured spectrum shape. Hence a new approach is proposed which also includes scission neutron emission.

The data of the IRDF-2002 dosimetry library and experimental average cross sections were used to validate the presently measured prompt fission neutron spectrum. Only the threshold reactions which provide good agreement between calculated and experimental data for ^{252}Cf were used. Together with the average cross sections, the average energies of the reaction response were calculated. The results were compared to the same calculations based on the ENDF/B-VII evaluation. A conclusion can be drawn that the measured prompt fission neutron spectrum of U – which is in excellent agreement with microscopic experimental data from literature – cannot describe the integral experiments.



Comparison between the present results averaged over all detectors and the ones of Starostov et al. *The data are shown as a ratio to a Maxwellian distribution with mean energy of 1.988 MeV.*



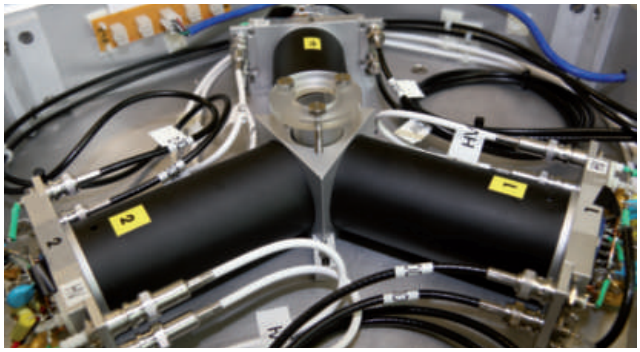
Ratio of calculated to measured values for different prompt fission neutron spectra for ^{235}U, versus the average energy of the reaction response.

Discussion has started with theoretical groups at Los Alamos National Laboratory and at Commissariat à l'énergie atomique (CEA) in Cadarache to identify the problems in the model description. Further experiments are planned at the Van de Graaff facility using digital signal acquisition to shed light on the existence of the angular anisotropy as well as the Budapest reactor to verify certain integral reaction cross sections.

6.3 New spectrometer for primary activity standardisation

JRC-IRMM has recently designed and constructed a triple-to-double coincidence ratio spectrometer to complement measurements currently being carried out using the so-called CIEMAT/NIST 3H-efficiency tracing method and to be used as an additional technique for primary activity standardisation.

The triple-to-double coincidence ratio (TDCR) method is a method in liquid scintillation counting (LSC) developed for the determination of the activity of pure β-particle and electron capture radionuclides. It is a direct method for radioactivity measurements; there is no need for calibration with radionuclide standard sources as in the conventional LSC methods.



The triple-to-double coincidence ratio (TDCR) liquid scintillation spectrometer.

However, the decay scheme data for a given radionuclide are necessary for the detection efficiency calculation. The method itself is based on a physical and statistical model of the distribution of scintillation photons and their detection probability in a three-photomultiplier counter.

The three photomultipliers of the system are connected to voltage divider bases specifically developed with direct access to the focusing grid to allow for easy defocusing and anode and 9^{th} dynode outputs to simultaneously acquire signals for counting and for spectrum analysis. The electronics chain consists of fast amplifiers and constant fraction discriminators, while the double and triple coincidences are formed by a four-fold logic unit. The data acquisition is based on the Modular Multiparameter Multiplexer and the DAQ2000 system developed earlier at JRC-IRMM. Computer programs to control the instrument, data acquisition and analysis are also developed and tested. The instrument offers a versatile platform for further improvement and development.

Interest in the method continues to increase amongst national metrology laboratories, especially as it has been recently adopted as an alternative to the CIEMAT / NIST efficiency tracing method of LSC for the extended international reference system for pure β-emitters.

The system has been used recently for the activity standardisation of a tritiated water solution in the frame of an international comparison under the auspices of the Bureau International des Poids et Mesures (BIPM).

6.4 Measurement of single uranium particles by thermal ionisation mass spectrometry

Within the world's nuclear non-proliferation regime, the safeguards system of the International Atomic Energy Agency (IAEA) functions as a confidence-building measure, an early warning mechanism, and the trigger that sets in motion other responses by the international community if and when the need arises.

As part of the Additional Protocol[2], environmental sampling and particularly the analysis of uranium particles has become an important tool for the detection of non-declared nuclear activities. Under the protocol, micrometer-sized uranium particles with an isotopic composition characteristic for the processes at the inspected facility are collected, identified and analysed. Considering the potential consequences of the analytical results, these measurements need to be subjected to a rigorous quality management system.

Opposite page: Assembly of the cooling system of a new target used for neutron production in the Geel electron linear accelerator (GELINA). The uranium disk that will be bombarded by electron beams can be seen at the top of the picture.

2 The Additional Protocol (INFCIRC/540) is a legal document granting the IAEA complementary inspection authorisation to verify the absence of undeclared nuclear activities in all parts of a state's nuclear fuel cycle as well as any other location where nuclear material is or may be present.

Loading of a single particle on the TIMS filament with a micromanipulator.

