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COMPUCEA 2nd generation performance evaluation

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Abstract. COMPUCEA (**C**ombined **P**rocedure for **U**ranium **C**oncentration and **E**nrichment **A**ssay) is used for on-site analytical measurements in support of joint Euratom-IAEA inspections during physical inventory verification (PIV) campaigns in European Low-Enriched Uranium (LEU) fuel fabrication plants. The analyses provided on site during the PIV involve the accurate determination of the uranium element content and of the U-235 enrichment in verification samples (uranium product samples of solid form, i.e. powders, pellets) selected by the Safeguards inspectors. These samples are dissolved and then measured by energy-dispersive X-ray absorption edge spectrometry (L-edge densitometry) to obtain the uranium elemental content and gamma spectrometry with a Lanthanum-bromide detector for the U-235 abundance determination. The second generation of COMPUCEA equipment is compact, rugged and ready-to use directly after transport, no cooling of the detectors with liquid nitrogen is required. A software package for comfortable instrument control and data handling has been implemented. The paper describes the technique, setup and calibration procedure of the instrument. Results from PIV campaigns and comparisons between COMPUCEA results with data obtained by remote analysis with a qualified primary analytical method are presented, which demonstrate the performance of the technique. The achieved uncertainties are well within the international target values. First results obtained with a sandwich detector configuration for enhanced detection efficiency of the passive gamma spectrometry and a small separate X-ray fluorescence unit for the pre-screening of the samples for their Gd content are discussed.

1. Introduction

The **C**ombined **P**rocedure for **U**ranium **C**oncentration and **E**nrichment **A**ssay (COMPUCEA) is a measurement technique for the uranium element and ²³⁵U-enrichment assay routinely applied to the analysis of uranium product materials (uranium oxide powders and sintered uranium oxide pellets). The analyses are performed with mobile equipment in different European fuel fabrication plants for **L**ow-**E**nriched **U**ranium (LEU) fuels during the nuclear material physical inventory verification (PIV) activities of international nuclear safeguards authorities (Euratom, IAEA). The samples are selected by the Safeguards inspectors and directly measured by ITU analysts on-site during the PIV week.

The technical know-how and expertise for the COMPUCEA technique have been developed at ITU. DG ENER, the IAEA and ITU therefore agreed that ITU takes over the responsibility for the in-field measurements with COMPUCEA (i.e. ITU sends qualified analysts in field during the PIV campaigns), and for the COMPUCEA technique in general (i.e. preparation of equipment for the missions, maintenance and upgrades or new developments). The equipment itself is procured and owned by DG ENER, who also handles the logistics for instrument transportation. Sampling and interpretation of the analytical results is the responsibility of the Safeguards inspectors (DG-ENER and IAEA, jointly).

A typical PIV campaign in a LEU fuel fabrication plant is carried out within a period of 1 week. On average, ITU sends 2 analysts to each site to perform the in-field analyses during this week. For a timely and conclusive termination of the PIV, the analytical results have to be handed over to the inspectors by

Thursday of the respective week at the latest. In order to keep this strict deadline, the ITU analysts sometimes arrive on site 2-3 days in advance for instrument preparation and calibration.

The missions made with COMPUCEA are unique because they represent, to our knowledge, the only example in the measurement practice of Safeguards, where high-accuracy analytical measurements for accountability verification are directly performed in field with mobile analytical equipment. The benefits of this practice are obvious: analysis results are immediately available for a timely conclusion of the PIV, observed discrepancies can be investigated and (in most instances) solved directly on the spot, and sample shipment to remote Safeguards laboratories is avoided or at least significantly reduced.

