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JRC irmm

Institute for
Reference Materials and Measurements

ANNUAL REPORT 2005

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institute for reference materials and measurements

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EUROPEAN COMMISSION
DIRECTORATE-GENERAL
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Mission

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

European Commission

Directorate-General Joint Research Centre
Institute for Reference Materials and Measurements

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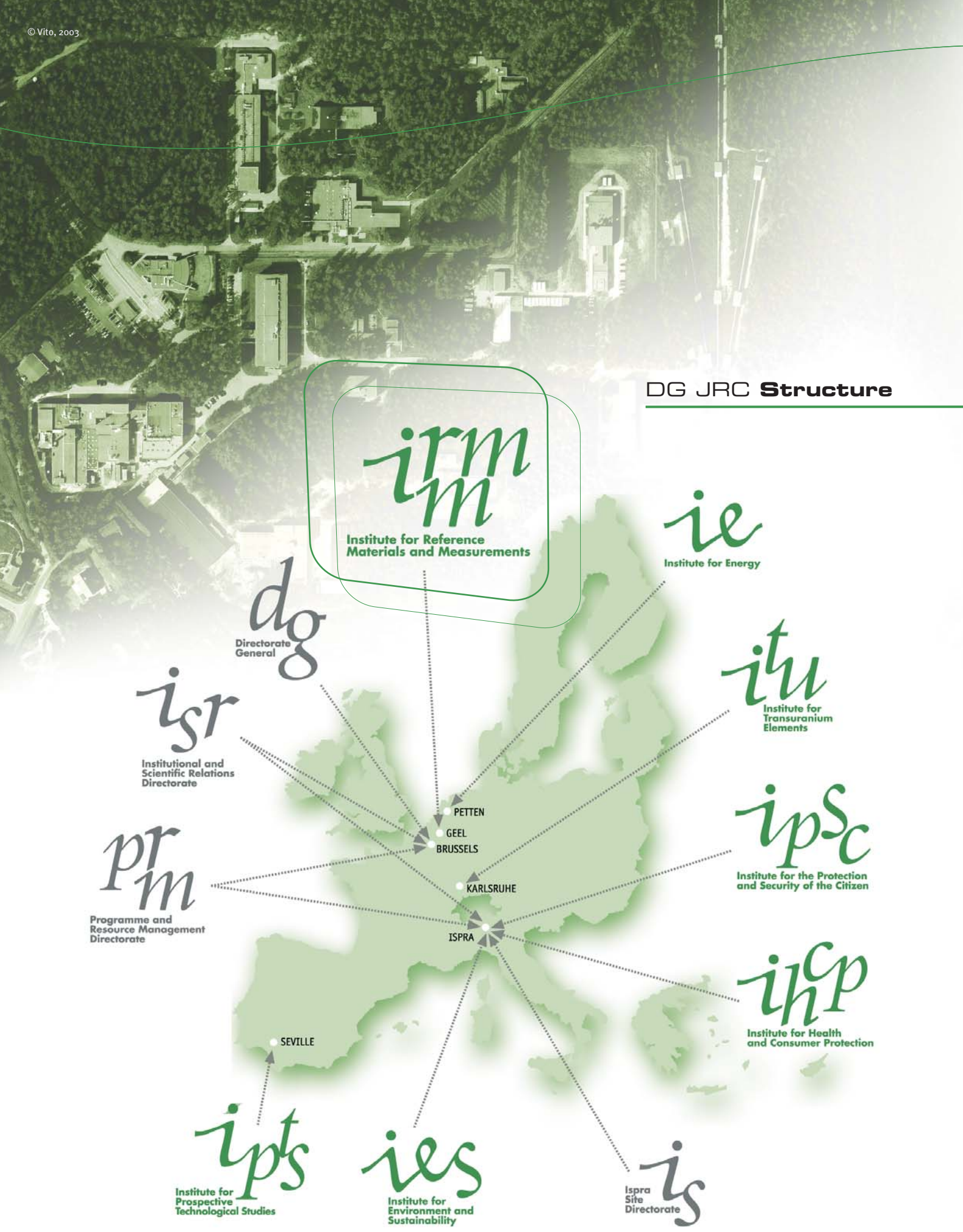


Message from the **Director**

Message from the Director

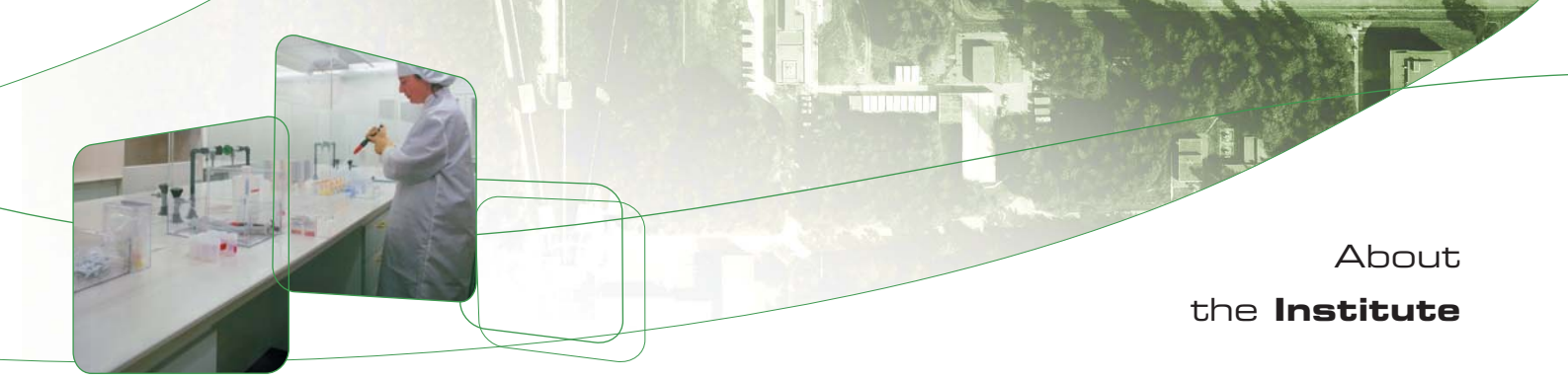
We all agree that testing results should be reliable. It is often taken for granted though, not knowing what all has to be done to ensure it. The reliability of testing routines is built on validated testing methods, reference materials and interlaboratory comparisons for instance, and standardisation sets the wider scene. Building a system of recognised reliability and comparability of measurement results is a global effort. It is the task of JRC-IRMM to help building this confidence in measurements. This task was given to JRC-IRMM when it was founded 45 years ago, and is still timely.

Work at JRC-IRMM covers a diversity of fields in science that are relevant in our everyday life. In this report we have highlighted some of our projects. For instance, in 2005 new rapid BSE/TSE tests have been evaluated. Three new series of reference materials for the analysis of genetically modified ingredients were released. A peanut test material kit is a new concept for JRC-IRMM offering the analyst new tools to improve measurement capabilities. JRC-IRMM has completed ongoing and commenced several new interlaboratory comparisons, studied the possibility of distinguishing organically grown crops from the products of conventional agriculture and the effects of heat treatment on the detection of peanut allergens. The many new and improved data provided by the JRC-IRMM neutron physics team help to model the processes. Also starting in 2005, external research teams can now apply for funding to do measurements at the JRC-IRMM accelerators. To maintain its knowledge base JRC-IRMM allocates a certain amount of resources to projects exploring technical and scientific problems. In 2005 ten research projects of exploratory nature were selected. All have yielded excellent results and contribute to robust science for policy making.

The inauguration of a new storage building for reference materials in October is a major milestone in a technically demanding project. It is a key strategic investment for JRC-IRMM's future and the new record on distribution of reference materials demonstrates the growing demand for JRC-IRMM products. The Community Reference Laboratory is now fully operational supporting the authorisation process of feed additives in the EU. Encouraged by the positive experiences, JRC-IRMM decided to apply for three other Community Reference Laboratories and writing this, we already know that they will be placed at JRC-IRMM. In 2006 those three will be set up to assist the European Food Safety Authority in the analysis of trace elements, polycyclic aromatic hydrocarbons (PAHs) and mycotoxins.

It is only fitting that in 2005 JRC-IRMM filed for the trademark 'IRMM–Confidence in measurements®'. The staff of JRC-IRMM has worked hard to fulfill this promise to provide the basis for a sound measurement system in Europe and will continue to do so. I thank my staff for that and with the many successes of year 2005, we can look forward to a new exciting year with our collaborators.

Alejandro Herrero
Director JRC-IRMM



About the **Institute**

The Institute for Reference Materials and Measurements (IRMM) is one of the seven Institutes of the Joint Research Centre (JRC), a Directorate-General of the European Commission, providing independent scientific and technical support to Community policy-making. The JRC-IRMM was founded in 1957 under the Treaties of Rome and started operation in 1960 under the name of the Central Bureau for Nuclear Measurements (CBNM). Today JRC-IRMM is an expert adviser in food safety and quality and bioanalysis, a valued provider of reference measurement data, and one of the world's largest reference material producers.

Mission and tasks

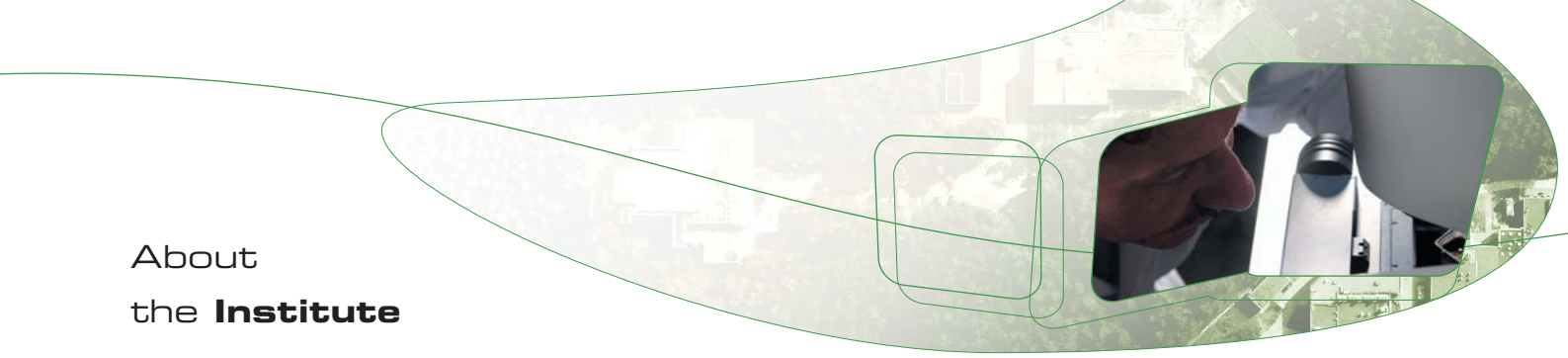
JRC-IRMM promotes a common and reliable European measurement system in support of EU policies. The primary task of JRC-IRMM is to build confidence in the comparability of measurement results by the production and dissemination of internationally accepted quality assurance tools. JRC-IRMM develops and validates analytical testing methods, produces reference materials, organises measurement evaluation programmes, and provides reference measurements.

As a recognised metrology institute 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 continuously contributes to improving 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). In 2004 JRC-IRMM started its operation as the Community Reference Laboratory for Feed Additives Authorisation.

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, and External Relations. All Commission departments 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 and authorities, universities as well as with industry. JRC-IRMM has over 60 formal collaboration agreements with 80 research partners, and numerous other partners via informal and competitive activities. In 2005, for instance, the collaboration between JRC-IRMM and RIKILT Institute of Food Safety was formalised and the collaboration agreement with the European Co-operation for Accreditation (EA) was renewed.

Mr Alejandro Herrero, the Director of the JRC-IRMM, and Mr Daniel Pierre, the Chairman of EA, met on the 29 November 2005 in Geel, to sign the new five-year agreement. This arrangement aims to demonstrate the reliability and comparability of chemical measurements.



About the **Institute**

Core competencies

The core competencies of JRC-IRMM are development and production of reference materials, development and validation of methods for food and feed analysis, bio-analysis, 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 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.

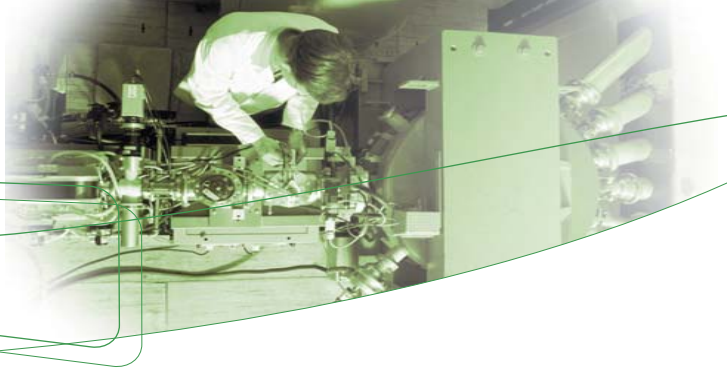
JRC-IRMM is one of world's largest reference material producers.

