Activities at Forschungszentrum Jülich in Safeguards Analytical Techniques and Measurements

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Abstract. The application of safeguards by the IAEA involves analytical measurements of samples taken during inspections. The development and advancement of analytical techniques with support from the Member States contributes to strengthened and more efficient verification of compliance with non-proliferation obligations. Since recently, a cooperation agreement was established between Forschungszentrum Jülich and the IAEA in the field of analytical services. The current working areas of the Forschungszentrum with IAEA SGAS are: (i) Production of synthetic micro-particles as calibration standard and reference material for particle analysis, (ii) qualification of the Forschungszentrum Jülich as a member of the IAEA network of analytical laboratories for safeguards (NWAL), and (iii) analysis of impurities in nuclear material samples.
With respect to the synthesis of particles, a dedicated setup for the production of uranium particles is being developed, which addresses the urgent need for material tailored for its use in quality assurance and quality control measures for particle analysis of environmental swipe samples. Furthermore, Forschungszentrum Jülich was nominated as a candidate laboratory for membership in the NWAL network. To this end, analytical capabilities at Forschungszentrum Jülich have been joined to form an analytical service within a dedicated quality management system. Another activity is the establishment of analytical techniques for impurity analysis of uranium-oxide, mainly focusing on inductively coupled mass spectrometry. This contribution will present the activities at Forschungszentrum Jülich in the area of analytical measurements and techniques for nuclear verification.

Keywords: Safeguards, Destructive Analysis, Environmental Sampling

1. R & D for International Safeguards at Forschungszentrum Jülich

In the development of methods and techniques for its safeguards mission, the IAEA relies on support from member states. In the analytical regime support may be provided in form of in-kind donations, analytical services or through consultancy and delegation of experts. The strengthening measures for Safeguards have led to an increasing degree of sophistication of analytical techniques and procedures used nowadays, which are not only applied for the purpose of verifying declared nuclear material flows and inventories, but also for acquisition of information, e.g. from environmental samples. The scope extension of analytical measurements as part of strengthened safeguards leads to a plurality of techniques and methods and an increased complexity. Research institutions can provide support in the development and adaptation of techniques towards application in Safeguards.

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As a member of the Helmholtz-Association, Germany's largest scientific research organisation , Forschungszentrum Jülich represents one of the major research facilities and works "towards comprehensive solutions for the grand challenges facing society in the future in the three fields of energy and environment, brain research and information technology, thus laying the foundation for future key technologies" [1]. Forschungszentrum Jülich contributes to research topics in the area of nuclear safety research which is part of the national energy research programme. The research topics covered by the subdivision *IEK-6: Nuclear Waste Management and Reactor Safety* of the *Institute for Energy and Climate Research* deal with material science aspects of nuclear waste management and applied research topics, including International Safeguards. *IEK-6* is responsible for the scientific coordination of the German Support Programme to the IAEA on behalf of the Federal Ministry of Economic Affairs and Energy.

The IAEA performs the analysis of safeguards samples 'in-house' via the Safeguards Analytical Service (SGAS), but also relies on the Network of Analytical Laboratories (NWAL), i.e. laboratories that have qualified to conduct analysis tasks for the Agency. The German Support Programme to the IAEA contributes to the development and advancement of safeguards and has become increasingly engaged in providing support in the area of analytical measurements for safeguards. In a joint initiative, three departments of Forschungszentrum Jülich, *IEK-6* (Institute for Nuclear Waste Management and Reactor Safety), *S* (Department of Safety and Radiation Protection), *ZEA-3* (Central Institute for Analytics), joined their analytical capabilities to provide support to IAEA safeguards analytical services. In mutual visits between representatives from Forschungszentrum Jülich and the IAEA areas of collaboration could be identified.

The three organizational units offer their combined capabilities (see overview in *FIG. 1*), where *IEK-6* brings in expertise in chemistry and material science involving actinide-elements. This expertise is complemented by a series of analytical techniques, in particular micro-analytical methods like scanning electron microscopy, focussed ion-beam but also Raman-spectroscopy and x-ray diffraction. Furthermore, *IEK-6* is licensed to handle bulk amounts of uranium and small amounts of plutonium. The department of Safety and Radiation Protection *S* is an accredited laboratory for radiochemical analyses according to ISO/IEC 17025:2005 one area of competency being ultra-trace analysis of U/Pu isotopes. The Central Institute for Analytics (*ZEA-3*) is a central department offering a wide range of analytical techniques in support of all research institutes within Forschungszentrum Jülich, but also to external customers. *ZEA-3* provides analysis of non-active samples, i.e. with radioactivity levels below the exemption limit.

The consortium of the three units *IEK-6*, *S* and *ZEA-3* has agreed to work towards a harmonized procedure regulation for providing the joint services to the IAEA-SGAS. The department *S* has a flexible accreditation according to ISO/EC 17025:2005 and may subcontract the services of *IEK-6* and *ZEA-3*. This way, analytical services can be provided within a cohesive quality management system.