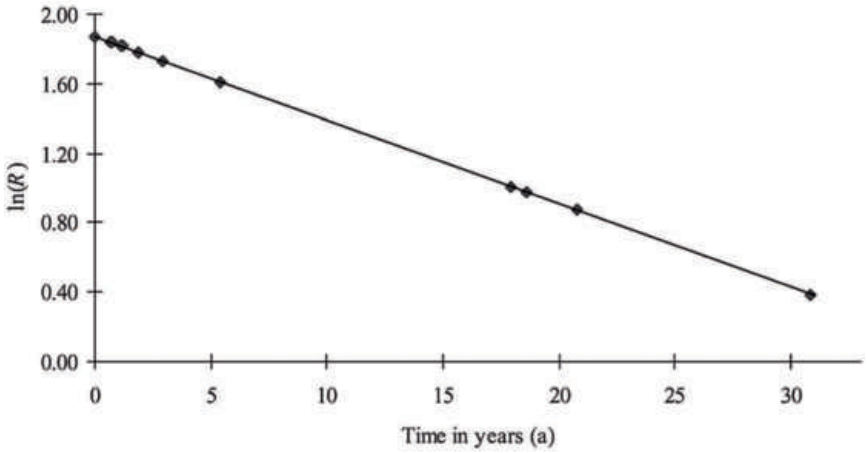
Recently, JRC-IRMM carried out measurements on single uranium particles applying thermal ionisation mass spectrometry (TIMS). Uranium particles are transferred using a micro-manipulator mounted within a scanning electron microscope (SEM) onto a carburised filament placed in a specially constructed filament holder. By the application of the so-called carburisation technique, the ionisation efficiency for uranium isotope ratio measurements is increased, so that the major and the minor isotope ratios in single uranium particles even smaller than 1 micron can be analysed by TIMS. This technique will be further applied to the certification of a uranium particle reference material, as requested by the IAEA and its Network of Analytical Laboratories (NWAL).

The development of single uranium particle analysis by TIMS improves further JRC-IRMM's capabilities for nuclear safeguards and so-called environmental sampling.

6.5 A new evaluation of the half-life of plutonium-241

Amongst the plutonium isotopes materials recovered from irradiated nuclear fuel, ^{241}Pu has the shortest half-life (around 14.3 years) and therefore affects the measurement of the amount of plutonium for accounting purposes to a much greater extent than other isotopes. Any correction of the decay of ^{241}Pu thus makes a considerable contribution to the overall uncertainty in the total amount of plutonium, especially after storage for several years. Therefore the value of the half-life together with its associated uncertainty needs to be known with a high degree of confidence to nuclear safeguards authorities.

The plutonium oxide (originally supplied from Oak Ridge) with a 92.7% abundance of ^{241}Pu (1976) has been carefully stored at JRC-IRMM and the isotopic ratios of the material were measured over a time period corresponding to two half-lives, which is unique. A new value of the ^{241}Pu half-life was evaluated by state-of-the-art mass spectrometric reference measurements of the isotopic ratio at JRC-IRMM. The measurements were designed to eliminate first order mass bias effects.



Natural log of the isotopic ratio-of-ratios, used to determine the new half-life value.

The new value published by JRC-IRMM is $\tau_{1/2}$ (^{241}Pu) = 14.325 a ± 0.024 a (k = 2), which is slightly shorter than the established value of 14.35 years recorded in the 7th edition of the Karlsruher Nuklidkarte.

6.6 Improved decay data for selected radionuclides

Accurate knowledge of selected nuclear decay data is critical in a wide range of fields, such as radiopharmaceutical production, radiotherapy and diagnostics, safeguards investigations, environmental monitoring, theoretical physics and radioactive waste disposal. In metrology, accurate decay data for certain radionuclides are required for the efficiency calibration of detectors used in X-ray and γ-ray spectrometry.

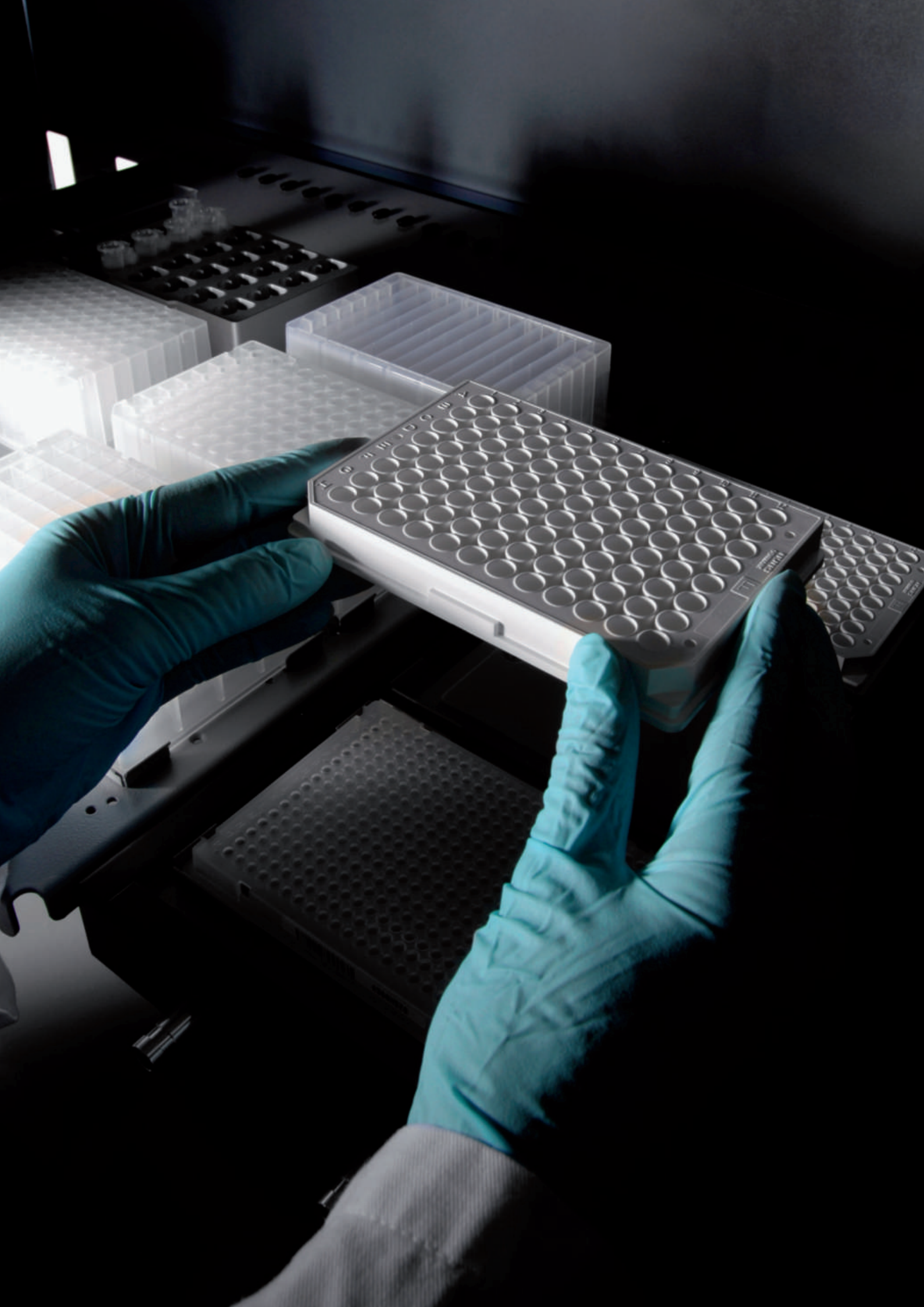
In 2009, JRC-IRMM published new measurement results of the half-lives of ^{54}Mn and ^{124}Sb. Standardised sources of these radionuclides are useful for calibration measurements of γ-ray spectrometers, and better knowledge of their respective half-lives extends the period over which the activity of the reference sources can be calculated accurately. For ^{124}Sb, the emission probabilities have been determined for 100 γ-rays, thus enabling to use them to calibrate the energy dependence of the detector efficiency.