Special infrastructure

The research facilities include multi-functional and flexible laboratories for development and production of reference materials, advanced analytical laboratories 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).

Some reference materials require storing above liquid nitrogen to avoid degradation.

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, offer experimental conditions that are complementary to those of other installations in the world. As from 2005 the two accelerators can accommodate external users via a project on access to large scale facilities (NUDAME).



Highlights in **2005**

Opening a new storage building

JRC-IRMM has constructed a new storage building for its reference materials. The new facility is an important upgrading of JRC-IRMM's infrastructure in a key area of its mission–namely to promote a common and reliable European measurement system.

Producing a certified reference material may take many years to accomplish and the stock of a reference material has to remain stable for several years. During that time it has to be stored and monitored appropriately. JRC-IRMM has invested in an entire building for the storage of its reference materials. The 1550 m² building houses currently over 580 different certified reference materials, in total around 500 000 samples. It has compartments to store materials at temperatures from -70 to +180 °C at controlled humidity. Some are stored even at -160 °C in special containers. 113 of the materials carry the new label ERM®–for European Reference Materials–marking products accepted in accordance with the latest international standards, ISO Guides 34 and 35.

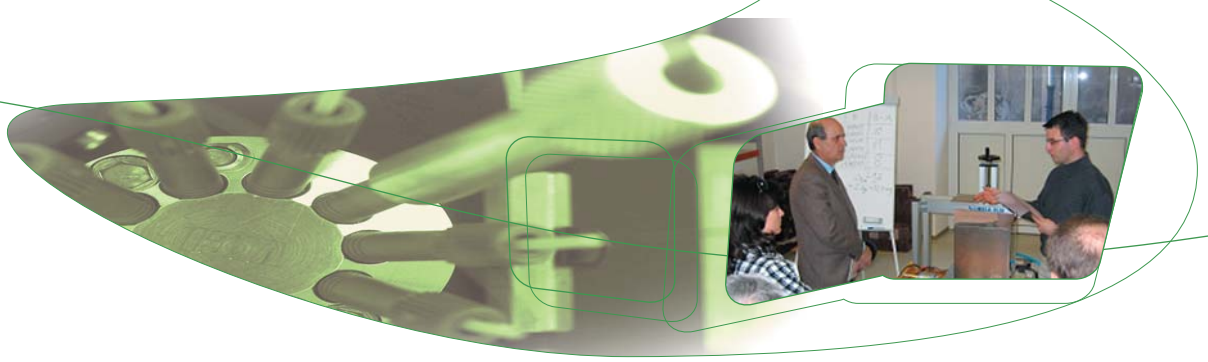
Samples are transferred from the low temperature chamber in the storage building through a lock.

Commissioner Potočnik officially opened the new building on the 20 October 2005. The opening was combined with a conference 'Confidence in Measurements' where the Nobel laureate Stanley Prusiner gave a keynote lecture.

Integration and enlargement

JRC-IRMM has actively supported the enlargement of the European Union since 2001 by providing assistance and training in its fields of expertise to help the Candidate Countries' integration to the European framework of legislation. This assistance is extended to the Stabilisation and Association Process and to include the Western Balkan Countries. JRC-IRMM helps to set up their measurement infrastructure, particularly in the fields of internal market, environment and food.

Earlier JRC-IRMM has helped representatives of the new Member States and Candidate Countries to map the status of metrology in chemistry in their countries. In 2005, a similar EU funded project was being carried out in Albania where metrology needs to meet new requirements. The establishment of a free trade area in the Balkans as well as a future free trade agreement with EU requires a functioning metrology system, which will also fulfil the requirements set on quality of environment and safety and quality of food. Within this project JRC-IRMM provides expert advice in capacity building, and helps upgrading the Albanian legal framework related to metrology. The project provides also for technical assistance and training, and helps in increasing public awareness. 150 local employees have been trained during their short-stay visits abroad and in Albania on topics like drafting of the legislation on metrology and organizing the national measurement infrastructure to meet European requirements, organising and managing regional verification offices to meet EU legal metrology requirements and implementation of EU legal metrology requirements and related quality assurance issues. The training was given by JRC-IRMM and national metrology institutes from Austria, Bulgaria, Czech Republic and Slovenia.



Training

Today's European directives and regulations can often only be implemented when reliable measurements are available. This requires a properly operating measurement infrastructure across the Member States, else implementation cannot be guaranteed. For this reason, JRC-IRMM has set up a training platform to improve the quality of analytical results by promoting and providing a European-wide harmonised practitioner training in metrology in chemistry via the TrainMiC programme, which is a network of national providers. JRC-IRMM has also set up a system of training trainers. In 2005, about 600 people were trained at 12 events in ten countries.

JRC-IRMM has also launched an initiative to create an European academic forum, where analytical chemistry lecturers can meet and share ideas and best practice to teach generic measurement science in analytical chemistry. In total, about 40 academic lecturers from 21 countries in Europe participated in a summer school in Rogaška Slatina (SI) in 2005.

JRC-IRMM is a partner of the Virtual European Radionuclide Metrology Institute (VERMI) together with the radionuclide metrology departments of Laboratoire National Henri Becquerel (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. VERMI has organised two training workshops on absolute and secondary standardisation of radionuclides for its own young staff. In 2005 JRC-IRMM organised the third VERMI Young Researchers Training Workshop on Standardisation of Radionuclides in Varna, Bulgaria. Eleven experienced lecturers from the four VERMI institutes addressed 39 participants during 5 1/2 days. 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. Primarily aimed at young researchers from Central and Eastern Europe, the workshop attracted participants from seven new Member States, the two Candidate Countries Bulgaria and Romania, the two Western Balkan Countries Albania and Croatia, as well as Georgia.

Access to JRC-IRMM accelerators –The NUDAME project

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 can now apply for measurement time at JRC-IRMM.

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).

The NUDAME project started in the spring of 2005 and has a total duration of three years. Any type of experiment in the areas of radioactive waste management, radiation protection and other activities in the field of nuclear technologies and safety can be proposed where the JRC-IRMM experimental infrastructure can offer a significant added value to the project. Selection is based on peer review by a programme advisory committee. Financial support to the approved proposals is provided by the European Commission through JRC-IRMM. 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.

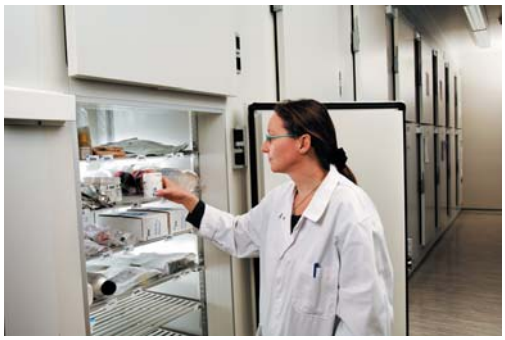
In the first meeting of the programme advisory committee in July 2005 four experiments were selected for the period September 2005-March 2006. The approved experiments deal with neutron fluence measurements for the calibration of spectrometers using the well defined neutron beams of the Van de Graaff facility, fission cross-section measurements of ^{234}Am, and high-resolution measurements on ^{nat}Hf and Cd.

The Community Reference Laboratory for feed additives authorisation

Feed additives are now authorised according to Regulation (EC) No 1831/2003, which introduced a new authorisation procedure and established a Community Reference Laboratory (CRL) on feed additives authorisation. The implementation of Regulation (EC) No 378/2005 nominates the JRC-IRMM as CRL. The main task of the CRL is to evaluate analytical methods proposed by the feed additive producer applying for authorisation that are suitable for the determination of the active substance in feed and, if applicable, also residues thereof in animal tissues or animal products. In addition, the CRL is responsible for the reception, storage and maintenance of the samples of the feed additive sent by the applicant. The CRL carries out its tasks in close cooperation with a network of national reference laboratories.

In 2005 the CRL and its network evaluated the analytical methods from 14 dossiers related to different types of feed additives, such as probiotics, enzymes and coccidiostats. For each dossier an evaluation report is made that is sent to the European Food Safety Authorisation (EFSA) to be appended to the overall evaluation. The implementation of the new evaluation procedure of the analytical methods was discussed with the representatives from the national reference laboratories, the Directorate-General for Health and Consumer Protection and EFSA in the two workshops that the CRL organised.

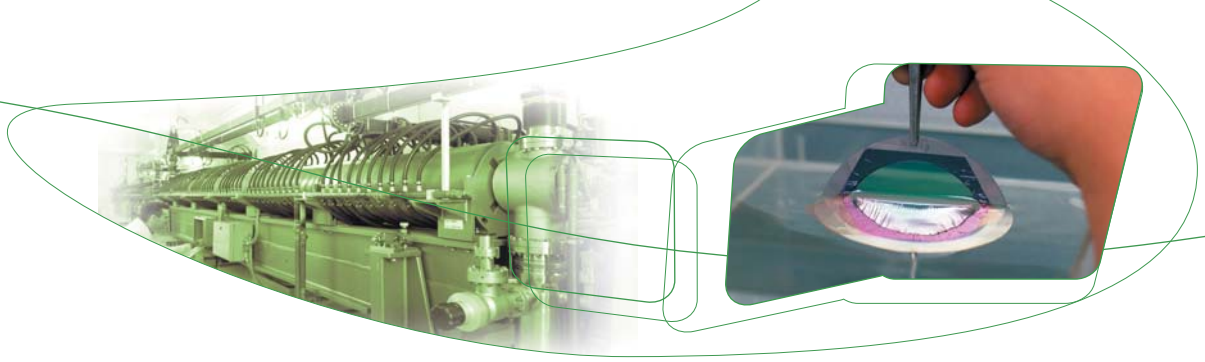
The CRL also supports the standardisation of analytical methods in the field of analysis of feed additives. In 2005 the CRL participated in the validation of a method for the determination of phytase activity in feed, which is currently under review in the Technical Committee on Animal Feeding Stuffs (TC 327) of the European Committee for Standardization (CEN).



JRC-IRMM is operating the Community Reference Laboratory for feed additives authorisation since 2004.

Recognised excellence

JRC-IRMM staff are members in numerous committees, their working groups and scientific boards of international organisations. In 2005 JRC-IRMM has taken over representing the European Commission in the steering committee of the Versailles project on Advanced Materials and Standards (VAMAS) and can now offer its expertise for the measurement communities in material sciences and generic concepts on the development, production and application of reference materials. Hendrik Emons was elected as the new vice-chairman of the Committee on Reference Materials of the International Organization for Standardization (ISO/REMCO). This policy committee develops and reviews ISO guides ensuring scientific underpinning and international harmonisation of the technical and quality characteristics of reference materials. The vice-chairman is responsible for the strategic planning, business plan and education activities of the committee.



Highlights in **2005**

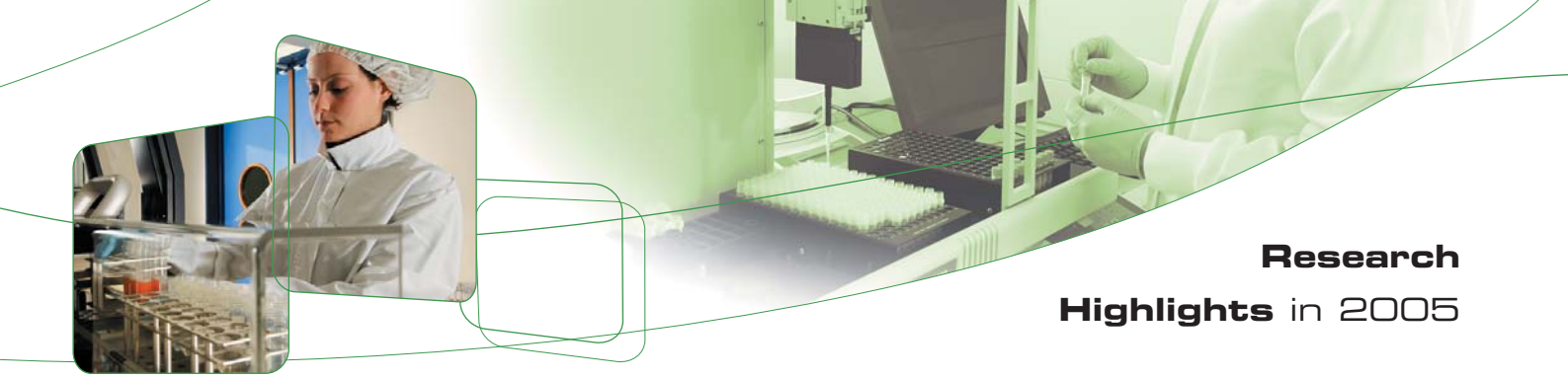
JRC-IRMM's work in the field of standardisation is widely recognised. For instance, the new revision of ISO Guide 35, published in 2005, contains concepts for certifying reference materials developed at JRC-IRMM. Various technical committees of ISO use expert advice of JRC-IRMM on reference materials 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 the European Committee for Standardization (CEN).