2. Analytical procedure and instrumental setup

2.1. Analytical procedure

The complete COMPUCEA analysis procedure represents a combined chemistry-spectrometry analysis involving accurate analytical steps (like quantitative sample dissolution, solution density measurements, quantitative aliquoting etc) combined with radiometric measurements. A more detailed description can be found in [1,2] The general scheme of analysis includes the following main steps:

- **Sample preparation:** The first step is to transform the solid uranium samples (powders or pellets) into a nitric acid solution of approximately constant acidity (3 M) and uranium concentration level, which is then characterised for its density and temperature. The nominal uranium concentration is set to be around 190 mgU/ml, which is close to the upper limit of the linearity range of the L-edge densitometry measurement [1]. The analytical tools needed for this sample preparation step (hot plate, density measurement device, glass ware, pipettes etc) are brought on site as part of the COMPUCEA equipment, but the use of operator facilities (fumehood) is also required at this stage. This also holds for the disposal of the (non-problematic) liquid waste in the form of pure uranyl nitrate solutions afterwards.
- **Radiometric measurements (L-Edge Densitometry and Gamma Spectrometry):** Aliquots are taken from the sample solution and subjected, without any further treatment, to parallel L-edge densitometry and passive gamma counting with a LaBr₃(Ce) detector. The two radiometric techniques are described in more detail below.
- **Data evaluation:** A software package for comfortable instrument control and data handling has been implemented. It is based on data acquisition with Canberra GENIE 2000 software, the analysis of the gamma spectra is performed with a specifically modified version of the NaGEM code, which can also handle the presence of ²³²U- decay products, as observed for samples of reprocessed uranium. In the final step of data evaluation, the different pieces of information obtained from the sample preparation and from the two radiometric measurements are combined to evaluate the uranium weight fraction in the original sample and the ²³⁵U weight fraction in the uranium material. It should be noted that the two radiometric measurements are interdependent, i.e. each technique requires input from the other for final data evaluation: the L-edge densitometry measurement needs the knowledge of the enrichment to accurately convert the measured uranium concentration into mass fraction, and the gamma measurement needs as input the knowledge of the uranium concentration. The evaluation of the final uranium concentration and enrichment is therefore made in an iterative manner.

2.1. Uranium concentration determination by L-edge densitometry

In the 2nd generation of the COMPUCEA equipment, a miniaturized 30 kV/100 μ A X-ray generator, and a Peltier-cooled, high-resolution 10 mm² x 0.5 mm Si drift detector are used to measure the energy-differential absorption at the L_{III}-shell absorption edge, which occurs for uranium at the energy of 17.17 keV. The sample cell consists of a fixed flow-through quartz cell with a path length of 2 mm, and a cell volume of 125 μ l.

The evaluation of the uranium concentration from the measured ratio of photon transmission across the L_{III} edge at 17.17 keV follows the proven analysis procedure adopted for K-edge densitometry with an X-ray continuum [3]. In this approach, the photon transmission as a function of energy, T(E), is measured relative to a blank spectrum from a nitric acid solution of representative molarity (3M), and then linearized in a representation $\ln(1/T)$ vs $\ln E$. Linear least-squares fits to the respective data on both sides of the absorption edge determine the photon transmission at energies slightly displaced from the absorption edge ('non-extrapolated fitting mode', $E_+ = 17.60$ keV, $E_- = 16.70$ keV), or directly at the absorption edge energy ('extrapolated fitting mode'). Fitting intervals ranging from 14.4-16.8 keV, and from 17.6-20.0 keV were

chosen for the evaluation of the transmission ratio across the L_{III} edge.

The uranium concentration derived in the 'extrapolated fitting mode' from the transmission ratio directly at the L-edge energy is virtually insensitive to matrix effects (for more detail, see [1]). The availability of two independent analysis results for the uranium concentration from the extrapolated and non-extrapolated fitting analysis therefore represents a very useful diagnostic tool. A statistically significant difference observed between the two results will immediately point to any sort of deviation in the matrix composition of the measurement sample from the assumed 3M HNO_3 reference matrix. This knowledge is not of immediate relevance for the L-edge densitometry measurement, but of practical help for the parallel enrichment measurement, where any deviation in the matrix composition will have a direct influence on the gamma attenuation behaviour of the sample. A practical example refers to the analysis of uranium samples containing a significant amount of gadolinium, which has been added as burnable neutron poison. In this case, the difference observed between the uranium results from the non-extrapolated and extrapolated fitting analysis can be used for an estimate of the Gd content, provided the discordance between the two evaluated uranium results can be reasonably attributed to the presence of this additional element alone. The knowledge about the Gd content then allows calculating corresponding correction factors for the enrichment measurement.