New α-emission probability values were also published for ^{240}Pu, which resolved some discrepancies observed in the past. Primary standardisation measurements were performed on ^{177}Lu, an increasingly important radionuclide for medical therapy, thus adding a value to the very few reference data hitherto available in the international key comparison scheme.

Insertion of a thin, vacuum evaporated source into an α-particle spectrometer.

Radionuclides with very low emission rates are measured in JRC-IRMM's underground laboratory, located 225 m underground in the HADES facility of the Belgian Nuclear Research Centre (SCK·CEN). In 2009, JRC-IRMM published new half-life values for two such radionuclides, namely ^{180}Ta^{m} (> 2.0 $\times 10^{16}$ y) and a newly discovered decay branch of ^{115}In (4.4 $\times 10^{20}$ y). Tantalum-180m is the rarest primordial isotope on earth and the longest living isomeric state. Knowing the value of the half-life is important to settle dispute on its production in stellar nucleosynthesis. Indium-115 has a newly discovered decay branch with the lowest β^- decay energy known to man (0.15 keV) and can thus be used in future studies aiming at measuring the mass of the neutrino.

Optical distance measuring device for α-particle counting at a defined solid angle. This is a primary standardisation technique for radioactivity and the determination of nuclear decay data.



Food safety and quality

Introduction

JRC-IRMM's food safety and quality activities aim at providing fit-for-purpose, validated analytical methods to testing laboratories. The institute also organises inter-laboratory comparisons to benchmark the capabilities of official and private food control laboratories across the EU. Together, these activities aim at ensuring the comparability of testing results to provide the basis for the harmonised implementation of EU food and feed safety regulations. Measurement-related expert advice is given to other Commission services dealing with food and feed safety and quality which complements the laboratory based activities.

One of the measures for ensuring that legislation is properly implemented across EU countries was the setting up of a number of European Union Reference Laboratories[3], or EU-RLs. Four EU-RLs are now operated by JRC-IRMM: feed additives, mycotoxins, polycyclic aromatic hydrocarbons, and heavy metals in feed and food.

In the food safety domain, JRC-IRMM has built up facilities and expertise to engage in the rapidly developing area of the "omics" sciences. Genomics, proteomics, metabolomics and metabonomics are used as a basis for the development of methods for the detection of allergens in food and the authentication of foodstuffs to protect the well-being of consumers and give them the opportunity of an informed choice when purchasing foodstuffs.

Extracting polycyclic aromatic hydrocarbons (PAHs) from chocolate.

7.1 Method to measure vegetable fats in chocolate becomes ISO standard

In 2009, a method developed by JRC-IRMM to measure vegetable fats in milk chocolate became the first such method to be adopted as an international standard by the International Organization for Standardization (ISO). The method was developed to enable the enforcement of the so-called Chocolate Directive.

The Chocolate Directive (Directive 2000/36/EC) allows the addition of up to 5% of vegetable fats other than cocoa butter in chocolate products. When these fats are added to chocolate, European legislation requires that consumers be informed by appropriate labelling of the product. The threshold of 5% is also an essential requirement for these products to move freely within the internal market.

Prior to the development of JRC-IRMM's method, no validated methodology existed in this field. It was therefore not straightforward to check whether manufacturers were correctly reporting the amount of vegetable fats other than cocoa butter in milk chocolate. The chemical composition and physical properties of certain vegetable fats resemble those of cocoa butter

3 European Union Reference Laboratories were formerly called Community reference laboratories (CRLs), but the name was changed with the entry into force of the Lisbon Treaty.

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very closely, thus making them extremely difficult to quantify or even detect. This left the door open for disputes and uncertainty as to whether or not milk chocolate products fulfilled legal requirements.

JRC-IRMM had been working on the problem since the entry into force of the Chocolate Directive in 2003, in close contact with the European Commission's Directorate-General for Agriculture and Rural Development. As a result, reliable analytical methods were successfully developed to detect and quantify so-called cocoa-butter equivalents (CBEs) in milk chocolate.