In 2005, Philip Taylor was awarded the Dr Jerzy Fijalkowski prize by the Atomic Spectrometry Commission of the Polish Academy of Sciences at the bi-annual meeting in Ustron (PL). The prize was awarded in recognition of his achievements in the field of reference measurements using mass and atomic spectrometry as well as for his efforts made in knowledge dissemination regarding chemical measurements during and after the accession of Poland to the European Union.

Within the JRC the Excellence Awards are given every year in recognition of the efforts and passion the JRC staff have put in their everyday work. This year, Carlos Chaves and Thierry Gamboni from JRC-IRMM won the prize for the realisation of the high-intensity/stability beam line at the JRC-IRMM Van de Graaff neutron physics laboratory. Wolfgang Philipp and Heinz Schimmel were awarded for their contribution to the European BSE/TSE monitoring programme. Since the outbreak of BSE in the UK, Wolfgang Philipp and Heinz Schimmel and their teams have carried out a number of studies evaluating all BSE/TSE tests proposed for approval in the EU. In 2005 alone the approval of seven new rapid post mortem BSE tests in the EU was based on the evaluation reports of JRC-IRMM.

In addition, JRC-IRMM best young scientist award was given to James Snell, and the JRC-IRMM award for best peer-reviewed scientific paper was given to Stefaan Pommé, Timotheos Altzitzoglou, Raf Van Ammel and Goedele Sibbens for the article 'Standardisation of ^{125}I using seven techniques for radioactivity measurement' published in Nuclear Instruments and Methods in Physics Research A 544 (2005) 584–592. Piotr Robouch, Steluta Duta, Ewa Bulska, Nineta Majcen and Emilia Vassileva received a special recognition for their contributions to the TrainMiC programme.

Thierry Gamboni, Heinz Schimmel, Carlos Chaves and Wolfgang Philipp won JRC Excellence Awards in 2005.



The work programme of the JRC is divided in four core areas and JRC-IRMM contributes to all of them. The research at JRC-IRMM covers a large variety of topics i.e. priority areas under the core areas.

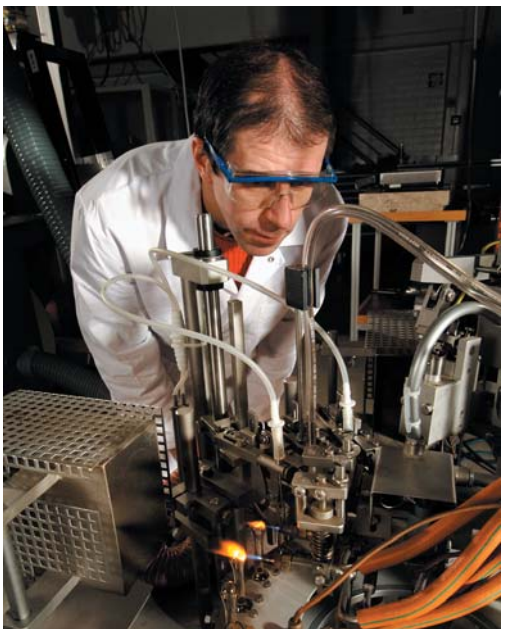
Core area horizontal activities: specifically *reference materials and measurements*, which due to the nature of the work, are also contained in other core areas;

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 2005 is presented here under these headings.



Ampouling of candidate reference material solutions.

Reference materials and measurements

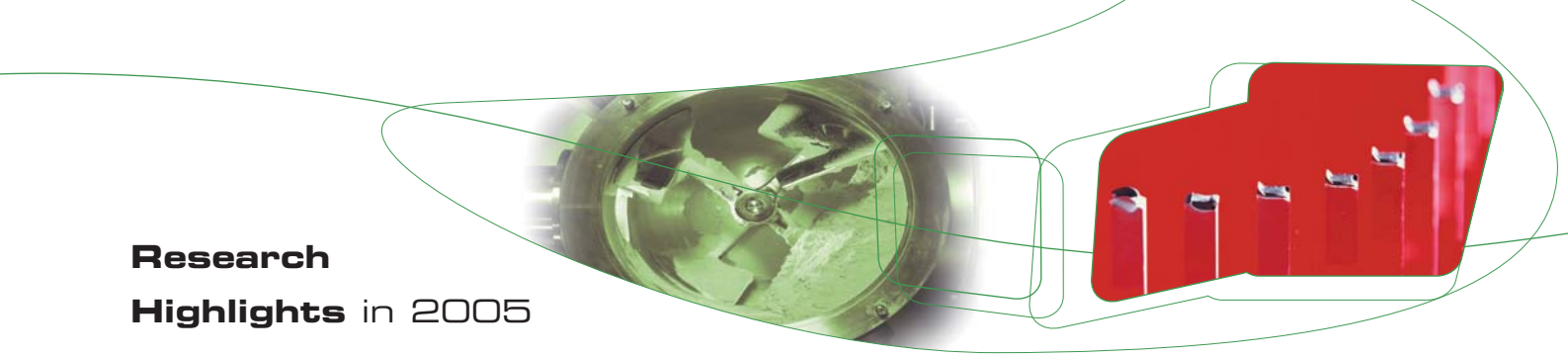
One of the key elements of cross border comparability and reliability of testing results is the availability of reference materials. Reference materials are needed for developing, calibrating and validating the methods of analysis. Today, reference materials are also needed to fulfill the requirements of present standards for the accreditation of testing and calibration laboratories, and demonstrating technical competence makes participation in interlaboratory comparison schemes often necessary.

JRC-IRMM is one of the world's largest reference material distributors and offers a large variety of certified reference materials for food and environmental analysis, for biotechnology, health related and industrial measurements, and nuclear safeguards. The Reference Materials unit of JRC-IRMM is accredited in accordance with the requirements of two international standards, the ISO Guide 34 and the ISO 17025.

JRC-IRMM organises interlaboratory comparisons to evaluate the equivalence of accreditation across Europe. 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.

Calibration solutions for mycotoxin analysis

The Fusarium fungi are probably the most prevalent toxin-producing fungi of the northern temperate regions. They are commonly found on cereals grown in those areas (the temperate regions of America, Europe and Asia). A variety of Fusarium fungi, which are common soil fungi, produce toxins of the class of trichothecenes such as T-2 toxin, HT-2 toxin, deoxynivalenol and nivalenol, and some other toxins like zearalenone and fumonisins.



Research Highlights in 2005

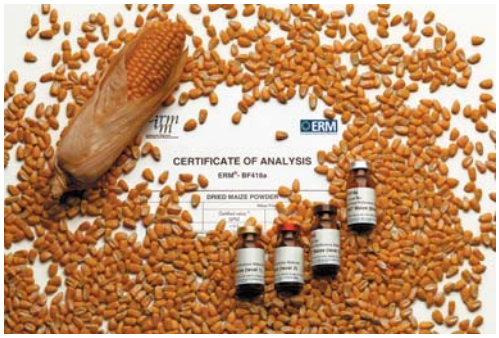
Controlling mycotoxin levels in food and feed is essential for protecting the health and well-being of European citizens. Legislation in the EU sets maximum levels for a number of mycotoxins in food and feed. Recently a statutory limit has been established for deoxynivalenol. The authorities in the Member States are tasked with enforcing the regulations. To enhance the reliability of trichothecene measurements the JRC-IRMM has developed and certified reference materials as calibration solutions for deoxynivalenol and nivalenol. These solutions can be used by laboratories to calibrate their instruments. Correct calibration is a key element for obtaining reliable and comparable measurements.

First certified reference material for a stacked GMO event in maize

Legislation in the European Union regulates the labelling of food products containing more than 0.9% genetically modified organisms (GMOs). For the characterisation of each GMO authorised in Europe, certified reference materials for the control and calibration of quantification methods like the real-time polymerase chain reaction technique are available. Lately, a trend towards applying stacked GMOs has been observed.

In 2005 the JRC-IRMM certified the first reference material for a stacked maize event, i.e. the incorporated DNA sequence and its location. ERM-BF417 has been certified for its mass fraction of the breeding stack MON 863 x MON 810. Due to the nature of the stacking, there are currently no methods available to distinguish between a breeding stack like MON 863 x MON 810 maize and a mixture of the single events MON 863 maize and MON 810 maize, unless analysis of the individual seeds is possible. The verification of the GM status of the seeds used for the production of a reference material is crucial for the certification in general. In the particular case of a stacked event material the verification needs to be carried out for both breeding partners of the stacked event.

ERM-BF417 in combination with the other materials produced at the same time for the single event MON 863 (ERM-BF416) and the one produced earlier for the single event MON 810 (ERM-BF413) will facilitate research on stacked GMO events. They will also allow to verify the performance of event-specific methods validated for the individual single events on stacked material.



New GMO reference materials were certified in 2005.

Certified reference materials for impact tests

The Charpy pendulum impact test is a method to measure the impact resistance of a material. The test consists of breaking a sample with a swinging hammer, while measuring the energy absorbed by the sample during the fracture process (KV). In Europe, materials used to construct pressure equipment must satisfy a minimum requirement in terms of KV as defined in the Pressure Equipment Directive (97/23/EC).

The measured KV depends on the dynamic behaviour of the Charpy pendulum impact machine, which must be regularly verified. The verification consists of breaking certified reference materials, and comparing the measured KV values with the certified values. For over 10 years now, JRC-IRMM has produced certified steel reference materials for instrument verifications, in accordance with the ISO 148 and EN 10045 standards. In 2005, the process of certifying the Charpy reference materials at JRC-IRMM obtained ISO Guide 34 accreditation.

The Charpy specimens can also be used to assess the uncertainty of the measurements. In 2005, JRC-IRMM convened and chaired a working group on measurement uncertainty of the corresponding ISO Technical Committee (TC 164/SC4p), where the annexes to the existing ISO 148 standards were drafted and revised. The proposed procedure to assess the uncertainty of the result of a Charpy pendulum impact test is implemented in the JRC-IRMM Charpy laboratory, which in 2005 obtained ISO 17025 accreditation.

Interlaboratory comparisons for benchmarking and validation

Interlaboratory comparisons can be run on different levels. Some involve the best laboratories in the world while others may test the robustness of a method by assessing as large a range of capabilities as possible. JRC-IRMM organises interlaboratory comparisons for many purposes and also participates in comparisons organised by other international organisations.

JRC-IRMM organises International Measurement Evaluation Programmes (IMEPs) to assess the measurement capabilities of testing laboratories.

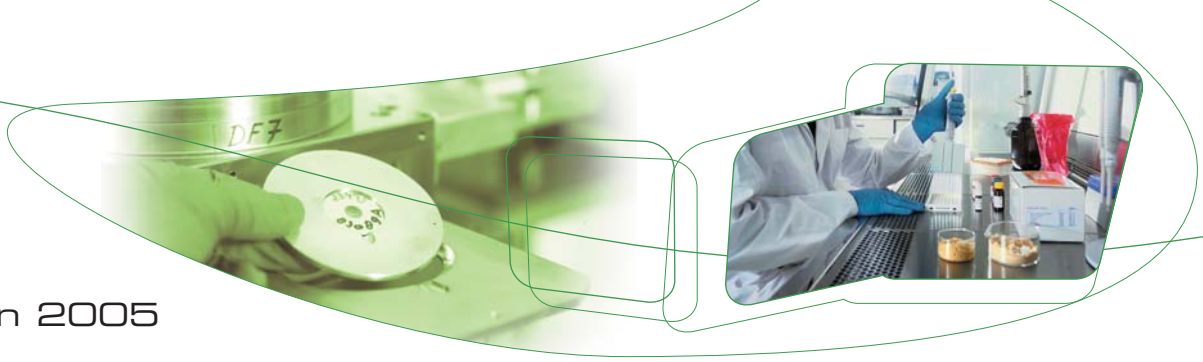
Many of the interlaboratory comparisons organised by JRC-IRMM are open to all laboratories that wish to participate but some are restricted to e.g. national reference laboratories or in the case of nuclear measurements to those that are licenced to keep and import radioactive samples. Some comparisons are run to certify reference materials and validation studies are organised regularly to validate methods of analysis. Often an interlaboratory comparison organised by JRC-IRMM was prompted by new or modified European legislation or policy.

JRC-IRMM runs an International Measurement Evaluation Programme (IMEP), a Regular European Interlaboratory Measurement Evaluation Programme (REIMEP) for nuclear measurements, and an Interlaboratory Measurement Evaluation Programme for Nuclear Signatures in the environment (NUSIMEP). In 2005, the IMEP team was running three campaigns. IMEP-18 on the analysis of sulphur in diesel was completed and IMEP-21 on the analysis of trace elements, polychlorinated biphenyls (PCBs) and polycyclic aromatic hydrocarbon compounds (PAHs) in sewage sludge has reached the evaluation stage. IMEP-22 on the analysis of sulphur in petrol, complementing the IMEP-18 campaign, has been started. In 2005, NUSIMEP and REIMEP were focussed

Milk powder samples were sent to 60 laboratories monitoring radioactivity in the environment.

on measurements of isotopic abundances of plutonium in plutonium nitrate samples and abundances of uranium isotopes in simulated urine.