2.2. ^{235}U enrichment determination with a $LaBr_3(Ce)$ detector

The ^{235}U enrichment measurement in the 2nd generation of COMPUCEA is based on the counting of the ^{235}U 186 keV gammas of a defined amount of uranium in solution in a well-defined counting geometry. The new detector replacing the previous HPGe well detector is a standard-type 2" x 1" cerium-doped lanthanum bromide scintillation detector – $LaBr_3(Ce)$. This being a detector operating at room-temperature offers the following main practical advantages for in-field applications: The need for detector cooling with liquid nitrogen, previously supplied by the operator on-site, is eliminated and the detector is directly ready for use after unpacking of the equipment. The relatively simple gamma spectrum of ^{235}U allows accurate enrichment measurements also at the lower energy resolution of the $LaBr_3$ compared to HPGe (FWHM @ 186 keV about 9 keV for the $LaBr_3$ detector compared to a value of 1.3 keV obtained with the previous HPGe well detector) [3].

Since the only recently developed $LaBr_3(Ce)$ scintillation detectors were not available in the form of well-type configurations, the sample counting geometry was changed, the sample is now placed on top of the detector. This measurement geometry is less efficient than counting in a well detector, therefore the sample volume for the measurement with the $LaBr_3(Ce)$ detector was increased to 10 ml (previously 2.5 ml).

The evaluation of the measured gamma spectrum for an accurate enrichment determination involves a two-step process: (1) analysis of the gamma spectrum itself for the extraction of the 185.7 keV net peak counts, and (2) the calculation of appropriate correction factors for the extracted peak counts accounting for the impact of variable sample parameters. The software for spectrum analysis is based on a modified NaIGEM analysis code [4]. It determines the net peak counts for the most prominent ^{235}U gamma line at 185.7 keV with associated uncertainty. Additional information is provided on the quality of the response function fitting, and on the full width at half maximum value determined for the 185.7 keV line. The fitting code has recently been adapted to handle gamma spectra from recycled uranium materials which show an additional gamma ray at 238.6 keV originating from the ^{232}U descendent ^{212}Pb . For those spectra, the analysis code also reports the peak area with associated uncertainty for the 238.6 keV line.

In the second step of the analysis, correction factors for the evaluated 185.7 keV net peak counts are calculated from relevant sample parameters. They are calculated relative to a standard configuration. The main contributors are the following:

- Variation of the concentration of the uranium solution (standard concentration: 190 gU/l). Here, a correction for the self-attenuation effect of uranium needs to be made for variations of the uranium concentration around the adopted standard concentration.
- Gd presence in the solution, which leads to self-attenuation of the sample depending on the Gd concentration (standard configuration: no Gd).
- Container bottom thickness (standard configuration: 1.10 mm). The bottom thickness of the sample containers represents a crucial parameter, because even small variations will slightly change the sample-to-detector distance, and hence also the measured 186 keV count rate. Prior to the in-field campaigns, the bottom thickness of each container is measured with a thickness gauge with an accuracy of ± 0.01 mm.
- Interfering gamma rays, i.e. the daughter products ^{234}Pa and ^{234m}Pa from ^{238}U

The corresponding correction factors can be obtained in 2 different ways: using separate correction factors

for each of the major contributors according to data obtained from experimental studies (= "traditional approach") and (ii) by a Monte Carlo (MC) calculation routine, which calculates the detection probability for the 185.7 keV photons for the given measurement configuration in dependence of the relevant sample parameters uranium concentration, Gd concentration and container bottom thickness relative to the standard configuration. The Monte Carlo calculation routine has been developed in-house [5]. The relative detection rates calculated within a runtime of 100 s for the Monte Carlo calculation have a statistical precision of ca. 0.03%.

