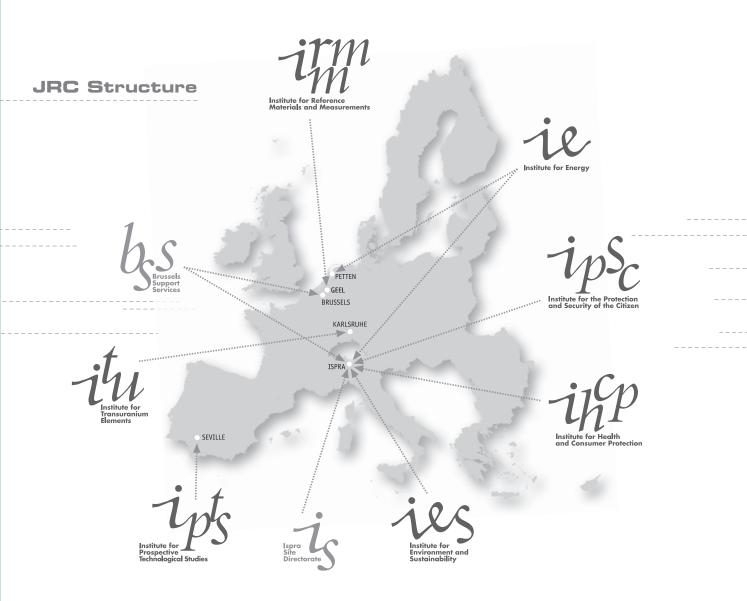


ANNUAL REPORT





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.



Institute for Reference Materials and Measurements

ANNUAL REPORT





European Commission Joint Research Centre Institute for Reference Materials and Measurements

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JRC-IRMM Annual Report 2008

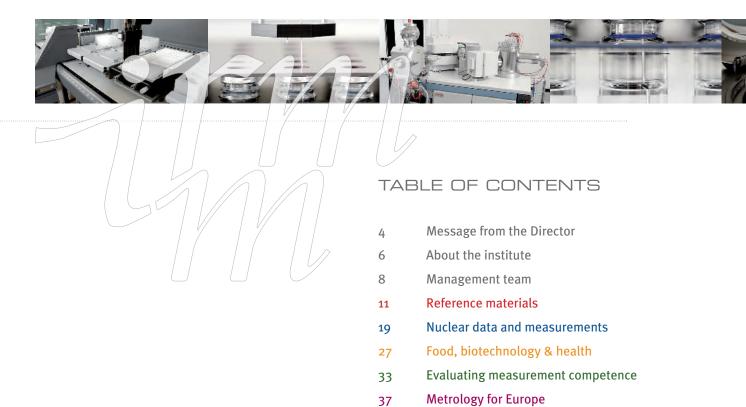
Community reference laboratories

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Message from the Director

In the European Union, an estimated €80 billion is spent on measurements annually, and accurate measurements are the foundation for global trade and a better quality of life. In 2008, JRC-IRMM continued to supply certified reference materials in core areas such as genetically modified food and feed, clinical chemistry and nuclear safeguards, but also turned its attention to new applications such as nanoparticles and biofuels, and developing new formats for microbiological applications.



In September 2008, melamine-tainted dairy products in China came to the attention of the general public. This latest food scare served as a reminder of the need to have a high-quality, well-functioning measurement infrastructure in Europe for food control. The ongoing food safety activities, the operation of four Community reference laboratories and the international comparisons organised by JRC-IRMM contribute to this effort.

Nuclear power currently provides around 30% of primary energy in the EU, and 2008 saw a marked increase of political interest in civilian nuclear power in view of its low-carbon credentials. The JRC-IRMM has a long-standing history of performing specialised measurements of neutron data, which are essential for the design of safe nuclear power installations, but new developments in nuclear technology bring additional demands for data and reveal gaps in our collective knowledge. Looking ahead, I am certain that JRC-IRMM will enhance its important role in the European nuclear research programme, particularly through the study of innovative concepts like nuclear waste transmutation and accelerator-driven systems.

An independent evaluation of the JRC's work performed under the 6th Framework Programme was published in 2008. This landmark evaluation was carried out by an expert panel led by Sir David King – the former Chief Scientific Advisor to the UK Government, and it confirmed that the JRC is an indispensable source of knowledge and expertise in support of the EU's political agenda.

Furthermore, the panel also provided recommendations for the future evolution of the JRC that reinforce the JRC's ongoing push towards a new corporate strategy. On this point, I was particularly pleased to note the three distinct activities recommended by the panel: i) scientific support to policy, ii) nuclear research and iii) reference materials and measurements. In view of our institute's contribution to all three of these areas, this evaluation provides independent and high-profile recognition of the value of our work, which in turn is credit to the abilities and dedication of JRC-IRMM staff.

Moving into 2009, I am confident that JRC-IRMM will profit from the momentum of the King evaluation. In line with the principle of continuous improvement, we will continue to deliver results and invaluable support to partners in our core areas, whilst proactively applying our expertise to new measurement challenges.

Finally, I would like to thank all our collaborators and customers for their continued support, which enables JRC-IRMM to carry out its mission to promote a common and reliable European measurement system in support of EU policies, and deliver on our trademark promise: IRMM – confidence in measurements®.

Alejandro Herrero

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.

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 production and dissemination of internationally accepted quality assurance tools. JRC-IRMM develops and validates testing methods, produces reference materials, organises measurement evaluation programmes, and provides reference measurements.

As a metrology institute of the European Commission, the 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 (EUROMET). 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 Standardization (CEN) and International Organization for Standardization (ISO). The JRC-IRMM now operates four Community reference laboratories.



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.

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 standardisation is widely recognised. 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 the AOAC International. Many testing methods validated by JRC-IRMM together with its collaborators have been approved as standards of CEN and ISO.



Reference materials are key tools for achieving traceability of measurement results, proving accuracy of methods and demonstrating proficiency of laboratories.

Isotopic reference materials are used to check and account for irradiated nuclear fuel.

Special infrastructure

The research facilities include multi-functional and flexible laboratories for development and production of reference materials, advanced analytical laboratories, mass metrology laboratory and an ultra-clean chemical laboratory. The JRC-IRMM analytical 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 materials 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. 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.

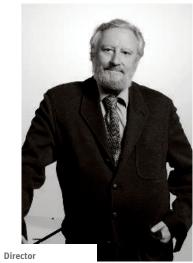
JRC-IRMM operates a 150 MeV linear electron accelerator (GELINA) and a 7 MV light-ion Van de Graaff accelerator. The two accelerators of JRC-IRMM, used for neutron production, are complementary in their experimental conditions and among the best such installations in the world. In the recent review of 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).



A new thermal ionisation mass spectrometer (Triton) was purchased and installed at JRC-IRMM in May 2008. The instrument was successfully validated and is used mainly for reference measurements of plutonium amount content and isotopic composition.

JRC-IRMM Annual Report 2008

Management team



Alejandro Herrero



Neutron physics Peter Rullhusen – *Head of Unit*



Reference materials HENDRIK EMONS – *Head of Unit*



Food safety and quality FRANZ ULBERTH - Head of Unit



Isotopic measurements PHILIP TAYLOR – *Head of Unit*

Management team

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Institute development and programme management DORIS FLORIAN – Head of Unit



Management support MARC WELLENS – Head of Unit



Infrastructure and site management Colin Woodward – Head of Unit



Informatics and electronics BARTEL MEERSMAN – Head of Unit



Radioprotection and security PIERRE KOCKEROLS – Head of Sector



Reference materials

Comparability of measurement and testing results from around the world depends on a framework of international measurement standards. Reference materials are needed for calibrating instruments and test kits, validating measurement procedures and monitoring the performance of laboratories. Today, reference materials are increasingly needed to fulfil the criteria of laboratory accreditation schemes.

JRC-IRMM is one of the world's leading reference material producers specialising in policy support. It runs a systematic development programme based on up-to-date knowledge of the needs of the testing laboratories and the demands of European legislation.

In 2008, JRC-IRMM continued to supply certified reference materials in core areas such as genetically modified food and feed, clinical chemistry and nuclear safeguards, but also turned its attention to new applications such as nanoparticles and biofuels, and developing new formats for microbiological applications.

Genetically modified organisms

ntroduction

Legislation in the European Union calls for food and feed products containing more than 0.9% of genetically modified organisms to be labelled accordingly¹. Implementing the legislation requires suitable quality assurance tools, and laboratories need certified reference materials for all genetically modified organisms authorised in Europe.

In 2008, JRC-IRMM produced a new series of matrix reference materials, ERM-BF410k, certified for their mass fraction of GTS 40-3-2 soya (also called Roundup Ready® soya). The new set of CRMs comprises four different mass fraction levels and replaces the set of CRMs with the code ERM-BF410. The timely replacement of these highly sought-after materials ensures that control laboratories can continue to carry out measurements according to ISO 17025 with regular quality control through the use of CRMs.

Human protein diagnostics

Measurements of serum proteins are used for the diagnosis of many conditions including infection, liver or kidney disorders, iron deficiency, malnutrition and the monitoring of autoimmune diseases, and they are amongst the most important measurements done in clinical chemistry. In 2008, JRC-IRMM released a new reference material certified for the mass concentration of twelve human serum proteins. The reference material, ERM-DA470k/IFCC, is the successor of the ERM-DA470, which is presently used worldwide as the serum protein standard.

European legislation requires traceability of calibrants and control materials to reference measurement procedures and/or reference materials of higher order². 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 discrepancies between laboratories for assays of serum proteins subsequently became substantially lower for most of the proteins certified. The new material will ensure continuity in the standardisation of serum proteins, which is crucial in clinical chemistry. It makes it possible for laboratories worldwide to use common reference ranges, and to compare results over time and between hospitals and countries.