Furthermore, a cooperation agreement was signed between Forschungszentrum Jülich (lead taken by *IEK-6*) and IAEA-SGAS as a framework for cooperation in the area of analytical measurements for safeguards.

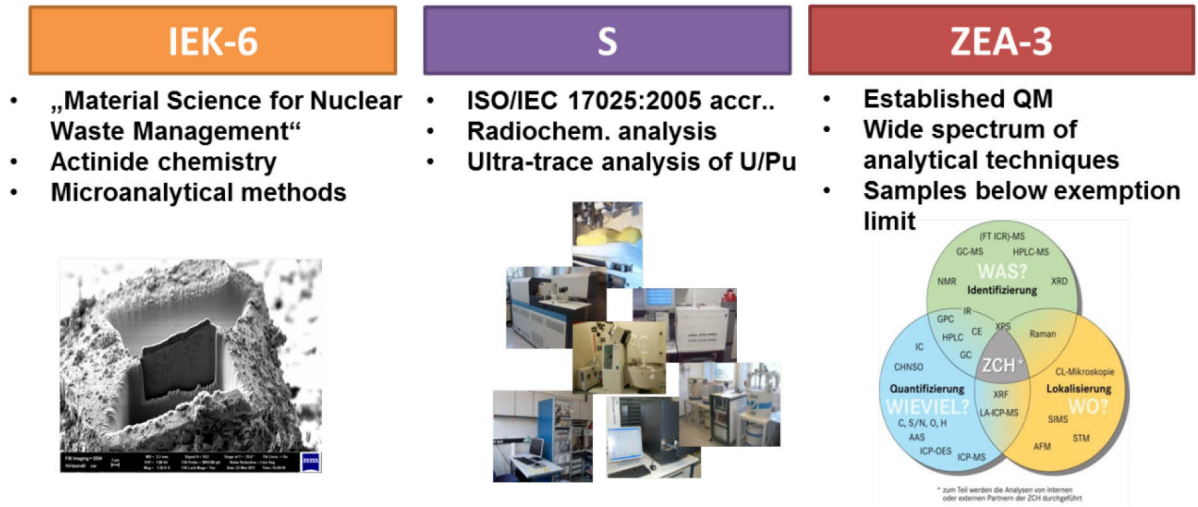

FIG. 1 Capabilities of the three departments at Forschungszentrum Jülich active in analytical measurements and techniques for safeguards.

2. Activities at Forschungszentrum Jülich

As a result of the arrangements described above, following activities of the Forschungszentrum Jülich in the area of analytical measurements for safeguards have emerged:

- Analysis of impurities in nuclear material samples,
- Production of micro-particles as reference material,
- Participation in the Network of Analytical Laboratories (NWAL).

These activities are outlined in further detail in the following sections.

2.1 Analysis of Impurities in Nuclear Material Samples

The aforementioned three organizational units established a process to address the analysis of nuclear material samples. In general, samples are dissolved and diluted and, if required, spiked at the radiochemistry laboratory of *IEK-6*. The diluted samples can be transferred to further analysis to the laboratories of the other departments for further analysis. For quantitative determination of uranium and impurity contents, ICP-MS is used for mass-spectrometry of samples taking advantage of stringent quality control routines established in the respective laboratories of *S* and *ZEA-3*.

The analysis of nuclear material samples is formalized in a procedure description describing responsibilities and the work flow between the three involved departments. In principle, the procedure may be included under the quality management system of the ISO/IEC 17025:2005 accredited laboratory of the Department for Safety and Radiation Protection *S*.

Currently, the three laboratories are focussing on of analysis of impurities in uranium paying specific attention to the evaluation of effects of sample preparation at *IEK-6*.

2.2. Production of Micro-Particles as Reference Material

In analytical measurements reference materials are needed for calibration of instruments, validation of methods and for quality control. In particular since the introduction of highly

sensitive Large Geometry SIMS analysis and the routine analysis of the uranium minor