JRC-IRMM submitted its milk chocolate testing methods to ISO. After a 2-year independent peer review process, the method was adopted by ISO as standard ISO 11053:2009.

Two other JRC methods to determine foreign fats in dark chocolate were previously adopted as international standards in 2007. This new method for milk chocolate took longer to develop because of the increased complexity of the measurement, as the milk fats in milk chocolate interfere with vegetable fats.

In 2009, a method developed by JRC-IRMM to measure vegetable fats in milk chocolate became the first such method to be adopted as an international standard by the International Organization for Standardization (ISO).

The international adoption of the JRC method for determining CBEs in milk chocolate marks the satisfactory completion of the JRC's work on foreign fats in chocolate.

7.2 Review of methods to measure phthalates in food

In 2009, JRC-IRMM published a report summarising methods used in Europe to measure phthalate contamination in food. Phthalates are mainly used as plasticisers (substances added to plastics to increase their flexibility), but they are being phased out of many products in the European Union and worldwide over health concerns.

The study found that the major difficulty in the analysis of phthalates is linked to the ubiquitous presence of the most prominent members of this class of 19 compounds (with respect to potential food contamination). Analysts have to continuously deal with "measurement blank" problems. The application of plastic materials for sample handling and sample preparation has to be avoided in phthalate analysis. Additional measures to reduce blank values can be taken, such as thermal treatment of glassware, redistillation of organic solvents, or rinsing

In 2009, JRC-IRMM published a report summarising methods used in Europe to measure phthalates in food.

of glassware with solvents. From the study, the application of thermally-cleaned aluminium oxide was found to be very efficient for the cleaning-up of apolar solvents. However, blank problems might also be caused by the analytical instrument.

The study noted that the validation of analytical methods for determination of phthalates in food is additionally hampered by a lack of suitable certified matrix reference materials. Therefore, particular importance should be placed on the participation in interlaboratory comparison tests, in order to evaluate the comparability of the results of analysis.

JRC-IRMM carried out the study upon the request of the Directorate-General for Health and Consumers, in order to evaluate the measurement capabilities of European food control laboratories.

7.3 Validated method to determine a marker in animal by-products

European legislation on animal by-products states that certain raw materials and processed products (category 1 and 2 materials) must not enter the feed or food chain. For this reason, these materials must be permanently marked in order to ensure identification and traceability of products destined for disposal, and to eliminate the risk of fraud.

JRC-IRMM previously identified glyceroltriheptanoate (GTH) as a suitable marker, and its use to mark category 1 and 2 materials in rendering plants at a minimum concentration of 250 mg/kg became mandatory as of 1 July 2008, by virtue of Regulation (EC) No. 1432/2007.

In 2009, JRC-IRMM published the results of a collaborative study to determine whether an analytical method to determine the GTH marker in animal by-products was fit for purpose.

In 2009, JRC-IRMM published the results of a collaborative study to determine whether an analytical method to determine GTH in animal by-products was fit for purpose. The method comprised three main steps: i) extraction of GTH from the samples, ii) clean-up of the extracts using solid phase extraction cartridges, iii) determination of GTH by gas chromatography using flame ionisation detection or mass spectrometry.

Twelve different test materials were used in the study, and these were processed under realistic rendering conditions and contained meat and bone meal and fat from categories 1 and 2. The levels of GTH in the test materials ranged from 61 to 455 mg/kg, ensuring that the laboratories had to measure at below, around and above the legally binding minimum concentration of 250 mg/kg.

The obtained relative standard deviation of repeatability (RSD_r) varied from 3.4 to 7.8%, and the relative standard deviation of reproducibility (RSD_R) varied from 9.0 to 16.5%, corresponding to HORRAT values that were in all cases equal to or below the critical value of 2.0. The estimated trueness expressed in terms of average concentration obtained in the study compared to the target concentration of GTH in all the test materials varied from 95 to 107%.

Based on the results of the study – in which 19 laboratories from 11 Member States participated – it was concluded that the method is indeed fit for official control purpose to determine GTH in processed animal by-products from categories 1 and 2.

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7.4 Influence of mycotoxin binders on testing methods

Upon the request of the Directorate-General for Health and Consumers, JRC-IRMM investigated the potential influence of mycotoxin binders on the performance of mycotoxin testing methods. Mycotoxins are toxic secondary metabolites produced by several fungi, and it is estimated that around 25% of harvested crops are affected by mycotoxins worldwide. The economic consequences of mycotoxin contamination are profound, and often crops with large amounts of mycotoxin have to be destroyed.

One way of trying to reduce the uptake of mycotoxins from contaminated feed is the use of mycotoxin binders. The aim of these additives is to inhibit the update of mycotoxins by an animal in vivo. These adsorbent materials are intended to act like a chemical sponge and adsorb mycotoxins in the gastrointestinal tract, thus preventing the uptake and subsequent distribution to target organs.

However, it remained an open question as to whether such mycotoxin binders could influence the performance characteristics of methods used to determine mycotoxins. If such an influence existed, it would open the door to binders being used to fraudulently mask the presence of non-compliant consignments.

Maximum levels of mycotoxins are set in European legislations, e.g. 0.005-0.02 mg/kg for aflatoxin B1 for feeding stuffs. Commonly used methods to detect mycotoxins include thin layer chromatography (TLC), high-performance liquid chromatography (HPLC), gas chromatography (GC) and immunochemical methods such as enzyme-linked immunosorbent assays (ELISA).

Verification of instrument settings for mycotoxin analysis using high performance liquid chromatography (HPLC).

The statistical analysis of the results of the study did not show any significant influence of mycotoxin binders on the capacities of the selected analytical methods to determine mycotoxins. Thus, it could be concluded that the tested binders had no effect on the level of mycotoxins, and it is therefore not possible to use the binders to mask feed contaminated with mycotoxins.

