

## **JOINT SAMPLE ANALYSIS ON SELECTED URANIUM ORE CONCENTRATES AND NUCLEAR FORENSICS LIBRARY EXERCISE**

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### **Abstract**

The IAEA's recent publication on Development of a National Nuclear Forensics Library: A System for the Identification of Nuclear or Other Radioactive Material out of Regulatory Control, reemphasizes the rationale for the development of a national nuclear forensics library (NNFL) and addresses how a country may use such a national system in investigations of nuclear and other radioactive material out of regulatory control. According to this publication, it is important for a country to determine whether a nuclear forensics sample is consistent with its domestic nuclear material holdings. As a system for the identification of nuclear or other radioactive material, a national nuclear forensics library, can facilitate interpretation of findings and assist in this determination.

Lawrence Livermore National Laboratory (LLNL), through the U.S. Department of Energy's Office of Nuclear Smuggling Detection and Deterrence (NSDD), has partnered with the Republic of Kazakhstan's Institute of Nuclear Physics (INP), the Japan Atomic Energy Agency (JAEA), and the Centre for Energy Research (MTA EK) in Hungary on a joint sample analysis involving a set of uranium ore concentrate (UOC) samples. The sample set contained five UOC powder samples of known origin and a sixth UOC sample of unknown origin (blind sample). The objective of the joint sample analysis exercise was to characterize the UOC samples according to a well-developed analytical plan and use the measured material characteristics to populate a nuclear forensics library. This library is then used to establish potential links between the blind sample and the samples of known origin. The four participating laboratories compared data and analysis methods, and shared best practices on the implementation of an NNFL.

As the largest producer of uranium in the world, Kazakhstan has a targeted interest in understanding the measurable characteristics associated with the UOC it produces. The planned Kazakhstan NNFL will include data resources and expertise on

the wide range of nuclear and radioactive materials present in Kazakhstan. UOC is signature-rich, thus making it a good material to target for inclusion in an NNFL. The first data resource for the Kazakhstan NNFL will therefore be UOC.

## 1. INTRODUCTION

Over the last few decades, nuclear forensics has become an essential component in the fight against illicit trafficking of nuclear and other radioactive materials. Nuclear forensic analysis can provide information to law enforcement agencies regarding the origin, process history and suspected use of the interdicted nuclear material. Typically employed alongside “traditional” forensics (e.g. fingerprints, crime scene analysis, etc.), nuclear forensic analysis involves the measurement of the physical, chemical, elemental, and isotopic characteristics of nuclear material in order to answer questions from law enforcement and nuclear security officials [1-2]. Nuclear forensics has been prominently featured in the work plans and communiqués from all four Nuclear Security Summits. The Summits recognized that effective nuclear security requires international cooperation, i.e. the “no country is an island” approach in nuclear security. By encouraging all countries to develop and maintain a minimum set of nuclear forensics capabilities, the international community can ensure that every country has the capability to identify nuclear material outside of regulatory control and support the prosecution of traffickers.

In this context, and under the auspices of the 2006 Joint Communiqué on counter trafficking of nuclear and radioactive material, DOE/NNSA’s Nuclear Smuggling Detection and Deterrence Office (NSDD) has partnered with the Kazakhstan’s Institute of Nuclear Physics (INP) to advance the technical and analytical skills relevant to nuclear forensic analyses. INP and DOE/NNSA have a long-standing partnership in nuclear forensics that formed in the early 2000s. The joint sample analysis described in this work is a follow-on activity to an earlier joint sample analysis on a UOC reference material (CUP-2, 2016), a DOE/NNSA workshop on the development of a National Nuclear Forensics Library (NNFL) (Almaty, September 2018), as well as in-depth and hands-on training on a wide range of analytical techniques, including gamma spectrometry and inductively-coupled mass spectrometry. Similarly, INP is engaged in bilateral nuclear forensics partnerships with the Japan Atomic Energy Agency (JAEA), who also participated in the 2016 joint sample analysis between INP and DOE/NNSA, and the Centre for Energy Research (MTA EK) in Hungary, who recently hosted several visits of INP’s nuclear forensics staff. Both JAEA and MTA EK provided analytical data as part of this recent joint sample analysis with INP and DOE/NNSA.

## 2. PARTICIPATING LABORATORIES

A total of five laboratories in four different countries participated in the nuclear forensics analysis of a set of six commercially obtained UOC samples, each having a different level of experience analysing UOC materials. The participating laboratories and their role in supporting national nuclear security, and nuclear forensics more specifically, are described below.