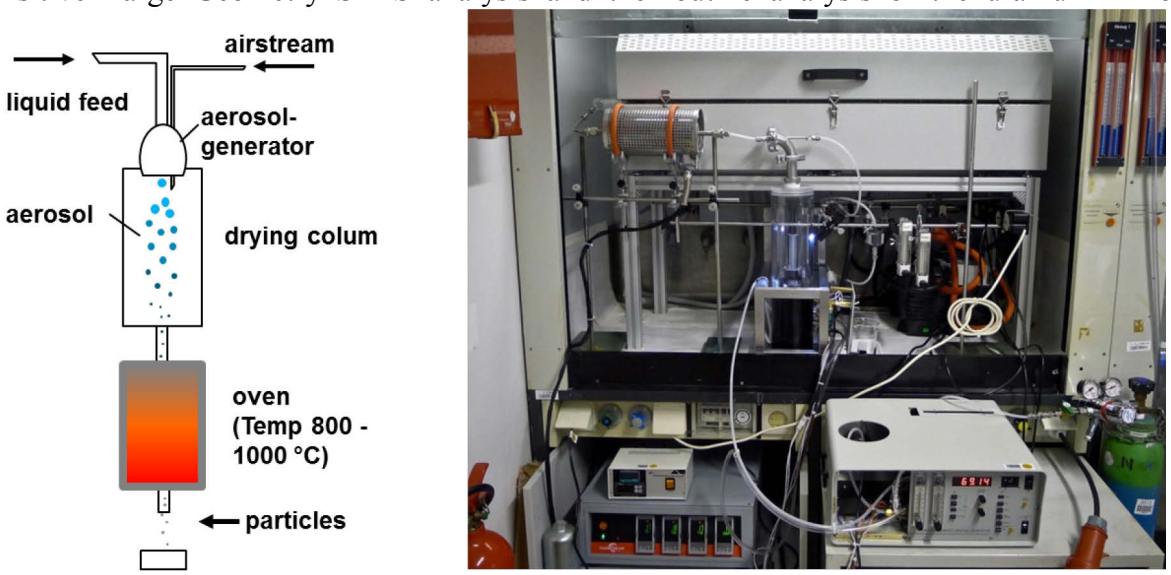

FIG. 2. Left: Schematic view of the particle production process for generation of reference particles. Right: Photograph of the particle production setup at Forschungszentrum Jülich.

isotopes, there is a need for customized reference material for particle analysis of environmental samples.

In the past, several approaches were made for production of particles to be used as a reference standard in particle analysis. Ideally, all particles would be uniform in the characteristics relevant to the analytical application. Generally this means that the particles should be identical in physical and chemical properties. In a joint project between the Forschungszentrum Jülich and the IAEA, an approach to particle production was chosen which allows the synthesis of uranium particles with a determined amount per particle and isotopic composition [2]. This approach is based on the generation of an aerosol from a liquid feed composed of uranium solution diluted in water-ethanol mixture. The aerosol is generated by a vibrating orifice aerosol generator which under certain operating conditions creates mono-disperse droplets, i.e. the droplets all have a uniform diameter. After evaporation, the droplets form a solid uranium-compound from the non-volatile component of the aerosol. The particles are transported via a carrier air-stream to a heating zone, where particles are heated to a temperature of up to 1000 °C and are therefore oxidized. This way, particles are sufficiently stabilized and can be collected on a suitable carrier (see *FIG. 2* and [3]).

The principle allows determination of the amount of uranium amount per particle by adjusting the U-concentration of the feed accordingly. The isotopic composition of the particles is determined by that of the feed solution. The chemical form of the particles depends on various parameters of the production process, e.g. temperature and dwell times of particles in the various stages of the process. The production setup (*FIG. 2*) was installed at *IEK-6* in collaborative effort with the IAEA and with support by the German Support Programme. First production of U particles started in 2014 where particles with 1 µm diameter have been successfully produced.

The characterization of particles is performed at *IEK-6* using particle search routines of a scanning electron microscope in order to characterize size, morphology and elemental composition of the produced particles. These results are needed to determine particle characteristics of produced particles as a quality check of the production run. If particles are

to be used as reference materials, particle characteristics of a subsample will have to be determined using "destructive methods" in order to gain additional information on morphology (e.g. porosity), chemical composition and crystal structure as well as quantification of the uranium amount per particle and the isotope content. To this end, Forschungszentrum Jülich is cooperating with EC Joint Research Centre – Institute for Reference Materials and Measurements on characterization of particles for determination of uranium content and isotopic composition (see e.g. [4]). Besides particle production, an area of research currently pursued at Forschungszentrum Jülich is the preparation of particles in a suitable form and investigation of particle stability.

2.3. Qualification as Member of NWAL

Forschungszentrum Jülich was nominated as a candidate for membership in NWAL early 2013, with the initial scope of nuclear material analysis. So far the efforts at Forschungszentrum Jülich focussed on quality management and qualification of analytical methods for analysis of safeguards samples. Due to a recent shift of priorities, technical discussions are ongoing on the involvement of Forschungszentrum Jülich as a laboratory for provision of particle reference materials. This would require a fully controlled production process and specified user requirements on particle characteristics.

3. Summary

In the past few years, Forschungszentrum Jülich has become engaged in the area of analytical techniques in the domain of International Safeguards. Analytical measurements and techniques for safeguards applications benefit from involvement of scientific research institutions. In particular with the high degree of sophistication of methods and instrumentation used in laboratories for safeguards, research and development of techniques and methods provides valuable support to the advancement and improvement of safeguards.

4. Acknowledgements

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