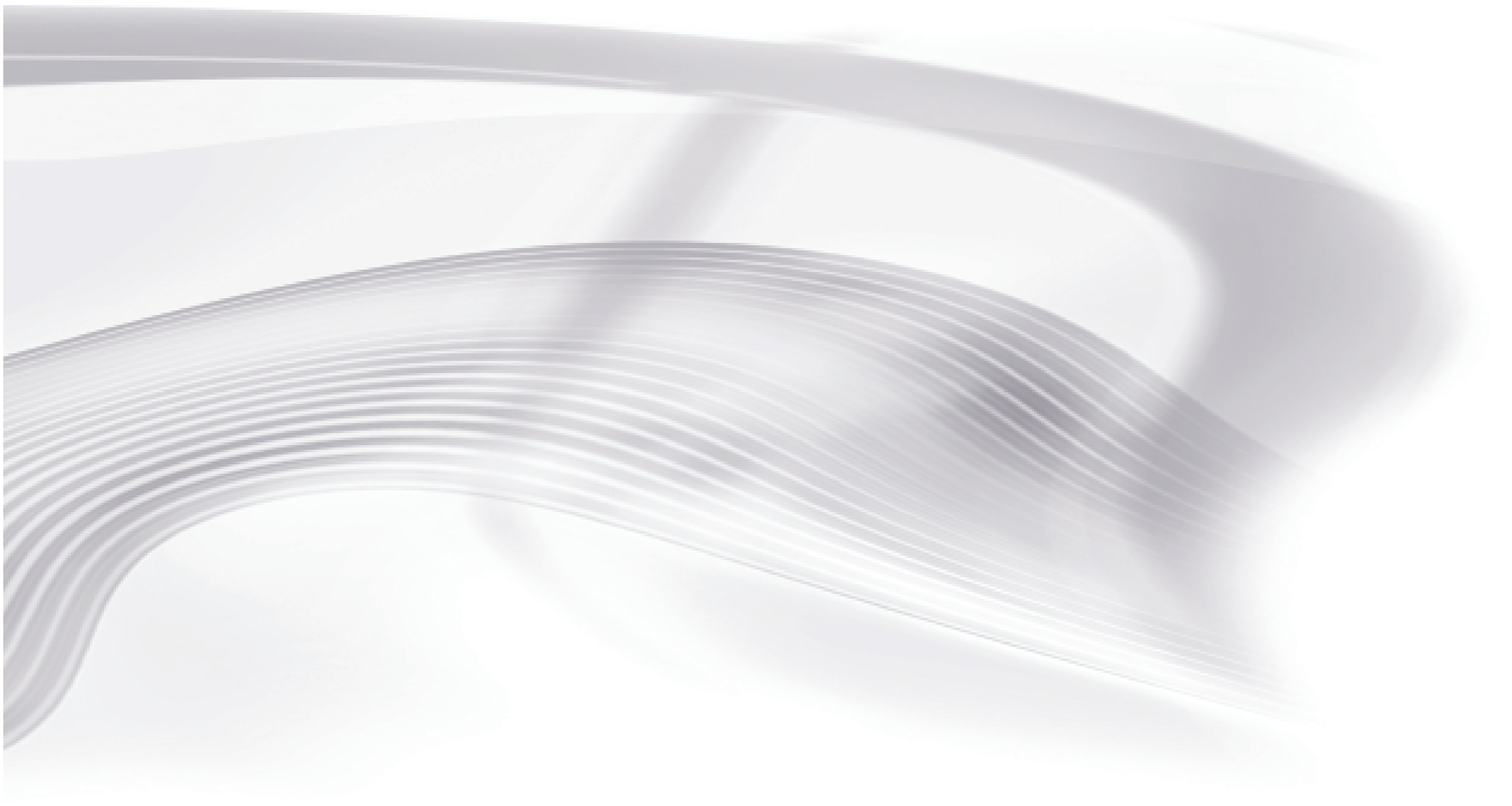

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Institute for Reference Materials and Measurements

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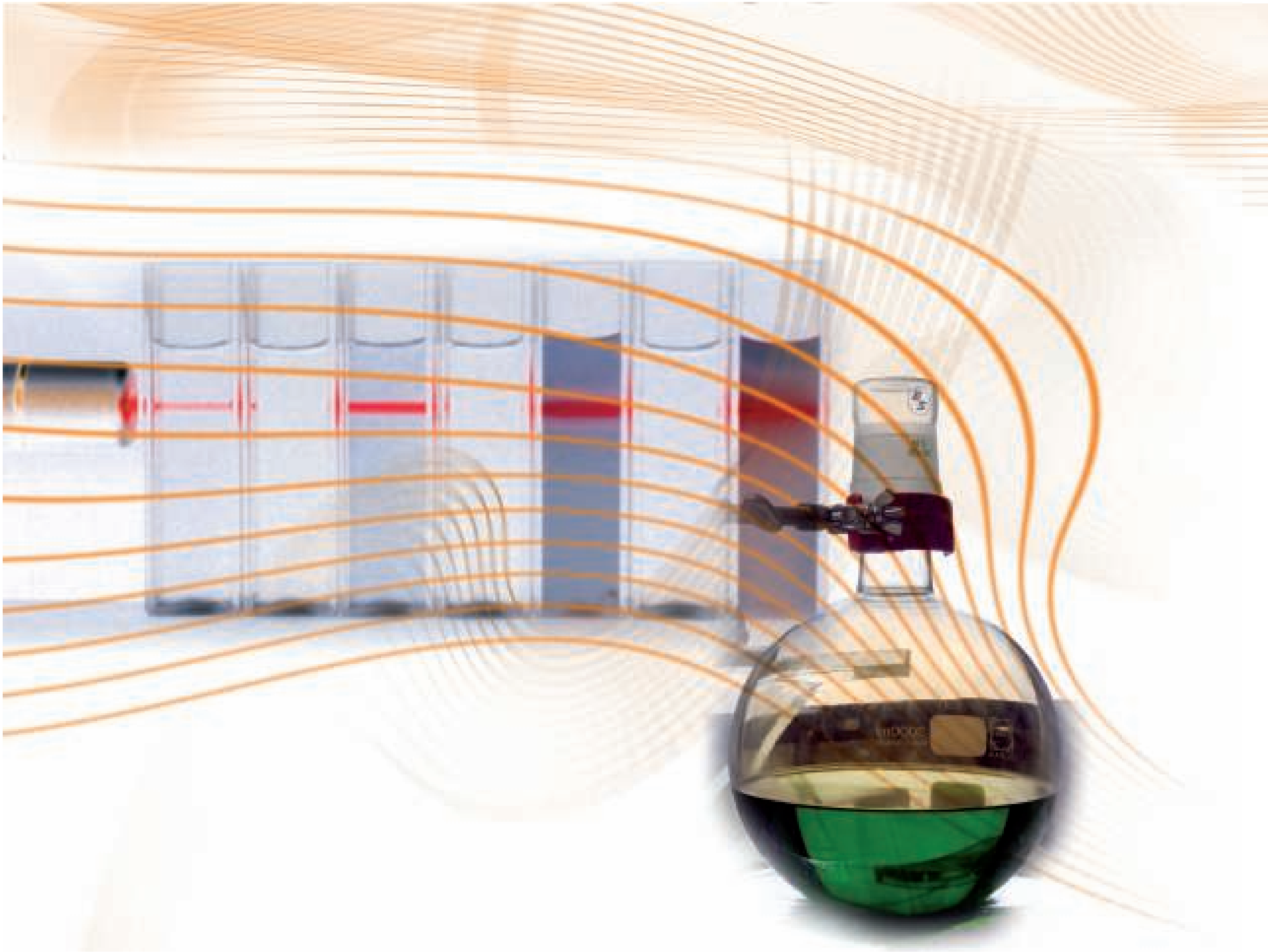


Message from the **Director**

The JRC-IRMM has a long history in validating methods of analysis, producing measurement standards and organising comparative and proficiency tests. Today, these are the core activities in its work programme that is based on the needs for scientific and technical support of other Directorates-General of the Commission.

This kind of expertise is also key for operating Community reference laboratories (CRLs). In 2004, the JRC-IRMM became the CRL for feed additives' authorisation. In 2006, its mandate was extended to cover also the control function, and the JRC-IRMM was designated as the three new Community reference laboratories (CRL) for heavy metals in feed and food, for mycotoxins and for polycyclic aromatic hydrocarbons (PAHs). These CRLs ensure that the testing of those substances is performed to a reliable standard across the food chain. In addition, the JRC-IRMM was appointed chair of the coordinating board of experts for monitoring water in poultry, similar in function to the CRLs.

Operating CRLs is a recent development at JRC-IRMM. The basis for such support function is built in the many research projects of the JRC-IRMM work programme. These projects span over several research fields from developing new reference materials for genetic testing to the analysis of semicarbazide in food and complex organic pollutants, measuring neutron cross sections and radioactivity in the environment or studying how to distinguish organically grown crops from those grown conventionally. Common for all is the drive to create confidence in measurements. In 2006, over 30 new sets of reference materials were added to the catalogue, and hundreds of laboratories participated in the interlaboratory comparisons organised by our institute.

Almost 50 years after signing the Treaties of Rome it is good to recall that the roots of the JRC-IRMM are in the nuclear research. The need for nuclear standards and nuclear data has not vanished, though, rather the new technologies have added to the requirements and revealed gaps in our knowledge. The NUDAME project that was started in 2005 to open access to JRC-IRMM accelerators for scientists of other organisations has yielded 18 experiments of shared interest, and seven of them were already executed by the end of 2006.

In August 2006, we said goodbye to Elke Anklam, the head of the Food Safety and Quality Unit who took up the position of the Director of our sister institute, the Institute for Health and Consumer Protection in Ispra. I would like to thank her for the efforts she put in setting up the food laboratories at the institute and starting up of new activities. Without a doubt we will continue to share experiences and best practices in the future.

2006 was also the last year of the 6^{th} Framework Programme for research. Taking stock of our work during those four years, I am proud of what the JRC-IRMM staff have achieved. The 7^{th} Framework Programme, however, will bring new challenges for us and our collaborators and I am looking forward to a fruitful 5-7 years ahead of us with many exciting projects to be realised.

Last but not least, I would like to thank our partners and customers for a successful year and all our staff for their efforts and dedication.

Alejandro Herrero

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 largest reference material producers and a provider of reference measurements and data.

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 operates now four Community reference laboratories.

Work programme

The research programme of JRC-IRMM is focussed on areas of policy relevance, and serves many Directorates-General e.g. for Health and Consumer Protection, Environment, Agriculture, Energy and Transport, Enterprise and Industry, Trade, Taxation and the Customs Union, Enlargement and External Relations. All Commission services can also request JRC-IRMM to carry out specifically targeted research projects outside its annual programme under a special administrative arrangement. As an institute of one of the Directorates-General of the European Commission JRC-IRMM performs tasks complementary to those of the Member States, especially where an independent approach at European level is needed. It works in close collaboration with national and international research organisations, metrology institutes, authorities, universities as well as with industry. JRC-IRMM has over 60 formal collaboration agreements and numerous other partners via informal and competitive activities. In 2006, the JRC-IRMM and the Flemish Institute for Technological Research (VITO NV) signed a collaboration agreement concerning scientific research on food and feed safety and environmental quality assessment.

Towards FP7

2006 was the last year of the 6th Framework Programme of the European Community for research, technological development and demonstration activities and of the European Atomic Energy Community (Euratom) for nuclear research and training activities. In December 2006 the European Parliament approved the 7th Framework Programme (FP7) for Community research for 2007-2013, and of the Euratom for 2007-2011.

The recoil proton telescope at JRC-IRMM is used for neutron fluence measurements

The specific programme of the JRC in FP7 is structured in policy themes and agendas. Due to the horizontal nature of JRC-IRMM's work, its activities fall under many of them. For instance under the policy theme 1 "Prosperity in a knowledge intensive society", the agendas on competitiveness and innovation, the European Research Area, life sciences and biotechnology are relevant for JRC-IRMM. Policy theme 2 "Solidarity and the responsible management of resources" contains topics like agriculture and climate change where tasks on analytical method development, validation and analytical quality assurance are assigned to JRC-IRMM. The policy theme 3 "Freedom, security and justice" contains research on food and feed safety and quality, that are core activities of the JRC-IRMM. The nuclear work programme of JRC-IRMM is included in the specific programme for the nuclear actions of the JRC.

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 in 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 Association of Analytical Communities (AOAC) International. Many testing methods validated by JRC-IRMM together with its collaborators have been approved as standards of CEN and ISO.

JRC-IRMM has upgraded its instrumentation in the underground laboratory

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 and small amounts of radioactive substances can be studied in the underground laboratory of JRC-IRMM located at the Belgian Nuclear Research Centre SCK•CEN in Mol (BE).

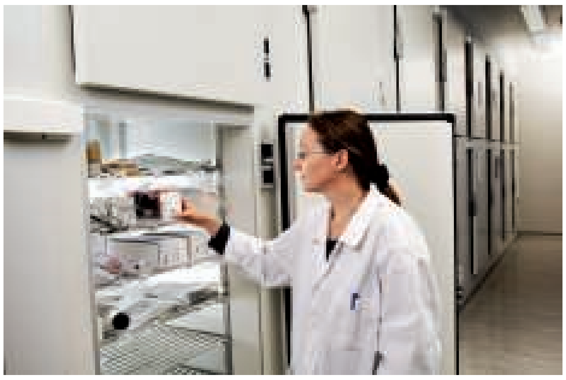
JRC-IRMM operates a 150 MeV linear electron accelerator (GELINA) and a 7 MV light-ion Van de Graaff accelerator. The two accelerators of JRC-IRMM, used for neutron production, are complementary in their experimental conditions and among the best such installations in the world. Since 2005 the two accelerators can accommodate external users via a project on access to large scale facilities (NUDAME).