The study was carried out by the European Union Reference Laboratory (EU-RL) for mycotoxins.

7.5 Polycyclic aromatic hydrocarbons (PAHs) in sausage meat

In 2009, JRC-IRMM published the results of the third interlaboratory comparison organised by the European Union Reference Laboratory (EU-RL) for polycyclic aromatic hydrocarbons (PAHs).

Polycyclic aromatic hydrocarbons (PAHs) constitute a large class of organic substances. PAHs may be formed during the incomplete combustion of organic compounds and can be found in the environment. In food, PAHs may be formed during processing and domestic food preparation, such as smoking, drying, roasting, baking, frying or grilling.

The interlaboratory comparison focussed on the determination of the so-called 15+1 EU priority PAHs in canned sausage meat and acetonitrile, and it was conducted along the lines of the international harmonised protocol for the proficiency testing of analytical chemical laboratories.

The test materials were prepared from certified reference materials. Standard stock solutions of each analyte were produced by substitution weighing of undiluted substances on a microbalance and dissolution in toluene. These stock solutions were added to gravimetrically determined amounts of acetonitrile and edible oil.

All 25 participants reported results for the two materials sent. For the sausage meat sample, 88% of the reported values were assigned z-scores ≤ |2|, indicating that most of the participating laboratories performed satisfactorily with respect to internationally accepted standards. However, in some cases, bias and /or a high variability were discovered, and some analytes consistently caused specific problems, and these issues should be investigated.

7.6 Heavy metals in mineral feed

Animals require certain basic nutrients for growth, reproduction and good health. Such nutrients include minerals, namely sodium chloride, calcium, phosphorus, sulphur, potassium, magnesium, manganese, iron, copper, cobalt, iodine, zinc, molybdenum and selenium. However, when consumed in excessive amounts, some minerals can be toxic.

To avoid problems associated with excessive metal content in feed, maximum levels for trace elements in several commodities have been laid down in Directive 2002/32/EC, and a network of laboratories has been built up to ensure quality and comparability in official controls throughout the European Union.

With the aim of expanding a previously organised study to a wider variety of feed matrices, the European Union Reference Laboratory (EU-RL) for heavy metals in feed and food organised an interlaboratory comparison for the determination of total cadmium, lead, arsenic and extractable cadmium and lead in mineral feed.

Thirty-one participants from 25 countries registered for the exercise. The test samples were derived from commercially available mineral feed for piglets, which was processed by JRC-IRMM (milling, particle size measurement, sieving, homogenisation and packaging).

Animals require certain basic minerals for growth, reproduction and good health. Some minerals can be toxic in excessive amounts, however, and maximum levels for trace elements in several commodities have been laid down in Directive 2002/32/EC (© 2001 image100 ltd).

From the results of the proficiency test, it was concluded that the selection of the reference material for method validation is critical. Using reference materials that do not match the type of matrix of the test material introduces a significant underestimation of some of the measurands, in this case of total lead. A second conclusion is that the concentration of total and extractable cadmium (according to Directive 2002/32/EC) can be considered identical when analysing mineral feed.

The EU-RL for heavy metals in feed and food also carried out an interlaboratory comparison on the determination of cadmium, lead, arsenic and mercury in food supplements (i.e. concentrated doses of nutrients in capsule or pill form, intended to supplement the normal diet). It was found that the national reference laboratories had considerably improved their performance compared to a similar exercise in 2006, although there was still some room for improvement in the evaluation of measurement uncertainties.

Evaluating measurement capabilities

Introduction

It is important for a testing laboratory to know that the measurement results it produces are reliable. It is equally important to be able to demonstrate these competences against reliable benchmarks. Many laboratories have acquired accreditation of their services/activities and increasingly the laboratory accreditation schemes require proof of proficiency. The testing laboratories can demonstrate their capability by participating in proficiency testing exercises organised by recognised national or international providers.

JRC-IRMM hosts four European Reference Laboratories: feed additives, mycotoxins, polycyclic aromatic hydrocarbons, and heavy metals in feed and food. One of their tasks is to organise comparative studies and proficiency tests for the networks of national reference laboratories. It also organises interlaboratory comparisons open to all laboratories that wish to benchmark their capabilities and pilot and key comparisons of the committees of the International Committee for Weights and Measures (CIPM) in its competence areas.

The JRC-IRMM runs an International Measurement Evaluation Programme (IMEP), a Regular European International Measurement Evaluation Programme (REIMEP) for nuclear measurements, an International Measurement Evaluation Programme for Nuclear Signatures in the environment (NUSIMEP) and an evaluation programme on the comparability of data collected by the JRC Institute for Environment and Sustainability from laboratories measuring radioactivity in the environment.

Below are some examples from 2009 of measurement campaigns organised by JRC-IRMM to benchmark measurement competence and proficiency. Further examples can be found in this report in the section on food safety and quality.

8.1 Mineral oil in sunflower oil

Following the discovery of contaminated sunflower oil imported from Ukraine (April 2008), JRC-IRMM invited 55 analytical laboratories from 17 EU Member States plus Switzerland and Ukraine to benchmark their capability to measure levels of the contaminant, mineral oil, in sunflower oil. The results showed that around 80% of laboratories performed satisfactorily.

The contaminated sunflower oil was reported by France to the European Commission and EU Member States via the Rapid Alert System for Food and Feed (RASFF) in April 2008, and the Commission subsequently imposed restrictions on the importation of sunflower oil from Ukraine (June 2008). JRC-IRMM was asked by the Commission's Directorate-General for Health and Consumers to investigate the capabilities of official control laboratories and industrial food laboratories to measure mineral oil in sunflower oil.