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¹ Regulation (EC) No 1830/2003 of the European Parliament and of the Council of 22 September 2003 concerning the traceability and labelling of genetically modified organisms and the traceability of food and feed products produced from genetically modified organisms and amending Directive 2001/18/EC.

The International Federation of Clinical Chemistry and Laboratory Medicine (IFCC), which actively supports standardisation in clinical chemistry, collaborated with JRC-IRMM on the development of this new reference material.

Liver enzyme aspartate transaminase

A new reference material for the liver enzyme aspartate transaminase (AST), also called aspartate aminotransferase (ASAT), was developed in 2008. As an important enzyme involved in amino acid metabolism, AST serves as a biomarker of liver damage (hepatitis, cirrhosis, etc.).

The measurement of the catalytic activity concentration of this enzyme is therefore of utmost importance in laboratory medicine, but at the same time also very challenging. The catalytic activity of an enzyme is a functional property measured via the catalyzed rate of a chemical reaction under specific experimental conditions. Consequently it is very difficult to standardise this parameter.

JRC-IRMM collaborated with the International Federation of Clinical Chemistry and Laboratory Medicine (IFCC), and involved twelve expert laboratories from EU and non-EU countries. The development of this reference material required particular processing techniques such as a robotic filling and freeze-drying in a semi-sterile laboratory (ISO class 5). In order to characterise the material, a specially designed study was organised with the twelve laboratories, using an IFCC Reference Measurement Procedure. Each laboratory characterised the material independently and agreement was found between the results. On this basis the property value could be assigned to the reference material, which has also been accepted as a European Reference Material (ERM) with the code ERM-AD457/IFCC.



New facility for large-scale mixing of powders (up to 550 L or 400 kg) during reference material preparation.

Aspartate transaminase (AST) serves as a biomarker of liver damage.

The development of this reference material is a good example of high-level and efficient international collaboration. The new reference material will play a key role in the metrological traceability chain for the standardisation of the measurements of enzymatic catalytic activity.

Nanoparticles

To better understand the potential effects of nanoparticles, accurate measurements of their physicochemical characteristics are needed. Currently, the key routine measurement is the analysis of the particle size - not only for risk assessment but also for production and subsequent trade of nanoparticle materials. Unfortunately, measurements using the currently available techniques are often difficult to compare, because the results depend on the measurement technique, on the data analysis and even on the way the data is presented.

In response to this need for better characterisation of nanoparticles, JRC-IRMM produced a new reference material for quality control of particle-sizing (IRMM-304). The reference material

consists of silica nanoparticles with a nominal diameter of 40 nm suspended in an aqueous solution. It can be used for checking the performance of instruments and methods that characterise the particle size distribution of nanoparticles suspended in a liquid medium. Three different particle size values were assigned to IRMM-304, which correspond to different measurement techniques (dynamic light scattering and disc sedimentation), and to different methods of data analysis.

With the release of this nanoparticle reference material, the JRC-IRMM increases its range of particle-size reference materials from gravel-like materials for sieve analysis down to the nanoscale.

Laser beam scattered through successive nanoparticle suspensions of increasing particle concentration.

material for accurate particle sizing.

Nitroimidazole veterinary drugs in meat

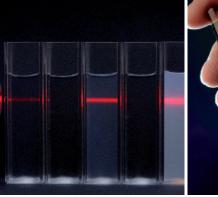
prohibited under EU legislation.

The monitoring of food for the possible presence of banned veterinary drugs is of major importance for efficient consumer protection. Nitroimidazoles have been used in the past in poultry and pigs to treat protozoan diseases and to combat bacterial infections. However, these veterinary drugs are prohibited under EU legislation³ following toxicological studies that support the suspicion that nitroimidazoles have mutagenic and carcinogenic properties.

JRC-IRMM released a new reference material, ERM-BB124, which will enable food-testing laboratories to reliably determine low levels of nitroimidazole veterinary drugs in meat. The new reference material fills an important gap in the range of veterinary drug reference ma-

Nitroimidazole veterinary drugs are

terials available from JRC-IRMM. The new reference material is an incurred pork muscle material certified for six nitroimidazole compounds (three parent drugs and three metabolites) at concentrations in the sub to low $\mu g/$ kg-range. These concentrations are in line with the guidelines of the responsible Community reference laboratory of 3 µg/kg per nitroimidazole compound.





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Nanoparticle reference

The reference material – which was processed and certified in accordance to ISO Guides 34 and 35 – is intended to be used for method validations (including trueness estimation) and method performance control, and shall contribute to enhance the quality and reliability of nitroimidazole measurements in food testing laboratories.



In 2008, JRC-IRMM released its first microbiological reference materials in the BioBall[™] format.

Microbiology

The control of food and feed for microbial organisms is essential to ensure the safety and quality of food and feed. Human and animal wellbeing and the probability of outbreaks of food- or feed-borne pathogens depend largely on the efficiency of microbial testing schemes. Reoccurring outbreaks of food-borne pathogens highlight the need to maintain an adequate level of microbiological control and method performance.

Microbiological certified reference materials (CRMs) are indispensable tools for the internal and external quality control in the area of microbiological analysis. JRC-IRMM released its first CRMs in the BioBallTM format, which are intended to be used for the development and validation of microbiological detection methods for the food pathogens, *Escherichia coli* O157 (*E. coli*) and *Salmonella enteritidis* (*S. enteritidis*), in particular for presence/absence tests. They offer a major advantage for laboratories since the absence of zero counts eliminates the need for testing a considerable number of samples.

JRC-IRMM already offers CRMs as spray-dried contaminated milk powder, covering six different food and water micro-organisms. However, measurement effects at low colony-forming-unit (cfu) level encouraged JRC-IRMM to consider alternative approaches for the development of certain reference materials. JRC-IRMM cooperated with BTF in Australia on the production of materials with a precise number of bacterial cells into a single BioBall[™] using a modified flow cytometer.

The new CRMs are IRMM-351 (containing *E. coli O157*) and IRMM-352 (containing *S. enteritidis*). The certified values (around 5 cfu) are based on an interlaboratory characterisation study, and the uncertainty budget for the certified values was estimated by applying a new statistical approach considering the low cfu level.

Another new CRM format has been developed for the authentication of bacteria. It is based on agarose inserts containing genomic DNA of bacteria and can be used for analysis by pulsed field gel electrophoresis, which is an indispensible tool for molecular typing or authentication of micro-organisms. The first two CRMs of this type, IRMM-311 and IRMM-312, contain genomic DNA of *Bacillus licheniformis* and *Bacillus subtilis*, respectively. Both *Bacillus* strains are authorised in the EU as probiotic feed additives.

Strict legislation on the use of probiotics as feed additives exists in the EU, and compliance is checked by official control laboratories in the Member States. These new CRMs will allow these laboratories to taxonomically identify the approved *Bacillus* strains and to discriminate them from other, possibly unapproved, probiotic *Bacillus* strains.

Reference materials

New activity on biofuels

The international trade of biofuels is anticipated to grow steadily, and a need for further harmonisation of biofuels standards was identified in the recent White Paper⁴ authored by a tripartite task force from Brazil, the EU and the US. Reliable and comparable measurements of biofuels are also required in the frame of EU legislation⁵ concerning biofuels for transport.

Technical standards defining the quality requirements for biofuels are of vital importance to biofuel producers, suppliers and consumers for quality assurance and allowing the free movement of these goods. In 2008, JRC-IRMM launched a comprehensive feasibility study for the production of a biodiesel reference material, as biodiesel is the most common biofuel in Europe. Currently, the minimum quality requirements for biodiesel and the related test methods are described in the international standard, EN 14214. Comprehensive homogeneity, stability

and characterisation studies have been designed for the 20 different parameters set out in EC 14214, such as the fatty acid methyl esters content, water content, methanol content and oxidation stability. Together with NIST (US), INMETRO (Brazil) and other EU partners (VSL, NPL and LGC), JRC-IRMM also participates in the recently launched FP7 project, BIOREMA, aiming at evaluating the potential of current analytical methods for analysing and characterising biofuels.

These activities support the development of quality assurance tools for the growing biofuel market, in particular for calibrating instruments, validating measurement procedures and monitoring the performance of laboratories in this field.

Large-sized dried uranium and plutonium spikes

The JRC-IRMM produced a new batch of large-sized dried (LSD) spikes. Dried spikes are isotopic reference materials, of certified uranium and plutonium composition, and they are a fundamental part of the fissile material control of irradiated nuclear fuel. These spikes are applied to measure the uranium and plutonium content of dissolved fuel solutions using isotope dilution mass spectrometry. Prepared under carefully controlled conditions, the new LSD series of spikes, IRMM-1027k, is the first to be dispensed by a new automated robot system at IRMM – allowing production in large batches. A light cellulose layer is dried on the spike material in a penicillin-type ampoule to retain the spike at the bottom of the vial during transport to where it is to be used.

In 2008, an administrative arrangement between JRC-IRMM and the Directorate-General for Energy and Transport was signed for the supply of large-sized dried spikes. The Directorate-General for Energy and Transport coordinates regular inspection activities throughout Europe to make sure that plant operator's book-keeping and material accountancy is correct. JRC-IRMM large-sized dried spikes enable on-site laboratories (for example at Sellafield and La Hague) to achieve the required level of detection probability, and there is no other regular supplier of these spikes with the required accuracy.

Considering that a nuclear plant like that in La Hague has a reprocessing capacity of 1700 tonnes of spent nuclear fuel per year, corresponding to about 17 tonnes of plutonium, JRC-IRMM's spikes have a direct impact on nuclear security.