Following food safety crises including BSE and the dioxin contamination, EU food law was substantially reformed (White Paper on Food Safety COM(1999)719 final) and as part of this a system of specialised Community reference laboratories has been created, cooperating closely with their national counterparts.

Community reference laboratories (CRLs) are analytical laboratories designated by Community directives and regulations. They are an integral part of European risk management system and help make the EU regulatory system more efficient and effective, with the same high level of laboratory performance across the EU. Their duties contain e.g.:

- setting up EU-wide standards to ensure comparability of testing results
- organising comparative tests
- training analysts from national laboratories
- providing scientific and technical assistance to the European Commission
- coordinating a network of national reference laboratories

The CRLs are also required to have knowledge of international standards and practices and keep lists of reference substances and reagents and their suppliers. With their networks of national laboratories the CRLs provide a platform for experts to share knowledge and best practices.



JRC-IRMM is operating the Community reference laboratory for feed additives since 2004

In 2006, three new CRLs started operation at JRC-IRMM and deal with testing of heavy metals, mycotoxins and polycyclic aromatic hydrocarbons (PAHs) in food and animal feed. Substances in these three groups can cause cancer and other serious illnesses or damage the central nervous system. In addition, in 2006 the mandate of the CRL assisting the European Food Safety Authority (EFSA) in the authorisation of feed additives was extended to cover activities related to control.

Community Reference Laboratory for Feed Additives

Since 2004 feed additives have to be authorised according to the procedure laid down in Regulation (EC) No 1831/2003. The procedure is designed on the basis of a strict separation between scientific assessment of the feed additive, which falls under the responsibility of EFSA, and the actual authorisation for placing the product on the market granted by the European Commission. The assessment of the feed additive also includes a close evaluation of the analytical methods that are proposed by the applicant to determine the active substance in various matrices such as animal feed. This evaluation and some other tasks are entrusted to the Community reference laboratory, which according to Regulation (EC) No 1831/2003 is the European Commission's Joint Research Centre. Within the JRC the IRMM has taken up this task. In addition, the Regulation (EC) No 882/2004 assigned tasks to this CRL that are related to the official food and feed controls.

The main task of the CRL is the evaluation of the analytical methods submitted by the applicant, establishing whether these methods are suitable for official control purposes. Analytical methods are for instance required to determine the active substance of the feed additive in animal feed and – if applicable – residues in animal tissue. The CRL is supported by a consortium of 35 European national reference laboratories (NRLs), which contribute to the evaluation procedure with their expertise on specific analytical methodologies. In case the submitted analytical methods fulfil the requirements for the intended purpose a favourable opinion will be given to EFSA without performing experiments. If necessary, the CRL may also test the method in its own or an NRL laboratory, or it may organise an interlaboratory comparison study to validate it.

In 2006, the CRL submitted final reports on the assessment of analytical methods for 20 dossiers to EFSA, and participated in proficiency tests to demonstrate its competence in producing reliable results as specified in its accreditation. It created expert groups to establish performance criteria for single-laboratory validated analytical methods. The other tasks of the expert groups are recommending analytical methods suitable for official control and contributing to the method data bank. Ultimately, experts' recommendations for performance criteria could provide the backbone for legislation on analytical methods relevant to feed, akin to the already existing Decision (EC) No 2002/657 on residues in food of animal origin. The CRL for feed additives also maintains a repository of reference samples of all authorised additives and keeps a database of methods for the analysis of specific feed additives.

Community Reference Laboratory for Heavy Metals in Feed and Food

Heavy metals are present in all foodstuffs. Some are important for our nutrition, but others, such as lead, cadmium and mercury, have no nutritional value and can in some cases contribute to serious illnesses such as cancer, or damage the central nervous system.

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. The Community reference laboratory for heavy metals was created to implement Regulation (EC) No 882/2004 on official controls performed to ensure the verification of compliance with the feed and food law, animal health and animal welfare rules. The task of the CRL for heavy metals is to facilitate the implementation of Regulation (EC) No 1881/2006 and Directive 2001/22/EC establishing the maximum levels of heavy metals such as lead, mercury and cadmium in different foods. The three types of matrices covered by the CRL are wild caught fish, food of plant origin and animal feed.

One of its core tasks is to organise interlaboratory comparisons for the appointed national reference laboratories. These campaigns are run within the International Measurement Evaluation Programme (IMEP®) of JRC-IRMM but then only the nominated laboratories can participate. In 2006, the CRL already organised one campaign for the national reference laboratories where their performance in measuring Cd, Pb and Hg in bread was evaluated. Development and validation of standardised analytical methods can also be carried out if the national reference laboratories ask for it, as well as training on the relevant European legislation.

Community Reference Laboratory for 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.

To ensure that results from food and feed monitoring fulfil the requirements of legislation, any method that is used for analysis of goods must meet established and accepted performance criteria. This is especially important in view of legal actions and trade specifications such as rejection of imports due to contamination, as well as monitoring and risk assessment studies. Both rapid screening methods and confirmatory methods are needed.

Animal feed extracts are being filtered before analysis of mycotoxins

This CRL at JRC-IRMM co-ordinates activities related to the development and improvement of methods of analysis for the official control of maximum levels and organises comparative tests for the network or national reference laboratories. The aim of the CRL is to facilitate the implementation of current and future European legislation for mycotoxins, e.g. Commission Regulation (EC) No 1881/2006 setting maximum levels for certain contaminants in foodstuffs. The JRC-IRMM has already validated a method for determination of patulin at the new legislative level of 10 ng/g in baby food and developed and validated methods for the determination of Fusarium toxins (such as deoxynivalenol, zearalenone and T-2/HT-2 toxins) in baby food and animal feed. In 2006 the CRL organised an interlaboratory comparison for the analysis of aflatoxins B1, B2, G1 and G2 in acetonitrile to assess instrument calibration.

Community Reference Laboratory for Polycyclic Aromatic Hydrocarbons

Polycyclic aromatic hydrocarbons (PAHs) are a group of about ten thousand compounds. Some of the PAHs are of particular concern for human health due to carcinogenicity and mutagenicity or since they may enhance the adverse effects of another compound. We are exposed to PAHs via air and drinking water, but mostly by intake of food. Contamination of food by PAHs largely arises from production practices. For instance, grains and raw products for the production of edible oils may be contaminated with PAHs through drying or food from animal origin can be contaminated through charcoal grilling, roasting, or smoking.

In 2006 the JRC-IRMM was appointed as CRL for PAHs. The CRL assists the national laboratories of the Member States by developing and validating methods of analysis for these substances, and by helping harmonise official controls. The CRL coordinates a network of national reference laboratories across the EU, organises proficiency tests for the assessment of the comparability of analysis data, and identifies needs for method development.

These activities refer in particular to Commission Regulation (EC) No 208/2005 setting maximum levels of benzo[a]pyrene (BaP) in various types of food, to Commission Directive 2005/10/EC laying down sampling and analysis measures for the official control of the levels of BaP in foodstuffs, and to Commission Recommendation 2005/108/EC, which proposes additional studies on the levels of PAHs in certain foods. The CRL for PAHs is also setting up a database on research projects related to PAHs in food. Its purpose is to help researchers across Europe to identify potential partners and to help authorities to address scientific questions related to PAHs to the respective experts. In 2006, the CRL organised an interlaboratory campaign on the analysis of "15+1 EU priority PAHs" * in acetonitrile to help the national reference laboratories in assessing instrument calibration.

Analysis of PAHs by solid phase extraction high performance liquid chromatography

** New European legislation was introduced in early 2005 to respond to food contamination problems, based on data collected by the EU Member States and assessment by the Scientific Committee on Food (SCF) in 2002. The SCF assessed 33 PAHs and recommended 14 of them be monitored in addition to benzo[a]pyrene (BaP), which was the only compound required that time. This is reflected in Commission Recommendation 2005/108/EC. In 2005, the Joint FAO/WHO Expert Committee on Food Additives (JECFA) brought up another PAH that is not covered by the Commission Recommendation 2005/108/EC. Therefore, EFSA and the European Commission have drawn up a priority list of the 15 PAHs mentioned in the recommendation complemented with the one specified by JECFA. The EU Member States are requested to provide monitoring data on these 16 PAHs to enable long-term exposure assessment. To distinguish this set of 16 PAHs from another set of 16 PAHs that was prioritised by the US Environmental Protection Agency (US EPA), the term "15+1 EU priority PAHs" is used.*

Integration and enlargement

Since 2001 the JRC-IRMM has actively supported the enlargement of the European Union by providing assistance and training in its fields of expertise to help the Acceding and Candidate Countries in taking up EU legislation. This assistance supports the Stabilisation and Association Process for the Western Balkan Countries. In 2006, the JRC-IRMM collaborated with a new research centre in Slovenia that was officially opened by European Science and Research Commissioner Janez Potočnik. The Slovenian-European Natural Sciences Research Centre (SENARC) provides training and opportunities to share knowledge for scientists from across South Eastern Europe. It focuses specifically on the field of the chemical and bioanalytical measurements required for the implementation of EU legislation in areas such as environmental monitoring. SENARC is funded from the EU's Instrument for Pre-Accession Assistance and Structural Funds, among other sources. It has evolved from collaboration of JRC-IRMM with 7 Slovenian research partners.

JRC-IRMM is also involved in EU funded projects (CARDS) carried out in Albania and Croatia. The establishment of a free trade area in the Balkans as well as a future free trade agreement with the EU requires a functioning metrology system, which will also fulfil the requirements set on quality of environment and safety and quality of food. The JRC-IRMM provides expert advice in capacity building and helps upgrading the legal framework related to metrology. These projects provide also for technical assistance and training and help increase public awareness. For instance, within the CARDS Albania project in 2006 a summer school on metrology in chemistry was held in Maribor, a training course on quality systems in Tirana and the Albanian metrology authority was advised in drafting strategies. Templates for technical documents needed for certification or accreditation were set up. In Croatia the projects aim to define national priorities for metrology and conformity assessment and assist in developing the quality infrastructure in Croatia.

Training

Passing on the best practices and experience is essential in all areas where JRC-IRMM is working. Training courses are regularly organised by the specialists of the institute and JRC-IRMM hosts a large number of PhD students, post doctoral fellows and young and senior visiting scientists, national experts and trainees every year.

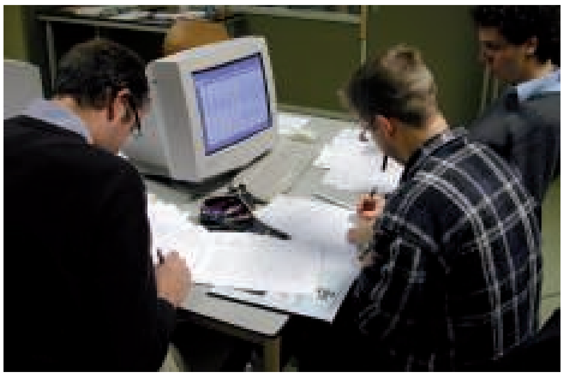
The TrainMiC programme of JRC-IRMM was started in 2001 to provide a platform for improving the quality of analytical results by promoting and providing a Europe-wide harmonised practitioner training in metrology in chemistry. In 2006, 662 practitioners participated in the 19 courses organised in ten countries. The JRC-IRMM has also initiated a AcadeMiC platform where European analytical chemistry lecturers can meet and share ideas and best practice to teach generic measurement science in analytical chemistry. Today, the organisations EUROLAB, EUROMET and EURACHEM as well as in total about 40 academic lecturers from 21 countries in Europe have signed the Rogaška Slatina declaration, which was drafted in the summer school of 2005 and calls for strengthening of university curricula in this field.

TrainMiC training session

The JRC-IRMM organises annually an advanced course on accelerators and time-of-flight measurements for students of the Belgian Nuclear Higher Education Network (BNEN). In 2006, six BNEN students followed the course, visited the accelerators and experimental set-ups and carried out practical experiments and measurements using the time-of-flight technique. The BNEN was created in 2001 by five Belgian universities and the SCK•CEN to maintain and further develop a high quality education programme in nuclear engineering in Belgium.

BNEN organises a one-year 60 credit academic programme called "Master of Science in Nuclear Engineering". The objective is to offer future professionals and researchers a solid background in the different disciplines of nuclear engineering.

In 2006, JRC-IRMM has enhanced contacts with local educational institutions. Collaboration agreements on scientific research were signed with the Katholieke Hogeschool Kempen (KHK) and the XIOS Hogeschool Limburg aiming to facilitate working together and the exchange of research results. The benefits of combining technical education with an opportunity to work with a research team in designing and constructing specialised equipment were highlighted when the seventh grade welding, pipe-fitting and construction class of VITO Hoogstraten, a school for professional education, ranked second in the 2006 competition for the Belgian GRAM prize*. The high ranking was based on making an aluminium array for measurements at JRC-IRMM's linear electron accelerator facility. The array is used at JRC-IRMM for the measurement of γ-emission probabilities associated with neutron induced reactions.



The JRC-IRMM organises annually an advanced course on accelerators and time-of-flight measurements for students of the Belgian Nuclear Higher Education Network (BNEN)

JRC-IRMM is a partner of the Virtual European Radionuclide Metrology Institute (VERMI) together with the radionuclide metrology departments of Laboratoire National Henri Becquerel (LNE-LNHB, FR), Physikalisch-Technische Bundesanstalt (PTB, DE) and National Physical Laboratory (NPL, UK). With the generation of scientists in nuclear research now changing, VERMI aims to maintain and develop the quality of European radionuclide metrology. Therefore VERMI organises training for young researchers in radionuclide metrology. The fourth VERMI wokshop was organised in 2006 in Varna (BG) and 34 staff from national metrology institutes, national authorities and research institutions participated. Topics covered statistics and uncertainty estimation as well as the major measurement techniques starting from the basics and ending in a detailed discussion of the state-of-the-art, difficulties and limitations of those techniques.