*FIG. 1. Participants of the joint sample analysis data review meeting hosted at the Nuclear Security Training Center (NSTC) at the Institute of Nuclear Physics (INP) in Almaty, Kazakhstan.*

## **2.1. Institute of Nuclear Physics (INP)**

The Republic State Enterprise "Institute of Nuclear Physics" (RSE INP) of the Ministry of Energy of the Republic of Kazakhstan, established in 1957, is the leading scientific organization of the Republic of Kazakhstan in nuclear physics. The main activities of RSE INP include: fundamental and applied research in nuclear physics, accelerator physics, solid state physics, materials science, nuclear and radiation safety, radiochemistry; development of the methods and technologies for radiation processing of materials, the ion-plasma synthesis of the coatings, EPR-dosimetry, the analysis of composition, structure and properties of materials, radioactive waste management; radioecological monitoring of radiation levels in the environment of Kazakhstan (including the Semipalatinsk Nuclear Test Site and other facilities located in Northern and Western Kazakhstan), development of the technologies for handling the ionizing radiation sources, radioactive waste, radiation sterilization, designing the nuclear energy facilities, training of the specialists in the area of radiation safety; production of radioisotopes, radiopharmaceuticals, sealed radioactive sources for industrial and medical application, radiation-crosslinked polymeric materials. The scientific, technical and production capacity of the Institute has been formed over the years. The Institute operates the research reactor WWR-K, the charged particles accelerators and cyclotrons. RSE INP includes the certified Centre of Complex Radio-Ecological Research, which provides the analysis of nuclear materials detected outside the regulatory control. RSE INP has recently made major investments in its isotope production facilities (including the new cyclotron and several hot cells) for medical and industrial application.

## **2.2. Lawrence Livermore National Laboratory (LLNL)**

LLNL has been at the forefront of nuclear forensics since the early 1990s. As one of only two U.S. laboratories designated to perform operational analysis of bulk special nuclear material for nuclear forensics, LLNL is responsible for the generation of reliable, high-quality analytical results that are legally defensible in a court of law. Through its experience as an operational nuclear forensics lab, LLNL has been instrumental in the advancement of nuclear forensics signatures research aimed at providing clues into the origin and history of material found outside of regulatory control. Through the Office of Nuclear Smuggling Detection and Deterrence (U.S. DOE/NSDD), LLNL is supporting nuclear forensics collaborations with almost twenty different partner countries across the world. These bilateral and multilateral partnerships focus on scientific skill development and stakeholder coordination in response to a nuclear forensics event.

### **2.3. Japan Atomic Energy Agency (JAEA)**

The Integrated Support Centre for Nuclear Non-proliferation and Nuclear Security (JAEA/ISCN) initiated research for nuclear forensics in 2011. Under the collaboration with US-DOE and EC/JRC, JAEA/ISCN has developed fundamental technologies for nuclear forensics and prototype of a NNFL. JAEA/ISCN also has been improving their capabilities through participation in the Collaborative Material Exercise (CMX) and the virtual exercise for Nuclear Forensics Laboratories organized by the Nuclear Forensics International Technical Working Group (ITWG). The Ninyo-toke Environmental Engineering Center (JAEA/NEEC) conducts research in the field of the front end of the nuclear fuel cycle. ISCN and NEEC initiated a collaboration to perform UOC analysis for nuclear forensics.

### **2.4. Centre for Energy Research (MTA EK) in Hungary**

MTA EK is one of the Technical Support Organizations of the Hungarian Atomic Energy Authority and the Nuclear Security Support Centre of Hungary. MTA EK was nominated to the Collaborating Centre of the International Atomic Energy Agency for Nuclear Forensics based on long-term experience in this field and long relationship with the IAEA in this field back to the early 2000 years. MTA EK is actively participating in combating illicit trafficking of nuclear materials in Hungary and in the Region since the beginning of 90'. Nuclear forensics as official task is delegated to MTA EK by a Governmental Decree from 1996. The Research Centre started to participate in collaborative material exercises and Galaxy Serpent virtual exercise for Nuclear Forensics Libraries organized by the Nuclear Forensics International Technical Working Group from the beginning. In between, MTA EK established the first prototype of NNFL in Hungary in 2015. As the Collaborating Centre of the IAEA, MTA EK organizes and hosts national and international training courses in the field of Nuclear Forensics and Radiological Crime Scene Management, as well as initiated bi-lateral cooperation with several countries as Romania, Serbia, Kazakhstan and the United Arab Emirates.

## **3. EXERCISE OBJECTIVES**

Joint sample analyses are a valuable tool in building a path to continuous advancement of the analytical capabilities in the international community and help establish long-term partnerships with strategic countries in the area of nuclear forensics. A joint sample analysis is typically the first step in establishing - or further advancing - a nuclear forensics capability with a partner country, and offers an informal way to practice existing capabilities, processes and procedures on a nuclear material sample. In a collaborative manner, an analytical plan, including an associated analysis timeline, is developed by all participating organizations, and the analytical work is conducted as if the sample was a nuclear forensics sample, in accordance to internal standard operation procedures, international guidance and good practices.