To calculate the final ^{235}U enrichment, the correction factor for Interfering gamma rays (the daughter products ^{234}Pa and $^{234\text{m}}\text{Pa}$ from ^{238}U) is applied for both approaches.

A comparison of the ^{235}U enrichment correction factors (Container bottom thickness + uranium concentration in solution + Gd) with the two different methods was done using the measurement data from 3 COMPUCEA in-field campaigns in 2010. 41 unknown samples (4 with gadolinium) were used for the comparison. The ratios of uranium enrichment obtained by the traditional approach relative to the enrichment derived using the Monte Carlo routine were calculated. These values ("traditional/Monte Carlo") are displayed in Fig. 1 for all in-field samples; an average value of 0.99965 with a standard deviation of 0.03% (identical to the statistical uncertainty of the Monte Carlo calculation) can be calculated. The differences between the 2 factors are small compared to the overall uncertainty of the technique, so the 2 factors can be considered identical and an additional bias due to choice of one method or the other is negligible. It has to be kept in mind, however, that for the data presented here, all relevant sample parameters were very close to the standard configuration. For samples deviating stronger from the standard conditions, the differences between the traditional approach and the Monte Carlo calculation still need to be studied and compared to experimental data.

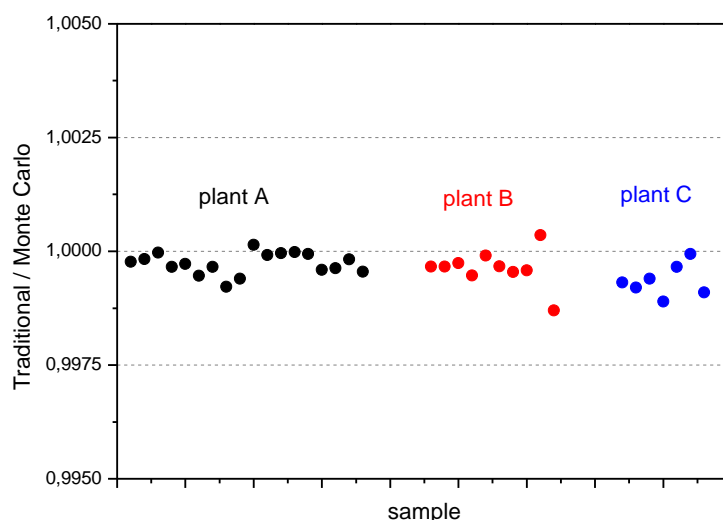


Figure 1: Comparison of ^{235}U enrichment correction factors obtained by the traditional approach and by Monte Carlo simulation

3. COMPUCEA calibration

Both measurement techniques in COMPUCEA require an instrument calibration. The calibration approach has been revised and simplified in the sense that for each technique only a *single calibration factor* needs to be determined. In order to arrive at this favourable situation, measurement and instrument properties considered as being relevant for measurement performance and calibration have been carefully studied during the instrument development stage [1].

Prior to the in-field measurements, all COMPUCEA systems are calibrated at ITU with a set of suitable reference solutions. With this pre-calibration, combined with the quantitatively known correction factors to be applied, calibration in field is reduced to the measurement of two calibration samples for a verification or re-normalisation of the basic calibration factors determined at ITU.

3. 1. Reference materials

The reference materials available for calibration consist of a set of sintered UO_2 pellets with different

enrichment grades. The UO_2 pellets were taken from the production batches of a uranium fuel fabrication plant, and subsequently characterized by primary analytical methods for the uranium element content and isotopic composition. The analytical measurements for material characterisation were independently carried out by ITU and IAEA-SAL. A set of such pellets with enrichment grades 0.72%, 2% and 4% ^{235}U has been available for several years, a new set of 100 pellets with 3% ^{235}U and 100 pellets of 4.4% ^{235}U has recently been purchased, the joint characterisation by ITU and IAEA-SAL is ongoing, each of the labs verifies the uranium elemental content (by titration) and ^{235}U -enrichment (by Thermal Ionization Mass Spectrometry, TIMS) independently.

In addition, 200 certified reference pellets (CRM 125-A), obtained from NBL, USA, are available.