Local fire brigades practised situations requiring radiation and contamination measurements during an exercise at JRC-IRMM

Organising courses on the use of reference materials has now become an established activity of the institute. These courses provide participants with the scientific basis for the estimation of measurement uncertainty and establishing traceability. They are designed for laboratory managers and practitioners in analytical laboratories who use reference materials for statistical quality control, method validation and calibration. In 2006, 30 practitioners participated in the course.

In 2006 the JRC-IRMM has also shared its expertise on biosafety with the local fire brigades and carried out joined exercises with several teams of local fire brigades to practise situations requiring radiation and contamination measurements during the intervention. Additionally a course was given on transport issues and on the use of personal dosimeters.

** The GRAM prize (Gilde voor Revaluatie Arbeid en Motivatie) is an initiative that aims at motivation of students and stimulation of professional education through a competition for sixth and seventh grade classes throughout Flanders.*

JRC-IRMM connected to GÉANT

As the first JRC institute the JRC-IRMM is now connected to the GÉANT network. GÉANT is the world's leading research and education network providing networking services to researchers across Europe. GÉANT is co-funded by the European Commission and Europe's national research and education networks. The GÉANT directly interconnects more than 3 500 research and education institutions in 34 European countries.

The GÉANT network access offers a bandwidth of up to one gigabit per second (1Gbit/s). This high capacity allows communicating even faster than on a normal local area network. The JRC-IRMM has many research projects where such a network access can facilitate rapid transfer of large amounts of data or collaboration with research partners in real-time. For example, the tens of gigabits of neutron data measured at the JRC-IRMM can be rapidly transferred to the collaborators who can then analyse the data in their home institution after the experiment.

Access to JRC-IRMM accelerators – the NUDAME project

The JRC offers its nuclear research facilities to European research teams investigating topics such as radioactive waste management, radiation protection and nuclear technologies and safety. JRC-IRMM has two accelerators that are used for accurate neutron data measurements. Within the framework of the Euratom Transnational Access programme research teams could apply for measurement time at JRC-IRMM until the end of 2006. The NUDAME project started in the spring of 2005.

Experiment in the areas of radioactive waste management, radiation protection and other activities in the field of nuclear technologies and safety can be supported. Proposals are reviewed by a programme advisory committee composed of experts in the fields of interest. Approved experiments are granted the necessary beam time and the same scientific, logistical and technical support as provided to all researchers of the Institute. The user groups are also supported for their travel and the subsistence costs during the time required conducting the experiment. The users perform their experiments in close cooperation with an JRC-IRMM contact person and if needed, may use the experimental set-ups and data acquisition systems of JRC-IRMM. In total, 3000 hours of data acquisition is reserved for the project.

In December 2006, the committee selected the experiments to be carried out during the last year of the project i.e. the experimental period ending in March 2008. These experiments deal with capture cross sections of isotopically enriched Hf samples, the ^{235}U(n,n'γ) and ^{235}U(n,2nγ) reaction cross sections, neutron cross sections of the tungsten isotopes, population of the super deformed ground state in ^{235}U, very short-lived activation cross sections from inelastic scattering on ^{206,207}Pb and testing and calibration of neutron dosimeters for radiation protection.

Over the three years, the requested beam time largely exceeded the time that could be made available. Despite the fact that the scientific quality of the proposed experiments was generally very high, only 43% of the beam time requested could be granted. Out of 22 proposals 18 experiments have been approved, but in many cases the beam time had to be reduced. By the end of 2006 seven experiments had been performed.

NUDAME

Neutron Data Measurements

As from 2005 the two accelerators at JRC-IRMM can accommodate external users via a project on access to large scale facilities (NUDAME)

Accreditations

In 2004 the Reference Materials unit of JRC-IRMM was accredited in accordance with the requirements of two international standards, the ISO Guide 34 and the ISO/IEC-17025. The ISO Guide 34 gives general requirements for the competence of reference materials producers, and the ISO/IEC-17025 standard for the competence of testing and calibration laboratories. Accreditation of the Reference Materials unit then covered reference materials production and several testing activities such as quantification of genetically modified organisms. In 2004, JRC-IRMM became the first organisation in Europe accredited for the production of reference materials, and the re-audit in 2006 was successful. The ISO/IEC-17025 accreditation was updated to the version of 2005 of this standard.

Later in 2006 the Food Safety and Quality unit of JRC-IRMM was accredited in accordance with the requirements of the ISO/IEC-17025 standard covering analysis of vitamin A and coccidiostats in feedingstuffs, Ca, Fe, Mg and Zn in mineral mixtures, and Mn and Zn in wholemeal flour. For each analyte – matrix combination the method of analysis is also specified. The scope of accreditation was extended at the end of 2006 to include testing activities relevant to the operation of the CRLs for mycotoxins and PAHs (aflatoxin B1 in peanuts, ochratoxin A in animal feed and the EU 15+1 PAHs in liquid smoke flavouring products).

Other activities were also audited in the autumn of 2006, and accreditations are now pending. The Isotope Measurements unit was audited against ISO/IEC-17025 for its isotopic measurements using inductively coupled plasma mass spectrometry (ICP-MS) and for its radionuclide primary standardisation, as well as its interlaboratory comparison activities against ISO Guide 43.

Research highlights in **2006**

The work programme of the JRC in the 6th Framework Programme was divided in four core areas and the JRC-IRMM has contributed to all of them. The research at JRC-IRMM covers a large variety of topics i.e. priority areas under those core areas.

- Core area horizontal activities: specifically **reference materials and measurements**, which due to the nature of the work, are also contained in other core areas.
- Core area food, chemical products and health: specifically **food chain, biotechnology and contributions to health**.
- Core area environment and sustainability: mostly under **protection of the European environment**.
- Core area Euratom programme: specifically **nuclear safety and security**.

A selection of JRC-IRMM's activities in 2006 is presented here under these headings.

Reference materials and measurements

The JRC-IRMM promotes the principle of "once measured, accepted everywhere" as established by the Mutual Recognition Arrangement of the International Committee for Weights and Measures (CIPM), under the international Treaty of the Metre Convention. The reliability and comparability of analytical and testing data across Europe – and globally – greatly depend on quality assurance tools like the certified reference materials and schemes for evaluating laboratory performance that the JRC-IRMM provides.

Reference materials are needed for developing, calibrating and validating the methods of analysis. Today, reference materials are also needed to fulfill the requirements of present standards for the accreditation of testing and calibration laboratories. JRC-IRMM is one of the world's largest reference material providers and offers a large variety of certified reference materials. In 2006, its reference materials programme resulted in over 30 new sets of certified reference materials that were released for distribution.

Measuring Charpy specimens

JRC-IRMM also organises interlaboratory comparisons to evaluate the equivalence of accreditation across Europe and the performance of testing laboratories. 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 specific ones for laboratories nominated for a Community reference laboratory, for instance. All these evaluation schemes enable analytical laboratories to benchmark their performance and in some cases to gain accreditation. At the same time they bring together measurement results from different geographical locations, from participants using various analytical methods and with different scopes and experience, and make an overall evaluation of measurement capabilities at various levels of the measurement chain possible. The measurement capabilities of JRC-IRMM in turn are benchmarked against the best achievable measurements via the key comparisons of committees of the International Committee for Weights and Measures (CIPM). JRC-IRMM also organises such key or pilot comparisons for the committees of CIPM. JRC-IRMM's own capability to perform reference measurements is a key factor in all these activities.

Soy bean plants are grown for experiments

Reference materials for the quantification of genetically modified organisms

In the EU genetically modified products need authorisation and according to the EC regulations food or food ingredients containing more than 0,9% GMOs must be labelled appropriately. Certified reference materials are needed for calibrating the methods used to quantify the content of genetically modified organisms (GMOs) and for controlling the quality of these measurements. Implementing the EU legislation on labelling of food and feed products containing GMOs largely depends on the accuracy and reliability of those measurements. For each GMO event authorised for food and feed use in Europe, certified reference materials need to be available.

The JRC-IRMM was the first to produce reference materials for the analysis of GMOs, and is the only one accredited under ISO Guide 34 for the production of certified GMO reference materials. The GMO reference materials of JRC-IRMM are produced under the European Reference Materials (ERM®) label that guarantees the use of best practices in production and certification. In 2006, the JRC-IRMM has developed and produced five new sets of certified reference materials for the analysis of GMO events. The new series include certified reference materials for the analysis of GM cotton, sugar beet, starch-modified potato, and two new GM maize events, namely MIR604 and 59122. The release of these new products has brought the number of certified GMO reference material sets to 14.

The making of the starch-modified potato materials, a set of sugar beet materials and a set of cotton seed materials required development of new processing techniques and certification strategies. Grinding techniques using liquid nitrogen and freeze-drying were applied to achieve stable and homogenous powders with a defined GMO content.

The genetically modified EH92-527-1 potato is, according to the application for authorisation, primarily intended for starch processing into industrial products and the use of the by-products in animal feed. The EH92-527-1 potato is an amylopectin-type starch potato with strongly reduced amylose levels. The samples for laboratory analysis are mainly whole potatoes, which will be qualitatively assessed. The reference material powders were prepared from GM potatoes and from non-GM potatoes. To prevent accidental contamination, the GM and non-GM raw materials were processed separately. The purity of the potato batches was tested on individual tubers: a chip was cut from the surface of each tuber and stained with iodine to demonstrate the presence of amylose. This way of testing clearly reduces the uncertainty of the eventually certified values. Also event specific polymerase chain reaction (PCR) analysis was performed to confirm the identity of the non-GMO and GMO potatoes. Following the freeze-drying and milling in a cryogenic mill, the hygroscopic potato powders were packed in vials using an automatic filling line in a closed glove box maintaining an inert atmosphere. The reference materials are certified for their EH92-527-1 potato number fraction and identity. The identity of the EH92-527-1 specific modification was confirmed by nucleotide sequence analysis of the junction region between the plant DNA and the genetic insertion.

Cotton seed oil is a common additive in food and feed products. The genetically modified stacked cotton event 281-24-236 x 3006-210-23 has been genetically engineered to provide resistance against certain lepidopteran pests. The reference material for event 281-24-236 x 3006-210-23 was produced from cotton seeds, using the grinding techniques at liquid nitrogen temperature to cope with the high fat content of the raw materials. The powders were prepared from GM and non-GM seed batches thoroughly checked for their purity. Beside the two pure materials two mixtures were prepared and certified for their mass fraction of event 281-24-236 x 3006-210-23. The value assignment approach is based on gravimetric preparation of materials containing different mass fractions of event 281-24-236 x 3006-210-23. The approach was afterwards verified using a real-time PCR method.

Certified reference materials released by JRC-IRMM in 2006		
Code	**Material**	**Certified property**
BCR-277R	estuarine sediment	content of trace elements
BCR-280R	lake sediment	content of trace elements
BCR-320R	channel sediment	content of trace elements
BCR-348R	progesterone in human serum	high progesterone
ERM-BF419a-b	sugar beet event H7-1	GMO mass fraction
ERM-BF421a-b	potato event EH92-527-1	GMO number fraction, identity
ERM-BF423a-d	MIR604 maize	GMO mass fraction
ERM-EF422a-d	cotton seed event 281-24-286 x 3006-210-23	GMO mass fraction
ERM-EF424a-d	59122 maize	GMO mass fraction
ERM-FA013av	Charpy V-notch reference test pieces of 30 J nominal absorbed energy	impact toughness
ERM-FA015t	Charpy V-notch reference test pieces of 80 J nominal absorbed energy	impact toughness
ERM-FA016as	Charpy V-notch reference test pieces of 120 J nominal absorbed energy	impact toughness
ERM-FA016au	Charpy V-notch reference test pieces of 120 J nominal absorbed energy	impact toughness
ERM-FA0415j	Charpy V-notch reference test pieces of 150 J nominal absorbed energy	impact toughness
ERM-FA013aw	Charpy V-notch reference test pieces of 30 J nominal absorbed energy	impact toughness
IRMM/IFCC-490	plasmid DNA for prothrombin wildtype (homozygous)	DNA sequence
IRMM/IFCC-491	plasmid DNA for prothrombin mutation (homozygous)	DNA sequence
IRMM/IFCC-492	plasmid DNA for prothrombin mutation (heterozygous)	DNA sequence
IRMM-018a	silicon dioxide	isotope amount ratios
IRMM-315	4-deoxynivalenol	mass fraction
IRMM-316	nivalenol	mass fraction
IRMM-447	genomic DNA of Lysteria monocytogenes	identity
IRMM-449	genomic DNA of Escherichia coli	identity
IRMM-468	thyroxine T4	purity
IRMM-469	3,3',5 triiodothyronine T3	purity
IRMM-540R	uranium doped oxide glass (15 ppm nominal)	uranium mass fraction
IRMM-541	uranium doped oxide glass (50 ppm nominal)	uranium mass fraction
IRMM-007	set of Zn synthetic mixtures (7 units)	isotope amount ratios
IRMM-651	^{nat}Zn isotopic reference material	isotope amount content
IRMM-652	^{64}Zn isotopic reference material	isotope amount content
IRMM-653	^{67}Zn isotopic reference material	isotope amount content
IRMM-652	^{68}Zn isotopic reference material	isotope amount content
IRMM-3702	Zn delta zero material	isotope amount ratio
IRMM-1027i	Pu/U dried spike	isotope amount content

The genetic modification in sugar beet H7-1 provides tolerance to Roundup® herbicides containing glyphosate. Since quantification of GMOs is based either on the detection of the modified DNA sequences or on the newly expressed proteins, it cannot be carried out on the final product, the sugar. Presence or absence of the GMO event is therefore tested on the sugar beet roots before they enter processing. Statistical evaluation of data then allows estimating the GMO concentration in a batch of sugar beet root. The reference material has also been produced from roots and a pure GM and non-GM powder have been made available to allow reliable detection of the H7-1 event. They have been certified for their mass fraction of event H7-1, verified independently using a real-time PCR method.