⁴ White Paper on Internationally Compatible Biofuel Standards, Tripartite Task Force Brazil, European Union & United States of America, 31 December 2007, http://www.nist.gov/public_affairs/biofuels_report.pdf

⁵ Directive 2003/30/EC of the European Parliament and of the Council of 8 May 2003 on the promotion of the use of biofuels or other renewable fuels for transport.



Uranium isotopic reference materials

Isotopic reference materials are extremely important for nuclear safety and security. They are indispensable tools to establish traceability of a measurement result to a common reference and provide comparability of measurement results. They play a vital role in verifying the non-diversion of nuclear materials from their intended and declared peaceful use.

In 2008, JRC-IRMM added two new uranium isotopic reference materials to its range. The "double spike" series (IRMM-3636) has an isotope amount ratio of ²³³U/²³⁶U of 1:1, and is particularly suitable to perform internal mass fractionation corrections during a mass-spectrometric measurement. It can also be used as a spike to measure the uranium content of the sample by isotope dilution mass spectrometry. The single highly-enriched ²³⁶U spike isotopic reference material (IRMM-3660) is very suitable for measuring the uranium content of samples by isotope dilution mass spectrometry, due to the extremely low abundance of ²³⁶U in nature.

The development of these new isotopic reference materials allows the European Commission to fulfil the legal requirements⁶ concerning nuclear reference measurements, as well as responding to the requirements of the International Atomic Energy Agency to improve the existing analytical applications for nuclear safeguards.

These reference materials are of major importance for isotopic "fingerprinting" of various uranium materials in nuclear safeguards. However, their use is extending to earth sciences applications – helping laboratories in the geochemistry field to improve the quality and comparability of nuclear measurements on environmental samples.



Glove box for vacuum deposition of ²³⁵U.



Nuclear data and measurements

Accurate neutron data are required for the assessment of safety aspects of nuclear power installations or for the study of innovative concepts like nuclear waste transmutation or accelerator-driven systems. A fundamental parameter is the neutron cross section, which is related to the probability that an interaction of a given kind will take place between a nucleus and an incident neutron.

Databases of neutron-induced reaction cross sections are maintained by the Nuclear Energy Agency (NEA) of the Organisation for Economic Co-operation and Development (OECD). The NEA databank maintains a high-priority request list of the most important nuclear data requirements. In particular, the Joint Evaluated Fission and Fusion project of evaluated neutron data hosts a comprehensive database for routine applications in various areas of science and technology.

JRC-IRMM uses specialised infrastructure – comprising a 150 MeV linear electron accelerator (GELINA) and a 7 MeV light-ion Van de Graaff accelerator – to measure neutron cross sections and improve knowledge of reaction data, focussing on the following applications:

standards

ntroductio

- safety of operating reactors
- handling of nuclear waste and waste transmutation
- design of new and innovative types of reactor systems and fuel cycles

In November 2008, a new European project, EUFRAT, was launched to enable researchers to perform experiments at the two accelerator facilities at JRC-IRMM. Through the EUFRAT project, researchers can apply for a total of 4500 hours experimental time for neutron data measurements at the accelerators at JRC-IRMM. 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 call for proposals is permanently open, and proposals can be submitted at any time until 31 August 2011 through the EUFRAT website⁷.

Identification of a shape isomer in uranium-235

The plot of potential energy against nuclear deformation of a heavy nucleus exhibits a doublehumped barrier shape, which inhibits spontaneous fission of the nucleus. The intermediate valley is known as the shape isomeric valley from which shape isomeric fission can occur.

This picture has been very successful in explaining a number of phenomena observed in nuclear reaction data, e.g. the intermediate structure in sub-threshold fission cross sections. The decay mode and half-life of a shape isomer directly probes the fission barrier height and penetrability, which both are important parameters for nuclear reaction modelling.

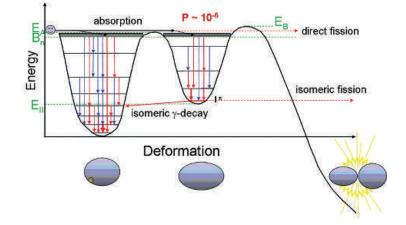
Using a beam chopper device at one of the beam lines of JRC-IRMM's Van de Graaff accelerator, the shape isomer in ²³⁵U was identified at a production level less than 10⁻⁵ per fission, and the half-life of 3.6 ± 1.8 ms was determined. The experimental results confirmed for the first time the existence of a super-deformed shape isomer in odd-numbered uranium isotopes.

Opposite page: Filament carburization device for environmental nuclear mass spectrometry.

7 EUFRAT: http://irmm.jrc.ec.europa.eu/eufrat, contract no. 211499, funded under the Seventh Framework Programme of the European Atomic Energy Community (Euratom) for nuclear research and training activities (2007 to 2011).

The experiments were carried out by researchers from the University of Örebro, Sweden, in collaboration with JRC-IRMM in the frame of the previous transnational access programme, NUDAME⁸. The work was published in Physical Review Letters⁹ and received the JRC Excellence Award 2008 for best peer-reviewed publication.

The nuclear energy landscape after absorption of a neutron. The fission barrier connects the ground-state deformed actinide (lefthand side) to the nuclear fission point (two separated ellipsoids at the right-hand side). The fission barrier parameters are determined from the half-life of the state located at the bottom of the intermediate valley, the so-called shape isomer, and the branching ratio of its isomeric fission and γ -decay mode.



Neutron inelastic scattering on lead-208

In the design of the new generation of nuclear power reactors, liquid lead or lead–bismuth eutectic are being considered as fast reactor coolants or as spallation targets for accelerator driven systems. For an accurate estimate of reaction rates in such systems, the kinetic energy distribution of neutrons must be known precisely.



Installation of a measurement sample with the germanium array for inelastic neutron scattering (GAINS).

The kinetic energy distribution is strongly influenced by neutron inelastic scattering, which is the process of a neutron bouncing off a nucleus while leaving it in an excited state. A neutron that undergoes inelastic scattering off a heavy nucleus loses much more energy than an elastic encounter – which reduces its kinetic energy by only about 1%. Sensitivity studies have shown that the probability for inelastic scattering off lead must be known with an uncertainty of less than a few percent to derive sufficiently accurate reaction rates.

To achieve this precision, accurate measurements were performed at the GELINA facility of JRC-IRMM using a new gamma-detector array for inelastic scattering. An unprecedented neutron energy resolution of 1.1 keV at 1 MeV (35 keV at 10 MeV) and a total uncertainty of about 5% was achieved for the integral gamma production cross section of the most intense transition in ²⁰⁸Pb. The results showed

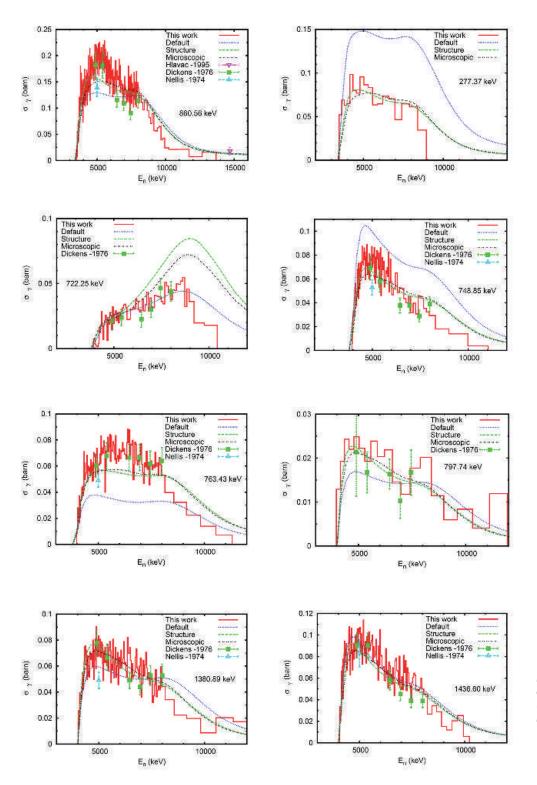
good agreement with the existing experimental data from the literature and with recent model estimates. The present work greatly expands the number of measured gamma production cross sections and the level cross sections derived for this nucleus, and the results were published in Nuclear Physics A¹⁰.

⁸ NUDAME, contract no. FP6-516487, funded under the Sixth Framework Programme of the European Atomic Energy Community (Euratom) for nuclear research and training activities, also contributing to the creation of the European Research Area (2002 to 2006).

⁹ Oberstedt A, et al., Identification of the shape isomer in ²³⁵U, Physical Review Letters 99; 2007, p.042502-1 042502-4.

L.C. Mihailescu, et al., A measurement of (n, xnγ) cross sections for ²⁰⁸Pb from threshold up to 20 MeV, Nuclear Physics A, Volume 811, 1 October 2008, Pages 1-27, ISSN 0375-9474.

Nuclear data and measurements



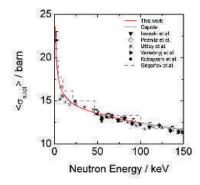
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Neutron inelastic scattering on ²⁰⁸Pb: experimental results obtained at GELINA (red curves) compared to previous measurements and model calculations.