In 2005 JRC-IRMM has also organised an interlaboratory comparison that evaluates the analysis of PAHs in edible oils, several campaigns to validate methods for the analysis of mycotoxins, acrylamide and semicarbazide, and a proficiency test for laboratories analysing semicarbazide in baby food and in egg. The annual exercise to evaluate



the comparability of data collected from laboratories measuring radioactivity in the environment by the JRC Institute for Environment and Sustainability was started by sending milk powder samples to 60 participants.

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.

Interlaboratory comparisons organised by JRC-IRMM in 2005

Topic	Type of comparison
sulphur in petrol	international measurement evaluation–IMEP-22
sulphur in diesel fuel	regional key comparison–EUROMET project 785
trace elements, PCBs and PAHs in sewage sludge	international measurement evaluation–IMEP-21
uranium isotopes in a simulated biological matrix	international measurement evaluation–NUSIMEP-4
uranium isotopes in a simulated biological matrix	pilot study CCQM-P48 for CIPM
uranium, plutonium and cesium isotope ratios in saline medium	international measurement evaluation–NUSIMEP-5
analysis of PAHs in edible oils	proficiency test
specific activity of the radionuclides ^{40}K, ^{137}Cs and ^{90}Sr in milk powder	annual evaluation of comparability of data collected from Member States monitoring radioactivity in the air, water and soil
allergens; detection of peanut in biscuits using dip sticks	method validation by way of collaborative trial
screening and identification of antibiotics in feed	method validation by way of collaborative trial
screening and identification of growth promoters in feed	method validation by way of collaborative trial
feed additives; determination of phytase activity in feeding stuffs	method validation by way of collaborative trial
determination of animal DNA in feed by PCR technique	pre-validation study by way of collaborative trial
determination of semicarbazide in egg	method validation by way of collaborative trial
determination of semicarbazide in baby food	method validation by way of collaborative trial
determination of semicarbazide in egg and baby food	proficiency test
determination of acrylamide in bakery and potato products	method validation by way of collaborative trial
determination of the mycotoxin deoxynivalenol in animal feed and baby food	method validation by way of collaborative trial
determination of the mycotoxin zearalenone in animal feed and baby food	method validation by way of collaborative trial
determination of the mycotoxins T-2 & HT-2 toxin in grains	method validation by way of collaborative trial
determination of the fumonisin mycotoxins in breakfast cereals and baby food	method validation by way of collaborative trial
PCBs in organic solution	interlaboratory comparison–EUROMET/METCHEM project 833
impact of extraction methods on DNA quantification for GMO analysis	pilot study CCQM-P60 for CIPM

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

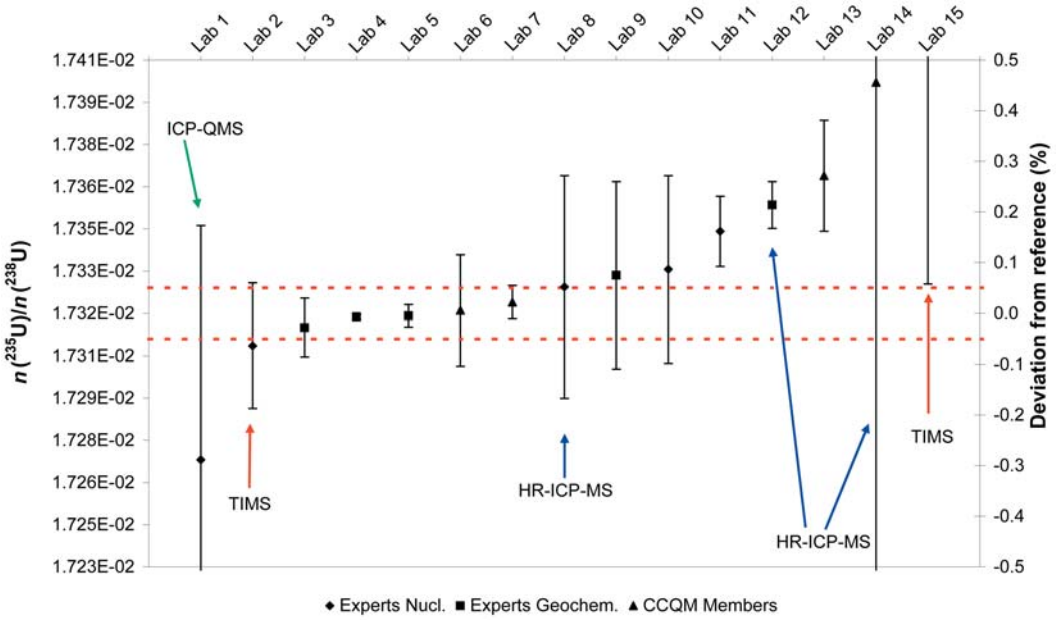
Benchmarking organic analysis capabilities

The reliability of the international system for chemical and biochemical measurements is strengthened through interlaboratory comparisons organised by the Consultative Committee for Amount of Substance–Metrology in Chemistry (CCQM) of the CIPM. The key comparisons allow to benchmark the analytical capabilities of the participating organisations. These activities are mandated by the International Metre Convention and an arrangement for the mutual recognition of national measurement standards and of calibration and measurement capabilities known as the Mutual Recognition Arrangement (MRA) has been signed by most national measurement institutes, and JRC-IRMM.

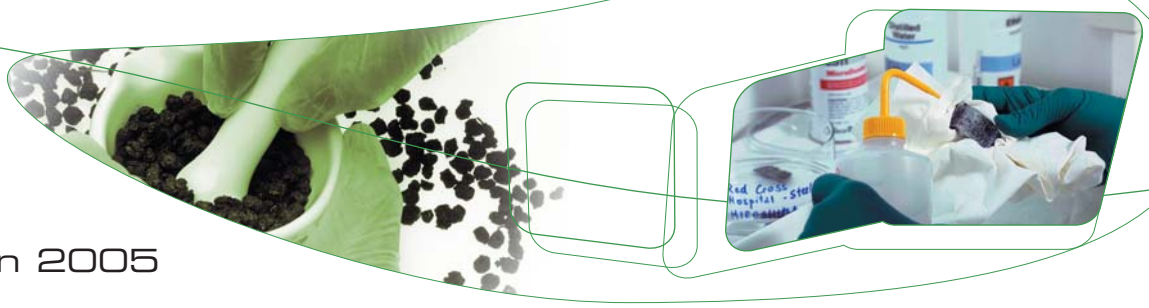
The Organic Analysis Working Group of the CCQM organised in 2005 a number of international pilot studies and key comparisons where JRC-IRMM participated. As a result 24 claims submitted by JRC-IRMM for organic analytes such as creatinine, aflatoxin M1 or PCB congeners in various matrices, were approved by the International Committee for Weights and Measures (CIPM) and were included in the Annex C to the MRA in 2005. Several claims for the analysis of PAHs and chlorinated pesticides were submitted in 2005. Such demonstrated measurement capabilities integrate participants into the global network formed by the world's national measurement institutes.

Comparison of uranium isotope ratio measurements

Uranium can be released into the environment by accidents, illegal dumping of nuclear scrap or waste, or releases of traces from declared or clandestine sources for instance. The use of ammunition of depleted uranium during military conflicts (e.g. Gulf war, Kosovo) has recently resulted in the generation of dusts containing depleted uranium. The need to study the effects to health and environment has also created a need to develop and improve measurement capabilities for uranium isotope ratios and uranium amount contents in environmental and biological samples.



Results of the CCQM-P48 interlaboratory comparison. Dashed red lines mark the reference range.



Research Highlights in 2005

Although very important political, economical, military or medical decisions may be based on such measurement results, sound and realistic uncertainty estimations associated with the measurement result are often lacking, making the comparison between different published sets of results difficult.

To demonstrate the capability of laboratories experienced in the field of measuring uranium isotopes in environmental samples, JRC-IRMM has organised the pilot study 48 of the CCQM of CIPM in 2005. This is an international comparison between 15 renowned laboratories worldwide in the field of chemical metrology, geochemistry and nuclear measurements. Test materials consisted of four isotopic mixtures of uranium in a simulated biological matrix, produced at JRC-IRMM and prepared at 5 µg U g^{-1} and 5 ng U g^{-1}. Participants reported results for the $n(^{234}U)/n(^{238}U)$, $n(^{235}U)/n(^{238}U)$ and $n(^{236}U)/n(^{238}U)$ isotope ratios, measured by thermal ionisation or inductively coupled plasma mass spectrometry.

The $n(^{235}U)/n(^{238}U)$ results could be compared to the reference values measured at JRC-IRMM during the preparation of the uranium isotopic mixtures and to those obtained in a parallel exercise in the NUSIMEP-4 campaign using same test materials. Although the emphasis of the two campaigns was different, a comparison of the results shows the qualitatively better values measured by the participants of the CCQM-P48. The results are being studied and will point the way to further interlaboratory comparisons for measurements of isotopic mixtures of uranium. This was the first CCQM comparison of isotope ratio measurements.

Confidence in radioactivity measurements

Iodine-125 isotope is used for clinical tests and in particular to diagnose thyroid disorders. As an Auger-electron emitter, ^{125}I is an attractive alternative to beta emitters for cancer therapy, in particular since it can be placed in close proximity to nuclear DNA. Its ability to deposit energy in extremely small volumes, in the range of cubic nanometers, makes it a valuable probe of radiobiological phenomena. As ^{125}I is also used for photon detector calibration at the low end of the energy scale, accurate radioactivity measurements of reference sources are therefore needed.

A recent key comparison with national metrology institutes of twenty countries has revealed that many primary standardisation laboratories have great difficulty in determining the activity of a ^{125}I solution accurately. JRC-IRMM has standardised the ^{125}I solution for radioactivity by means of seven primary techniques, of which four are unique in the world. These measurements demonstrated the consistency of six methods and the fact that one commonly used method is significantly biased. Moreover, by pushing the precision of each individual method to the limit and by combining their results JRC-IRMM succeeded at reaching a record low overall uncertainty.

One of the crystals of the CsI(Tl) sandwich spectrometer that is used for primary standardisation of activity.

Underground search for the decay of ^{180m}Ta

Tantalum-180m is the rarest isotope in nature and the only primordial isotope in nature that is not in its nuclear ground state. But the radioactivity of ^{180m}Ta has not yet been observed. Previous attempts to measure the half-life of ^{180m}Ta have been performed using detectors located above ground. At JRC-IRMM the radionuclide metrology team and its collaborators measured a 606 g Ta disk of natural isotopic composition for 170 days in the 225 m deep underground laboratory HADES located at SCK•CEN in Mol (BE). The radioactivity could not be detected but the lower bounds for the half-life could be significantly improved. It is now $1.7 \cdot 10^{16}$ years for the electron capture decay and $7.7 \cdot 10^{15}$ years for the β-decay. The new lower bound of the total half-life is a factor of five higher than the previous best value obtained by a research group at Brookhaven National Laboratory (USA).

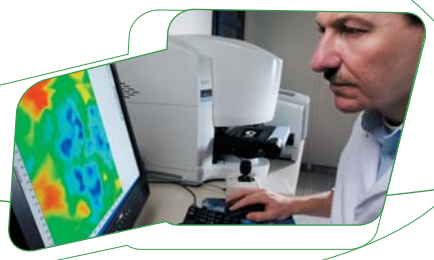
JRC-IRMM has now designed an experiment that should allow improving the lower bound by a factor of 20. The discovery of the radioactivity of ^{180m}Ta would elucidate questions of its production during nucleosynthesis in stars.

Measurements at the underground laboratory have improved the lower bound for the half-life of ^{180m}Ta.

Food chain, biotechnology and contributions to health

Food scares undermine public confidence in that the public authorities and the food industry are able to ensure that food is safe. Therefore, food safety is one of the top priorities of the European Commission. Since 2000 the framework of rules in Europe covers the whole food chain. To implement the European Commission's food safety policy though, means for the control are needed e.g. for monitoring compliance with labelling rules or for measuring the contents of contaminants or additives in food and feed. Developments in biotechnology and other life sciences bring about new needs for assuring the quality of testing result, also in the field of health applications.

JRC-IRMM produces new reference materials, and develops, validates and tests analytical methodologies for food and feed analysis. In the area of biotechnology and health related research, JRC-IRMM develops and produces reference materials for bioanalysis and clinical measurements, explores the requirements set by biotechnology research, and has an advisory role in measurement related matters. 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 is increasingly giving scientific and technical support to the Directorates-General (DGs) that develop food and feed legislation, to the European Food Safety Authority (EFSA), to Community Reference Laboratories and to national food and feed control laboratories, and operates a Community Reference Laboratory for feed additives authorisation.



Research Highlights in 2005

Contaminants in food

The JRC-IRMM develops and validates methods by way of collaborative trials for the analysis of both natural and anthropogenic contaminants in food and feed products for the Health and Consumer Protection DG. To support Regulation (EC) No 466/2001 and its later amendments on contaminants, JRC-IRMM organised in 2005 collaborative trials to validate analytical methods for the determination of the mycotoxins deoxynivalenol and zearalenone in baby food and animal feed, and T-2 and HT-2 in grains. In addition, suitable and simple alternatives for currently used sampling schemes for mycotoxins in grains have been investigated.