Ampouling of candidate reference material solutions

The ICP-MS laboratory at JRC-IRMM

Isotopic reference materials for Zn analysis

Isotope ratios of zinc can be used as indicators of metabolic, geochemical and environmental processes. The isotopic variations monitored in such studies are usually expressed relative to a standard on a so-called delta scale. The Commission on Atomic Weights and Isotopic Abundances of the International Union of Pure and Applied Chemistry (IUPAC) concluded in 2002 that having isotopic reference materials of zinc would improve comparability of isotope ratio measurements. To facilitate that JRC-IRMM has prepared a natural-like zinc material as a solution (IRMM-3702) to serve as common “delta-0” material. JRC-IRMM has also produced a series of certified isotopic reference materials for the analysis of Zn. These include solutions containing natural-like Zn, gravimetrically prepared Zn isotope mixtures, and isotope “spikes”.

New developments in reference material processing

The stability of many biological reference materials depends strongly on their water content. However, an efficient and reliable on-line method for measuring the water content in powders during processing could not be realised until recently. In 2006, a new instrumental method has been adapted at JRC-IRMM, validated and implemented for on-line water measurements in glass vials filled with dried biological powders. It is based on high speed acousto-optical tuneable filter near infrared spectrometry (AOTF-NIR). By using 18 universal meat powder calibrants with 0,5 to 8% water (m/m) and a specific statistical model it has been possible to screen several different material types allowing the efficient production control of › 50 000 samples. The AOTF-NIR equipment is now installed in an automatic capping and labelling machine that provides synchronization between the spectrum number recorded by the spectrometer and the sample identification number on the labelled unit.

During 2006 the processing team has further developed the working methods for the use of a new cryogenic mill, which has considerably increased the daily output of finely ground biological materials. Previously only batch-wise ball-milling could be performed, whereas now on-line milling is possible with the new inert milling equipment. In this way, the resulting materials are not contaminated by the mill and can therefore be certified for most elements even at trace levels. The low temperature also protects thermally unstable analytes like nitroimidazoles used as veterinary drugs, from being destroyed in the milling step. The possibility for rapid re-milling of a first milling portion for further reduction of the particle size, following a rapid check of the particle size, is another attractive feature.

Some candidate reference materials are milled at liquid nitrogen temperature

For making of new reference materials of the ERM® consortium for the analysis of sulphur in petrol a solution to how to fill and keep the samples had to be found. Therefore, development of a reliable method for containment of petrol in flame sealed glass ampoules was one of the first and most challenging tasks of this project. Firstly, filling and sealing stations were separated physically for safety and bulk petrol was kept outdoors. Secondly, the vapour pressure of the petrol was decreased by super-cooling filled ampoules in liquid nitrogen to avoid problems when the glass melts in the sealing area. Pre-heating and sealing steps were also recorded using an infrared camera in the range -40°C – 1500°C. This way monitoring the change in temperature over every mm^2 on the ampoule as a function of time was possible and optimal settings could be found. The sealing method was specifically developed for glass with very low thermal conductivity and allowed to keep the petrol below its flash point of -40°C.

Interlaboratory comparisons for benchmarking and validation

JRC-IRMM organises interlaboratory comparisons for many purposes. Interlaboratory comparisons are the preferred way to validate methods of analysis or to certify a reference material according to internationally agreed guidelines. They are also used to evaluate the performance of laboratories in specific testing activities. Many of the interlaboratory comparisons organised by JRC-IRMM are open to all laboratories that wish to participate but some are restricted to e.g. national reference laboratories, expert laboratories for certification of reference materials, or in the case of nuclear measurements to those that are licenced to keep and import radioactive samples. Often an interlaboratory comparison organised by JRC-IRMM was prompted by new or modified European legislation or policy.

IMEP® test samples from different campaigns

In 2006, JRC-IRMM was running evaluation campaigns on the analysis of sulphur in petrol, on the analysis of trace elements, polychlorinated biphenyls (PCBs) and PAHs in sewage sludge, and on measurements of isotopes of plutonium and uranium. Other interlaboratory comparisons dealt with PAHs in edible oils and primary smoke condensate, semicarbazide in baby food and in fresh egg and egg products, and evaluation of the comparability of data collected by the JRC Institute for Environment and Sustainability from laboratories measuring radioactivity in the environment. Operating three new Community reference laboratories requires JRC-IRMM to organise more proficiency test type campaigns for the nominated laboratories, according to their mandate. The JRC-IRMM also participates in comparisons organised by other international organisations. For instance JRC-IRMM experts participated in a CCRI(II) key comparisons analysing ^{60}Co and ^{55}Fe, and in CCQM comparisons measuring lead in wine (CCQM-K30) and mercury in water (CCQM-P100).

Consultative Committee on the Amount of Material (CCQM) of the International Committee for Weights and Measures (CIPM)
Consultative Committee for Ionising Radiation (II) (CCRI(II)) of the International Committee for Weights and Measures (CIPM)

Interlaboratory comparisons organised by JRC-IRMM in 2006
(started, on-going or completed; reference material certification campaigns not included)

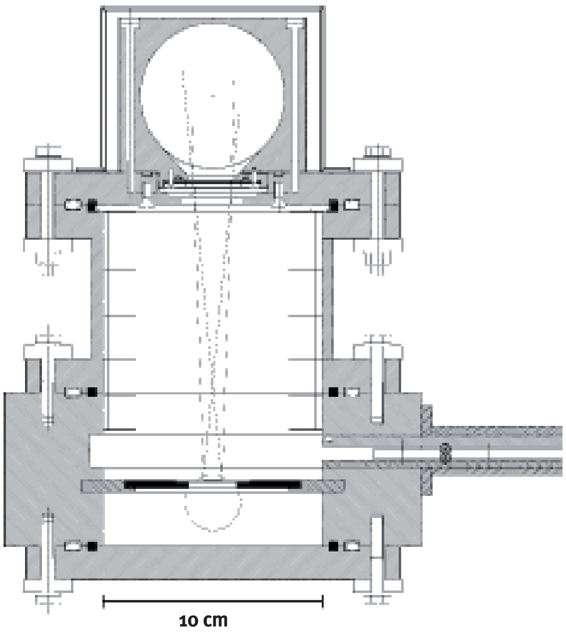
Topic	Type of comparison
sulphur in petrol	international measurement evaluation – IMEP-22
trace elements, PCBs and PAHs in sewage sludge	international measurement evaluation – IMEP-21
activity concentration of the radionuclides ^{40}K, ^{137}Cs and ^{90}Sr in milk powder	evaluation of comparability of data collected from Member State laboratories monitoring radioactivity in air, water and foodstuffs
detection of meat and bone meal by PCR	method validation by interlaboratory comparison
heavy metals in food of plant origin	proficiency test for national reference laboratories – IMEP-101
EU 15+1 PAHs in acetonitrile	proficiency test for national reference laboratories
aflatoxins B1, B2, G1, G2 in acetonitrile	proficiency test for national reference laboratories
quantitation of PAHs in primary smoke condensate	proficiency test
analysis of PAHs in edible oils	proficiency test
quantitative determination of cocoa butter equivalents in milk chocolate	method validation by interlaboratory comparison
determination of a mixture of authorised and non-authorised sweeteners in food	method validation by interlaboratory comparison
determination of animal DNA in feed by PCR technique	interlaboratory comparison for pre-validation
uranium, plutonium and caesium isotope ratios in saline medium	international measurement evaluation – NUSIMEP-5
isotopic abundances of uranium	international measurement evaluation – REIMEP-18
analysis of methionine	pilot study CCQM-P75 for CIPM
analysis of sewage sludge	pilot study CCQM-P70 and key comparison CCQM-K44 for CIPM
PCBs in sewage sludge extract and in sewage sludge	interlaboratory comparison for EUROMET/METCHEM project 833

PCB – polychlorinated biphenyl
PAH – polycyclic aromatic hydrocarbon
PCR – polymerase chain reaction

^{55}Fe half-life and activity measurements

The radioactive iron-55 disintegrates mainly through electron capture decay to the ground state of ^{55}Mn. Having X-ray emission at 5,9 keV makes it very useful for calibrating low energy photon detectors. The half-life of ^{55}Fe has not yet been well established as ionisation chambers are insensitive to the emitted radiation, and the published data obtained by means of low energy detectors are inconsistent. At JRC-IRMM, experimental work was undertaken to resolve the ambiguity on the ^{55}Fe half-life value. The decay of a pure ^{55}Fe source was followed by repeated high precision activity measurements by X-ray counting at a defined solid angle. As a result, a new half-life value was determined with unmatched accuracy. The study also revealed neglected uncertainty components in data analysis based on least squares fits, providing an explanation and remedy for apparent underestimations of uncertainty commonly found in the scientific literature, in particular concerning half-life measurements.

A recent key comparison with national metrology institutes of fifteen countries has revealed that most primary standardisation laboratories have great difficulty in determining the activity of a ^{55}Fe solution accurately. The JRC-IRMM has submitted a consistent set of activity values obtained by means of three independent primary standardisation methods for this key comparison.



Schematic design of the defined solid angle X-ray counter, a tool for primary standardisation of activity

From the Avogadro constant to a kilogram

The kilogram is the only remaining base unit in the International System of Units (SI) still defined in terms of a material artefact. A possible way of redefining the kilogram is via the mass of a specified number of atoms. The Avogadro constant (N_A) could be a key to a new definition of the kilogram, yet it is not known within sufficiently small uncertainty to make the transition from the old definition to a new one. The variation of the international (prototype) kilogram has been shown to be a few parts in 10^8. For its redefinition therefore, an uncertainty on N_A of $2 \cdot 10^{-8}$ relative or better, is required. The JRC-IRMM is participating in the international Avogadro project to realise the concept.

To count atoms, this research consortium starts with a perfect crystal of pure silicon. Its dimensions are measured, its volume calculated and divided by the volume of one atom in the atomic lattice, which can be measured by X-ray crystallography. Knowing the number of atoms in the crystal and the mass of the crystal, and knowing the mass of one mole of silicon (about 0,028 kg) enables the calculation of the number of atoms in one mole, and thus the calculation of N_A.

For many years the research was centred on the problem of how far the perfection of a real crystal is away from the ideal state. At present, it is widely accepted that within the limits of the target uncertainty, the lattice parameter and the unit cell volume of silicon can be seen as an invariant quantity when the influence of e.g. impurities is taken into account. It has also been shown that up to a relative measurement uncertainty of a few parts in 10^7, the molar volume is constant, too. The combination of data from several independent measurements of the unit cell and the molar volumes has led to a value for the Avogadro constant of N_A = 6,022 1353 (18) x 10^{23}/mol in 2005.

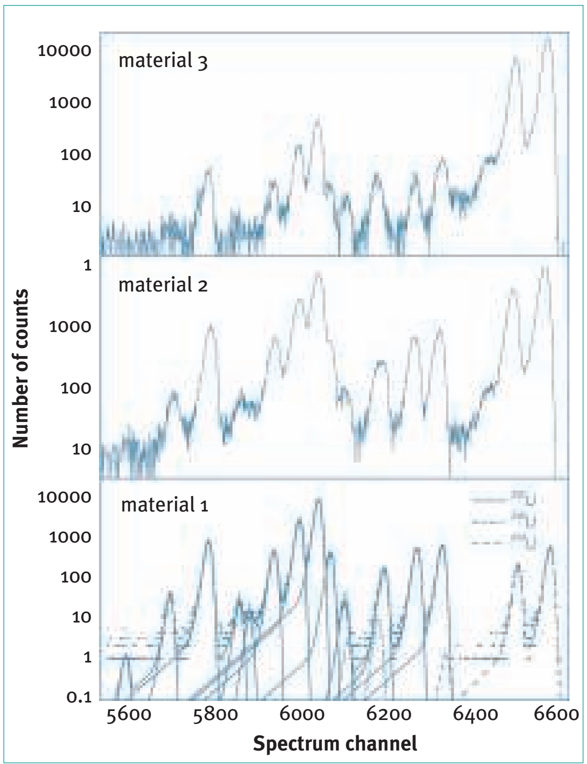
To continue reducing the uncertainty of N_A, an improvement in the molar mass determination is needed. This can be realised by fabricating a sphere out of silicon that consists ≥ 99,99% of ^{28}Si, and where the ^{29}Si and ^{30}Si abundances are in the order of 0,005%. A relative combined uncertainty of ≤ 1% on each of these abundance corrections contributes a relative uncertainty on the molar mass value of the highly enriched ^{28}Si of ≤ $3 \cdot 10^{-8}$. Using this concept, the task is now to measure the small ^{29}Si and ^{30}Si abundances in the highly enriched ^{28}Si, instead of the isotope ratios in Si of natural isotopic composition as was done before. That results in very small corrections to the molar mass value of ^{28}Si and these corrections can be directly measured.

This concept has become feasible because of a source of very highly enriched Si isotopes in Nizhny-Novgorod (RU). The Institute of Chemistry of High-Purity Substances of the Russian Academy of Sciences has demonstrated its capability to produce ^{28}Si with the required level of enrichment and the amount needed to make a 5 kg crystal. The isotopic homogeneity and purity has to be assured during the complete process. This is where JRC-IRMM comes in with its expertise in measurements of isotope ratios. In 2006, the JRC-IRMM has measured the isotope ratios of ^{28}Si samples at the different production steps. In pursuit of improvements the dynamic range of the dedicated mass spectrometer at JRC-IRMM has been upgraded from 10^4 to 10^8 and the preparation of a new set of synthetic isotope mixtures for calibration has been initiated.