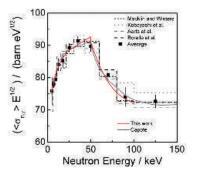
Nuclear data for the thorium-uranium fuel cycle

Past developments in nuclear technology have focused on the use of uranium in thermal reactors and fast reactors to improve the utilisation of natural uranium resources. However, there is increasing interest in innovative fuel cycle concepts based on thorium. A thorium-based nuclear fuel cycle offers many advantages. World reserves of thorium are larger than those of uranium and thorium fuel results in less long-lived higher actinides – which are the main source of long-term residual radioactivity in nuclear waste. JRC-IRMM evaluated neutron-induced reactions of ²³²Th in the unresolved resonance region from 4 to 100 keV. This work was performed as part of a coordinated research project organized by the International Atomic Energy Agency (IAEA) to evaluate nuclear data for the thoriumuranium fuel cycle¹¹.

The evaluation was based on a combined analysis of experimental average cross-section data (including the most recent capture cross-section data obtained at the time-of-flight facilities GELINA and n_TOF), and results of dispersive coupled-channel optical model calculations. Experimental data on transmission and self-indication measurements, as well as integral benchmark calculations, were used for validation.



Average capture cross-sections on ²³²Th, compared to previous results.

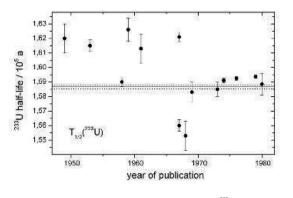


Total cross-sections on ²³²Th, compared to previous results

The evaluation resulted in average total and partial cross sections completely consistent with those in the high-energy region. Average parameters and their covariance matrix were deduced. These parameters can be used for uncertainty calculations, calculation of self-shielding factors, and for sampling resonance ladders in Monte Carlo simulations. The results were published in the Annals of Nuclear Energy¹².

Half-life of uranium-233

Accurate half-life data for long-lived uranium isotopes, such as ²³³U, ²³⁵U and ²³⁸U, are of crucial importance in different scientific applications, such as α -particle spectrometry, reactor physics, geochronology and radiological dating. However, knowledge of the half-lives of these uranium isotopes is still based on a few measurements performed decades ago.



Overview of the published measured values of the ²³³U half-life and their year of publication. The horizontal line corresponds to the ²³³U half-life (with combined standard uncertainty) recently obtained by JRC-IRMM and collaborators.

In collaboration with three European metrology institutes, JRC-IRMM has taken the initiative to determine the half-lives of ²³³U, ²³⁵U and ²³⁸U using state-of-the-art techniques. These experiments involve the combination of SI-traceable mass metrology for establishing the amount of material, mass spectrometry to determine the isotopic composition of the highly enriched uranium materials and finally primary standardisation techniques to measure the specific activity of a known amount of the radionuclide. JRC-IRMM holds a unique position in that it hosts all the necessary facilities and know-how, and additional measurement confidence was provided by the complementary measurements performed by the collaborating metrology institutes.

A new value for the half-life of 233 U of 1.5867 (14) x 10⁵ years was determined. A thorough treatment of the uncertainty budget was performed, which was not always the case in the past. The new value provides additional evidence to exclude previously-measured outlying values.

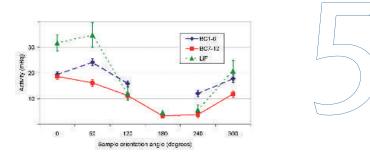
12 Sirakov I, et al., An ENDF-6 compatible evaluation for neutron induced reactions of 232Th in the unresolved resonance region, Annals of Nuclear Energy, 35; 2008, p.1223-1231.

11

CRP on evaluated nuclear data for thorium-uranium fuel cycle, http://www-nds.iaea.org/Th-U/public.html

In-vessel plasma diagnostics in JET

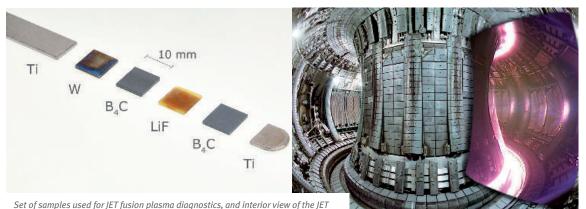
JRC-IRMM is collaborating with researchers at the Joint European Torus (JET) operated under the European Fusion Development Association (EFDA), to develop a new technique to enable in-vessel measurement of the leakage of charged particles from the fusion plasma. It is ultimately this leakage that determines how well a plasma can be contained.



The activity of ⁷Be per sample at the end of the last plasma pulse as a function of the sample position, for three different types of samples. The value without uncertainty bars is a decision threshold, i.e. an upper limit.

Six sets of samples facing six different directions were inserted inside the Tokamak. By measuring the very low level of activity induced by charged

particles and neutrons in these samples, it was possible to extract information that will lead to improved plasma diagnostics and ultimately better plasma control. This was the first time a probe could determine the angular distribution of leaking protons from fusion plasma.



vacuum vessel with a superimposed image of an actual JET plasma (Image: EFDA-JET).

The main proton-induced radionuclides detected were ⁷Be (in B₄C and LiF samples) and ⁴⁸V in Ti samples. Furthermore, an additional 11 radionuclides (mainly neutron-induced) were determined. The gamma-ray spectrometry measurements were performed by JRC-IRMM in the HADES laboratory – located 225m underground at the site of the Belgian Nuclear Research Centre SCK-CEN. In order to determine relatively short-lived radionuclides such as ⁴⁸V (T₂ = 16 d) and ⁴⁷Sc (T₂ = 3.4 d), some samples were measured simultaneously in two other underground laboratories that are members of the network CELLAR (Collaboration of European Low-level underground LAboRatories): Gran Sasso (INFN, Italy) and ASSE/UDO (PTB, Germany). The results were published in Nuclear Instruments and Methods in Physics Research A¹³ and Review of Scientific Instruments¹⁴.

Detecting polonium and uranium in drinking water

The geological setting strongly influences the occurrence of natural radionuclides in drinking water. Most radionuclides are at levels that are low enough to not be considered a public health concern. There are, however, areas where higher levels of naturally occurring radionuclides are found. Drinking water sources could also be contaminated from man-made nuclear materials. Measurements of their levels in drinking water are therefore important to reduce the potential exposure of the public.

¹³ J.S. Elisabeth Wieslander, et al., Low-level gamma-ray spectrometry for analysing fusion plasma conditions, Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment, Volume 591, 21 June 2008, Pages 383-393, ISSN 0168-9002.

¹⁴ Georges Bonheure, et al., In-vessel activation monitors in JET: Progress in modeling. Review of Scientific Instruments, Volume 79 (October 2008), 10E505.

European legislation¹⁵ requires public authorities to monitor microbiological, chemical and radioactive content in drinking water surveys. An upcoming revision of this legislation is anticipated to define reference values for more individual radionuclides than at present, and will require several radioactivity parameters to be monitored.

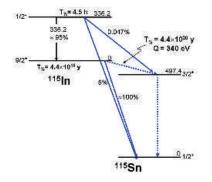
For this reason, JRC-IRMM developed a novel radiochemical method for the sequential determination of low activity levels of polonium and uranium in drinking water using α -particle spectrometry. The determination of ²¹⁰Po in water, food and in bioassay samples requires special attention due to its short half-life of 138 days and its high radiotoxicity, based on its high solubility in water and the risks of internal doses from α -emission after ingestion.

Two different approaches for the sequential determination of low-levels of ²¹⁰Po and uranium radioisotopes by alpha particle spectrometry were developed and compared. Two different preconcentration methods for polonium and uranium were used; after addition of ²⁰⁸Po and ²³²U tracers, the radionuclides were preconcentrated from water by co-precipitation with Fe(OH)₃ or MnO₂ at pH 9 using an ammonia solution. The ²¹⁰Po source for alpha counting was prepared by spontaneous deposition onto a copper disk before and alternatively after uranium separation using a UTEVA column. The uranium source for alpha counting was prepared by micro co-precipitation with CeF₃. The results in terms of measured activity concentration show no difference between the preconcentration and separation methods of radionuclides. A higher recovery for polonium, however, was obtained when the preconcentration with manganese dioxide was combined with the procedure where polonium is deposited onto a copper disk before the separation of uranium. On the other hand, the overall recovery for uranium depends on the duration of mixing in order to ensure the quantitative adsorption on the precipitate.

With the new method, a detection limit for 210 Po of 0.1 mBq·L-¹ was obtained, and the results are in press, to be published in Applied Radiation and Isotopes.

Measurement of the lowest known decay energy

A very rare decay branch in the isotope ¹¹⁵In was detected when a 2.5 kg piece of ultra-pure indium was measured for 77 days using gamma-ray spectrometry in the 225m-deep underground laboratory, HADES. The detection was possible thanks to the extremely low background radiation, which is 10,000 times lower than that of a normal gamma-ray detector above ground.



The decay scheme of ¹¹⁵In with the inclusion of the newly discovered decay branch (blue dotted line).

The 497.4 keV gamma-ray following the beta decay to the first excited state in ¹¹⁵Sn was detected, and the partial half-life of the decay was calculated to be $4.1(6) \times 10^{20}$ years. From the most recent mass evaluation, the decay energy is given as 1.6 ± 4.0 keV. It was thus debatable whether the decay could occur at all. JRC-IRMM collaborated with scientists from Jyväskylä University, Finland, who performed a new mass determination of ¹¹⁵In and ¹¹⁵Sn and calculated the decay energy for the newly discovered transition to be 0.35 ± 0.17 keV.

At 7.4 times lower than the previously lowest known decay energy in ¹⁸⁷Re of 2.6 keV, this is the lowest decay energy ever encountered. The result, beyond being spectacular in itself, may have an effect on setting limits for the anti-neutrino mass.

Opposite page: Pre-concentration of polonium and uranium isotopes from water by co-precipitation with Fe(OH)3 (back), with MnO2 (right) after adding KMnO4 and MnCl2 at pH 9.