Test samples comprising both naturally-contaminated and 'spiked' sunflower oil were dispatched to the laboratories, and between 78% and 85% of the laboratories were able to measure satisfactorily, depending on the test material.

High levels of mineral oil were discovered in sunflower oil imported from Ukraine. JRC-IRMM was asked by the Commission's Directorate-General for Health and Consumers to investigate the capabilities of official control laboratories and industrial food laboratories to measure mineral oil in sunflower oil.

Mineral oil is a by-product of the distillation of petroleum. Food may come into contact with mineral oil, such as lubricants or binding agents, during harvesting, storage, processing or packaging. However, the levels of mineral oil measured in the imported sunflower oil from Ukraine were much higher than what could be expected from atmospheric or other background sources of contamination.

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8.2 Melamine in milk powder and bakery products

Melamine is a nitrogen-rich organic compound, with the chemical formula $C_3H_6N_6$, which is normally used as an industrial chemical in plastics and glues. It is sometimes fraudulently added to food and feed products to increase the apparent protein content, as protein concentrations are typically measured by analysis of nitrogen. Intake of melamine has been linked to kidney stones and other health problems.

The 2008 health scare in China over powdered milk raised concerns about possible melamine contamination in products on the European market. Although the EU does not import milk or other dairy produce from China, processed foods such as biscuits and chocolates might contain traces of milk powder. The European Commission consequently decided that composite products, including feed, that contain milk products originating in or consigned from China shall be checked, including laboratory analysis (Commission Decision 2008/798/EC). Products containing more than 2.5 mg/kg were to be immediately destroyed.

JRC-IRMM carried out a proficiency test to benchmark laboratories ability to detect melamine in food and feed. Laboratories from 31 countries participated in the test, including Australia, China, India, Japan, New Zealand, the United States of America, as well as 21 of the 27 EU Member States.

Upon request from the Commission's Directorate-General for Health and Consumers, JRC-IRMM has reviewed existing analytical methods for the detection of melamine in food and feed, and carried out a proficiency test to benchmark laboratories ability to detect melamine.

Laboratories from 31 countries participated in the test, including Australia, China, India, Japan, New Zealand, the United States of America, as well as 21 of the 27 EU Member States. The response to the call for participation was so overwhelming that the test was fully subscribed within days and the registration had to be closed prematurely.

The results of the study were that 74% of the 114 results for milk powder and 73% of the 112 results for the baking mix were within the acceptable range, as defined by common international laboratory proficiency guidelines. The values reported by the laboratories were also accompanied by values of measurement uncertainty, which is extremely important when measuring close to a legal limit. Here there was some scope for improvement, as around a quarter of the uncertainty values (23% milk powder, 22% baking mix) were underestimated.

The study also compared the laboratories' results with the methods they used to reveal which measurement technique works best. In this case, isotope dilution mass spectrometry with a stable isotope labelled melamine was generally more accurate.

In 2009, JRC-IRMM organised a proficiency test to benchmark laboratories measuring eight heavy metals whose safety limits are set out by the Toy Safety Directive and specified in the harmonised European Standard EN71-3:1994 (© Radius images).

8.3 Heavy metals in toys

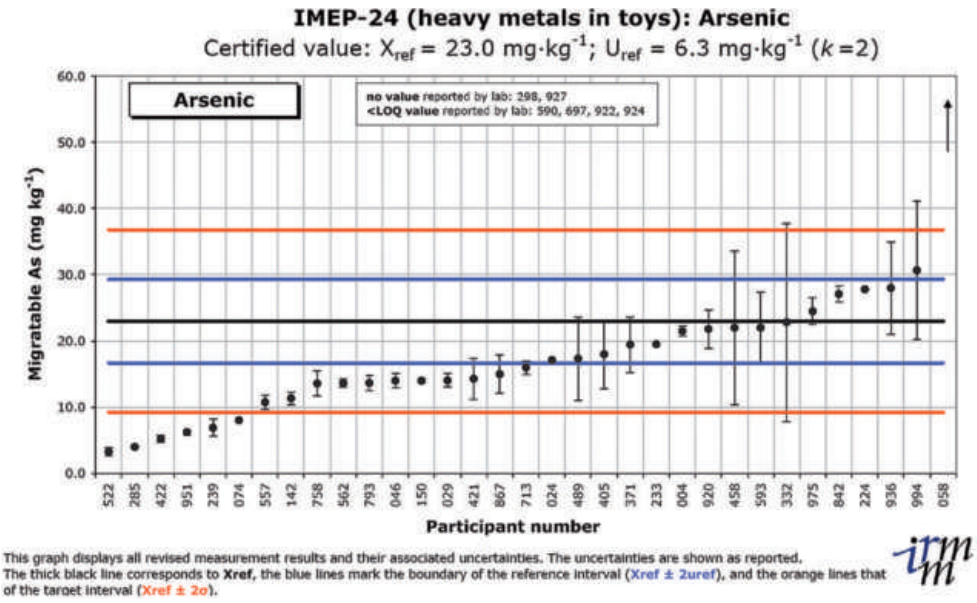
The EU's Toy Safety Directive (88/378/EEC) aims at protecting children from harmful substances in toys, as well as harmonising standards to enable the free movement of toy products.

Upon request of European Accrediation (EA), JRC-IRMM organised a proficiency test in the frame of the International Measurement Evaluation Programme (IMEP). The test aimed at benchmarking laboratories measuring eight

heavy metals whose safety limits are set out by the Toy Safety Directive and specified in the harmonised European Standard EN71-3:1994.