Nuclear decay data for geochronology

Radiometric techniques can be used to determine the age of rocks, sediments and fossils. They are based on the decay rates of naturally occurring isotopes. The decay of ^{238}U to ^{206}Pb and of ^{235}U to ^{207}Pb, form the basis of one of the oldest and most important methods of geochronology. This is partly due to the relatively long half-life of ^{238}U and ^{235}U, in the order of 10^9 years, comparable to the age of planet Earth. The applicability and accuracy of U-Pb dating greatly depends on the accuracy of the nuclear decay constants. The currently assumed values are mainly based on only one precise experiment performed in 1971. The geochronology community therefore is in need of precise measurements of the ^{238}U and ^{235}U half-lives.

JRC-IRMM together with some European laboratories, has launched a project to measure these half-lives directly, involving mass spectrometry and primary activity measurements on highly enriched uranium materials. In parallel with these ongoing direct measurements, information on the half-lives has been extracted from high-resolution alpha spectra of three uranium materials enriched in ^{235}U, using the $^{234}U/^{235}U$ activity ratio as a probe for the $^{238}U/^{235}U$ half-life ratio. The isotopic composition of the material was quantified by mass spectrometry and, as an intermediate link, the half-life ratio of $^{234}U/^{238}U$ was derived from published mass spectrometric analyses of uranium in "secular equilibrium". The resulting half-life ratio $T_{1/2}(^{238}U)/T_{1/2}(^{235}U) = 6{,}351 \pm 0{,}031$ is in agreement with the commonly adopted half-life values. Future experiments will narrow down the margin of uncertainty.



Measured α-spectra of three ^{235}U enriched materials. Contribution of ^{234}U peaks can be seen at the right hand side

Food chain, biotechnology and contributions to health

Food safety is one of the top priorities of the European Commission. The "from farm to fork" concept of the European legislative framework on food safety covers the whole food chain addressing issues from traceability of cattle to contaminants in baby food. Life sciences research and biotechnology make new exciting applications a part of our everyday life, whether concerned with food or health.

To implement the European Commission's food safety policy or to assure the quality of testing results in the field of life sciences, means for the control are needed. Monitoring compliance with labelling rules or with the regulatory limits of contaminants or additives in food and feed must be done on a reliable basis.

In response to those needs, the JRC-IRMM produces new reference materials, develops, validates and tests analytical methodologies, and explores the testing requirements set by biotechnology research. Many of the methods developed and validated in the projects of JRC-IRMM are later reviewed by the international standardisation bodies and turned into standards. JRC-IRMM operates four Community Reference Laboratories (see p. 8) and also supports others that form an important part of the risk management in the European Union.

Studies on semicarbazide

Semicarbazide is a metabolite of nitrofurazone, an antibiotic that is banned from use within the EU. It was measured to detect illegal use of nitrofurazone until it became known that nitrofurazone is not the only source of semicarbazide in processed foods. It may also be formed by other reactions e.g. during disinfection by hypochlorite treatment, or it may be transferred from gaskets used in jars. Semicarbazide is a weak genotoxic and carcinogenic agent but its occurrence in baby food is a considerable concern. Certain gasket materials have, however, already been phased out to avoid this problem.

When the problem was acute, the JRC-IRMM and its collaborators developed sensitive methods to detect semicarbazide in different types of baby food, eggs and egg powder at levels as low as 10 ng/g. These methods were validated by interlaboratory comparisons, and in 2006 a proficiency test organised by JRC-IRMM showed that European laboratories' capabilities in measuring semicarbazide are satisfactory. A validated method was used to monitor semicarbazide in 107 different baby food products produced in 11 European countries, purchased at local supermarkets in 14 Member States showing that the semicarbazide content ranged from below 0,2 ng/g to 50 ng/g.

CoCal-2 for analysis of foreign fats in milk chocolate

Cocoa butter in chocolate can be replaced by other vegetable fats, the so-called cocoa butter equivalents (CBEs). Directive 2000/36/EC allows the addition of up to 5% of vegetable fats other than cocoa butter. If CBEs are added, consumers have to be informed by appropriate labelling. But assessing compliance of chocolate products with the labelling provisions cannot be done without appropriate methods of analysis. Therefore, the JRC-IRMM has developed a reliable analytical approach to detect and quantify CBEs in dark and milk chocolate.

Preparing chocolate powder samples for the analysis of fats

The CoCal-1 (Cocoa butter Calculation) toolbox for the analysis of dark chocolate was published in 2004, and the building of CoCal-2 for milk chocolate was completed in 2006. Both CoCal toolboxes are built around validated methods, and advanced statistical evaluation algorithm and a calculation sheet. A certified cocoa butter reference material, which is needed for calibration of instruments, is also available from JRC-IRMM. The analytical approach is based on triacylglycerol profiling by high resolution gas liquid chromatography. CoCal-1 and CoCal-2 were subjected to validation in international collaborative trials organised by JRC-IRMM. The methods for the detection and quantification of CBEs in dark chocolate have become standardised methods of the American Oil Chemists Society (AOCS) and the International Organization for Standardization (ISO).

JRC-IRMM is also carrying out a survey on the correct labelling of foreign fats in dark and milk chocolate samples acquired from the European market using CoCal-1 and CoCal-2.

Testing food allergens

The presence of food ingredients derived from commonly allergenic foods needs to be declared on the product label, as requested by European legislation (Directive 2000/13/EC as amended by Directive 2003/89/EC). This is to help the increasing number of sensitised people to avoid allergic reactions, some of which can be life-threatening. The work on food allergens that takes place at the JRC-IRMM aims to support the implementation of the labelling Directives by providing suitable methods of analysis of allergens in food products.

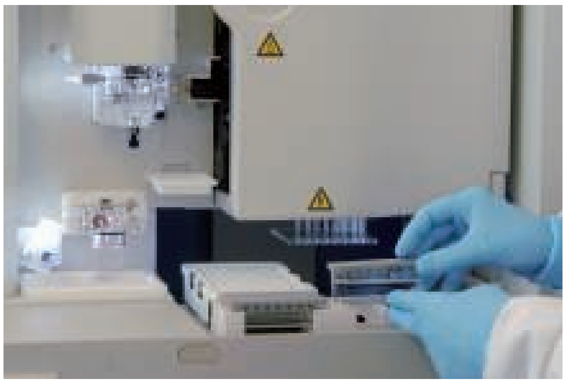
To ensure an appropriate labelling of food products, food allergen detection methodology needs to be developed. The JRC-IRMM currently develops protein and DNA based methods for allergen detection and studies the effect of food processing like heating of dairy products and roasting of peanuts on the sensitivity of the assays commonly used for allergen detection. In addition to this, validation studies have been performed to determine the performance of allergen detection methods. In 2006, the robustness of dipsticks designed to detect peanut traces in food products was assessed in a collaborative trial.

The implementation of the labelling requirements as set out in the European legislation has prompted the JRC-IRMM to analyse the labels of more than 550 different cookies and chocolates with regard to the declaration of the allergenic ingredients. All these products have been analysed to determine their peanut and hazelnut content, allowing a comparison of the analytical results with the information provided on the food labels.

The allergens project at JRC-IRMM is also making this research known in schools. A project carried out in cooperation with the European School in Mol (BE) gave the pupils of the school an opportunity to visit the laboratories of the JRC-IRMM and to gain hands-on experience with allergen detection methods. Earlier a questionnaire-based survey has been carried out with and Austrian secondary school (BG/BRG Tulln), the European School in Mol, and the research institute IFA Tulln (AT) to gain an insight in the prevalence of food allergies in the families. 268 families from the European School in Mol completed the questionnaire. According to this study almost 9% had food allergies and more than 80% pay attention to food labels.

New reference materials for life sciences

In 2006, the JRC-IRMM has released for distribution its first certified reference materials for genetic testing. These reference materials complement the series of health related reference materials that the JRC-IRMM has in its catalogue. The three certified reference materials are meant for the detection of the G20210A mutation in the human prothrombin (Factor II) gene (IRMM/IFCC-490, IRMM/IFCC-491 and IRMM/IFCC-492), and are suitable for the quality control of polymerase chain reaction (PCR) -based methods. The IRMM/IFCC-490 contains the wild-type sequence, the IRMM/IFCC-491 contains the G20210A mutation, and the IRMM/IFCC-492 contains equal amounts of IRMM/IFCC-490 and IRMM/IFCC-491. The materials were processed and certified according to ISO Guides 34 and 35. The certification covers the sequence of the 609 *bp* human prothrombin gene fragment included in a plasmid.



DNA sequencing for analysis of pathogens

Other new releases in 2006 were two new certified genomic DNA reference materials for the analysis of food-borne pathogens. These reference materials can be used as positive control materials for the detection of *Escherichia coli* O157 and *Listeria monocytogenes* by diagnostic PCR methods, or for harmonising and validating those methods. JRC-IRMM has also extended its range of clinical reference materials by a new hormone material. BCR-348R, containing approximately 8 µg/L lyophilised endogenous progesterone in human serum, has been certified for its progesterone concentration. It is suitable for *in vitro* diagnostics manufacturers and reference laboratories to calibrate quantitative measurement procedures.

TSE testing

Testing for bovine spongiform encephalopathy (BSE) i.e. the mad cow disease is compulsory in the EU for all cattle older than 30 months and for cattle of certain risk groups. More than 11 million rapid BSE tests are carried out annually in hundreds of laboratories in the European Union. In the EU, the performance of these tests is evaluated by JRC-IRMM. The evaluation of 20 different rapid BSE tests by JRC-IRMM in the past has led to a total of 12 tests approved under Regulation (EC) No 999/2001. Also rapid tests for the diagnosis of scrapie have received approval. Approving more tests has a direct effect on the consumer confidence and opens competition in the TSE diagnostics market. A considerable drop in costs has already been observed in several Member States.

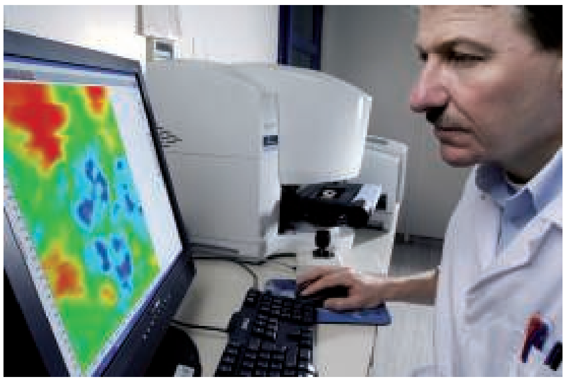
In 2006, JRC-IRMM has evaluated a live animal BSE test. This is the first time such an evaluation has been made. The test was selected to enter JRC-IRMM's evaluation programme in the frame of a call for the expression of interest published by Health and Consumer Protection DG in 2003. The test of the company DiaSpec (DE) is based on a sophisticated method using Fourier transform infrared spectroscopy and applies chemometric methods for data analysis. Blood serum of the animal is analysed and the spectrum obtained is compared to specific reference features of serum of BSE infected or BSE free animals. The report from JRC-IRMM concludes that the test demonstrated a good, but not sufficient performance to be recommended for approval by the European Commission. This statement was agreed on by the European Food Safety Authority's working group on TSE testing in their final report.

Meat and bone meal studies

To prevent bovine spongiform encephalopathy (BSE) from spreading, the European Commission has taken several measures. The most important ones are the removing of specified risk material from the food chain, applying appropriate rendering conditions for the production of animal by-products and the ban of meat and bone meal (MBM) from mammals in compound feed for ruminants, which was extended in 2001 to cover feed for almost all farmed animals.

Still today, monitoring of feed that would allow lifting the extended ban is hampered by the lack of appropriate animal specific tests, i.e. selective methods for detecting mammalian proteins in the presence of proteins from other animals. The only official method in the EU is microscopy, which does not yield precise information about the origin of the detected bones. Other methods are applied to the analysis of feed samples for the presence of banned processed animal proteins, such as methods based on polymerase chain reaction (PCR), immunoassay and near infrared microscopy (NIRM).

To evaluate the performance characteristics of three selected real-time polymerase chain reaction (PCR) methods, the JRC-IRMM has conducted a pre-validation study for the detection of animal DNA in feed using PCR. An important outcome of this study is, that sensitivity and specificity of the methods have been significantly improved making PCR a promising technique for the identification of animal species in feed.



Feed analysis with microscope

A marker for animal by-products

Regulation (EC) No 1774/2002 lays down rules to enable safe use of animal by-products that are fit but not intended for human consumption. It sorts animal by-products into three categories according to associated risk, and requires that materials of different categories are kept separated during collection, handling and transportation. Also, 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 would be visible and detectable by its olfactory properties, non-toxic, safe for handlers, commercially available, inexpensive, stable, recoverable and easy to analyse. It would ensure identification and traceability of products to be disposed of and eliminate risk of fraud.

The Directorate-General for Health and Consumer Protection has asked JRC-IRMM to evaluate the characteristics of substances that may be suitable for this purpose. The JRC-IRMM proposed to use glyceroltriheptonate (GTH) as a chemical marker. It meets the requirements of the legislation, and in addition could be used in the animal by-product industry as it withstands extreme sterilisation conditions. An implementation study at ten European rendering plants has demonstrated that GTH added in rendering plants is a suitable marker for animal by-products from category 1 and 2. In the frame of that implementation study the JRC-IRMM has developed a method for the detection of GTH in meat and bone meal (MBM) and rendered fat. The method is based on gas chromatography coupled to mass spectrometry (GC-MS) and was validated in a single-laboratory study. It was also used to analyse the samples collected from the rendering plants.

Organic food

Authenticity of organic food is one of the issues where the consumer's trust is put on trial. Currently the only possibility to check whether a farmer's production system complies with organic farming is to have on-site inspections and audit trails covering all stages in the production process. However, the verification of the authenticity of end products at the point of sale (whole sale, distributor, supermarket) is difficult due to lack of analytical methodologies giving conclusive answers. So far no single testing method for authentication of organic food or for distinction from conventionally produced food has been proven suitable and validated.