Animal feed extracts are filtered before immunochemical clean-up.

To facilitate the implementation of Regulation (EC) No 208/2005 amending Regulation (EC) No 466/2001 in its part on polycyclic aromatic hydrocarbons (PAHs) and the Recommendation 2005/108/EC concerning the monitoring of PAHs in food products, the JRC-IRMM has helped EFSA to establish a European database on PAHs by assessing the suitability of analytical methods currently available, and has also organised a workshop on methods of analysis with the Health and Consumer Protection DG and EFSA.

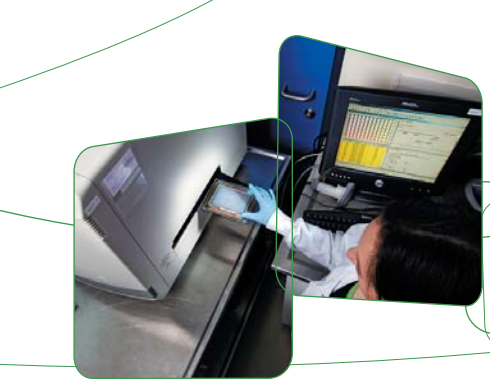
In collaboration with expert laboratories the JRC-IRMM has also investigated suitability of alternative markers for the banned veterinary drug nitrofurazone, validated an analytical method for the determination of semicarbazide in baby food and monitored its contents in bottled European baby food products.

Smoke flavourings

Food smoking is one of the oldest food technologies. About a hundred years ago, liquid smoke flavour prepared from a primary smoke condensate was invented. Motivated by the ease of application, the smoking of food has been progressively replaced by adding liquid smoke flavourings during the last three decades.

Smoke flavourings are produced on an industrial scale and are widely used to impart the smoky flavour to foods such as meat, fish or snacks. From a consumer protection point of view, using smoke flavourings has the advantage over direct smoking in that the content of toxic compounds such as polycyclic aromatic hydrocarbons (PAHs) can be measured and controlled more easily.

Today, European legislation (Regulation (EC) No 2065/2003) lays down a system of safety assessment conducted by EFSA, and authorisation by the Directorate-General for Health and Consumer Protection of the primary products, which are the raw materials for the production of smoke flavourings. The regulation on smoke flavourings sets maximum permitted concentrations in primary products for two PAHs and requires characterisation of those primary products.



The JRC-IRMM staff have reviewed the literature to provide an overview on the state-of-the-art of the analysis of the chemical composition of smoke flavourings. The former Scientific Committee on Food of the Health and Consumer Protection DG has indicated 15 PAHs relevant to human health. Therefore in 2005, JRC-IRMM developed and validated methods for the analysis of the 15 EU priority PAHs in liquid smoke flavourings by organising a collaborative trial. JRC-IRMM also assists a specific working group of EFSA in evaluating applications of the smoke flavouring producers for authorisation of their products in the EU.

Smoke flavourings are used in foods to impart a smoky flavour.

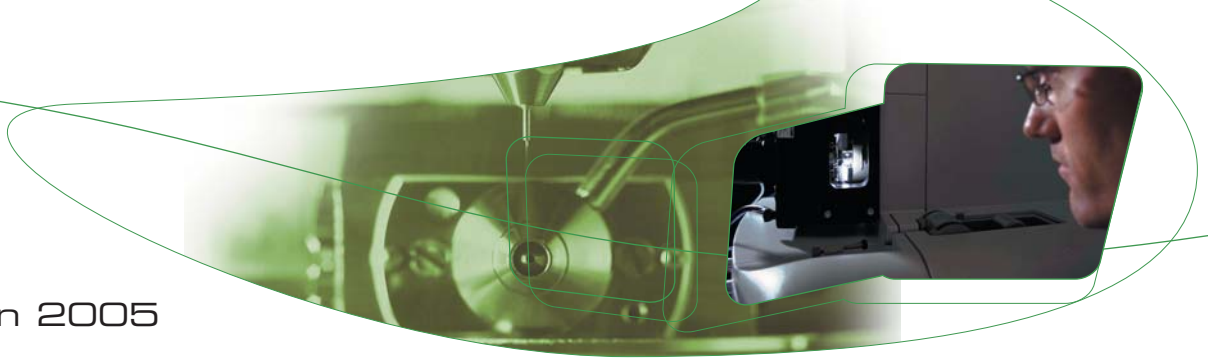
Testing food allergens

Food allergens, particularly peanut allergens, can cause severe symptoms in persons who are allergic and therefore European legislation sets rules for labelling of food products containing known allergens. But the present means to detect, identify and quantify them are still very limited. Developing better analytical methods and harmonising them across Europe and the world requires appropriate test materials.

The work of JRC-IRMM on food allergens aims to facilitate the implementation of the Labelling Directive 2003/89/EC by providing suitable methods of analysis for detection and quantification of allergens in food products. JRC-IRMM validates immunological methods for analysis of traces of peanut in biscuits and chocolate. These validated methods have been forwarded to the European Committee for Standardization (CEN) for preparing European standard methods. In addition, JRC-IRMM investigates the suitability of DNA based methods for peanut analysis.

Peanut test material kit was launched in 2005.

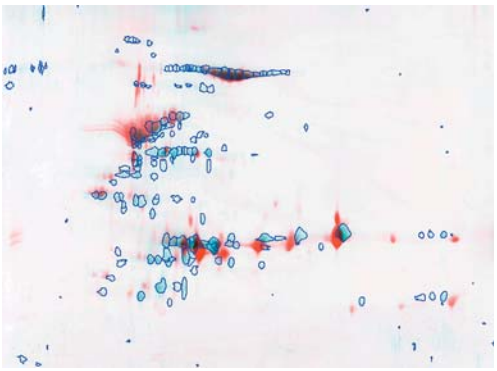
Standardisation of methods is further facilitated by JRC-IRMM by means of a peanut mixture material. With the production of this peanut test material kit, JRC-IRMM has introduced a new concept in its range of products. The JRC-IRMM set of peanut test materials contains four varieties of peanut powder pre-treated in five different ways. Instead of a reference material, the kit provides a reference matrix for analysts who wish to conduct research on a similar set



of samples. No parameter in this material is certified but a gravimetrically prepared mixture of the five different materials is also provided.

In 2005 JRC-IRMM has improved methods for peanut protein extraction so as to obtain better yields, and investigated the impact on food processing (e.g. heating) on the proteins responsible for peanut allergy.

Work on milk allergens has been initiated and scientific literature has been reviewed. The food allergen research within the JRC-IRMM is performed in close collaboration with a global network of food allergen experts.



2D gel electrophoresis of peanut proteins is used to detect allergens in foodstuffs.

Toxic vs. beneficial substances in biscuits

For almost three years now, researchers around the world have studied the formation of the compound acrylamide in food. Acrylamide is a carcinogenic substance and a natural contaminant in heat treated food, always formed when carbohydrate rich food is heat processed e.g. by the so called Maillard reaction. In parallel with the scientific research, the food industry has devoted considerable effort to reducing the production of acrylamide during processing.

To ensure good quality of acrylamide testing results in heat treated food, the JRC-IRMM organises proficiency tests and has started the validation by way of collaborative trial of two analytical methods for potato and bakery products. JRC-IRMM also hosts the European acrylamide monitoring database. To date there are more than 6800 data in the database.

Apart from the generation of the carcinogenic acrylamide during the Maillard reaction, substances with positive effects on human health, such as antioxidants, are also formed. JRC-IRMM has evaluated the correlation between the levels of acrylamide and the antioxidant activity in biscuit test samples prepared according to cooking book recipes under household conditions. A direct correlation was found between the concentration of acrylamide and the antioxidant activity. An increase in baking time increased the concentrations of both parameters together with the darkness of the biscuits. The use of fructose, which is frequently used in dietary biscuits, instead of sucrose i.e. the normal sugar, enhanced the formation of acrylamide and of compounds with antioxidant activity. This study demonstrates that suppressing the Maillard reaction to avoid formation of acrylamide can also lead to loss of substances considered to be beneficial.

JRC-IRMM hosts an European acrylamide monitoring database that contains information about acrylamide content in different foods.

TSE testing

Regulation (EC) No 999/2001 lays down rules for the prevention, control and eradication of certain transmissible spongiform encephalopathies (TSE). To implement and monitor compliance with the regulation, rapid tests for the diagnosis of bovine spongiform encephalopathy (BSE) i.e. the mad cow disease in cattle, and scrapie in sheep and goats are needed, and the means for monitoring the ban of animal by-products need to be developed, improved or validated.

TSE tests are approved by the European Commission following recommendation of EFSA. JRC-IRMM evaluates the performance of the rapid tests for EFSA. The evaluation of 20 different rapid BSE tests by JRC-IRMM in the past and in 2005 has so far led to a total of 12 tests approved under Regulation (EC) No 999/2001. In 2005, evaluations by JRC-IRMM led to the official approval of seven new rapid post mortem BSE tests for Regulation (EC) 260/2005. For the first time, also rapid tests for the diagnosis of scrapie will be receiving EC approval. Three of these tests were evaluated by JRC-IRMM in 2005 and six were assessed already in 2004. In 2005 JRC-IRMM has started the evaluation of a live animal BSE test.

But JRC-IRMM does more than evaluating tests. JRC-IRMM has developed well characterised reference materials for the quality control of both rapid BSE and scrapie tests. Three different materials of BSE positive homogenates are now available for rapid BSE tests. They are subject to strict production control and undergo regular stability monitoring. These materials cover the whole range of currently approved BSE tests and are widely used by Member States authorities, national reference laboratories and test producers.

The measures introduced in the EU have significantly reduced the number of BSE cases. The JRC-IRMM work contributes essentially to the implementation and the monitoring of a safe and reliable large scale BSE/TSE test monitoring programme in the European Union. The quality control materials offer for the first time a harmonised way to perform quality control of approved rapid BSE and scrapie tests throughout the European Union. Approving more tests has a direct effect on the consumer confidence and opens competition in the TSE diagnostics market. A considerable drop in costs for the more than 11 million tests carried out annually in the EU has already been observed in several Member States.

Banned feed additives in feed

The enforcement of the ban of some antibiotics and growth promoters that were used as feed additives in the past, requires reliable analytical methods. The JRC-IRMM has participated in the European project 'Screening and identification methods for official control of banned use of antibiotics and growth promoters in feedingstuffs (Simbagfeed)' aimed at the development and validation of appropriate methods, which can be applied for screening and confirmatory purposes regarding the presence of the banned substances in feedstuffs. In this project that now has come to its end, JRC-IRMM has been responsible for the organisation of the collaborative trials and the treatment of data.

During the project JRC-IRMM prepared a range of feed samples (cattle, pig, poultry and calf) containing the banned substances and distributed them along with the pure substances for method validation to partners within the project. Several hundreds of units of the samples were distributed to the 40 laboratories that participated in the trials for validation of methods for screening and/or for confirmative analysis.

In 2005 the JRC-IRMM completed the validation of four methods by inter-laboratory studies. The validation experiment included (1) a screening method for the antibiotics spiramycin, tylosin and virginiamycin by thin layer chromatography, (2) a post-screening method for the growth promoters carbadox and olaquindox by high performance liquid chromatography (HPLC) coupled to an

ultraviolet detector, and (3) two confirmatory methods based on HPLC coupled to a mass spectrometric detector for the above mentioned compounds and the antibiotics avoparcin and zinc-bacitracin. These methods are currently under review within CEN for standardisation purposes.

Organic food

Food traceability and organic food authenticity are important as consumers buying organic food should be able to do so with confidence. However, so far no method for authentication of organic food or for distinction from conventionally produced food has been proven suitable and validated.

Together with laboratories from public institutions and the food industry, farmer associations and consumer organisations, the JRC-IRMM designs field experiments and develops and validates suitable analytical

Among other methods JRC-IRMM investigates the use of so-called biocrystallisation as a technique for distinguishing organically grown crops from conventionally grown crops.

methods aiming to distinguish organically grown crops from the conventionally grown. A controlled experimental field has been designed with a Belgian organic farmer association, a technical centre and certified farmers. A farm comparison approach was selected to control parameters such as farm location, soil type, cultivar or physiological stage in crops.

In 2004 and 2005, one environment per crop was selected in the Walloon region of Belgium where one variety per crop was sown on the adjacent organic and conventional fields. For each field, soil samples were extracted and their physical, chemical and biological parameters were analysed together with the crops under investigation. In 2005 carrot, horse bean, potato and winter wheat samples were analysed in the JRC-IRMM laboratories for dry matter, total nitrogen, proteins, gene expression, trace elements, radionuclides, stable isotopes, phenolic acids (plant metabolites), antioxidant and anti-mutagenic activities. The validity of a so-called bio-crystallisation method is also investigated. The preliminary analysis of the data collected so far indicates several possible approaches for specific crops that can be pursued further.