The exercise raised concerns about how the participants would interpret their results to approve or reject a toy product for entering the market. In around a third of cases, participants were interpreting their own measurement data incorrectly either by erroneously accepting a value or by rejecting it unnecessarily.

JRC-IRMM carried out two other IMEP campaigns in 2009: one on the analysis of total Cd, Pb and As and extractable Cd and Pb in mineral feed, and the other on the determination of total Cd, Pb, As and Hg in food supplements.



International Measurement Evaluation Programme (IMEP) for heavy metals in toys. Each dot on the graph is a measurement value determined by an individual laboratory. The graph shows the spread of values around the reference value, in this case for arsenic.

8.4 Monitoring of radioactivity in the environment

To assess the radioactive exposure of the population as a whole, Member States are required by European legislation[4] to monitor radioactivity levels in certain environmental and food samples. Since 2003, JRC-IRMM has supported the Directorate-General for Energy and Transport to gain insight into the measurement methods and the quality of the values reported by the Member States. JRC-IRMM organises comparison exercises among those Member State laboratories regularly monitoring radioactivity.

In the most recent exercise, the laboratories had to determine low levels of activity concentrations (about 10 mBq/L to 100 mBq/L) of the naturally occurring α-decaying radionuclides ^{226}Ra, ^{234}U and ^{238}U and the natural β-decaying nuclide ^{228}Ra in mineral waters. Reference values for all samples were established at JRC-IRMM with traceability to the SI system of units, exploiting JRC-IRMM's special radionuclide measurement infrastructure.

As observed in previous campaigns, the coherence of laboratory measurement results with the reference value does not depend on a particular method of measurement or radiochemical separation applied for the determination of these radium and uranium nuclides. As the figure reveals for ^{226}Ra, several of the participants' results deviate by a factor of more than two from the reference values.

Since these samples had rather low activity concentrations – around the detection limits required by future EC legislation which is in preparation – and since not all laboratories are routinely analysing water for these radionuclides yet, such unsatisfactory comparison results could have been expected, in particular for ^{226}Ra and ^{228}Ra.

4 Commission Recommendation (2000/473/Euratom) of 8 June 2000 on the application of Article 36 of the Euratom Treaty concerning the monitoring of the levels of radioactivity in the environment for the purpose of assessing the exposure of the population as a whole.

The comparison clearly demonstrates, however, that a number of monitoring laboratories need to improve their analysis procedures, in particular for radium, to correctly identify drinking water sources for which remedial action needs to be taken with respect to their natural radioactivity concentration. Erroneous analysis results may lead to the omission of necessary remedial action or to economic loss by an unjustified action.

In anticipation of new European requirements for monitoring radioactivity in drinking water, JRC-IRMM compared the capabilities of laboratories to determine low concentrations of natural radioactivity in three commercially available mineral waters.

8.5 Pilot study on DNA analysis

The provision of reference materials to the biological community is a recent area of active research in several National Metrology Institutes (NMIs). The quantification of the relative amount of DNA sequences extracted from a biological tissue remains a complex analytical procedure and relies on the availability of such reference materials. Quantitative real-time polymerase chain reaction (QRT-PCR) is currently the most common measurement method to identify and quantify DNA sequences in a biological sample.

During a previous study, several NMIs were able to demonstrate their ability to use QRT-PCR to quantify a defined plasmid DNA while calibrating with the same plasmid DNA. The same measurement method was later used to quantify genomic DNA extracted from a plant tissue mixture calibrated with genomic DNA extracted from the pure plant tissue. Nevertheless, all previous studies were performed using matching calibrants for which a reference value had been assigned.

A new CCQM study[5] was therefore organised by JRC-IRMM in the frame of the programme on metrology in bioanalysis of the International Committee on Weights and Measures (CIPM). It aimed at investigating how far 14 participating NMIs were able to quantify relative amounts of DNA sequences present in a biological tissue using independent calibration systems.

Opposite page: Loading of proteins on a polyacrylamide gel prior to electrophoresis, which then migrate according to their respective size.

The methodology requires extraction and purification of genomic DNA from a plant tissue and accurate detection and quantification of the relative amount of two defined DNA sequences present in the extracted genomic DNA of transgenic maize 1507. Four maize samples with two different levels of transgenic sequences had to be analysed. The copy number percentages for samples 1 and 3 were chosen to be at 1/18 of the European labelling threshold for GMO in food and feed. In other words, the NMIs had to demonstrate their ability to detect and quantify very low absolute numbers of transgenic copies present in the samples.

The average relative expanded uncertainty at those low levels (0.05 (cp/cp) % of maize 1507) was 44 %, and could be reduced to 26 % at the level of 0.5 (cp/cp) % of maize 1507. The study has indicated the intrinsic limit of quantification for low numbers of DNA targets by QRT-PCR and enabled the evaluation of the performance of NMIs in this new field of metrology.

5 CCQM is an acronym for Comité Consultatif pour la Quantité de Matière (Consultative Committee for Amount of Substance), established in 1993 by the Comité International des Poids et Mesures (CIPM).

9 Other policy support activities

9.1 Pre-accession assistance to Turkey in the field of metrology

A new three-year project was launched in 2009 under the stewardship of JRC-IRMM, which will bring together European and Turkish experts in measurement science. The project is funded by the European Union under the instrument for pre-accession assistance (IPA), and it aims at providing Turkey's citizens an increased quality of life through an enhanced measurement infrastructure in areas such as environment, health, food safety and consumer protection. A well-functioning measurement infrastructure is also necessary for the adoption of the body of European law known as the *acquis communautaire*, thus supporting the accession of Turkey to the EU.