The JRC-IRMM is investigating the combined use of several physico-chemical and biological methods to discriminate the organically grown from the conventionally grown crops. Controlled experimental field tests have been designed with a Belgian organic farmers association and certified farmers. Crop samples (winter wheat, horse beans, potato and carrots) produced under the same climatic and pedological conditions but under different production systems i.e. organic vs. conventional, have been collected from the 2004, 2005 and 2006 harvests. Along with the crops soil samples were taken from the fields, and their physical, chemical and biological parameters were analysed.

Organic carrots (cv. Népal)

About 20 different methods have been applied to analyse dry matter, total nitrogen, proteins, gene expression, trace elements, radionuclides, stable isotopes of light elements, phenolic acids (plant metabolites), antioxidant and anti-mutagenic activities. In 2006, two-dimensional difference gel electrophoresis (2-D DIGE) was added to the suite of methods. This approach enables accurate analysis of differences in abundance of individual proteins between organically and conventionally grown samples. Statistical data evaluation techniques have been applied to find suitable variable combinations for distinguishing between the organically and conventionally grown crops. Some samples will be analysed to evaluate the robustness of the selected analytical methods within the network of experts established by JRC-IRMM.

PROTECTING THE EUROPEAN ENVIRONMENT

The implementation of legislation on environmental protection is closely linked with measurement results. The monitoring data are the basis for decisions that can have significant consequences for the environment or the industry. Therefore those results need to be reliable and comparable.

The JRC-IRMM's work is in particular related to the implementation of the Water Framework Directive (2000/60/EC), Drinking Water Directive (98/83/EC), RoHS Directive (2002/95/EC), Nitrate Directive (91/676/EEC), Air Quality Directive (96/62/EC and 2004/107/EC), the Thematic Strategy for Soil Protection (COM(2002)179 final), and Articles 35 and 36 of the Euratom Treaty. The JRC-IRMM provides testing laboratories with reference materials and proficiency testing schemes and develops and validates methods of analysis.

Investigating how isotopic techniques can be used for identifying sources of pollution has been a research topic at JRC-IRMM for the last three years. It is commonly known, that processes occurring in nature e.g. evaporation, condensation or oxidation, affect the relative abundances of isotopes. This isotopic information could be used to identify and quantify pollution sources and to assess the effectiveness of environmental management plans. The JRC-IRMM studies the analysis of isotopes in nitrates, benzene, toluene, ethylbenzene and xylene, PAHs, CO_2 and N_2O. Its intention is to make these research tools more accessible to those that implement and monitor the implementation of directives.

Isotope techniques can also be applied to study kinetic effects in fuel cells. In 2006, the JRC-IRMM and the JRC Institute for Energy (IE) have applied $^{13}C/^{12}C$ analysis in demonstrating the behaviour of fuel cell anodes in proton exchange membrane (PEM) cells.

Tracing sources of nitrate pollution

Nitrate isotope analysis ($\delta^{15}N$ and $\delta^{18}O$ values) enables to discriminate mineral fertilizers, organic nitrogen from soil and domestic wastewater or animal manure as potential nitrate sources. In 2006, the isotope techniques developed at JRC-IRMM have been put in use.

Nitrate concentration in surface waters has to be monitored by the Member States and reported to the European Commission every four years. In Flanders (BE), for instance, a network of 800 surface water sampling points has been established, and sampling is carried out regularly. Despite the measures taken to reduce the nitrate pressure from agriculture the monitoring results have shown trends evolving in the opposite direction. Since the knowledge of nitrate concentrations does not allow to identify the sources, isotope techniques were applied to find some of the answers.

To identify the source of nitrate the JRC-IRMM measured samples collected by the Flemish Environment Agency (VMM) from six sampling locations in the northern region of Flanders (BE). The aim was to investigate whether other potential sources than manure e.g., greenhouse or domestic wastewater discharges, mineral fertilizers, or nitrogen in soil, significantly contribute to the observed nitrate pollution. Combined $\delta^{15}N$ and $\delta^{18}O$ analysis of nitrate in water samples confirmed that in four of the six sampling points nitrate pollution could be almost completely attributed to animal manure application. In two of these points, the isotope data indicated a significant contribution of mineral fertilizers or greenhouse discharges (20-90%) at several sampling dates. In two of the six points, $\delta^{15}N$ and $\delta^{18}O$ data indicate that both animal manure and domestic wastewater contributed to the observed nitrate pollution. Additional analysis of boron isotopes would enable to discriminate between those two sources so the study continues by carrying out $\delta^{11}B$ analysis.

Analysis of trace elements in environmental samples at JRC-IRMM

Analytical quality control for the Water Framework Directive

The Water Framework Directive 2000/60/EC sets out a long-term perspective for the management and protection of waters. It requires that all inland and coastal waters within defined river basin districts must reach good status by 2015. To implement the Water Framework Directive the Member States need to design monitoring programmes. The effectiveness of the implementation will greatly depend on the ability of Member States' laboratories to measure chemical, biological and ecological changes of the quality of the waters.

As these monitoring data are the basis for regulatory decisions and measures required to achieve the environmental objectives of the Directive, analytical quality assurance and control measures have to be established across all EU laboratories involved in monitoring. The European project EAQC-WISE (European Analytical Quality Control in support of the Water Framework Directive via the Water Information System for Europe) addresses these issues. Its aim is to develop a sustainable system to ensure comparability of data for the needs of the Directive.

The project in which the JRC-IRMM acts as scientific coordinator, first assesses the current status of quality assurance and control tools like proficiency testing, reference materials, validated or standardised methods, and training and research in the field of water monitoring. Based on that assessment a "blueprint" is developed. Such a system should provide confidence in the whole analytical process, from sampling to reporting, for chemical and biological parameters from monitoring at river basin scale as well as at European scale. The system should make use of existing structures and best practice.

New reference materials

Three new certified reference materials for the analysis of sediments were released for distribution in 2006. BCR-277R, BCR-280R and BCR-320R replace the exhausted batches of an estuarine sediment, a lake sediment and a channel sediment. These materials are designed for assessing method performance and for quality control of sediment measurement results. They are certified for their content of As, Cd, Co, Cr, Cu, Hg, Ni and Zn. The contents of Fe, Mn, Sc, Th, Tl, U and V in BCR-320 have also been certified. Indicative values for Se and Sn are available for all three materials. They help monitoring laboratories in the Member States in obtaining reliable and comparable data for sediment monitoring in the frame of the Water Framework Directive 2000/60/EC.

The JRC-IRMM offers a wide range of certified reference materials for the analysis of environmental samples

The Air Quality Framework Directive 96/62/EC and its first (1999/30/EC) and fourth (2004/107/EC) Daughter Directives require the monitoring of As, Cd, Ni, Pb and some polycyclic aromatic hydrocarbons (PAHs) in particulate matter of an aerodynamic diameter of less than 10 µm (so-called PM10). At present no suitable reference material certified exists for those elements and/or PAHs in PM10 that would support the monitoring laboratories in their analytical quality control. Now JRC-IRMM is testing the suitability of different sources of air particulate matter as a PM10 reference material. Also the processing technologies to obtain a sufficiently small particle size and good homogeneity of the material are investigated.

Nuclear safety and security

Many issues related to nuclear safety and security require accurate calculations using nuclear and isotopic data. Isotope analysis, absolute measurements of radiation i.e. radionuclide metrology, and measurements of neutron induced reactions and cross-section standards have been key activities of the JRC-IRMM since it started operation in 1960. For instance, the JRC-IRMM performs SI-traceable extremely accurate radioactivity measurements supporting the European and international metrology organisations. The JRC-IRMM provides certified isotopic reference materials for environmental analysis and so-called spikes for calibration of nuclear detection techniques in support of the Euratom safeguards. From the onset the JRC-IRMM has been a major provider of microscopic neutron data (cross sections) in Europe. Measurements carried out at its Van de Graaff and neutron time-of-flight facilities have played a significant role in establishing and improving the evaluated nuclear data file maintained at the databank of the Nuclear Energy Agency (NEA) of Organisation for Economic Co-operation and Development (OECD). Among others, the $^{10}B(n,\alpha)$ and several fission cross-section standards are still valid today. In line with the JRC's research goals in the 6th Framework Programme neutron data related to waste management has taken a large part in the JRC-IRMM research programme.

Large size dried spikes

Measurements for nuclear material accounting and control need reference materials as the foundation of the measurement processes. The JRC-IRMM specialises in isotopic reference materials and has, for instance, developed large size dried spikes. They are used extensively in safeguards and the nuclear industry for measuring the contents of uranium and plutonium in dissolved fuel. Approximately one batch of over 1000 units of these spikes is prepared at JRC-IRMM per year. A novelty was introduced in 2006: an automatic unit for the dispensing and ampouling of the large size dried spikes was installed and tested.

In 2006, the batch IRMM-1027i was completed. The materials – uranium and plutonium in metallic form – were dissolved completely already in 2005 and the solution was dispensed into individual vials, each engraved with an identifier, and dried carefully. A covering of a cellulose based material ensures the dried material is fixed on the base of the vials for transport.

The certification of amounts of spike in the individual vials was based on the masses of the metals dissolved, their certified contents, the total mass of solution and the mass of the amounts dispensed into each vial. The isotope content of the solution was verified by taking samples, spiking with a IRMM certified reference material (IRMM-049c) and after chemical sample treatment measuring the uranium and plutonium isotopic ratios by isotope dilution mass spectrometry. After dispensing and drying, the same procedure was carried out for a selection of the vials across the whole sequence. The 1027i batch is the 10th in line offered by JRC-IRMM since 1988.

An automated system for dispensing and ampouling of large sized dried spikes was installed at JRC-IRMM in 2006

Gas analysis for nuclear safeguards

The JRC has investigated the potential and limitations of such a technique. In the end of 1990s a feasibility study was performed by JRC-IRMM and the JRC Institute for Transuranium Elements investigating the potential of identifying reprocessing activities by means of isotopic measurements of xenon and krypton. Calculations were performed on the nuclear inventory of spent fuel for reactor operating scenarios used for electricity generation (high burn-up) as well as for scenarios suspected for production of weapons grade plutonium (low burn-up). During reprocessing of the spent fuels significant isotopic alterations of stable xenon and krypton are caused by dilution of the released noble gases with the atmospheric ones, which can be simulated as isotope mixing at different blending ratios.

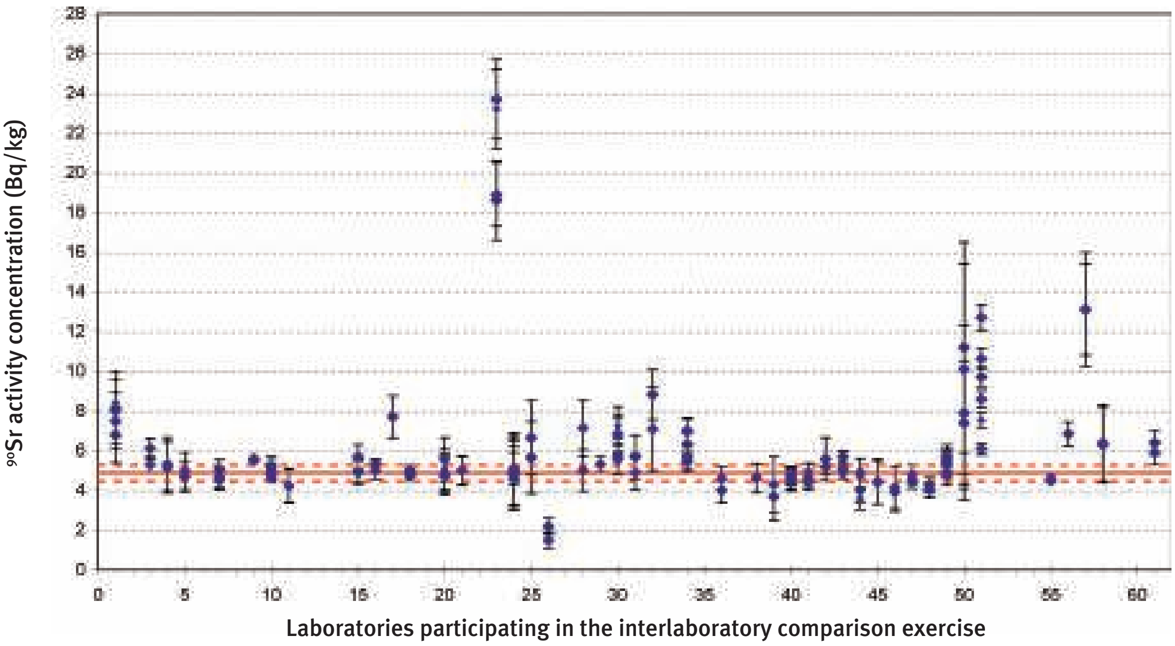
In 2006 the expert meeting on noble gas monitoring of the Department of Safeguards of the International Atomic Energy Agency (IAEA) reviewed the applicability of noble gas sampling, analysis and monitoring. The recommendation of the expert group was that use of stable xenon isotopes and ^{85}Kr in local noble gas monitoring should be explored. This could be used to confirm operator's declaration of reprocessed fuel at declared large reprocessing plants. Within some constraints the accumulated information gained from xenon signatures could also provide valuable information for nuclear safeguards verification of undeclared reprocessing activities. One requirement in view of quality control is to enable comparability of measurement results via traceability to noble gas standards of highest metrological quality. JRC-IRMM has prepared a unique set of primary isotopic gas standards certified for the isotopic composition and molar mass of xenon and krypton.

Radioactivity in milk powder

Based on Commission Recommendation 2000/473/ Euratom and Article 36 of the Euratom Treaty, the EU Member States are obliged to monitor radioactivity in the environment as well as in selected foodstuffs, and to report the monitoring results to the European Commission. To evaluate the comparability of those data, JRC-IRMM organises regular measurement comparisons where all participating laboratories receive a test sample certified by JRC-IRMM. The purpose of the latest comparison exercise was to obtain a realistic estimate of the accuracy of radioactivity measured in milk during routine monitoring.