As the largest uranium producer in the world, Kazakhstan has a targeted interest in understanding the measurable characteristics associated with the UOC it produces. UOC is signature-rich and is therefore a good material to target for inclusion in an NNFL, such as the one currently being established in Kazakhstan. The objective of the joint sample analysis was for the participating laboratories to 1) exercise their nuclear forensics capabilities by analysing a set of five UOC samples (A-E) according to a nuclear forensics analytical plan developed by each participating laboratory, and 2) develop tools to determine whether an additional “blind” sample (sample F) was similar to any of the other UOC samples in the set of known samples.

All participating laboratories developed and documented an analytical plan that outlined the sequence of techniques applied to the sample, in accordance with the guidance in IAEA Nuclear Security Series No. 2 Nuclear Forensics in Support of Investigations [3]. Standard Operating Procedures (SOPs) and other procedures were followed as if these samples were part of an actual nuclear forensics ‘case’ – starting with documenting sample receipt, performing dose rate measurements, followed by physical characterization, determination of the chemical form, uranium concentration and isotopic compositions, as well as major, minor, and trace elemental analyses. An example

of an analytical plan is shown in Fig. 2. Radio chronometry, or a ge-dating, of the UOC samples was outside the scope of this exercise. Several grams of material were available for the analysis (not sample limited).

Through these analyses, the participating laboratories both enhanced their experience working with UOCs, while increasing their understanding of the signatures associated with this type of material.

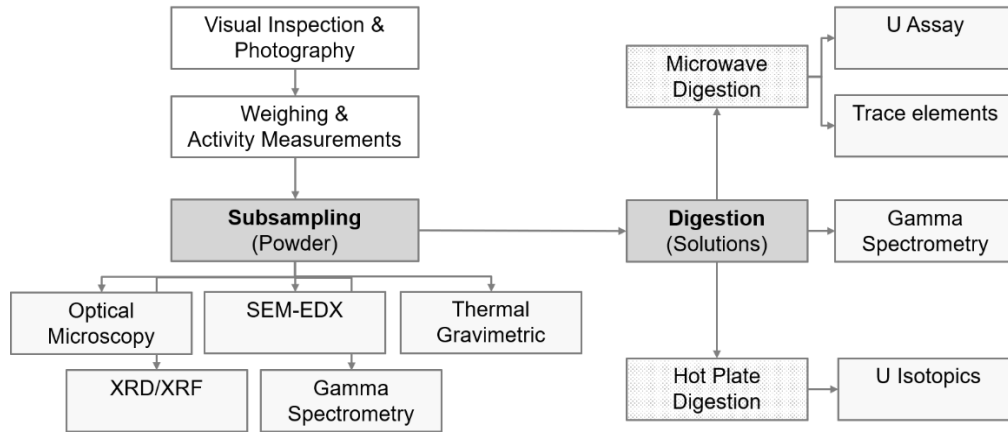


FIG. 2. Example of an analytical plan for the nuclear forensic examination of uranium ore concentrates (UOCs).

#### 4. SELECTED RESULTS

The INP in Almaty, Kazakhstan, hosted a data review meeting on 15-17 October 2019 to discuss the outcomes of the nuclear forensics joint sample analysis between INP, JAEA, MTA EK and LLNL. During this 2.5-day meeting with close to 30 participants, the participating laboratories described their analyses and presented their findings, which largely agreed with each other. The following sections provide an overview of some of the analytical results obtained on the UOC sample set. Due to wide range of analytical techniques applied by the participating laboratories on the sample set, only selected measurements will be reported here. A more detailed description of the results will be published elsewhere.

##### 4.5. Sample Receipt

The UOC sample set shipped to INP, JAEA and MTA EK was unpacked and the physical condition of the sample containers (dimensions, dose rate) was measured and documented. The samples were photographed in their original containers upon receipt (Fig. 3), and chain-of-custody was started. The samples were then re-labelled (samples A-F) and the analyses were started according to the analytical plan developed by each laboratory.