The reference solutions required for instrument calibration, either in field or at ITU, are prepared from these reference pellets, following exactly the same procedures for sample preparation as applied for the normal measurement samples. From each reference pellet, a single reference solution is prepared, which is used both for the calibration of the L-edge densitometer and of the gamma spectrometer.

4. COMPUCEA performance evaluation and validation

To evaluate the performance of the COMPUCEA 2nd generation equipment, the influence of relevant measurement parameters, such as working and linear range, matrix effects, counting precision, measurement reproducibility, gamma self-attenuation and counting geometry, was studied in detail [1]. With the identification and quantification of individual uncertainty components, it was then possible to present an estimate of the total uncertainty of the two analytical determinations made. This estimation also includes uncertainty components related to the sample preparation (sample weighing, dissolution and density measurement).

The estimations for the total measurement uncertainties of COMPUCEA 2nd generation are well within the International Target Values (ITV) for measurement uncertainties in the field of International Safeguards for nuclear materials (relative combined standard uncertainty: 0.25% for U concentration and 0.45% for ^{235}U abundance). The typical counting times with the 2nd generation of COMPUCEA are 3 x 1000s for the L-edge measurement and 3 x 2000s for the ^{235}U enrichment measurement, however, even with a reduced measurement time of 1000 s, the ITV's are safely met.

For method validation, the measurement performance was evaluated in three different ways by comparing the COMPUCEA results

- with results from parallel analyses made with a primary reference method,
- with well-specified reference values for the quantity of interest, and
- with data obtained in round robin tests

4.1. Comparison of COMPUCEA and primary reference methods

During the in-field measurements made with the 2nd generation of COMPUCEA in 2007, 2008 and 2009, a total of 173 uranium samples were analysed at 5 different locations. For a subset (taken at 4 of the 5 locations), parallel samples were taken for remote analysis with a qualified primary analytical method (potentiometric titration according to the method of Davies and Gray).

Figure 2 shows the results of the parallel analyses. The COMPUCEA results are in perfect agreement with the titration results (average difference of 0.055%). The combined uncertainty of both methods (0.11% for COMPUCEA and 0.05% for titration) calculates to 0.12%. The observed standard deviation of 0.11% for the differences between the analysis results is in agreement with this value.

The validation of the enrichment measurements made with the new LaBr_3 detector setup bases on a comparison with results obtained with a recognized and validated measurement technique for isotope abundance measurements, i.e. Thermal Ionization Mass Spectrometry (TIMS). During the in-field COMPUCEA measurement campaigns in 2007, 2008 and 2009 a total of 36 parallel samples of low-enriched uranium were taken and sent for off-site analysis by TIMS. The compared data are plotted in Figure 3.

The average difference for the whole set of data calculates to 0.07% with a standard deviation (1s) of 0.21%. For the underlying type of enrichment measurements made with a scintillation detector in the 2nd generation of COMPUCEA, the results demonstrate a remarkable level of performance. The observed overall uncertainty is well within the estimated uncertainty of the gamma measurement (about 0.25% for low-enriched materials), and also well within the uncertainty levels set by the ITVs.