Through the project, JRC-IRMM will provide consultancy, training and support to metrological institutes in Turkey, i.e. the National Metrology Institute (TÜBİTAK-UME) and to the Sarayköy and Çekmece Nuclear Research and Training Centers (SANAEM and ÇNAEM) of the Turkish Atomic Energy Authority (TAEK). JRC-IRMM will also provide training in the field of chemical and ionising radiation metrology to laboratories and universities, in order for them to optimise the use of services they obtain from the national metrology institute, and to help them incorporate metrology-related subjects into educational curricula.

(left to right): S. Can, Director TAEK-CNAEM, S. Süer, Acting Director TÜBITAK UME, K. Maruszewski, JRC Director and I. Tükenmez, Director TAEK-SANAEM, signing a letter of intent during the project kick-off (October 2009).

The project kicked-off in Ankara in October 2009, in the presence of approximately 100 participants from both the project partners and from project stakeholders like universities, accredited laboratories and private companies.

A workshop on the standardisation of radionuclides took place in Ankara in November 2009. The workshop was jointly organised by JRC-IRMM, the Turkish Atomic Energy Authority (TAEK) and the Virtual European Radionuclide Metrology Institute (VERMI). The lectures and exercises focused on primary standardisation techniques and the metrology system for radioactivity, and treated in depth the topics of gamma-ray spectrometry, liquid-scintillation counting, ionisation chambers and radionuclide calibrators.

JRC-IRMM has previously supported enlargement policy, including the development of metrology, standardisation, conformity assessment and accreditation in Croatia. That project was funded under the Community Assistance for Reconstruction, Development and Stabilisation programme (CARDS), and was concluded in 2009.

9.2 Stable light isotope research in support of environmental policies.

Nitrate contamination in water is an environmental problem worldwide. Increased input of reactive nitrogen is attributed to intensive land use, increased use of organic and inorganic fertilisers containing nitrogen, animal manure, discharge of human sewage and elevated atmospheric deposition of nitrogen. The World Health Organisation has set a limit of 10 mg/L NO_3-N (the content of nitrogen in solution due to nitrates) for drinking water. The implementation of the Nitrate Directive[6] in Europe established a detailed framework for prevention of nitrate pollution to waters.

In 2009, JRC-IRMM co-authored a review paper[7] on the present limitations and future prospects of stable isotope methods for nitrate source identification in surface and ground waters. The paper concluded that various potential nitrate sources have distinct δ^{15}N- and δ^{18}O-NO_3 values and a dual isotope approach is a powerful tool to identify nitrate sources in contaminated water. Reliable quantification and uncertainty assessment of a variety of nitrate source inputs via Bayesian framework estimation could provide evidence for nitrate source contribution. In addition, the "ion-exchange method" and the "bacterial denitrification method" are frequently used for δ^{15}N- and δ^{18}O-NO_3 measurement, but only a limited number of laboratories worldwide are capable of carrying out these analyses. Hence, accurate, efficient and broadly applicable isotope techniques need to be explored before nitrate source quantification using the isotope mixing model operating in a Bayesian framework can be implemented in large-scale water monitoring programmes.

In 2009, JRC-IRMM co-authored a review paper on the present limitations and future prospects of stable isotope methods for nitrate source identification in surface and ground waters. (© Image Source)

Through the CARIBIC project [8], JRC-IRMM performed atmospheric CO_2 isotope measurements, with a focus on understanding instrument effects, corrections and metrological aspects[9]. Measurements of CO_2 isotope measurements are necessary to better understand the atmospheric budget of CO_2, especially in the upper troposphere and lower stratosphere. They are also required to obtain a high-quality dataset of background tropospheric air, far from CO_2 sources and sinks.

The results indicated for the first time CO_2 isotope trends due to uplift and pole-wards transport of tropical air and stratosphere-troposphere exchange. It was demonstrated that $^{18}O/^{16}O$ (CO_2) in the upper troposphere and lower stratosphere region can be used as a new, additional tracer of transport and air mixing. The work also led to a methodological paper on CO_2 isotope measurements, with recommendations concerning the unification of raw CO_2 isotope data treatment.

JRC-IRMM performed atmospheric CO_2 isotope measurements on air samples collected on a Lufthansa Airlines passenger aircraft at altitudes of 9 to 12 km, as part of the CARIBIC project.

6 Council Directive of 12 December 1991 concerning the protection of waters against pollution caused by nitrates from agricultural sources (91/676/EEC)

7 Xue D, Botte J, De Baets B, Accoe F, Nestler A, Taylor P, Van Cleemput O, Berglund M, Boeckx P. *Present Limitations and Future Prospects of Stable Isotope Methods for Nitrate Source Identification in Surface- and Groundwater.* WATER RESEARCH 43 (5); 2009. p. 1159-1170. JRC49525

8 Civil aircraft for the regular investigation of the atmosphere based on an instrument container (CARIBIC), http://caribic-atmospheric.com

9 Assonov S, Taylor P, Brenninkmeijer C. *A System for High-quality CO_2 Isotope Analyses of Air Samples Collected by the CARIBIC Airbus A340-600.* RAPID COMMUNICATIONS IN MASS SPECTROMETRY 23 (9); 2009. p. 1347-1363. JRC48443

10 Annex: Selected publications in 2009

10.1 Articles in peer-reviewed periodicals[10]

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European Commission

EUR 24246 EN – Joint Research Centre, Institute for Reference Materials and Measurements

Annual Report 2009

Editor: D. Anderson

Luxembourg: Publications Office of the European Union, 2010

2010 – 48 pp. – 21.0 x 29.7 cm
EUR 24246 – Scientific and Technical Research series – ISSN 1018-5593, 1681-5084
ISBN 978-92-79-15010-4
Catalogue number LA-NA-24246-EN-C

Abstract
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LA-NA-24246-EN-C

ISBN 978-92-79-15010-4

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