JRC-IRMM

Food, biotechnology & health

To implement food safety policies, and to ensure the quality of testing in the field of life sciences, validated and reliable testing methods need to be developed. For this reason, JRC-IRMM develops, validates and tests analytical methodologies and explores the testing requirements set by biotechnology research. In some cases, also the development of a reference material for quality assurance of testing results may require development and validation of alternative or very accurate methods for characterisation.

The research topics range from identifying suitable markers for animal by-products not intended for human consumption to harmonising and improving the quality of genetic testing. Many of the methods developed and validated by JRC-IRMM with its collaborators are reviewed by international standardisation bodies and subsequently adopted as standards.

Melamine in Chinese milk powder

Jtroductio

Melamine is a nitrogen-rich organic compound, with the chemical formula C₃H₆N₆, 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.

In 2007, melamine was detected in vegetable protein products of Chinese origin destined for pet food manufacture, and in September 2008, melamine-tainted dairy products in China came to the attention of the general public. Concerns were raised 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 have traces of milk pow-

der. The European Commission consequently decided¹⁶ that composite products, including feed, that contain milk products originating in or consigned from China shall be checked, including laboratory analysis. Products containing more than 2.5 mg/kg melamine are to be immediately destroyed.

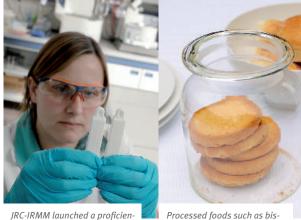
In response to this issue, JRC-IRMM set up a website to disseminate information about existing analytical methods for the detection of melamine in food and feed. Upon request of Directorate-General for Health and Consumers, JRC-IRMM launched a proficiency test to get an overview of the testing capabilities of laboratories in Europe, North America and the Asia-Pacific region. More than 100 participants from the EU, USA, New Zealand, Australia, Taiwan and China registered, and the results will be announced in 2009.

Contaminated sunflower seed oil

Food may come into contact with mineral oil, such as lubricants or binding agents, during harvesting, storage, processing, and packaging. Jute and sisal fibers used for manufacturing bags are treated with batching oils consisting of brown mineral oil. Printing inks used on food packaging materials may contain paraffin oils, which can migrate from the packaging into the food. Even in untreated plant material, traces of paraffin oils can be found as a result of atmospheric contamination due to soot deposition from vehicles, domestic heating systems and industry.

cv test in 2008 to benchmark laboratories' ability to measure melamine in food.

Processed foods such as biscuits and chocolate miaht have traces of melamine-tainted milk powder (© imageshop).





Ultra performance liquid chromatography: loading the autosampler tray.

High levels of mineral oil were detected in sunflower seed oil imported from Ukraine in the first half of 2008. The paraffin oil content in those imports was much higher than what could be expected from atmospheric or other background sources of contamination. The EU consequently introduced a screening level of 50 mg/kg of mineral paraffins in Ukrainian sunflower oil. JRC-IRMM supported official European food control laboratories by organising a training workshop on suitable methods for the quantification of mineral paraffins, together with the Directorate-General for Health and Consumers and the Official Food Control Authority of Zurich. Following this, JRC-IRMM organised an interlaboratory study to find out whether current methods to test imports were sufficient to comply with the screening level. More than 50 laboratories participated and the majority of laboratories proved to be competent to carry out the test.

Food processing contaminants

Industrial and domestic processing of food, in particular heating, creates a multitude of compounds that impart aroma, taste, and colour to food products. However, several of the generated substances may exert a negative impact on human health, such as acrylamide, furan and benzene.

European legislation requires the generation of reliable EU-wide data on levels of furan in heat-treated foodstuffs, so that the European Food Safety Authority can carry out proper risk assessments¹⁷. To ensure that testing laboratories provide valid and comparable data, JRC-IRMM first collected information regarding the applied analytical methods, then organised a proficiency test for European laboratories. The high volatility of furan makes its analysis a real challenge. Frequent monitoring at JRC-IRMM of the test material proved that its furan content did not change throughout the duration of the comparison. Nearly three quarters of the participating laboratories reported values that were in acceptable agreement with the reference value of the test material, demonstrating their proficiency in furan analysis.

Similarly, European legislation requires the monitoring of acrylamide in foodstuffs throughout the EU¹⁸. To provide an external benchmark for European laboratories, JRC-IRMM organised a proficiency test for acrylamide measurements in potato crisps. Available exposure data indicates that roasted coffee contributes significantly to acrylamide uptake. As roasted coffee is a difficult matrix for acrylamide analysis, JRC-IRMM developed and validated a method for the determination of this compound in coffee by ring-trial. The scope of the already standardised testing method, which was also validated by JRC-IRMM, will be enlarged to include roasted coffee.

Validated methods for mycotoxin analysis

According to the EU-wide early warning system for food toxicants (Rapid Alert System for Food and Feed), mycotoxins are among the most frequently found toxins in our food supply. The EU has set up an extensive regulatory framework to protect animal and human health from the effects of mycotoxins. In order to implement the system effectively, validated test methods, certified reference materials and external quality assurance systems must be made available to the official food and feed control authorities in the Member States.

¹⁷ Commission Recommendation 2007/196/EC of 28 March 2007 on the monitoring of the presence of furan in foodstuffs.

¹⁸ Commission Recommendation of 3 May 2007 on the monitoring of acrylamide levels in food.

In 2008, JRC-IRMM validated methods by collaborative studies for the quantitative determination of:

- ochratoxin A in animal feed
- fumonisins in animal feed, breakfast cereals and baby food
- T2/HT2 toxins in animal feed, cereals, and baby food

All methods were found to be fit-for-purpose as judged by internationally accepted criteria such as the HORRAT-value. The method protocols were submitted for formal standardisation to the technical committees within the European Committee for Standardisation (CEN) dealing with food and feed analysis.

Marker for unfit animal by-products

European legislation sets out rules for the safe use of animal by-products that are not intended for human consumption¹⁹. Animal by-products are sorted into three categories according to the associated risk, and materials in different categories must be kept separate during collection, handling and transportation. Furthermore, products derived from category 1 and category 2 need to be permanently marked, because they must not enter the feed and food chain. The ideal marker must be visible and detectable by its olfactory properties, non-toxic, safe for handlers, commercially available, inexpensive, stable, recoverable and easy to analyse.



Sample divider for homogenous distribution of material.

Bio-robot sample preparation.

Previous work by JRC-IRMM led to the proposal of glyceroltriheptanoate (GTH) as a suitable marker. As of 1 July 2008, European legislation requires that GTH has to be added to processed animal by-products that must not enter the feed and food chain²⁰. Towards the end of 2008,

19 Regulation (EC) No 1774/2002 of the European Parliament and of the Council of 3 October 2002 laying down health rules concerning animal by-products not intended for human consumption.

20 Commission Regulation (EC) No 1432/2007 of 5 December 2007 amending Annexes I, II and VI to Regulation (EC) No 1774/2002 of the European Parliament and of the Council as regards the marking and transport of animal by-products.



the WTO distributed a document concerning the requirements for exporting meat products which specifically recognises the role of the JRC-IRMM²¹.

The objective of this new requirement is to safeguard product traceability and to ensure that these materials are disposed of in a proper way. The enforcement of this new provision by official feed laboratories requires the availability of appropriate analytical methods. For this reason, JRC-IRMM published a detailed protocol of the analytical method that has been developed, which official feed laboratories can use for their investigations. In addition, this method is currently validated by conducting an interlaboratory validation study based on internationally accepted protocols.

Human genetic testing

Human genetic testing is an emerging field for which many issues have to be further developed and harmonised, from basic genetics to legal aspects. JRC-IRMM shares its competence in measurement standards in the European project EuroGentest²², which aims to structure, harmonize and improve the overall quality of genetic testing services.

In 2008, JRC-IRMM published a guidance document on the use of reference materials in genetic testing (EUR 23256 EN) and organised the EuroGentest workshop on reference materials for new genetic testing technologies. Participants from various research institutes, the International Federation of Clinical Chemistry, industry and the US Centers for Disease Control and Prevention, addressed new technologies looking at several genes at a time, in



contrast to traditional approaches. Discussion topics included how to perform the quality control for microarrays, how to validate all features on an array and which reference materials are most appropriate for those techniques.

The ISO Committee on Reference Materials (ISO REMCO) is working under the coordination of JRC-IRMM staff on a new international standard for defining requirements and documentation needs of qualitative reference materials, such as the ones needed for genetic testing.

Variability of a reference gene in maize

The quantification of genetically modified organisms (GMOs) is commonly based on real-time PCR analysis of the number of copies of an event-specific target gene in relation to copies of

²¹ G/SPS/GEN/889: Rules related to the export of meat-and-bone meal to third countries in order to ensure the prevention and control of certain transmissible spongiform encephalopathies (TSES), WTO Committee on Sanitary and Phytosanitary, http:// docsonline.wto.org/DDFDocuments/t/G/SPS/GEN889.doc.

²² EuroGentest, contract no. FP6-512148, funded under the Sixth Framework Programme of the European Community for research, technological development and demonstration activities, contributing to the creation of the European Research Area and to innovation (2002 to 2006).

an endogenous reference gene. Different detection assays for endogenous reference gene have been developed and validated for maize, targeting the single copy genes hmg, zSSIIb, lectin or adh1. During the systematic monitoring of JRC-IRMM's certified reference materials (CRMs) containing the GM events GA21 and NK603, an abnormally lower GM percentage was measured compared to the certified gravimetric ratio.