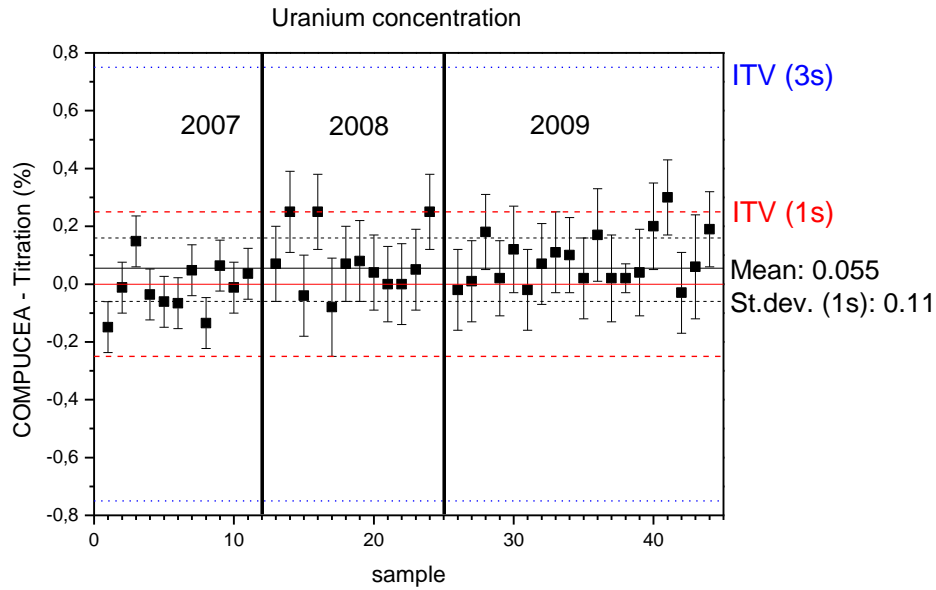


Figure 2: Percentage differences between COMPUCEA L-edge in-field analytical results and primary reference method titration

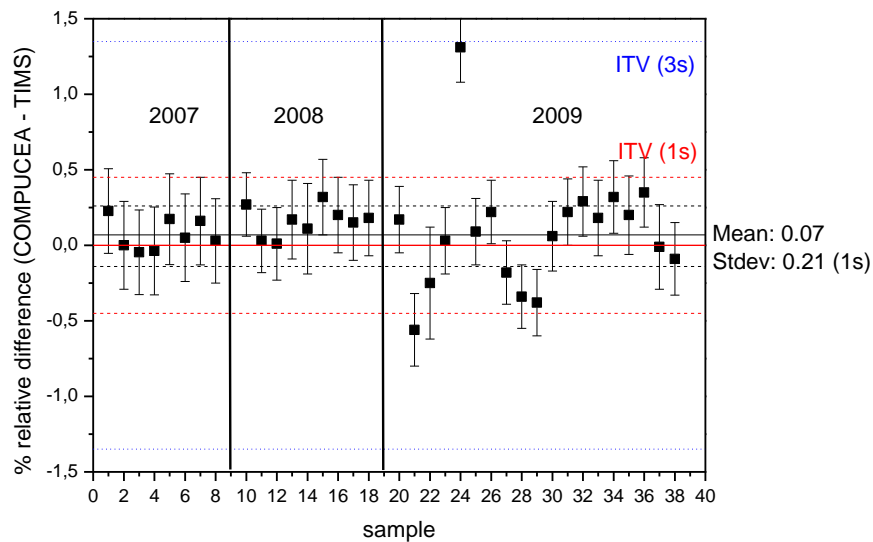


Figure 3: Percentage difference between ^{235}U enrichment values measured with COMPUCEA 2nd generation and TIMS.

4.2. Participation in round robin tests

A key element for measurement performance evaluation, and according to the Laboratory Guide to Method Validation [8] the preferred way of validating methods, is the external control through interlaboratory trials. An interlaboratory round robin exercise pertinent to the validation of COMPUCEA is provided by the programme "EQRAIN" (from the French acronym for "Quality Assessment of Analysis Results in the Nuclear Industry") conducted by CEA-CETAMA. Under the EQRAIN programme highly concentrated uranyl nitrate solutions ($> 200 \text{ gU/kg}$) of unknown concentration are distributed on a regular basis to interested parties for controlling their analytical methods for uranium analysis. COMPUCEA participated with the L-edge densitometer part in 4 round robin tests performed under EQRAIN 12 in 2008/2009. During each of the rounds, 2 or 3 sub-samples were analysed independently. The results obtained prove a high degree of reproducibility and, from a comparison with the reference values communicated after submission of the results, a high degree of accuracy with an average deviation as low as -0.023% (the individual data can be found in [2]).