This activity represents a follow up of the communication of the European Action Plan for Organic Food and Farming adopted by the European Commission in 2004. JRC-IRMM is also a partner in the European project TRACE –Tracing the Origin of Food.

Confectionary products

JRC-IRMM has developed the CoCal (Cocoa butter Calculation) toolbox to detect and quantify foreign fats in cocoa butter and chocolate. The CoCal toolbox contains validated methods, a calculation sheet for plain chocolate, and a certified cocoa butter reference material and helps to enforce the Chocolate Directive 2000/36/EC that allows addition of up to 5% of vegetable fats other than cocoa butter, so-called cocoa butter equivalents (CBEs), in chocolate. In 2005, the methods developed and validated by the JRC-IRMM have been accepted by the American Oil Chemists Society (AOCS) and the Internal Organisation for Standardisation (ISO) and will become international standardised methods. Now JRC-IRMM has collected and analysed a wide array of European milk fats by the same analytical approach to develop a similar toolbox for milk chocolate.

Another topic studied at JRC-IRMM is the analysis of sweeteners. The use of sweeteners is regulated by European directives and their amendments. The list of regulated substances contains authorised sweeteners with restricted concentration limits like sucralose, acesulfame K or aspartame, and those not authorised like tagatose, neotame or alitame. On request of the Technical Committee on Food (TC 275) of CEN, JRC-IRMM has started developing an approach using methods for the simultaneous analysis of both authorised and non-authorised sweeteners in beverages, dairy and confectionery products.

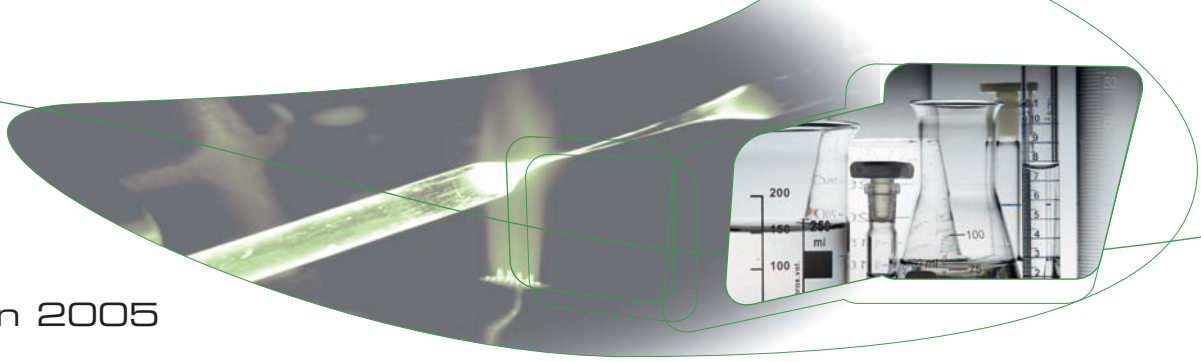
JRC-IRMM analyses sweeteners in confectionary products.

Focus on quality of testing results for life sciences research

Applications of life sciences are an integral part of our everyday life, even if we are unaware of that. Applications in areas such as food production, drug development or diagnostics are generally considered to have a substantial potential for improvements in the production and monitoring of advanced products and in public health.

Life sciences research is usually coupled with the analysis of complex compounds of biological origin such as DNA and proteins. But even today, measurement results often lack reliability and comparability. Without reliable and comparable data the enforcement of legislation can be hampered. In addition, interpretation and exploitation of data in a research and development environment is limited, and can slow down further technical development.

The CCQM Bioanalysis Working Group aims at the development of quality assurance tools and a fundamental understanding of measurement concepts related to molecular biology that can be applied for the various measurement and production platforms. In 2005 JRC-IRMM has participated in the development of a roadmap for this working group to tackle the various measurement issues in the life sciences area in a systematic way. JRC-IRMM has also participated in pilot studies in the life sciences area with national metrology institutes around the world. The pilot studies are organised to explore the state-of-the-art of understanding the techniques used and to identify the potential problems before a key comparison will be carried out. In 2005, the pilot studies covered genetic profiling by specialised techniques, the quantification of plasmid DNA and the influence of extraction methods applied on quantitative DNA analysis using a maize GMO as a model. JRC-IRMM co-ordinated the study on extraction methods, provided the necessary materials for it and evaluated the measurement results from the participants.



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Protecting the European environment

The European legislation obliges the Member States to monitor and assess the quality of water, soil and air. The monitoring data are the basis for decisions that can have significant consequences for the environment or the industry. Those data need to be reliable and comparable. Therefore, the JRC-IRMM provides testing laboratories with reference materials and proficiency testing schemes, and develops and validates methods of analysis.

JRC-IRMM activities in the field of environmental analysis are focussed in particular to supporting the implementation of the Water Framework Directive. For the last two years JRC-IRMM has also focussed on investigating isotopic techniques for identifying sources of pollution. The efficiency of environmental management can be limited by the lack of knowledge on the sources of contamination and their relative contribution. It is known that processes occurring in nature e.g. evaporation, condensation or oxidation affect the relative abundances of isotopes. This isotopic information can be used to identify and quantify pollution sources. This information can be used to assess the effectiveness of environmental management plans for instance.

Analysis of waters

The Water Framework Directive 2000/60/EC sets the objective to prevent deterioration in status of all Community waters, surface and ground waters, and coastal waters throughout the EU. It is to ensure achieving and maintaining their good status by 2015. Reliability and comparability of the monitoring data is a key issue for the directive. Therefore, the Member States have to develop and implement a quality assurance system to ensure that all monitoring results meet target levels of accuracy. The quality assurance procedures may include standardisation and validation of sampling and analytical methods, and use of reference materials and laboratory accreditation schemes.

An European project 'Screening methods for Water data InFormation in support of the Water Framework Directive' (SWIFT-WFD) addresses these topics. The SWIFT-WFD project aims to review the existing quality control tools related to the parameters and water matrices to be characterised in each river basin. It also aims to produce reference materials for screening methods. JRC-IRMM, as a partner in the project, has the task of providing proficiency testing materials for the European laboratories participating in the proficiency testing schemes organised by the project consortium. The test materials cover the analysis of trace elements, major components, polycyclic aromatic hydrocarbons (PAHs) and pesticides. They address specific measures against contamination of water by pollutants known as priority substances that are recognised to be hazardous for the environment or human health.

Sampling of spring water in Montreuil-sur-Epte (FR) for preparing test materials.

Tracing the source of nitrate in natural waters

The rules for monitoring nitrate in waters in the EU are set in the Nitrate Directive (Council Directive 91/676/EEC). The Member States monitor the nitrate content of surface waters and groundwater at selected measuring points. This way the extent of nitrate pollution in the waters from e.g. agricultural sources can be established. Despite all efforts, however, sometimes the origin of the source cannot be identified and eliminated.

The analytical techniques for determining the nitrate content in water are well established. The origin of nitrogen containing nutrients in surface and ground waters, however, cannot always be identified based on those methods only. Combining the conventional methods with the analysis of the nitrogen and oxygen isotopes in nitrate, improves the possibilities of discriminating between the different sources and mechanisms in the nitrogen cycle such as animal manure or fertiliser, or fixing nitrogen from the air.

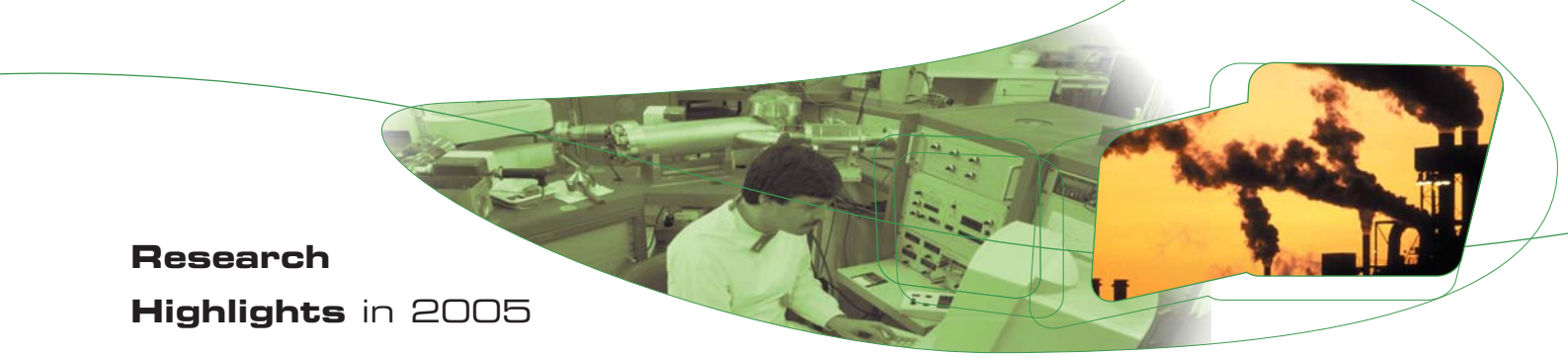
As yet, no reference method exists for the isotope analysis of ^{15}N and ^{18}O in nitrates. JRC-IRMM has now developed and validated methods that enable to measure the nitrogen and oxygen isotope ratios in the nitrate present in natural waters. This way it is possible to distinguish between nitrate originating from fertiliser or manure coming from chicken, cattle or pig farms. These methods will be applied to analyse waters from the Flanders region (BE) where nitrate constitutes an yet unresolved problem.

Source apportionment of particulate matter in air

The Council Directive 1999/30/EC aims to reduce emissions of particulate matter in air by setting limits for the emissions. Source apportionment i.e. differentiation and quantification of sources from diesel cars, power plants, industrial processes or incinerators for instance, could make environmental management more efficient. A method for source allocation of particulate matter in ambient air could be used for the assessment of the impact of the Directive and better implementation of the regulation. It would also support the European Environment and Health Strategy (Com (2003) 338).

Organic pollutants in soil, water and air can be analysed with a combination of gas chromatography and isotope ratio mass spectrometry.

Source apportionment of the particulate matter has been demonstrated to be possible by using a combination of elemental analysis and isotope ratio mass spectrometry. Additionally, compound specific isotope data on the PAHs absorbed on the particulate matter could be used to track the origin of the particulate matter. In 2005 JRC-IRMM started the testing of suitable methods for this kind of analysis. The work is linked to the Krakow project of the JRC Institute for Environment and Sustainability, in which a variety of analytical techniques are tested for their potential for source apportionment of aerosols.



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Nitrogen isotopes for environmental studies

Nitrous oxide (N_2O) is one of the greenhouse gases targeted by the Kyoto protocol. The predominant N_2O sources are known but the knowledge about individual sources is poor and lacks experimental proof. To better quantify the atmospheric nitrous oxide budget, many research groups are now investigating the possibility of measuring the stable isotopic composition of nitrogen and oxygen in N_2O.

Due to the lack of a reliable international measurement standard, however, the progress has come to a halt. Therefore, in 2005 JRC-IRMM has produced an isotopic reference gas that provides a possibility to link the measurement scales used by the research groups all over the world. JRC-IRMM is also studying the analysis of position of isotopes of oxygen and nitrogen in the N_2O molecule. The site preference could be used to differentiate processes of production and consumption of N_2O to eventually estimate the contributions of nitrification and denitrification processes to the total N_2O budget. As a result of this feasibility study the measurement techniques have been improved.

Nuclear safety and security

The nuclear activities of JRC aim to satisfy the research and development obligations of the Euratom Treaty. They also support the European Commission and Member States in the field of nuclear safeguards and non-proliferation, nuclear waste management, nuclear fuel cycle safety and radioactivity in the environment and radioprotection.

JRC-IRMM produces isotopic reference materials for quality assurance of measurements for nuclear safeguards, carries out research in the field of radionuclide metrology and investigates interaction of neutrons with matter.

Reference particles from UF_6

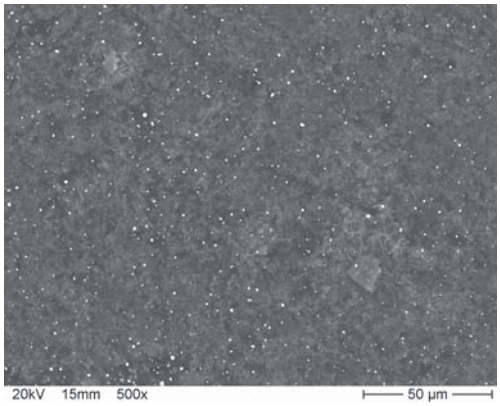
Environmental sampling is a powerful method used by safeguards inspectors of the International Atomic Energy Agency (IAEA) and the European Commission for the control of undeclared nuclear activities. Since its implementation in the early 1990's, environmental sampling has proven to be very sensitive and effective. It is based on collecting dust material by swiping surfaces in or around nuclear sites, including facilities for the enrichment of uranium. This dust will

Reaction chamber for production of uranium particles by hydrolysis of UF_6 gas.