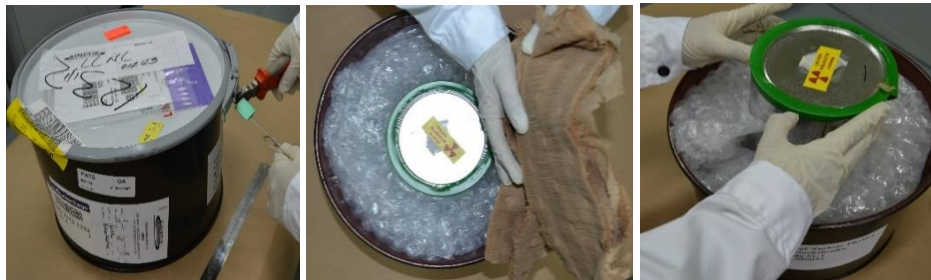


FIG. 3. Documenting the unpacking of the shipping container at INP.

#### 4.6. Physical Characterization

During the previous joint sample analysis on the CUP-2 reference material, there was some discrepancy in the description of sample colour between the participating laboratories. Even though sample colour can be a very important nuclear forensics signature for UOC materials, documenting sample colour was found to vary between analysts and observation conditions. For this joint sample analysis, LLNL made use of the Munsell colour chart to obtain a more objective determination of colour using three parameters: hue, value and chroma<sup>1</sup>. The colour determination results for the main phases are summarized in Fig. 4 and Table 1 below. All laboratories reported the presence of minor phases of a lighter colour in the optical microscopy images for all samples (Fig 5).

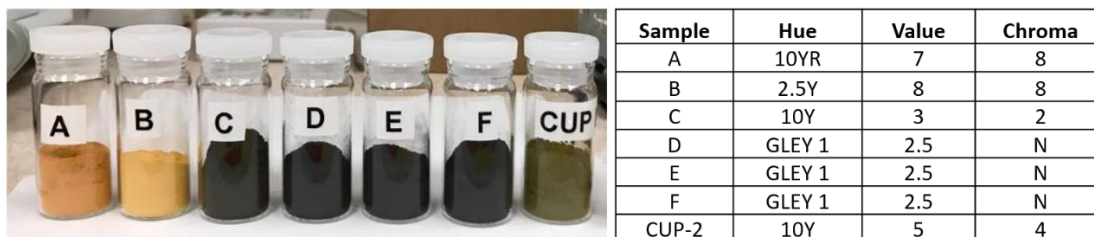


FIG. 4 and Table 1. UOC sample set (A-E) and one blind sample (F) with corresponding colour determinations using the Munsell chart. The CUP-2 material was used as a reference material for the exercise.

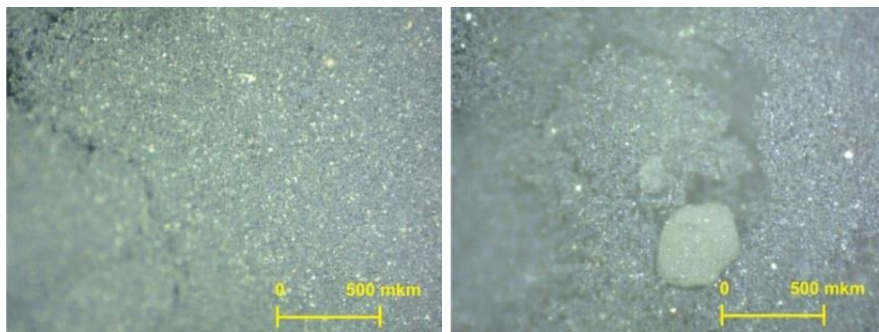


FIG. 5. Optical microscope images of sample C (left) and the blind sample F (right) showing multiple phases of different colour (images by INP).

The SEM-EDX analyses were performed by a wide range of microscopes and detectors across the participating laboratories, including a tabletop HITACHI TM3030Plus instrument recently installed at INP. Small aliquots of the powder were placed on a substrate suitable for SEM imaging in high vacuum (e.g. carbon or copper tape). SEM-EDX analysis showed a high degree of agglomeration for all samples analysed, with particle size ranging from submicron to several hundreds of micrometres for the largest agglomerates. JAEA performed an in-depth particle size distribution analysis of the particles using a commercial software tool (ImageJ v1.52a) and determined that the cumulative size distribution for the blind sample (sample F) was similar to that of sample E and sample D (Fig. 6).

<sup>1</sup> Hue, value and chroma assign a value to the color shade, darkness and purity, respectively.

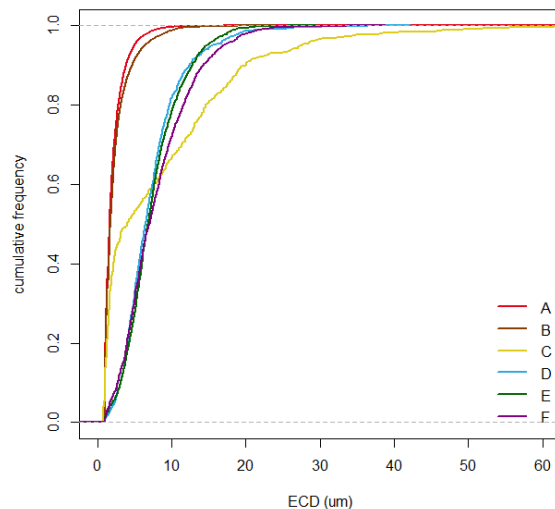


FIG. 6. Cumulative size distribution (based the particles' equivalent circular diameter (ECD) of the 6 UOC samples (A-F) obtained by JAEA using ImageJ software.