Another interlaboratory round robin exercise is offered by New Brunswick Laboratory (NBL), USA. Under

their Measurement Evaluation program, uranium samples (UO₂ pellets, U₃O₈ Powder, UF₆ samples) are distributed for destructive analysis (SME program). COMPUCEA participated during several rounds in the analysis of UO₂ pellet samples. The results of the first 2 rounds, where reference data is already available, are summarized in table 1. The COMPUCEA data are in good agreement with the reference values and well within the ITVs.

Table 1. COMPUCEA results obtained under the SME program by NBL. The uncertainties stated are 1s.

	U concentration (wt %)			U-235 wt %		
	Reference value	COMPUCEA	% difference	Reference value	COMPUCEA	% difference
1 st round	88.129	88.12 ± 0.13%	0.01	4.00823	4.010 ± 0.21%	-0.04
		88.12 ± 0.13%	0.01		4.016 ± 0.21%	-0.19
		87.98 ± 0.13%	0.17		4.007 ± 0.24%	0.03
		88.05 ± 0.14%	0.09		4.026 ± 0.30%	-0.44
		88.11 ± 0.13%	0.02		4.008 ± 0.21%	0.01
		88.13 ± 0.13%	0.00		4.000 ± 0.21%	0.21
		88.01 ± 0.13%	0.14		4.016 ± 0.23%	-0.19
2 nd round	88.129	88.05 ± 0.13%	0.09	4.00823	4.0005 ± 0.21%	0.19
		88.08 ± 0.15%	0.06		4.0065 ± 0.23%	0.04
		88.10 ± 0.13%	0.03		4.0025 ± 0.22%	0.14
		88.05 ± 0.15%	0.09		4.0045 ± 0.23%	0.09

4.4. COMPUCEA Upgrades: LaBr₃ sandwich detector and XRF unit

In order to further improve the measurement performance of the COMPUCEA equipment, an increase of sample throughput is of relevance during in-field operation, without loss of measurement performance. The limiting technique here is the gamma counting with the LaBr₃ detector. Depending on ²³⁵U enrichment, typically 3x2000s measurement time is necessary to achieve the desired counting statistics; for samples of natural uranium the counting time is increased to 3x5000s, these measurements are run over night. In order to increase the sample throughput in-field, a second separate LaBr₃ detector was taken on-site during the 2009 and 2010 campaigns. As an alternative, a counting configuration using two LaBr₃ detectors in a sandwich-type arrangement promises to enhance both the robustness of the counting configuration and the detection efficiency, allowing for shorter counting times and/or improved measurement precision. First experiments were performed using specifically designed sample containers, filled with 10, 12 and 13ml of sample solution. For data evaluation, the two spectra were evaluated separately and the number of counts determined for the 186 keV peaks summed up afterwards. For the 10, 12 and 13 ml samples, an increase of the overall counts of a factor of 2.5, 3 and 3.3, respectively, was obtained, compared to the counting configuration with one detector (and 10 ml sample volume). The reproducibility, both for several repetitions of measurements as well for a series of measurements where the sample container was taken out and put back into position, turned by 180°, was satisfactory, a repetition of 5 samples led to a random uncertainty of < 0.15%.

For the final evaluation of the ²³⁵U enrichment, it is necessary to determine the Gd content in the sample to accurately calculate the respective correction factor. A first value can be obtained from the L-edge data by using the extrapolated and the non-extrapolated results. However, this is only an indirect measure, as a difference in the 2 values only points to a general difference in matrix composition relative to the standard conditions, but no direct measurement of Gd itself is made. This can be achieved, for example, by an additional XRF measurement. First tests were done using a modified sample holder in the L-edge setup. Here, the measurement geometry required the covering of the sample with a thin foil to avoid spilling of the liquid. This led to problems with absorption effects and unsatisfactory reproducibility of the results.

Thus, a new independent unit for XRF measurements was designed, using the same type of X-ray tube and Si drift detector as for the L-edge setup. These components are typically taken in-field as spare parts, so any additional equipment is limited to the small aluminium holder that fits the X-ray tube, detector and sample container. The XRF setup is schematically shown in figure 4, together with an X-ray spectrum obtained from a Gd-containing uranium solution (10% Gd). The setup was designed in such a way that a sample

container with a liquid sample can be positioned in an upright geometry. This has the advantage that the container can be left open during the measurement, minimizing absorption effects in the X-ray beam paths (only air between X-ray tube, sample and detector).