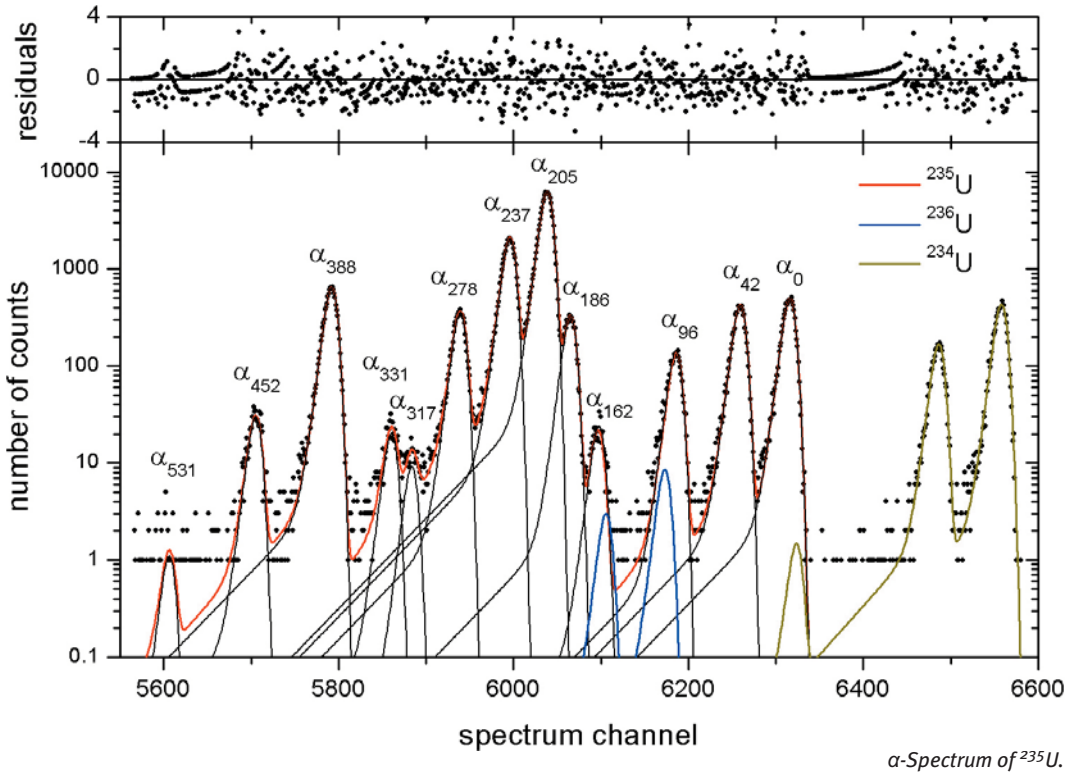
contain uranium particles with an isotopic composition characteristic for the process in the facility. In the case of enrichment facilities, these particles were formed from small leaks of UF_6 gas during manipulations of the connections to the plant.

The swipes with the sampled dust are then sent to a number of specialised laboratories for further analysis. Isotopic composition of the individual micrometer-sized particles is analysed e.g. using secondary ion mass spectrometry (SIMS). The measured uranium isotope ratio should match the declared range of values of the facility. Since the results of these measurements may have political consequences, they are subject to careful quality control. However, despite the recognized need for reference materials in the form of uranium containing particles, they are lacking at present.

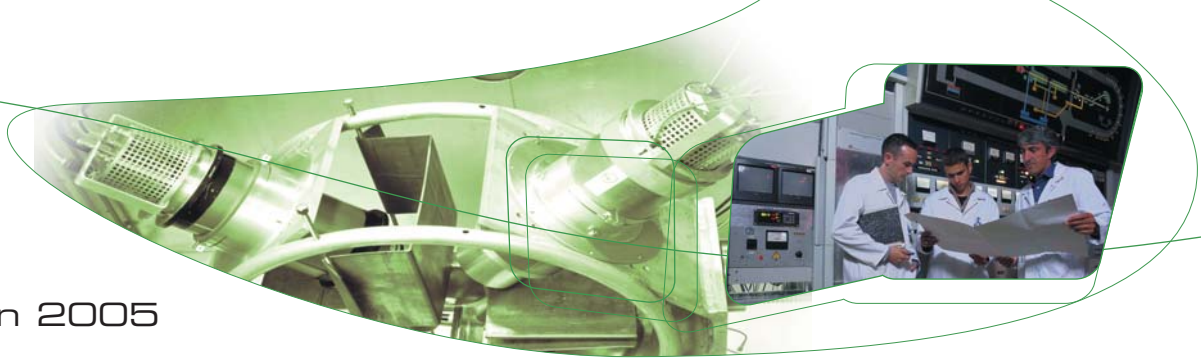
A new method is under development at JRC-IRMM for producing reference particles from certified UF_6 gas. This method allows realistic uranium containing particles with certified abundances to be prepared. Using the aerosol deposition chamber, UF_6 leaks are simulated on a small scale: a small amount of UF_6 is brought into a reaction vessel and hydrolysed in the gas phase. Particles are subsequently formed by aggregation. The particles are characterised by secondary electron microscopy and SIMS at the University of Antwerp and the JRC Institute for Transuranium Elements (DE).



Secondary electron micrograph of uranium particles (bright spots) on a collection plate.



α-Spectrum of ^{235}U.



Research Highlights in 2005

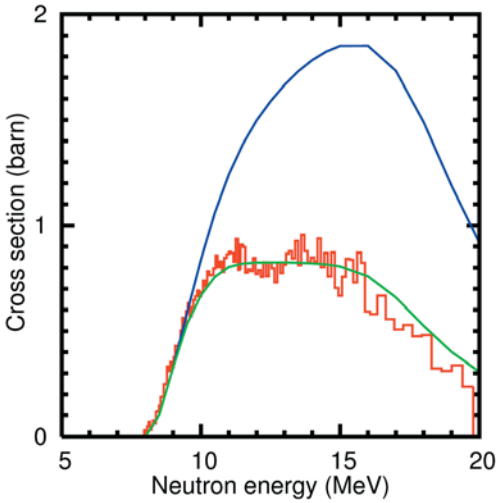
Nuclear decay data for ^{235}U

^{235}U is one of the most important nuclides in the nuclear fuel cycle. Reliable nuclear data for this nuclide are of great importance for nuclear safeguards, environmental monitoring and dating studies. A Coordinated Research Project of the IAEA on the measurement of transactinide isotope nuclear data has established the objective of measuring the α-emission probabilities of ^{235}U with an accuracy of 3% for the main lines. Until now published data did not meet these requirements.

Therefore, JRC-IRMM has prepared very thin uranium samples, yet possessing the required activity and sent them to the participants of a EUROMET cooperation project. The α-emission probabilities of ^{235}U were then measured in all laboratories using high-resolution α-spectrometry with silicon detectors. The use of improved measurement techniques and numerical analysis of spectra have resulted in a new set of decay data with improved uncertainties.

Improved neutron data support transmuter design

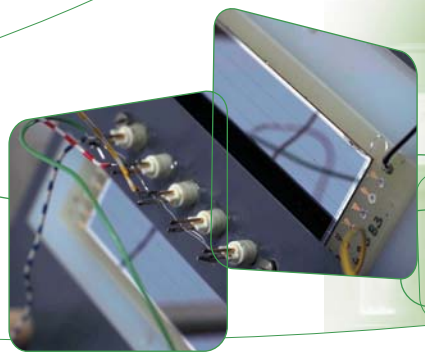
Minimising nuclear waste by transmutation can be done either by using accelerator-driven systems (ADS) or fast reactors. The goal is to transmute the long-lived radiotoxic fission products and minor actinides such as Np, Am, and Cm isotopes into short-lived or stable ones. For this purpose, very high fluxes of fast neutrons from so-called fast reactors are needed or neutrons from the spallation process in dedicated accelerator-driven systems can be used.



Recent results of the (n,2nγ) cross-sections on ^{208}Pb measured at the GELINA facility of JRC-IRMM in comparison with predictions from the TALYS code. Blue line: default TALYS calculation; green line: without intermediate population of the isomeric state in ^{207}Pb.

The selection of the most effective scheme and the design of appropriate facilities requires accurate knowledge of the underlying nuclear reactions. For instance, 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. Modelling of ADS would then result in unnecessarily large safety margins in the first demonstration facilities.

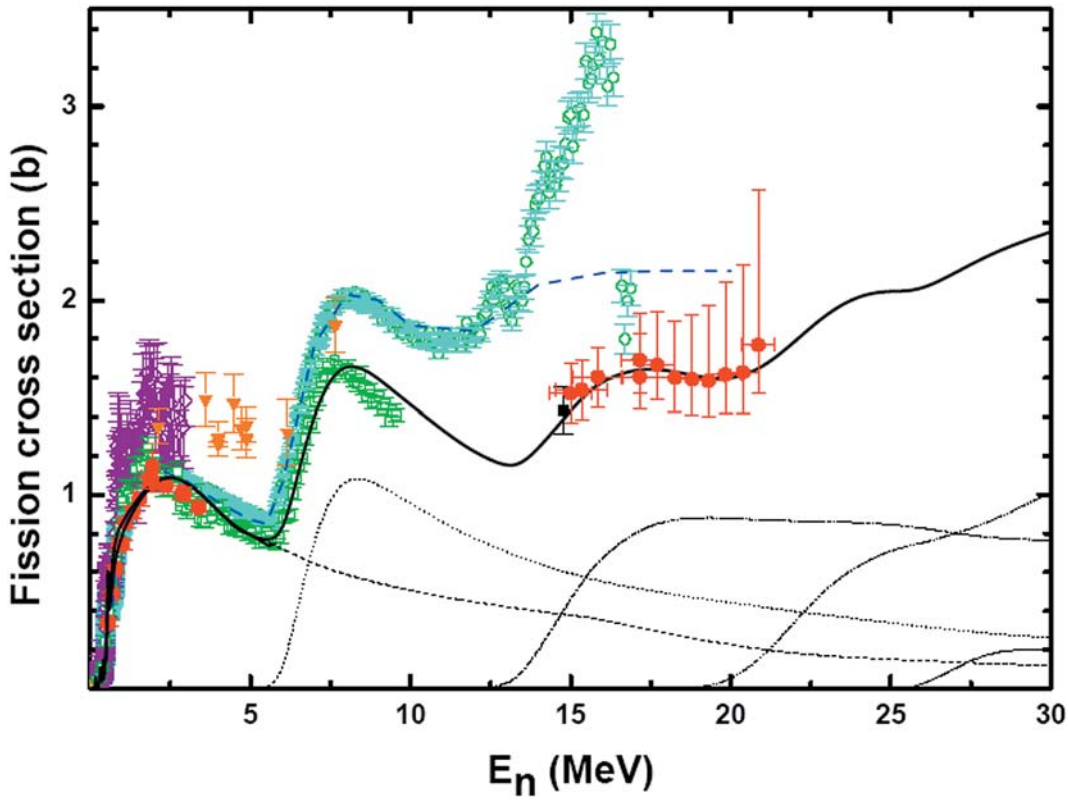
JRC-IRMM has now completed data analysis on ^{209}Bi for inelastic neutron scattering and (n,2n) cross sections with very high energy resolution of up to 18 MeV. Measurements have been completed also for ^{206}Pb and ^{208}Pb, complementing the measurements of ^{207}Pb and ^{209}Bi cross sections in 2004. More measurements of (n,2n) and (n,3n) cross sections are being carried out and a new setup has been developed for the measurement of the isomer ratio in radiative neutron capture on bismuth.



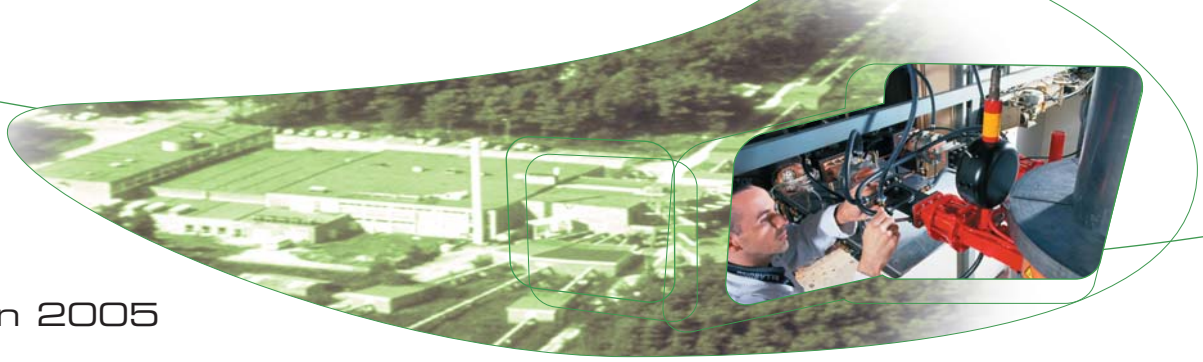
Neutron data for the thorium fuel cycle

If a Th-U fuel cycle was used for nuclear power production the amount of plutonium and minor actinides like neptunium and curium in the waste would be negligible. The Th-U cycle is hence very advantageous where radiotoxicity and waste handling are concerned. To develop this concept some major nuclear reactions need to be known more accurately.