High magnification imaging showed that the blind sample (sample F) had a very distinct surface structure with pore spaces that looked very similar to that of sample E (Fig. 7). Semi-quantitative analysis of the elemental composition by EDX showed that samples E and F were also similar in terms of impurities. Samples A and B had significantly more impurities (several weight % Fe, sub-% Al and Si) than samples E and F (sub-% Al and Si). MTA EK concluded the sample set consisted of three distinct groups (samples A-B; sample C; sample D-F) based on their SEM-EDX analysis results. This was consistent with the findings from JAEA, LLNL and INP.

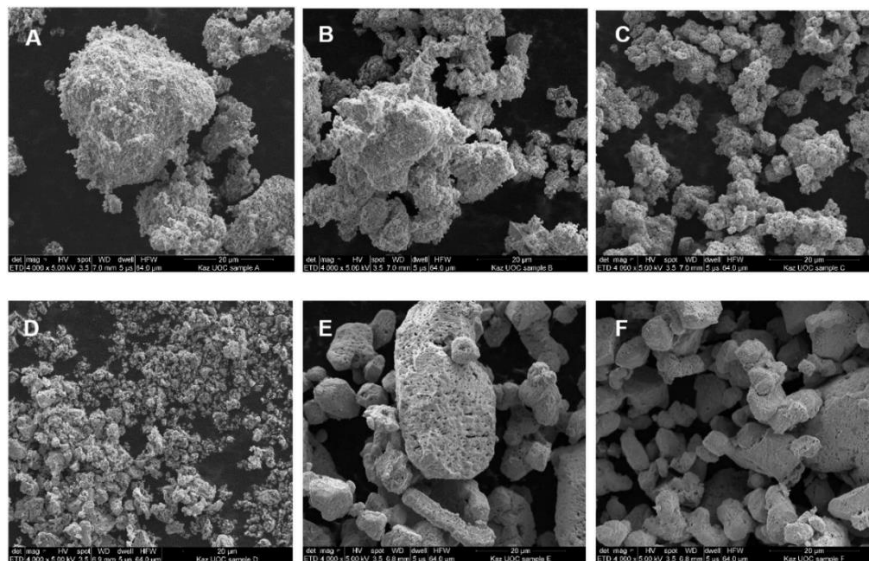


FIG. 7. UOC sample set (A-E) and one blind sample (F) sent to the different participating laboratories for a joint nuclear forensics sample analysis. The CUP-2 material was used as a reference material for the exercise.

#### 4.7. X-Ray Diffraction (XRD) and X-Ray Fluorescence (XRF)

The three laboratories that included XRD analysis in their analytical plan (INP, MTA EK, LLNL) used the same type of diffractometer instrument (Bruker AXS D8 ADVANCE) to analyse milligram amounts of the UOC

powders, top loaded on a low-background silicon plate. However, the interpretation of UOC XRD spectra is not always straight forward due to the presence of multiple phases and the limited extent of the library available for peak matching. MTA EK and LLNL identified the major phase of the blind sample (sample F) and sample E as uranium dioxide (UO<sub>2</sub>) (Fig. 8), while INP identified the major phase as U<sub>4</sub>O<sub>9</sub>. The analyses of samples A and B did not result in consistent results among the different laboratories, with some of the major phases identified as UO<sub>4</sub>, UO<sub>3</sub>, U<sub>3</sub>O<sub>8</sub>, U<sub>4</sub>O<sub>9</sub> and U<sub>2</sub>O<sub>5</sub>. Sample C was identified by all laboratories as U<sub>3</sub>O<sub>8</sub> (major phase).