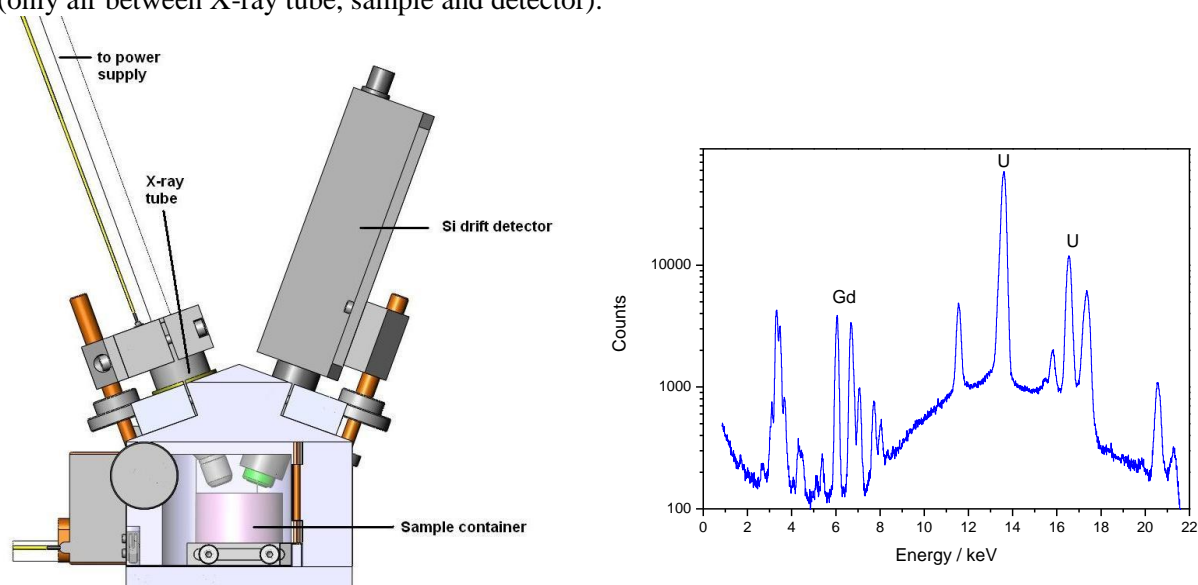


Figure 4: Setup for XRF (left) and spectrum obtained from a 10% Gd-containing uranium (190 g/l) solution (right), the main lines of U and Gd are indicated.

The setup also offers the possibility to perform XRF measurements directly on solid samples. A set of Gd-containing UO₂ pellets with different Gd concentration (2%, 4%, 6% and 8%) were measured. The ratio between the two L_{α1} peak areas of Gd (6.06 keV) and U (13.61 keV) showed a linear behaviour with increasing Gd content, so a calibration was performed, which can give a first estimation of the Gd content in an unknown sample. More accurate results are expected from the use of solutions, a detailed study is still ongoing.

5. Conclusion

COMPUCEA is a compact and transportable system which allows high-accuracy uranium elemental assay and enrichment determination starting from solid uranium samples. The 2nd generation COMPUCEA avoids radioactive sample transport, does not need transport of radioactive sources and attains excellent accuracy with an easily portable system. It is routinely applied in physical inventory verification campaigns at European LEU fuel fabrications plants. The 2nd generation system with a compact L-edge densitometer and LaBr₃(Ce) scintillation detection has been evaluated and validated, the performance is well within the International Target Values. New developments include a sandwich detector configuration with two LaBr₃ detectors for improved counting efficiency and higher sample throughput. A COMPUCEA demonstration exercise together with IAEA inspectors will be performed in a LEU fuel fabrication plant outside Europe (at Ulba Metallurgical plant, Kazakhstan) to evaluate the use of COMPUCEA for Safeguards purposes outside the EU.

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