The IAEA has organised a Co-ordinated Research Project to produce an improved evaluated nuclear data library for this cycle. The JRC-IRMM is responsible for the evaluation of the data in the unresolved region for ^{232}Th, which is the most relevant isotope in the cycle. In addition, 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. 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, yielding very low values and a challenge for theoretical modelling. Prompt fission neutron spectra and multiplicities of ^{233}Pa were also calculated using modelling based on the recent experimental results obtained at JRC-IRMM.



Recent measurements of the neutron induced fission cross section on ^{231}Pa at JRC-IRMM (red) in comparison with previous measurements and values from the JENDL data library (blue dashed curve). The black dotted curves indicate the contributions of higher-chance fission (n,xnf) to the total cross section.



Research Highlights in 2005

Better 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 pressurized water reactors (EPR) and future Generation-IV systems implies an increased demand for accuracy of the most important fission products.

In 2005, JRC-IRMM was engaged in a collaboration with the Oak Ridge National Laboratory of the US Department of Energy to improve the accuracy of nuclear data for fission products to the level that is required for criticality estimates of spent nuclear fuel. A measurement campaign was conducted for ^{55}Mn, and the study of ^{103}Rh and ^{133}Cs was initiated.

^{nat}Cd is a neutron absorber found both in nuclear reactors to control the power level and is used in many nuclear measurements to eliminate spurious thermal neutrons. With the increasing demands on nuclear data accuracy, the cross section of this absorber is being revised. The IAEA is carrying out a new set of total and capture cross section determinations with state-of-the-art techniques at the JRC-IRMM GELINA facility, supported by the NUDAME trans-national access scheme.

^{16}O(n,α)^{13}C reaction cross section for criticality predictions

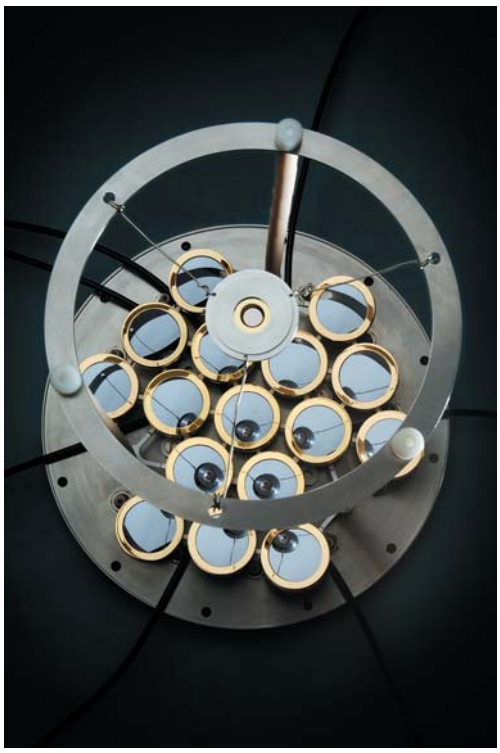
To improve criticality predictions of thermal and fast nuclear reactors, as well as the calculation of helium production in those reactors, a better knowledge of the ^{16}O(n,α) reaction in the energy range from 2.5 to 10 MeV is needed. The current uncertainty of the ^{16}O(n,α) cross section is >30% and an accuracy of better than 5% is required to essentially improve. In 2005, this triggered a data request within the High Priority Request List of the Nuclear Energy Agency of the Organisation for Economic Cooperation and Development (OECD-NEA).

In 2005, the feasibility study on measuring the ^{16}O(n,α) cross section was performed at the JRC-IRMM Van de Graaff accelerator. As a result, a 1-dimensional time-projection chamber will be used to analyse the ^{16}O(n,α) reaction products. The full power of the technique will be exploited using a 3-dimensional application, which is now being developed at JRC-IRMM.

New spectrometers at the Van de Graaff laboratory

For the measurement of the decay of metastable nuclear states with half-lives well below 1 s a new isomer spectrometer has been installed at the Van de Graaff accelerator. This instrument provides pulsed neutron beams with variable pulse width and repetition frequency that can be adapted to the particular experimental needs. The investigation of shape isomer decay in actinide nuclei will provide improved input data for nuclear reaction modelling.

To improve the quality of fission fragment detection and to attain a better understanding of prompt and delayed neutron emission, JRC-IRMM is constructing a new high resolution fission fragment two-arm time-of-flight spectrometer in its Van de Graaff laboratory. This detector will allow, for the first time, to obtain precise prompt neutron evaporation data as a function of the fission fragment properties. By detecting the masses and kinetic energies of the fission fragments with high accuracy, the precursors for subsequent delayed neutron emission can be identified.



High resolution fission fragment and delayed neutron precursor spectrometer in the Van de Graaff laboratory.

Low scattering setup for activation studies at the JRC-IRMM Van de Graaff laboratory.

Research Highlights in 2005

JRC-IRMM measurements incorporated in new evaluations and international databases in 2005

Cross section	Database	Field of application
high precision transmission and capture measurements of ^{103}Rh	JEFF-3.1 library	safety of nuclear reactors
activation cross sections for Mo, Zr and Ni	JEFF-3.1 library OECD-NEA EXFOR database	accelerator driven systems, fusion research community
^{10}B(n,α) branching ratios in the energy range from 1 keV to 2 MeV	IAEA project to be incorporated in the ENDF B-VII	standards, basis for most of the measurements
transmission and capture cross sections of ^{99}Tc	JEFF-3.1 library	transmutation of nuclear waste
total and capture cross sections on the long-lived fission product ^{129}I and stable ^{127}I	JEFF-3.1 library OECD-NEA EXFOR database	transmutation of nuclear waste
capture cross section of ^{nat}Fe	JEFF-3.1 library Nuclear Data Section of IAEA	safety of nuclear reactors
inelastic scattering and (n,2n) cross sections of ^{52}Cr	OECD-NEA EXFOR database	transmutation of nuclear waste
average capture cross sections of ^{232}Th	IAEA Th/U CRP EXFOR database	minimising nuclear waste
fission cross sections of ^{231}Pa	IAEA Th/U CRP OECD-NEA EXFOR database	minimising nuclear waste
total cross sections of ^{61}Ni	OECD-NEA EXFOR database	safety of nuclear reactors

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

Institute in figures

Human resources

At the end of the year 2005 the total number of staff at JRC-IRMM was 316 persons, of which 169 are Commission officials and the others are visiting scientists, PhD or post-doctoral fellows, or hold a temporary contract. JRC-IRMM has also been able to employ a considerable number of scientists from the new Member States and the Candidate Countries on short term contracts.

Since 1995 the number of women working at the Institute has increased significantly. In 2005 women made up 41% of all staff while 55% of the visiting staff and 31% of the core staff were women, including two unit heads out of eight.

Core Staff (end-of-year situation)

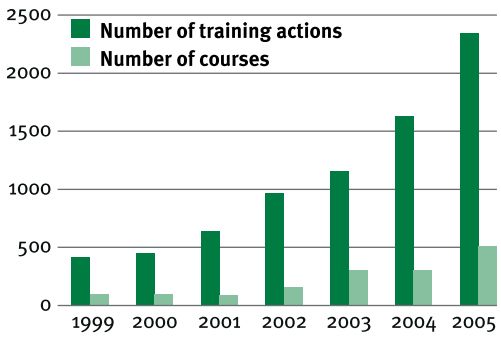
	2004			2005		
	M	F	Total	M	F	Total
OFFICIALS	123	40	163	122	47	169
TEMPORARY AGENTS ON 5-YEAR RENEWABLE CONTRACTS	0	3	3	1	3	4
TEMPORARY AGENTS ON NON-RENEWABLE CONTRACTS	10	8	18	20	14	34
TOTAL	133	51	184	143	64	207

Temporary Staff

	2004			2005		
	M	F	Total	M	F	Total
POST GRADUATE FELLOWS	10	6	16	10	7	17
POST DOCTORAL FELLOWS	5	6	11	17	18	35
SENIOR SCIENTISTS	3	2	5	9	5	14
VISITING SCIENTISTS	3	7	10	0	2	2
SECONDED NATIONAL EXPERTS	4	5	9	12	4	16
AUXILIARY STAFF	30	35	65	11	18	29
CONTRACTUAL AGENTS	0	1	1	7	23	30
TRAINEES	0	2	2	2	1	3
TOTAL	55	64	119	68	78	146

In the light of the reform of its services, the European Commission has highlighted the importance of training in developing the competences of its staff, and its importance as a central element of career development.

In 2005 JRC-IRMM continued its efforts and staff spent more than 3300 man-days in training with an average of 10 per person while the JRC average was 7. Training was mainly given in scientific and laboratory techniques, informatics, safety and security issues, financial regulations, soft skills, languages and implementing ISO standards.



Institute
in figures

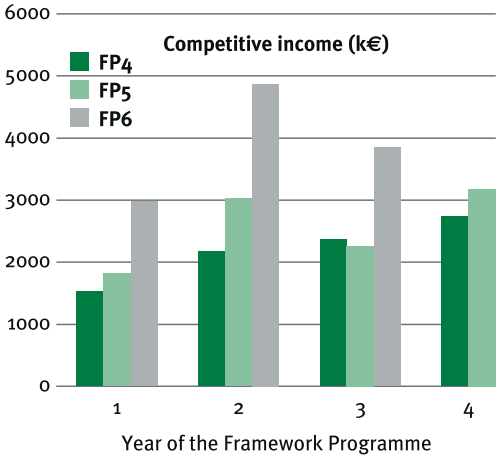
Budget

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, means of execution (technical and administrative support) and operational appropriations (direct scientific activities). 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.

About 9% of the Institute's budget is competitive income from the distribution of reference materials, from participation in projects of the EU Framework Programmes or work performed for customers mainly from other Directorates-General. The competitive income has increased throughout the three Framework Programmes (see figure on competitive income through FP4-FP6) and has reached a sustainable level. The income from reference material distribution shows a steady increase and has again reached an all-time record. Success rate in filing for competitive contracts was 56%.

Competitive Activities (k€)

2004	2005
COMPETITIVE CONTRACTS	
3088	1691
WORK FOR THIRD PARTIES	
190	335
REFERENCE MATERIAL DISTRIBUTION	
1587	1821
TOTAL	
4865	3847



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 are always published as an EUR report.

The JRC-IRMM staff has participated in conferences and meetings presenting over 170 scientific posters or oral presentations.

Publications

2004	2005
EUR REPORTS	
30	29
ARTICLES IN PEER REVIEWED PERIODICALS AND MONOGRAPHS	
89	63
CONFERENCE PROCEEDINGS	
5	48
SPECIAL PUBLICATIONS AND TECHNICAL NOTES	
6	6
TOTAL	
130	146

Events

In 2005 JRC-IRMM opened its doors to the public for the first time. On Sunday 2nd October, the 'Open bedrijvendag' in Flanders, 400 people visited JRC-IRMM. The visitors were welcomed by JRC-IRMM staff in six laboratories. They could attend demonstrations and follow presentations on topics studied at JRC-IRMM that are of concern to the European citizen. Answers to questions like why it is important to detect peanut allergens in biscuits or how JRC-IRMM helps in solving the nuclear waste problems were given. The visitors could test themselves if a sample biscuit contains traces of peanuts, were introduced to the ingredients and process of making chocolate, and learned how smoked ham gets its flavour. The purpose and importance of making reference materials and validating methods of analysis were explained, and the radioactivity in the dial of grandfather's watch was measured.

Operation of an accelerator was explained to the visitors during the open day at JRC-IRMM.

On the 20th of October 2005 the Science and Research Commissioner Janez Potočnik opened one of the world's largest storage buildings for reference materials at JRC-IRMM. In his speech he emphasised the importance of standardisation and the quality of measurement results. During a tour of the new storage building Mr Potočnik participated dispatching a mussel tissue reference material to an analytical laboratory that measures the quality of the environment. The same day JRC-IRMM organised a conference 'Confidence in Measurements' where examples of the importance of reference materials and measurements were presented. The presentations dealt with reliable bioanalysis, environmental measurement challenges, food and feed safety and quality and the issue of nuclear energy and environmental protection. The conference ended with a keynote lecture by the 1997 Nobel laureate Stanley Prusiner who presented studies on prions, the cause of bovine spongiform encephalopathy (BSE) of cattle, Creutzfeldt-Jakob disease (CJD) of humans, scrapie of sheep, and chronic wasting disease (CWD) of deer and elk.

The new storage building was opened by Commissioner Janez Potočnik.

In addition to the two major events, JRC-IRMM welcomed 300 visitors who attended meetings, workshops or guided laboratory visits. Altogether, 15 workshops were organised at the Institute and 37 seminar lectures were given during the year, promoting exchange of information and best practises. Among those were the meeting of the Working Group I of the Joint Committee of Traceability in Laboratory Medicine (JCTLM) and the meeting of the JRC fire brigades. The fellows at JRC-IRMM presented their work during a topical day and exploratory research projects were kicked off in an institute seminar.

Organigramme

status December 2005

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