Further investigations have shown a more than two-fold difference in 3'-adh1 copy numbers between the non-GMO and the pure GM raw materials used for RM production. A similar discrepancy of the number of 3'-adh1 copies was observed when screening other GM maize varieties. Using a multi-target plasmid as a calibrant in real-time PCR assays, it was confirmed that the ratio between the copy number measured by the 3'-adh1 assay and that measured by the other 3 reference assays was about 50 % in most maize varieties tested, and only 20 % in the varieties that had earlier shown a two-fold reduction in 3'-adh1 content using the genomic calibrant.

Based on the results of this study, it was suggested to revise the detection method for GA21 and NK603 maize and to agree on uniform taxon-specific reference gene assays presenting no allelic variation between the same taxon. The results of this research were published in the Journal of Agricultural and Food Chemistry in 2008²³.



JRC-IRMM has proposed improved detection methods for genetically modified maize (© image100 ltd).

Analysis with real-time polymerase chain reaction (PCR).

23 Broothaerts W, et al., A Single Nucleotide Polymorphism (SNP839) in the adh1 Reference Gene affects the Quantification of Genetically Modified Maize (Zea mays L.), Journal of Agricultural and Food Chemistry 56; 2008. p. 8825-8831.



Evaluating measurement competence

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.

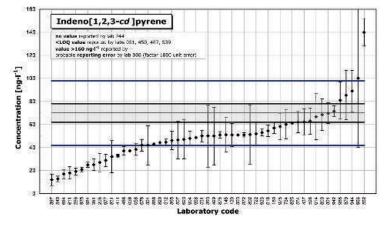
The JRC-IRMM organises proficiency tests for designated laboratories e.g. the national reference laboratories. It also organises inter-laboratory comparisons (open to all laboratories that wish to benchmark their capabilities), and pilot and key comparisons of the committees of the International Committee for Weights and Measures (CIPM) in its competence areas. The inter-laboratory comparisons organised by JRC-IRMM support new or modified European legislation and the monitoring of policy implementation across the EU.

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 2008 of measurement campaigns organised by JRC-IRMM to benchmark measurement competence and proficiency. Further examples can be found in this report in the sections describing the food safety activities and the Community reference laboratories.

Polycyclic aromatic hydrocarbons in water

The Water Framework Directive²⁴ provides a legislative framework for the protection of inland and coastal waters in the EU. In 2001, the European Commission decided²⁵ to establish a list of priority substances, and in 2008 it laid down maximum levels for these substances and certain other pollutants²⁶. The eight polycyclic aromatic hydrocarbons (PAHs) considered priority substances were the subject of an international measurement evaluation organised by JRC-IRMM in 2008.



The matrix used in the study was ground water with humic acid – which was added to simulate colloidal organic matter in surface water. The PAH concentrations were set close to the levels of the above-mentioned proposal when practically feasible. The presence of humic acid made the certification campaign very complicated. PAHs adsorb onto humic acid and this can lead

IMEP-23 addressed polycyclic aromatic hydrocarbons (PAHs) in ground water. The results shown are for indeno[1,2,3-cd] pyrene.

²⁴ Directive 2000/60/EC of the European Parliament and of the Council of 23 October 2000 establishing a framework for Community action in the field of water policy.

²⁵ Decision No 2455/2001/EC of the European Parliament and of the Council of 20 November 2001 establishing the list of priority substances in the field of water policy and amending Directive 2000/60/EC.

²⁶ Directive 2008/105/EC of the European Parliament and of the Council of 16 December 2008 on environmental quality standards in the field of water policy, amending and subsequently repealing Council Directives 82/176/EEC, 83/513/EEC, 84/156/ EEC, 84/491/EEC, 86/280/EEC and amending Directive 2000/60/EC of the European Parliament and of the Council.

to material losses that may remain undetected if no internal standard is used or the internal standard is given insufficient time to reach the adsorption equilibrium before further sample treatment. There are indications that a number of participating routine laboratories have overlooked this effect. A total of 59 participants from 27 countries reported their results, and z-scores were calculated with a target standard deviation of 20% of the reference value. The scores were satisfactory for approximately 80% of the participants. In summary, the measurement capabilities of those laboratories involved in routine PAH measurements in the frame of the WFD appear quite positive, despite some clear points for improvement.

International Comparison Scheme for Radioactivity Environmental Monitoring

In order to assess the radioactive exposure of the population as a whole, Member States are required by European legislation²⁷ to monitor radioactivity levels in certain environmental and food matrices.

Since 2003, JRC-IRMM helps the Directorate-General for Energy and Transport gain insight into the measurement methods and the quality of the values reported by the Member States, by organising measurement comparison exercises.

The Member States' laboratories who had participated in such comparisons met for the first time to discuss the results in a workshop hosted by JRC-IRMM. The comparison samples consisted of air filters, milk powder and three different mineral waters. A range of radionuclides had to be determined in these three exercises: γ -ray emitting ¹³⁷Cs and ⁴⁰K, α -decaying ²²⁶Ra, ²³⁴U and ²³⁸U and the β -decaying nuclides ⁹⁰Sr and ²²⁸Ra. Reference values for all comparison samples had been established at JRC-IRMM with traceability to the SI system of units, thanks to JRC-IRMM's unique radionuclide measurement infrastructure.

A detailed analysis of the results revealed that the coherence of measurement results with the reference value does not depend on a particular method of measurement or radiochemical separation applied in the corresponding laboratory. For any separation method used, there are laboratories which manage to determine values within the uncertainty limits of the reference value, but for almost all methods there are also those laboratories which produce largely deviating results. Clearly some of the participating laboratories need to improve their skills and implementation of the methods, and the further harmonisation of the European measurement system in radioactivity remains a challenging task.

Environmental sampling for nuclear safeguards

Environmental sampling has become an important tool for the detection of non-declared nuclear activities. In environmental sampling, micrometer-sized uranium particles with an isotopic composition characteristic for the processes at the inspected facility need to be collected, identified and analysed.

Considering the potential consequences of the analytical results, these measurements need to be subjected to a rigorous quality management system. This has created a demand for interlaboratory comparisons and quality control tools particularly designed for these types of analysis. Consequently, JRC-IRMM produced, characterised, certified and dispatched 30 test samples to participants for the NUSIMEP-6 comparison. It was the first interlaboratory comparison organised by JRC-IRMM for laboratories dealing with analysis of uranium in particles. Participating laboratories received a test sample of uranium particles on a graphite planchet with undisclosed isotope amount ratio values $n(^{234}U)/n(^{238}U)$, $n(^{235}U)/n(^{238}U)$ and

²⁷ Commission Recommendation 2000/473/Euratom 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.

NUSIMEP-6: Uranium isot

Certified value for n (235U)/n (206U)

7.74835-03

 $n(^{236}U)/n(^{238}U)$. The uranium isotope amount ratios were to be measured using their routine analytical procedures.

Fifteen institutes reported results using different analytical methods, seven of which were from the IAEA network of analytical laboratories. The participants' measurement results were evaluated against the certified reference values, and z-scores were calculated. The results of NUSIMEP-6 confirm the capability of laboratories in measuring the ratio $n(^{235}U)/n(^{238}U)$ and $n(^{234}U)/n(^{238}U)$ in uranium particles. Difficulties were observed particularly for the ratio $n(^{236}U)/n(^{238}U)$.

7.60743-03 (***U)/n(***U) 7.46651-03 7.32975-03 7.18493-03 7.04393-03 6 90000-00 6.76218 03 0.02135-03 6 /8012-03 6.39057-03 E 023 52 2115 2110 623 152 354 240 \$52 290 100

The NUSIMEP-6 campaign provided invaluable insight into the measurement capabilities of the IAEA network of analytical laboratories for environmental sampling, which is important for the detection of clandestine nuclear activities.

NUSIMEP-6 measurement results and their associated uncertainties. These uncertainties are shown as reported, with various coverage factors and levels of confidence. The grey band represents the reference interval ($X_{ref} \pm 2u_{ref}$).

nt ratios in uranium particles

0.007 043 9 ± 0.000 003 5 [U=k-u (k=2)

EVALUATION	DESCRIPTION
melamine in foodstuffs	two samples containing melamine: skimmed milk powder and baking mix
IMEP-27	analysis of total Cd, Pb and As and extractable Cd and Pb in mineral feed
IMEP-105	analysis of total Cd, Pb and As and extractable Cd and Pb in mineral feed
mineral oil in sunflower oil	determination of mineral oil in contaminated sunflower oil, spiked sunflower oil and solvent solution
deoxynivalenol (DON) in cereal products	analysis of DON in cereal products and solvent solution
PAHs in meat products	analysis of 15+1 EU priority PAHs in sausage and solvent solution
FURAN-01	analysis of furan in baby food
acrylamide in coffee	analysis of acrylamide in roasted coffee
IMEP-104	analysis of total Cd, Pb, As, Hg and MeHg in seafood
IMEP-103	analysis of total Cd, Pb, Hg and extractable Cd and Pb in feed
IMEP-23	eight WFD priority PAHs in ground water with added humic acids
ochratoxin in food	analysis of OTA in spices
acrylamide	analysis of acrylamide in crisps
PAHs in edible oil	analysis of 15+1 EU priority PAHs in edible oil and solvent solution
IMEP-102	analysis of total Cd, Pb and Hg in mineral water
aflatoxins in food	analysis of aflatoxins in a peanut material
ochratoxin in feed	analysis of OTA in animal feed
fumonisins in food	analysis of fumonisins in breakfast cereals and baby food
fusarium toxins in feed	analysis of deoxynivalenol, zearalenone, T-2 and HT-2 toxins in animal feed
fusarium toxins in food	analysis of deoxynivalenol, zearalenone, T-2 and HT-2 toxins in baby food
fusarium toxins in cereals	analysis of deoxynivalenol, zearalenone, T-2 and HT-2 toxins in cereals
sucralose in food	analysis of sucralose in soft drinks by thin-layer chromatography
aflatoxins in pure solvent	analysis of aflatoxins B1, B2, G1 and G2 in acetonitrile
sweeteners in food	analysis of eight sweeteners in various food matrices
PAHs in solvent	analysis of 15+1 priority PAHs in acetonitrile
PAHs in primary smoke condensate	analysis of 16 PAHs in a smoke condensate mixture
NUSIMEP-6	uranium isotope amount ratios in uranium particles
REIMEP-18	isotopic abundances of uranium

Inter-laboratory comparisons started, ongoing or completed by JRC-IRMM in 2008 (reference material certification campaigns are not included).