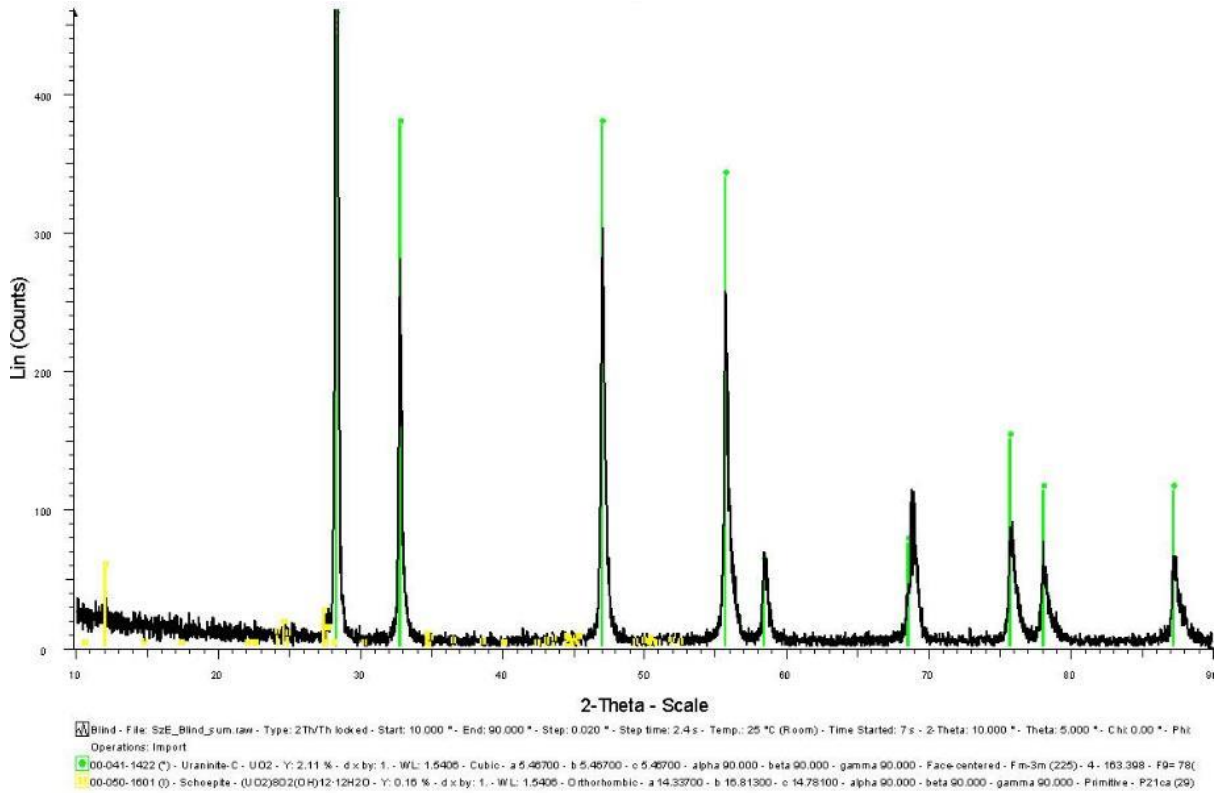


FIG. 8. XRD spectrum of the blind sample F where the major phase was identified to be UO<sub>2</sub> (spectrum by MTA EK).

XRF is typically not a very reliable method for measuring the uranium concentration of loose UOC powders but can be useful for semi-quantitative trace element analysis. Also, XRF can be useful for elements difficult to measure by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) without high resolution or collision cell capabilities, such as S, Si, P, Cl and Br. At INP and JAEA, XRF was used as an initial screening tool for uranium concentration and elemental composition. The impurity data (Fig. 9) were generally consistent with the SEM-EDX results.



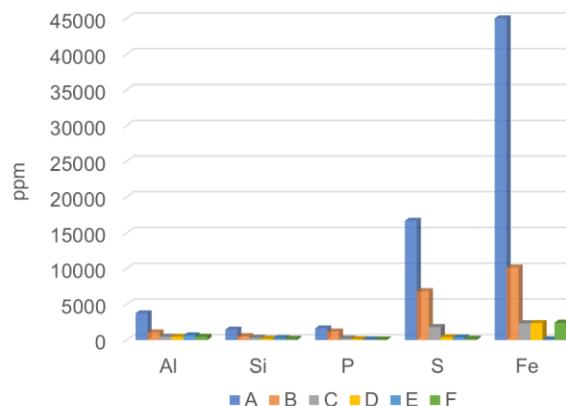


FIG. 9. Concentration of selected impurities for all 6 UOC samples as determined by XRF (data JAEA).

#### 4.8. Gamma spectrometry

Gamma spectrometry analysis was performed both in the solid phase (powder) and on the solutions (powder dissolved in HNO<sub>3</sub>) with different sets of detectors collecting a range of energy spectra. Assuming the <sup>238</sup>U is in secular equilibrium with <sup>234</sup>Th and <sup>234m</sup>Pa, the isotopes <sup>234</sup>U, <sup>235</sup>U, and <sup>238</sup>U daughters were detected and quantified. The CUP-2 UOC sample was measured as a reference to correct for systematic biases. The <sup>234</sup>U data had the highest uncertainty due to use of the low-energy and low-branching ratio 121 keV peak. No other radionuclides were detected in any of the samples A-F and the uranium isotope ratios of the samples were all confirmed to be natural uranium.