After distributing more than 130 units of milk powder to laboratories in all Member States and Candidate Countries, the laboratories were asked to determine the activity concentration of the radionuclides ^{40}K, ^{137}Cs and ^{90}Sr in this material. In 2006, the measurement results from the 59 participating laboratories from 30 countries were evaluated.

The reported ^{137}Cs and ^{40}K activity concentrations obtained by gamma spectrometry were generally in agreement with the reference activity concentration even though the measurement uncertainty was often underestimated. The analysis of results of ^{90}Sr activity concentration, however, pointed out a different problem. According to statistical tests,



44 of the 59 laboratories that participated in the interlaboratory comparison exercise measuring radioactivity in milk powder reported results for ^{90}Sr activity concentration. The red lines show the reference value (solid line) and the range (dashed line) about 1/3 of all reported ^{90}Sr results are not compatible with the reference value, and 10 laboratories deviate more than 30% from it. ^{90}Sr decays via emitting electrons (β^-) only and the analysis involves destructive sample preparation and radiochemical separation followed by counting in liquid scintillation or proportional counters. The results of this interlaboratory comparison show the need to improve those procedures in some of the laboratories.

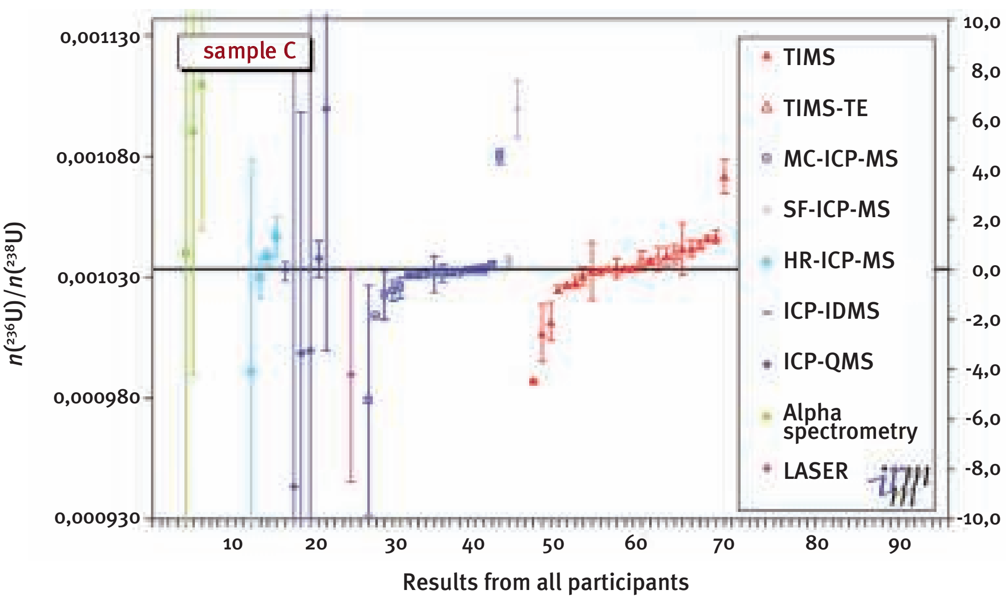
External quality control for nuclear laboratories: REIMEP-18

Regular European Interlaboratory Measurement Evaluation Programme (REIMEP) was started by JRC-IRMM in 1978 for carrying out external control of the quality of the measurements of the nuclear fuel cycle materials. In the REIMEP campaigns samples matching materials analysed routinely in the nuclear fuel cycle are sent to participating laboratories for measurements and to date 17 REIMEP campaigns have been completed, which involved safeguards laboratories throughout the world. Participation in such measurement campaigns is an integral part of the quality control required for nuclear safeguards laboratories worldwide.

The REIMEP-18, that was completed in 2006, has been the largest nuclear isotopic measurement campaign organised by JRC-IRMM so far. Samples were shipped to 85 registered participants, and the JRC-IRMM received results from 71 laboratories. The participating laboratories were asked to determine the isotopic composition of uranium ranging from depleted to slightly enriched uranium in four samples.

Certification measurements were carried out at JRC-IRMM using state-of-the-art mass spectrometric methodology. A UF_6 gas source mass spectrometer was used to determine the $n(^{235}U)/n(^{238}U)$ ratios and a TRITON thermal ionisation mass spectrometer for the minor isotope ratios $n(^{234}U)/n(^{238}U)$ and $n(^{236}U)/n(^{238}U)$. Verification measurements on ampouled samples performed prior to shipping showed good agreement with the certified ratios.

The results of the REIMEP-18 campaign confirm the excellent capability of nuclear safeguards and other participating laboratories in measuring isotopic abundances of uranium, although some problems were discovered for the measurements of the minor isotope ratios $n(^{234}U)/n(^{238}U)$ and $n(^{236}U)/n(^{238}U)$, and in calculating measurement uncertainties.



Results for the $n(^{236}U)/n(^{238}U)$ ratio for sample REIMEP-18C, measured by participants using various mass spectrometric techniques such as accelerator mass spectrometry (AMS), inductively coupled plasma mass spectrometry (ICP-MS), isotope selective laser ionisation mass spectrometry (LASER), thermal ionisation mass spectrometry (TIMS), and TIMS with total evaporation (TIMS-TE).
$n(^{236}U)/n(^{238}U)$ certified range ($\pm U=2u_c$): 0,00103326-0,00103414

Neutron dosimetry and spectrometry using activation

The increasing number of nuclear installations world-wide also increases the need for monitoring possible neutron radiation and doses in the environment. This and issues like personal safety, safety at work or benchmarking of existing monitoring techniques are sometimes directly linked to whether precise neutron fluence monitoring is possible. However, conventional techniques for detecting neutrons and measurement of neutron energy spectra in particular, often require use of bulky equipment with delicate electronics. Such measurements are also time consuming and when using spectrometry, data evaluations, analysis and data reduction at advanced level are usually necessary.

JRC-IRMM with its collaborators has tested an alternative approach. It is based on measuring the activity induced by a neutron field in metal disks. A rather simple passive detector device is placed at the measurement site while measurements of the activated disks are carried out in a specialised laboratory. The activation measurements are followed by spectrum unfolding to obtain a complete neutron energy spectrum. This detector has a number of distinct advantages:

- it is only sensitive to neutron radiation,
- it can be made small, it requires no electrical power on measurement site and it is robust,
- it can be placed at remote locations or locations with difficult access and/or in hazardous environments,
- it is energy sensitive over a large neutron energy area, and
- it is of relatively low cost.

There are numerous situations where better knowledge of the neutron spectrum would facilitate a better understanding of the origin of the neutrons, scattering effects or possible shielding effects. In this study, the detector is designed as a neutron spectrometer, but its extension to neutron dosimetry using a different design is possible.

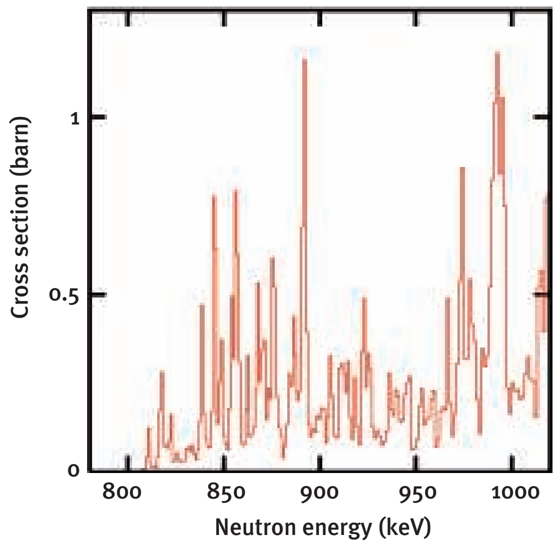
A passive detector used for neutron dosimetry is tested at JRC-IRMM

Lead and bismuth data for transmutation studies

Studies related to minimising highly radioactive waste need neutron data. For instance, lead and bismuth are candidate coolants for accelerator driven systems (ADS) optimised for waste transmutation and for certain fast reactors of Generation-IV type. Some uncertainties in calculations are due to lack of sufficiently accurate neutron data. To facilitate better reactor design, the data files need to be improved. Various studies have concluded that the uncertainties in the prediction of the neutron flux inside an ADS target are largely due to the lack of accurate data for inelastic neutron scattering and (n,2n) cross sections of lead and bismuth isotopes. Apart from that, these elements occupy a large volume fraction in lead or bismuth cooled reactors and are therefore responsible for the removal of a significant number of neutrons through capture reactions even though they have low capture cross sections. The ^{210m}Bi resulting of capture of a neutron by ^{209}Bi, has a very long half-life of $3 \cdot 10^6$ years and can thus be considered radioactive waste, while ^{210g}Bi decays with a half-life of five days to the radioisotope ^{210}Po.

In 2006, the measurements of $^{206,207,208}Pb$ and ^{209}Bi neutron inelastic scattering and (n,2n) cross sections were completed at JRC-IRMM. Exceptionally good energy resolution and accuracy were obtained. A measurement campaign to determine the ratio of production of ^{210m}Bi to ^{210g}Bi at neutron energies in the keV range was also carried out and ^{206}Pb neutron capture and transmission cross sections in the resonance range were measured.

These measurements can be used to improve the assessment of neutron removal in an ADS, and together with measurements of the neutron capture and total cross sections of ^{206}Pb of JRC-IRMM, will allow improved reactor calculations. JRC-IRMM participates in the European Research Programme for the Transmutation of High Level Nuclear Waste in an Accelerator Driven System (EUROTRANS project) that aims to the assessment and demonstration of the technical feasibility of transmutation using ADS. It coordinates the neutron data measurements at low and intermediate energy.



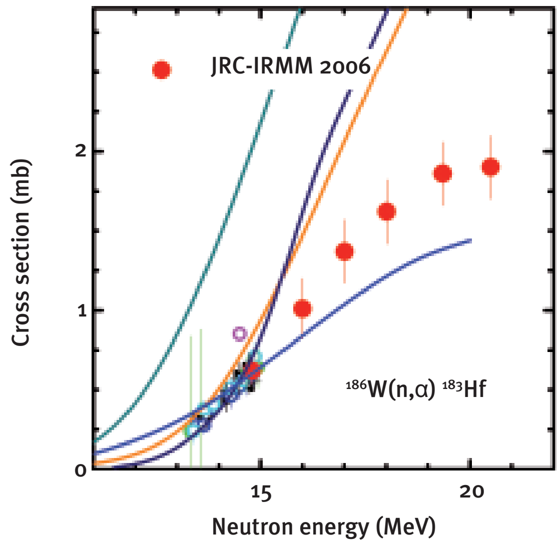
High resolution and high precision cross section for inelastic scattering of neutrons by ^{206}Pb, as measured at JRC-IRMM. The energy dependence of the cross section reflects the resonances characteristic of the interaction of a neutron with this isotope of lead

Neutron induced activation in structural materials

Materials subject to intense fields of neutrons undergo a number of changes as neutrons interact with atomic nuclei. Atoms are dislocated, foreign atoms are introduced, hydrogen and helium gas inclusions develop and radioisotopes grow in. As a result, materials swell, tensile properties and critical parameters such as breaking points change, and handling and reuse become subject to radiation safety restrictions.

Neutron induced activation cross sections are needed to fix the probabilities of the processes by which materials are modified by irradiation. Improved accuracies for such probabilities allow a better understanding of material damage and activation resulting in more reliable assessments of structural integrity and radiation safety. Such information is especially interesting for reactions where isotopes of hydrogen and helium are emitted, or which lead to radioactive final products.

Low activation steels are investigated because of their use in accelerator driven systems and in nuclear fusion test facilities. Cr, Cu, Ni, Ta and W are the main components that are interesting from activation point of view. In 2006, JRC-IRMM carried out experiments to determine the activation cross sections of Cr, Cu, Ni, Ta and W. For Cr, Ni and Cu three reactions were studied that concern rare pathways to the well-known medium half-life radioisotopes ^{48}V, ^{54}Mn, ^{56}Co and ^{59}Fe. Two of these reactions also produce tritium. Tungsten and tantalum are expected to have low activation cross sections but they are not well known. At JRC-IRMM the reaction cross sections in which isotopes of hydrogen or helium are produced, were determined. The results were adopted as a guide for improved evaluations in the latest version of the European Activation File (EAF2007).



The new measurements at JRC-IRMM of neutron-induced activation cross sections of the reaction producing He on one of the three major isotopes of tungsten are the first for energies above 15 MeV. Tungsten is commonly used in low activation materials and helium gas build-up can cause embrittlement. Predictions from various libraries scatter considerably emphasising the need for measurements to guide estimates for the production of helium gas. Full lines are predictions from major libraries (dark cyan: JENDL-3.1/JEFF3.1, dark blue: ENDF/B-VI, blue: EAF-2005, orange: TALYS-0.57), while the points around 14 MeV are from earlier measurements by others

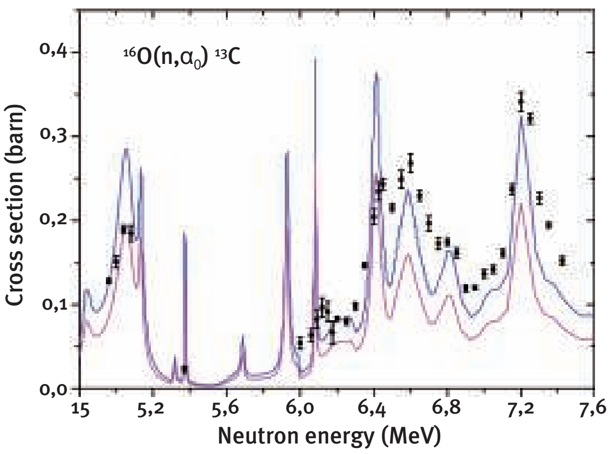
Better fission cross section data – safer nuclear energy production

Fission products are an important by-product of nuclear reactors, which have to be accounted for in criticality estimates of operating reactors as well as in criticality estimates of spent nuclear fuel. The strive towards higher burn-up in present Generation-III European pressurised water reactors (EPR) and future Generation-IV systems implies an increased demand for accuracy of the most significant fission products.