Metrology for Europe

European metrology research programme

European metrology research took a big step forward in 2008, when the European Commission adopted a proposal to set up a joint European research programme in the field of metrology under Article 169 of the EC Treaty²⁸. Funding of around €400 million is earmarked for the programme; half of this will come from the countries involved and the other half will be allocated by the European Commission. Its work will be coordinated by the European Association of National Metrology Institutes (EURAMET), of which the JRC-IRMM is an associated member.

The European Metrology Research Programme (EMRP) is a long-term plan for joint R&D amongst the top-level metrology community in Europe. The first phase of implementation is already underway with funding from the ERA-NET project, iMERA+.

JRC-IRMM contributes to the following three iMERA+ projects.

N _A h	The kilogram is the only one of the seven SI base units that still retains its 19th century definition. The aim of the N _A h project is to redefine the kilogram. JRC-IRMM contributes to the determination of the Avogadro number, N _A , with its expertise in isotopic measurements to count the atoms in a near-perfect sphere of ²⁸ Si.
Boltzmann constant	In the 'Boltzmann constant' project, researchers aim at determining an improved value of the Boltzmann con- stant for redefining the base unit of the kelvin by three fundamentally different primary methods: acoustic gas thermometry, Doppler broadening thermometry, and dielectric constant gas thermometry. JRC-IRMM contributes to this project by provision of its argon isotopic measurement expertise.
CLINBIOTRACE	Traceable measurement results in clinical chemistry are essential, and the aim of the CLINBIOTRACE project is to expand the available range of reference measurement procedures and reference materials of a higher order for bio-molecules of clinical significance. JRC-IRMM is contributing with its expertise in the production of reference materials for complex protein molecules and the standardisation of clinical chemistry measurements.

Supporting European legislation

Although European legislation is heavily dependent on measurements to set targets and monitor progress, there is currently no harmonised approach among the various Commission services to address in a systematic way the technical aspects of measurement quality.

In view of its metrology expertise, the JRC-IRMM took the initiative to set up a new inter-service group on "Measurement standards for European Legislation". The group is currently composed of representatives from the following Directorates-General: EuropeAid, Enlargement, Enterprise and Industry, Environment, Research, Health and Consumers, Taxation and Customs Union, Trade, and Energy and Transport. The group aims to provide clarity on measurement quality issues for a proper understanding, implementation and assessment of EU policies. A guidance document is being developed for this purpose. The group also aims to anticipate possible bottlenecks arising from the measurement requirements of new legislation.

The Water Framework Directive²⁹ is one example where JRC-IRMM has provided conceptual advice to policy regarding the quality of measurements. The quality and comparability of monitoring data is essential for the successful implementation of this Directive. JRC-IRMM was charged with the scientific co-ordination of the FP6 project, EAQC-WISE, which in 2008 delivered a blueprint of an efficient and potentially sustainable quality control system for



The Water Framework Directive is one example where JRC-IRMM has provided conceptual advice to policy regarding the quality of measurements.

²⁸ Article 169 of the EC Treaty enables the Community to participate in research programmes undertaken jointly by several Member States, including participation in the structures created for the execution of national programmes.

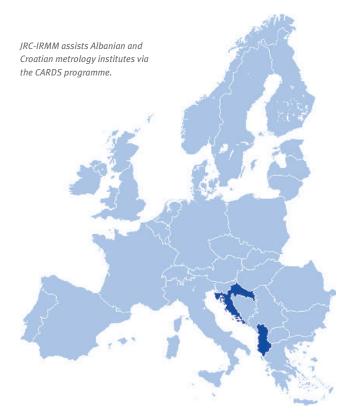
²⁹ Directive 2000/60/EC of the European Parliament and of the Council of 23 October 2000 establishing a framework for Community action in the field of water policy.

implementing the Water Framework Directive. The JRC-IRMM also drew on its expertise in measurement evaluation to deploy an interlaboratory comparison in support of the Water Framework Directive. Around 60 laboratories involved in monitoring water quality compared their ability to measure eight polycyclic aromatic hydrocarbons (PAHs) considered priority substances in the Water Framework Directive.

Technical support for accession countries

A key external relations' priority for the EU is to promote stability and peace in the Western Balkans. The establishment of a free-trade area in the Western Balkans and free-trade agreements with the EU require a functioning metrology system, which will also fulfil the requirements for environmental quality and food safety. For this reason, capacity building in the area of quality infrastructure is one of the activities receiving EU support under the Community Assistance for Reconstruction, Development and Stabilisation programme (CARDS)³⁰.

JRC-IRMM was requested by the Directorate-General for Enlargement to participate in the CARDS project: "Development of national metrology, standardisation, conformity assessment and accreditation system in Croatia".



Activities in 2008 focused on the development of a strategy for the Croatian accreditation agency (HAA), and helping to select reference laboratories in strategically important fields. Additionally, targeted support was provided to the Croatian Standards Institute (HZN) on the way to fulfil the CEN/CENELEC membership requirements. A number of workshops, seminars and working sessions involving local experts and practitioners were organised with the aim to provide training and raise awareness on crucial conformity assessment topics. Several important pieces of legislation relevant for the integration of the Republic of Croatia in the European Internal Market were revised, and interlaboratory comparisons in the food sector were organised.

Since 2007, EU funding for both candidate and potential candidate countries is channelled through a single, unified instrument called the instrument for pre-accession (IPA). This new instrument supersedes the previous pre-accession funding programmes like CARDS, Phare, ISPA and SAPARD. For the 2008-2010 multi-annual indicative programmes dealing with metrology, accreditation and standardisation projects, JRC-IRMM provided technical support for the IPA Quality Support Group and reviewed six project fiches with a total budget of €19.5 million.

Measurement Science in Chemistry

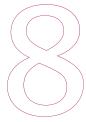
A high-quality, well-functioning measurement infrastructure in Europe depends not only on reference materials and measurements, but also on the availability of trained and competent practitioners. JRC-IRMM is very active in the area of knowledge transfer – striving to ensure the widest possible implementation of best practices in measurement science.

JRC-IRMM coordinates the Training in Metrology and Chemistry programme (TrainMiC[®]) which provides a harmonised interpretation of ISO/IEC-17025 – the main standard used by testing and calibration laboratories. It is organised as a network of national training providers dealing with topics like validation, calibration and uncertainty reporting. Three new teams started their activities in 2008: the former Yugoslav Republic of Macedonia, Albania and Bosnia-Herzegovina. Since 2001, a total of 4500 practitioners have been trained. In 2008, a User Licence Agreement was created and formalised in a Commission Decision of 17 Dec 2008. This document stipulates the rights and obligations of the authorised trainers within TrainMiC[®].

The promotion of measurement science advanced significantly in 2008, when a consortium of nine EU universities (coordinated by the University of Tartu, Estonia) offering a harmonised MSc programme, 'Measurement Science in Chemistry' was awarded the prestigious quality award from the European Chemistry Thematic Network Association (ECTNA). The consortium was originally brought together by JRC-IRMM, which continues to follow the consortium in a mentoring capacity.



Prof. J. Zupan presents the Euromaster quality label on behalf of ECTNA to the MSc coordinator, Prof. I. Leito, in the presence of European Commissioner for Science and Research, Janez Potočnik, and the Slovenian Minister for Higher Education, Science and Technology, Mrs M. Kucler Dolinar (Ljubjana, 26 May 2008).



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Community reference laboratories

Following a series of food safety crises in Europe, a system of Community reference laboratories (CRLs) was set up to support national authorities in their efforts to keep food and feed free from dangerous substances. Their main tasks are evaluating, developing and validating methods of analysis, producing documentary standards, as well as organising comparative studies and proficiency tests for the networks of national reference laboratories assisting the CRLs. The JRC-IRMM operates four CRLs (feed additives, mycotoxins, PAHs and heavy metals in food and feed), and this important mandate is set out in European legislation³¹, ³².

Community reference laboratory: feed additives

Feed additives are an integral part of modern animal husbandry, which aims to improve animal production, performance and welfare. Such additives may not be put on the market without authorisation by the European Commission. The authorisation can only be granted after a scientific evaluation demonstrating that the additive has no harmful effects on human and animal health and on the environment.



The information that applicants have to provide for the assessment and the authorisation of feed additives is set out in European legislation³³. In 2008, the CRL for feed additives helped the Directorate-General for Health and Consumers to completely revise the chapter concerning the analytical methods that applicants have to propose for the determination of the feed additive in feed and other matrices. A major objective of the revision was to ensure that the proposed method would fit the needs for official control, which was not the case in the former dossier guidelines from 2001. As suggested by the CRL, one of the major changes made was the inclusion of the concept of method verification. For its implementation, the

CRL prepared a guidance document for the applicants, to provide them with the necessary information for the experimental design of the verification exercise.