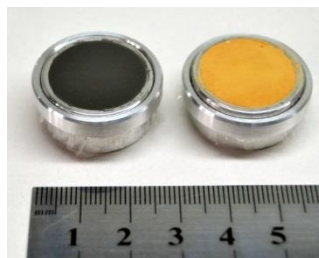


FIG. 10 and TABLE 2. Uranium content of the UOC samples obtained by gamma spectrometry at INP using standardized sample containers.

TABLE 2

Sample	U Content, %
A	68.8 ± 2.5
B	74.0 ± 2.7
C	78.5 ± 2.9
D	83.9 ± 2.9
E	84.3 ± 3.0
F	83.7 ± 2.9

Gamma spectrometry was also used to determine the total uranium concentration of the UOC samples. At INP, a set of identical sample containers were made (20.00 mm ± 0.05 inner diameter and 10.00 mm ± 0.05 depth) that were entirely filled with the sample to avoid geometrical differences between samples (Fig. 10). The AnaGamma software used for routine gamma-spectrometric analysis does not provide for correction of self-absorption in a very

heavy matrix, such as UOC. Therefore, this software package was used only to determine the specific activity of gamma lines. Calculation of the uranium concentration was performed by the relative method for  $^{235}\text{U}$  gamma radiation using an external standard, CUP-2 UOC. The measurement results in Table 2 showed that the concentration of the blind sample was the same as samples E and D, within uncertainty of the measurement. A more precise assay technique, or other signatures like trace elements, are required to identify the blind sample with higher confidence.

#### **4.9. Mass Spectrometric Techniques for Impurity Analysis and Uranium Isotopics**

INP, JAEA, MTA EK and LLNL all used different techniques in the determination of trace elements in the UOC sample set. A combination of inductively coupled plasma optical emission spectroscopy (ICP-OES), quadrupole and sector field ICP-MS was used by the four participating laboratories. The ICP-OES analysis applied an external standard calibration on a handful of major elements, whereas INP used standard addition on one sample for calibration of the other samples. The analysis was performed using an Agilent HP 4500 quadrupole ICP-MS. LLNL performed a matrix matched external calibration on a Thermo Scientific iCAP-Q quadrupole ICP-MS. The approach to sample digestion taken by each laboratory also varied: JAEA and MTA EK performed chemical purification (separation techniques) of the material to remove the uranium matrix prior to analysis. The UOC sample set contained uranium oxides that were much more pure than the CUP-2 UOC analysed in the previous joint sample analysis, and especially for the blind sample and sample E, some of the impurities were close to the detection limit of the mass spectrometers applied by the different laboratories.

Upon review it was determined that the main contributors to the differences seen in the analytical results between the participating laboratories were the differences in the way samples were analysed (different instruments, different uranium concentrations). However, small differences in overall concentration are not necessarily an issue when looking for signatures. When normalized to chondrite, the rare earth elements (REE) produced similar patterns (Fig. 11).

Each laboratory used a different method to determine the isotopic composition of uranium in the UOC materials. LLNL performed chemical separation chemistry to purify uranium in the material, then used a multicollector ICP-MS to quantify  $^{234}\text{U}$ ,  $^{235}\text{U}$ , and  $^{238}\text{U}$  ( $^{236}\text{U}$  and  $^{233}\text{U}$  below detection limit). INP used a dilute aliquot of the material to quantify  $^{234}\text{U}$ ,  $^{235}\text{U}$  and  $^{238}\text{U}$ , using a quadrupole ICP-MS. INP also measured uranium isotopics by alpha spectrometry to quantify  $^{234}\text{U}$  and  $^{238}\text{U}$  and calculate a  $^{234}\text{U}/^{238}\text{U}$  activity ratio. MTA EK used sample dilution, besides isotope dilution, as well as external calibration for determination of uranium isotopics measured by high-resolution sector field single collector ICP-MS, and JAEA performed total evaporation thermal ionisation mass spectrometry (TIMS) on a purified aliquot. The measured uranium isotope ratios for  $^{235}\text{U}/^{238}\text{U}$  agreed for all four laboratories, and were, within uncertainty, consistent with the isotopic composition of natural uranium (Fig. 12).



FIG. 11. Diagram of the rare earth elements (REE) for all UOC samples by ICP-MS (data LLNL). The chondrites were normalized using the mid ocean rich basalt (MORB) values from Pierce (1973) and Bevins (1984).