Within the collaboration agreement between the US Department of Energy (DOE, USA) and Euratom, the JRC-IRMM is performing a study of capture and transmission measurements with the Oak Ridge National Laboratory measuring isotopes of interest to criticality safety studies. Fission cross sections are investigated together with the Los Alamos National Laboratory. So far, the capture and transmission cross sections of ^{55}Mn, ^{103}Rh and ^{133}Cs have been obtained at the linear accelerator facility of JRC-IRMM.

The ^{235}U prompt fission neutron spectrum

In 2006, JRC-IRMM investigated the energy spectrum of neutrons emitted after fission of ^{235}U, which is the most important isotope for energy production in a reactor. The Working Party on Evaluation Cooperation of the OECD-NEA has requested the investigation as the accuracy of experimental data and theoretical modelling for this process are still insufficient. New measurements were started at the Van de Graaff accelerator of JRC-IRMM to obtain a better accuracy and statistical precision.



The $^{16}O(n,\alpha)^{13}C$ cross section measured at the JRC-IRMM Van de Graaff accelerator in 2006 (black; preliminary data) in comparison with the ENDF/B-VI (2001, blue line) and ENDF/B-VIIb2 (2005, purple line) evaluations

The new measurements have yielded a much better statistical accuracy than anything reported in the literature so far. Comparison with other experimental data show different levels of agreement within the statistical uncertainties given. A comparison with the theoretical models has revealed discrepancies in the emitted neutron energy range 1 to 4 MeV. By using two different detectors at different angles relative to the incoming neutron beam a probable angular dependence of the spectrum was discovered, which has been overlooked in existing evaluations. The goal in 2007 is to investigate this possible angular dependence in more detail with an improved set-up consisting of four detectors at different angles.

The $^{16}O(n,\alpha)^{13}C$ reaction cross section for criticality predictions

The OECD-NEA keeps a priority list of the nuclear data requirements. A subgroup of one of its working parties has investigated the under-prediction of reactivity of light water reactors with the most recent nuclear data evaluations, and suggested new measurements of the $^{16}O(n,\alpha)^{13}C$ cross section. In light water reactors, fuel elements are usually made of uranium oxide (UO_2) or mixed oxide (UO_2 – PuO_2) and water (H_2O) is used as a moderator and as coolant. To improve the reactivity predictions of thermal and fast reactors as well as the calculation of helium production in a reactor a better knowledge of the reaction $^{16}O(n,\alpha)^{13}C$ is necessary. Knowledge of the helium production is important for evaluating the performance of fuel pins and claddings since helium gas build-up causes embrittlement of the material. The $^{16}O(n,\alpha)^{13}C$ reaction makes up 25% of the total helium production and contributes 7% to its uncertainty.

In 2006, a measurement campaign at JRC-IRMM was started with a time projection chamber and a mixture of Kr (97%) – CO_2 (3%) gas sample at the Van de Graaff accelerator. The incident neutron energy range of 6 to 7,5 MeV and at 5 MeV was covered and the preliminary analysis showed that the JRC-IRMM data are close to the evaluated data in the ENDF/B-VI library for the 6,5 to 7,5 MeV range. There is disagreement with the most recent version ENDF/B-VIIb2, though. At 5 MeV the agreement is with ENDF/B-VIIb2 and the data disagree with ENDF/B-VI. The measurement programme will be extended in 2007.

Key measurements for the thorium fuel cycle

Using a Th-U fuel cycle for nuclear power production instead of the conventional U-Pu cycle would reduce the amount of plutonium and minor actinides like neptunium and curium in the waste. To develop this concept some nuclear reactions need to be known more accurately. JRC-IRMM carries out measurements on the capture and total cross sections of ^{232}Th, the fission cross section of ^{234}U in the resonance region and the fission cross sections for ^{231}Pa and ^{233}Pa in the fast neutron region. The new experimental data measured at JRC-IRMM can help improve the feasibility studies and risk assessment of advanced nuclear applications based on the thorium-uranium fuel cycle.

In 2006, the measurements and data evaluation of the capture cross section of ^{232}Th were completed producing also full covariance information for the entire energy range. These results were used in the new evaluation conducted by the Coordinated Research Project of the IAEA. The covariance information of the neutron capture measurements was adopted for the new ENDF/B-VII evaluation.

In 2005, the fission cross section of ^{231}Pa in the energy range of 15 to 21 MeV was measured directly for the first time, and in 2006 the JRC-IRMM investigated the ^{231}Pa(n,f) cross section at incident neutron energies below 6 MeV. Prompt fission neutron spectra and multiplicities of Pa isotopes up to 50 MeV incident neutron energy were also calculated with collaborators using modelling based on the experimental results obtained at JRC-IRMM.

JRC-IRMM measurements incorporated in new evaluations and international databases in 2006

Data	Database	Field of application
Temperature dependent transmission measurements on natural hafnium	JEFF-3.1 library OECD-NEA EXFOR database	safety of nuclear reactors
Temperature dependent transmission measurements for $^{240,\,242}$Pu	JEFF 3.1 library OECD-NEA EXFOR database	safety of nuclear reactors
^{231}Pa fission cross section data	IAEA Th/U CRP	minimising nuclear waste
$^{231,\,233}$Pa prompt fission neutron multiplicity and spectrum data	IAEA Th/U CRP	minimising nuclear waste
Total and capture cross sections of ^{206}Pb	JEFF 3.1 library	transmutation of nuclear waste
Resonance parameter evaluation for ^{206}Pb	JEFF 3.1 library	transmutation of nuclear waste
Covariance file for ^{232}Th in the unresolved resonance region	Nuclear Data Section of IAEA	transmutation of nuclear waste
High resolution measurements of ^{237}Np	JEFF 3.1 library OECD-NEA EXFOR database	transmutation of nuclear waste
Activation cross sections of isotopes of Zr, W, Ta and for specific reactions of Cr, Ni and Cu	European Activation File Project OECD-NEA EXFOR database	transmutation of nuclear waste

JEFF – Joint Evaluated Fission and Fusion File
OECD-NEA – Nuclear Energy Agency of the Organisation for Economic Co-operation and Development
EXFOR – EXchange FORmat
IAEA CRP – Coordinated Research Project of the International Atomic Energy Agency
ENDF – American Evaluated Nuclear Data File

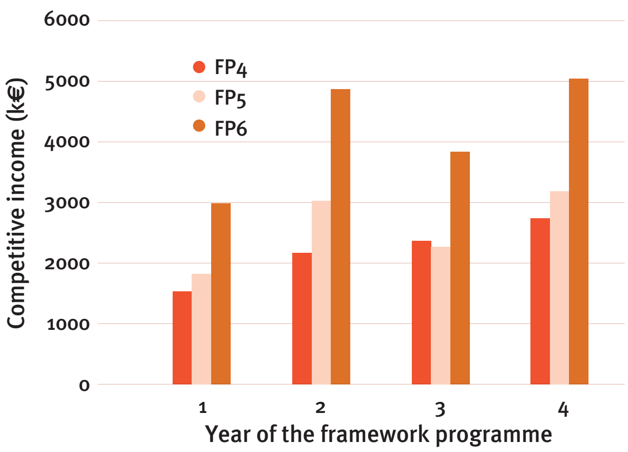
Budget

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 6th Framework Programme, and the competitive income. The institutional credits contain staff expenses, technical and administrative support and operational appropriations. The competitive income comprises shared cost actions under the Framework Programmes and other competitive contracts with the Commission, work for third parties and the distribution of reference materials.

10-15% of the Institute's budget is competitive income. It has increased throughout the three Framework Programmes (see below Figure on competitive income through FP4-FP6) and has reached a sustainable level. In comparison to 2005 an increase of 31% has been recorded. Success rate in filing for competitive contracts was 63%.

Competitive activities (k€) in FP6

	2003	2004	2005	2006
Competitive contracts	1299	3088	1691	2687
Work for third parties	281	190	335	403
Reference material distribution	1394	1587	1821	1949
Total	**2974**	**4865**	**3847**	**5039**



European projects and networks for research in which the JRC-IRMM participated in 2006	
Networks of excellence	
CASCADE	Chemicals as contaminants in the food chain
EUROFIR	European food information resource network
EUROGENTEST	Genetic testing in Europe
MONIQA	Monitoring and quality assurance in the food supply chain
NEUROPRION	Prevention, control and management of prion diseases
Integrated projects	
BIOCOP	New technologies to screen multiple chemical contaminants in foods
CO-EXTRA	GM and non-GM supply chains: Co-existence and traceability
EUROTRANS	European research programme for the transmutation of high level nuclear waste in an accelerator driven system
TRACE	Tracing the origin of food
Specific targeted research projects	
SAFEED-PAP	Detection of presence of species-specific animal proteins in animal feed
ANCIENT CHARM	Analysis by neutron resonant capture imaging and other emerging neutron techniques: new cultural heritage and archeological research methods
EAQC-WISE	European analytical quality control in support of the Water Framework Directive via the water information system for Europe
HORIZONTAL-ORG	Horizontal standards on organic micropollutants for implementation of EU directives on sludge, soil and treated bio-waste
SWIFT-WFD	Screening methods for water data information in support of the Water Framework Directive
QUOVADIS	Quality management organisation, validation of standards, developments and inquiries for solid recovered fuels
Integrated infrastructure initiatives	
ILIAS	Integrated large infrastructures for astroparticle physics
EFNUDAT	European facilities for nuclear data measurements
Coordination actions	
CANDIDE	Coordination action on nuclear data for industrial developments in Europe
IMERA ERA-NET	Implementing metrology in the European Research Area
EMERALD	Empowering the microarray-based European Research Area to take a lead in development and exploitation
Transnational access scheme (Euratom)	
NUDAME	Neutron data measurements at JRC-IRMM

Human resources

At the end of year 2006 the total number of staff at JRC-IRMM was 340 persons. The staff is composed of Commission officials and employees on fixed-term contracts. Approximately 30% of posts are non-permanent to ensure flexibility and the ability to host PhD students, post-doctoral fellows and visiting experts. In 2006 women made up 40% of all staff while 52% of the staff holding non-permanent positions and 33% of the core staff were women.

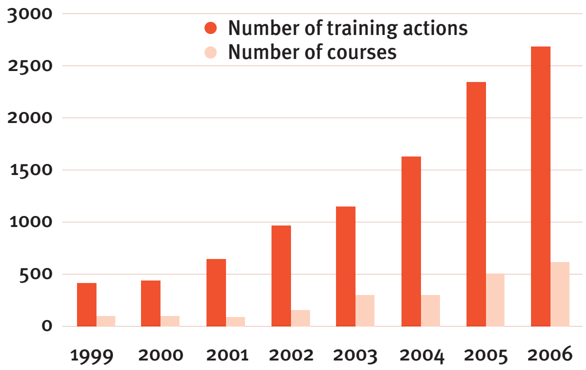
Core staff 2006

End-of-year situation	M	F	Total
Officials	122	55	177
Temporary agents on 5-year renewable contracts	1	3	4
Temporary agents on non-renewable contracts	19	11	30
Total	**142**	**69**	**211**

Staff on fixed-term contracts (in total during 2006)

Type of contract	M	F	Total
Post graduate fellow	4	1	5
Post doctoral fellow	6	7	13
Senior scientist	1	0	1
Visiting scientist	0	0	0
Seconded national expert	9	5	14
Auxiliary	0	1	1
Contractual agent	44	57	101
Trainee	2	0	2
Total	**66**	**71**	**137**

In the Commission, training is a central element of career development. The JRC-IRMM offers its staff a large variety of courses to help them develop their competences. Many of the courses given by JRC-IRMM deal with scientific and laboratory techniques, informatics, safety and security issues, but also financial regulations, soft skills, languages and implementing ISO standards are covered. Training delivered by internal trainers has been strongly encouraged. In 2006, JRC-IRMM staff spent 2700 man-days in training with an average of 9,1 per person while the JRC average was 7,6.



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 or results of many interlaboratory comparisons are published in the EUR series.

Publications in FP6				
	2003	2004	2005	2006
EUR reports	26	30	29	55
Articles in periodicals and monographs	71	89	63	86
Conference proceedings	42	5	48	55
Special publications and technical notes	7	6	6	1
Total	**146**	**130**	**146**	**197**

Events

In 2006, 19 workshops or training courses were organised at the Institute, and elsewhere. For instance, four CRL workshops for the national reference laboratories were held at JRC-IRMM, the 4th international workshop on water in food (EURO FOOD'S WATER 2006) and workshops on standardised methods for food and feed analysis with CEN in Brussels (BE) and NEMEA-3 workshop on neutron measurements, evaluations and applications in Varna (BG). JRC-IRMM was also promoting the ERM® label at Analytica 2006 in Munich (DE).

The 27 seminar lectures given during the year promote exchanging information and best practises with partners. JRC-IRMM welcomed over 650 visitors who attended meetings, workshops or guided laboratory visits. In addition to the scientific visitors, also the pupils of the European School in Mol had the opportunity to perform hands-on experiments in the JRC-IRMM laboratories and the fire brigades of the surrounding towns joined the Institute's voluntary fire brigade for exercises. To mark the change in EU presidency an Austrian-Finnish event was organised on 30 June. Also the annual barbecue for staff featured Austrian and Finnish themes with demonstrations of Finnish tango and Viennese waltz.

Pupils of the European School in Mol could benefit of the cooling assistance of the Institute' s fire brigade during a sports event

Fire exercise at JRC-IRMM

Appendix: **Organigramme**
status December **2006**

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