Community reference laboratory: polycyclic aromatic hydrocarbons

Polycyclic aromatic hydrocarbons (PAHs) are a group of about ten thousand compounds. Contamination of food by PAHs largely arises from production practices. Grains and raw products for the production of edible oils may be contaminated with PAHs through drying. Food from animal origin can be contaminated through charcoal grilling, roasting or smoking. Some of the PAHs are of particular concern for human health due to carcinogenicity and mutagenicity or because they may enhance the adverse effects of another compound.

In 2008, the CRL for polycyclic aromatic hydrocarbons (PAHs) organised an interlaboratory comparison on the determination of 16 PAHs³⁴ in sausage meat and acetonitrile. The test

34 Known as the 15+1 EU-priority PAHs

³¹ Regulation (EC) No 1831/2003 of the European Parliament and of the Council of 22 September 2003 on additives for use in animal nutrition.

³² Commission Regulation (EC) No 776/2006 of 23 May 2006 amending Annex VII to Regulation (EC) No 882/2004 of the European Parliament and of the Council as regards Community reference laboratories.

³³ Commission Regulation (EC) No 429/2008 of 25 April 2008 on detailed rules for the implementation of Regulation (EC) No 1831/2003 of the European Parliament and of the Council as regards the preparation and the presentation of applications and the assessment and the authorisation of feed additives.



materials used in this exercise consisted of canned sausage meat spiked with the 16 PAHs and a solution of the analytes in acetonitrile, respectively. The participants were free to choose the method for the analysis of the materials. The laboratories' performance was expressed as z-scores, which were calculated from the final reported results for the analytes' contents in the

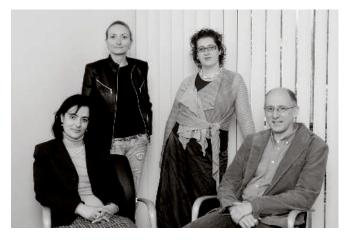


sausage meat material, based on gravimetrical preparation. For the sausage meat material, 88% of the reported values were attributed with z-scores ≤ |2|, indicating that most of the participating laboratories were performing satisfactorily with respect to internationally accepted standards. However, in some cases bias and/or a high variability were discovered, and some analytes consistently caused specific problems.

Community reference laboratory: heavy metals in food and feed

Heavy metals are present in all foodstuffs. Their amount in food and feed depends on the natural content and on the conditions under which food and feed are produced and processed. Some heavy metals have nutritional functions and are essential to the health. But others such as lead, cadmium and mercury have no nutritional relevance and can cause serious illnesses. To reduce the risk to human health associated with high heavy metal content in food and feed, maximum allowed limits in several commodities have been laid down in the European legislation.

In general, exposure to organic mercury can cause brain damage to a developing fetus. For this reason, the European Commission recommends pregnant women, breast-feeding women and children to limit their consumption of big fish predators in which high levels of methylmercury are known to be present. However, fully validated methods for speciation between organic and inorganic mercury compounds are currently not available. In order to improve this situation, and with a view to getting more precise data on methylmercury in fish, a proficiency test (IMEP-104) for the determination of total Cd, Pb, As, Hg and methylmercury in seafood was organised. Generally, the National Reference Laboratories (NRLs) taking part in the exercise performed well, and a high percentage obtained satisfactory scores. Four laboratories reported results for methyl-



mercury determination, with a satisfactory response, which should encourage the other NRLs to press ahead with the implementation of methods for methylmercury determination.

For farm animals, the basic nutrients required for growth, reproduction and good health include carbohydrates, proteins, fat, vitamins and minerals. Farm animals are often given a mineral feed to top up their dietary intake of essential minerals, such as sodium chloride, cobalt, iodine, zinc, molybdenum and selenium. However, too much of these minerals can be toxic, and



Community reference laboratory: mycotoxins

Mycotoxins are substances produced by certain fungi growing on food and feed. Estimates show that up to 20% of food products may contain mycotoxins, which can cause anything from mild to serious illness. To protect the consumers, European legislation sets maximum limits for the content of harmful mycotoxins in certain foodstuffs.

The CRL Mycotoxin organised a proficiency test for deoxynivalenol in cereal. The test materials used in this study were maize-based cereal flours, either free or naturally contaminated with deoxynivalenol. Laboratories determined the deoxynivalenol content by either enzyme linked immuno sorbent assay (ELISA), gas chromatography (GC) or reverse-phase high-performance liquid chromatography (RP-HPLC). Applying the Horwitz equation as a basis for the target standard deviation (19% in the case of this proficiency test), 27 out of 34 laboratories reported



values within the z-score limit of 2 after recovery correction of the result for the deoxynivalenol-positive sample. Twenty-five laboratories reported results within a z-score limit of 1. Thus, 79 % of the participating laboratories performed satisfactorily in the proficiency test.

Selected training events

The availability of suitably-trained researchers is a key challenge in many scientific domains today, particularly in the nuclear field. JRC-IRMM is active in several important knowledge transfer activities – a selection of which is described below.

Neutron measurements

In October 2008, the JRC-IRMM organised the fifth workshop on "Neutron Measurements, Evaluations and Applications" (NEMEA-5) in Ljubljana, Slovenia, in the frame of the FP6 project, CANDIDE³⁶. This important event for the nuclear data community – initiated by JRC-IRMM – was attended by 36 scientists from 15 countries. Participants presented and discussed the state-of-the-art and new developments of methods to produce high-quality nuclear data for advanced reactor applications. The findings of the workshop will be used to advise research programme managers on nuclear data projects for the short, medium and long term.

Mass spectrometry training

Upon request of the International Atomic Energy Agency (IAEA), JRC-IRMM gave mass spectrometry training for staff of the Safeguards Analytical Laboratory (SAL). The training started in 2008 and will continue into 2009. Through this activity, JRC-IRMM helps to strengthen the measurement capabilities of the Safeguards Analytical Laboratory, which has the mandate to analyse safeguards inspection samples of nuclear materials with the goal to verify that material is not diverted for non-peaceful purposes.

Minor isotope measurements in uranium

JRC-IRMM organised a workshop entitled "Measurements of minor isotopes in uranium in bulk and particle analysis" in Geel, 10-11 April 2008, to discuss the importance and applications of minor isotope measurements in uranium – also beyond nuclear safeguards purposes. The workshop aimed at reviewing the state of the art in measurement techniques and to identify future needs. The event was an excellent example of interinstitutional collaboration between JRC-IRMM and JRC-ITU. Both institutes support the European Safeguards Research and Development Association (ESARDA) by jointly chairing the ESARDA Working Group on Standards and Techniques for Destructive Analysis. Close cooperation between the two institutes extends to the supply of nuclear reference materials, the development of reference particles for environmental sampling and mutual training of JRC-IRMM and JRC-ITU staff.



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JRC-IRMM and JRC-ITU exchanged best practice on the set-up of a measurement procedure for minor isotope ratios in uranium.

Reference materials and the estimation of measurement uncertainty

Reference materials are key tools for achieving traceability of measurement results, proving accuracy of methods and demonstrating proficiency of laboratories. Since 2005, JRC-IRMM has given advanced training for external participants on the use of reference materials and the estimation of measurement uncertainty. During 2008, two such courses comprising lectures and practical exercises were organised. Participants included laboratory managers and practitioners in analytical laboratories who use reference materials for quality control, method validation and calibration. The courses were also attended by technical assessors of accreditation bodies performing audits for ISO 17025.

³⁶ CANDIDE: Coordination Action on Nuclear Data for Industrial Development in Europe, contract no. FP6- 036397, funded under the Sixth Framework Programme of the European Atomic Energy Community (Euratom) for nuclear research and training activities, also contributing to the creation of the European Research Area (2002 to 2006).

Facts & figures

Human resources

The total number of staff at JRC-IRMM at the end of 2008 was 299 persons. The staff is composed of Commission officials and employees on fixed-term contracts. Approximately 30% of posts are non-permanent to ensure flexibility, gaining of new knowledge and the ability to host PhD students, post-doctoral fellows and visiting experts. The staff on non-permanent posts are hired as contractual agents or seconded national experts. Around 40% of all staff are women.

CORE STAFF (END-2008)	
officials	191
temporary agents on 5-year renewable contracts	4
temporary agents on non-renewable contracts	24
FIXED-TERM STAFF	
PhD and post-doctoral fellows	21
seconded national experts	7
contractual agents	48
trainees	4
TOTAL	299

Budget

The Institute 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 Institute budget consists of institutional credits coming directly from the JRC budget for the 7th Framework Programme and the competitive income.

COMPETITIVE INCOME (K€) IN 2008 (% OF COMPLETION)	
Contracts with other DGs	2134
Participation in indirect actions, including CRLs	826
Work for third parties	435
Reference material distribution	2044
TOTAL	5439

The institutional credits contain staff expenses, technical and administrative support and operational appropriations. The competitive income comes from the distribution of reference materials, from participation in the indirect actions of the Framework Programmes and other competitive contracts with the Commission and work for third parties. Around 10-15% of the Institute's budget is competitive income.

Publications

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 EUR reports. For instance, reports on certification of reference materials and the results of many interlaboratory comparisons are published in the EUR Scientific and Technical Research series.

PUBLICATIONS 2008	
Article contribution to a monograph	3
Article contribution to a peer-reviewed periodical	58
Article contribution to other periodicals	4
JRC Scientific and Technical Reports	47
Contributions published in conference proceedings	61
PhDs	6
TOTAL	179

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Annex: Selected publications in 2008

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Abstract

The annual report of the JRC Institute for Reference Materials and Measurements describes the research highlights in 2008.

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