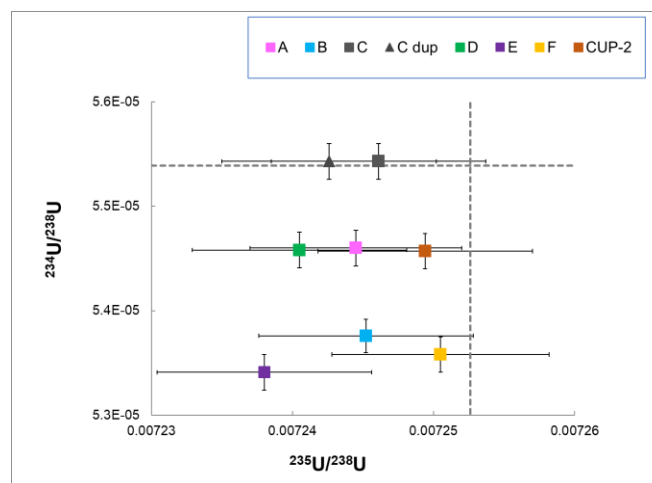


FIG. 12. Three isotope plot for the uranium isotope ratios for all UOC samples by ICP-MS (data LLNL).

## 5. CONCLUSIONS

As described earlier, the overall objectives of this joint UOC sample analysis were for the participating laboratories to 1) exercise their respective nuclear forensics capabilities by analysing a set of UOC samples, and 2) develop tools to determine whether an additional “blind” sample (sample F) was similar to any of the other UOC samples in the set. All participating laboratories developed their own analytical plan for the nuclear forensic analysis of the UOC sample set and used the CUP-2 UOC material from the previous joint sample analysis as a reference material. The results presented by the different laboratories during the October 2019 data review meeting largely agreed with one another. Although there were some significant differences in the XRD and XRF results, all laboratories concluded that, based on the physical characterization of the samples, in combination with several other non-destructive measurements, the blind sample (sample F) was similar to samples D and E. Further non-destructive analysis of the impurities indicated the blind sample was similar to sample E with high confidence.

Because of the limited size of this sample set, this determination could be done manually, by comparing the individual characteristics of the samples obtained by the different analytical techniques. For a larger data set however, a more rigorous approach would be required. JAEA and LLNL therefore implemented statistical evaluation tools to the data set generated from the UOC analyses. LLNL applied the Graded Decision Framework tool developed as part of the most recent Collaborative Materials Exercise (CMX-6) of the Nuclear Forensics International Technical Working Group (ITWG) [4] to evaluate consistency between (quantitative) sample characteristics, and the associated confidence levels of those findings. However, this tool assumes a normal distribution, and therefore JAEA opted to combine a set of statistical methods to evaluate similarities between the different samples depending on the nature of the data. In both cases, however, the importance of nuclear forensics expertise was recognized, not only to generate reliable data, but also to provide the most accurate interpretation of the results and weigh in on the relative importance of the different characteristics and signatures.

## 6. OUTCOMES AND PATH FORWARD

International capacity building through peer-to-peer engagements is considered a key pathway to building a sustainable nuclear forensics capability in a partner country. Activities such as joint sample analyses, mentorships and technical exchanges all contribute to the establishment of an informal network of nuclear forensics practitioners, while advancing the science of nuclear forensics. These scientific exchanges also lay the groundwork for the evaluation and interpretation of nuclear signatures associated with nuclear materials of interest. Due to the success of this joint UOC sample analysis, the nuclear forensics group at INP decided to use the outcomes and results of the joint sample analysis for a new scenario-based table-top exercise as part of the IAEA's Third Regional Seminar on Nuclear Forensics, which was hosted at INP in December 2019.

The development and exercising of the UOC component of the NNFL will be an important next step in the assembly of a comprehensive library capturing important nuclear and radioactive materials in Kazakhstan. Although it is possible to create an NNFL by incorporating all material types in a single data resource, many countries, including Kazakhstan, the U.S. and Japan, have taken a federated approach whereby different material types are captured in several data resources that can be accessed by the library. By adopting a federated approach to the NNFL, Kazakhstan will be able to include additional materials, expertise, and data resources in the NNFL as they become available. To that effect, representatives from Kazatomprom, the world's largest producer of uranium ore concentrates, the Institute of High Technology, the National Nuclear Centre Kurchatov (NNC) and the Committee of Atomic and Energy Supervision and Control of the Ministry of Energy (CAESC) were invited to the data review meeting as the Kazakhstan government further advances its development of the Kazakhstan NNFL and its holistic approach to nuclear forensics.

## ACKNOWLEDGEMENTS

The authors would like to thank the scientific staff and support staff involved in the joint sample analysis in their respective laboratories. This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

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