

TABLE 4a – Uranium Element Mass Fraction Measurements (DA)

NOTE: Description of the methods applicable for Table 4a: TITR, MC ICPMS, HKED, KEDG and XRF.
Description of GRAV and IDMS methods are in preparation.

Method/measurement system (including software): [Davies and Gray titration method \(TITR\)](#)

Application: [Measurement of uranium amount content](#)

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)
The modified Davies and Gray titration method is probably the most widely used analytical method for the potentiometric titration of uranium for determination of uranium amount content (U mass fraction or U assay) in nuclear materials. The method is widely applied to various uranium containing compounds - oxides, fluorides, nitrides metals, alloys, yellow cakes, carbides, scraps, mixed oxides etc. Nuclear material samples need to be dissolved and various dissolution procedures can be applied depending on the material type. Prepared stock solutions of known concentration are subsampled into aliquots by weighing, typically containing 20-60 mg of uranium. The amount of uranium in aliquots might be dependent on the concentration of titrant used and can be limited by the size of the burette that is used for accurate delivery of the titrant solution. Potassium dichromate of known amount content is used as titrant (NIST SRM136); its amount content is determined from NBL CRM 112-A (U metal) or other certified reference material with certified value with relative expanded uncertainty ($k=2$) $\leq 0.05\%$. The method is based on the reduction of U(VI) to U(IV) and subsequent redox titration of the U(IV) with potassium dichromate solution. First quantitative reduction of uranium (VI) to uranium (IV) is done in concentrated phosphoric acid medium by reaction with iron (II) in excess. The excess of iron (II) is subsequently oxidized to iron (III) by nitric acid in the presence of sulphamic acid and catalyzed by molybdenum (VI). Uranium (IV) is determined by redox titration with standardized potassium dichromate solution employing vanadium (IV) for enhanced endpoint detection. There are number of reactants to be added before the titration is started, and the timing of these additions also plays a role. In order to render the titration less dependent on human factors and with the aim of reaching high precision, a computer-controlled system set-up for titrant additions by weight (equipped with a micro-dispenser, capable of delivering the titrating solution by equal increments of 20 mg or less) and titration end point detection is recommended. Interference from other elements is low: fluoride, perchlorate, sulfate, Be, Si, Nb, Ti, Cr, Fe, Co, Ni, W, Cu, Sb(V), Pb, Pu, Np, Am, the rare earths and the alkaline earth metals do not interfere. Nitrate and peroxide will not interfere unless present in higher than normal (1 mol/L) concentrations.
Factors affecting the method performance
The main factors impacting the performance of the modified Davies and Gray titration depends on the technique used (manual or automatized), expertise of the analyst, equipment used and the experimental conditions in the laboratory. Typical repeatability and reproducibility standard deviation of 0.1% can be achieved in well controlled laboratory conditions. Major contributor to measurement uncertainty obtained by using the modified Davies and Gray method is dominated by weighing uncertainty - the mass of the sample solution (mass of the aliquot taken from stock solution) - the mass of the nuclear material sample that was dissolved to prepare sample stock solution- the determination of the endpoint detection. Additional sources of uncertainties might be dependent on environmental conditions (temperature variability or system performance) or sample characteristics (homogeneity of the sample and presence of

impurities¹). It is therefore recommended to apply systematic monitoring of the measurement performance and apply well defined QA/QC procedures using matrix matched certified reference materials as quality control samples.
Source of top-down value and ITV applicability
The ITVs were evaluated using a top-down approach. Evaluation was based on historical paired measurement data obtained from the Agency's measurements database. Data evaluation methodology included the following steps: data quality check, outlier detection and removal, application of the Analysis of Variance with Random Effects for the differences in paired data, calculation of random $u(r)$ and short-term systematic $u(s)$ uncertainties. The ITVs are applicable for measurements taken: for safeguards purposes, in safeguarded facilities, with measurement procedure and instrument specification equivalent to the one described in the "Short description" and the "Factors affecting the method performance".
Source of bottom-up value (only for prospective ITV not based on historical data)
N/A
Method references
1. W. Davies, W. Gray. Talanta, 11, 1203, 1964. Page 13 of 14 2. E. Kuhn, S. Deron, H. Aigner, A. Zoigner. Destructive Analysis of Safeguarded Materials. The NBL-potentiometric titration of uranium. Experience of the Safeguards Analytical Laboratory. IAEA/RL/62.

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¹ the amount content of Cr(VI) in the potassium dichromate titrating solution is also a significant source of uncertainty even if it has been prepared by weighing and verified against U CRM and its durability is also questionable if it is not properly stored.

Method/measurement system (including software): Multi-Collector Inductively Coupled Plasma Mass Spectrometry (MC ICPMS)

Application: Measurements of uranium and plutonium isotope ratios and assay in nitrate solutions of U and Pu and in dissolved solid samples (e.g., metals, oxides, mixed oxides)

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)

The first magnetic sector ICPMS with a multi-collector array was introduced in 1992 [1]. This instrument is dedicated for precise isotope ratio measurements for a large range of applications including the nuclear field. The source of this instrument is an argon plasma source (ICP) with the capacity to ionize with high efficiency all the elements of the periodic table. The energy dispersion of the source must be matched to that of the magnetic sector and this is done using an electrostatic filter located between the source and the magnet (double focusing mass spectrometer). A collision reaction cell can also be included on the most recent generation of MC ICPMS instruments. After separation of the ions, the detection is performed using a multi-collector system including Faraday cups and ion counters. Multiple collection allows the simultaneous measurement of the different isotopes of an element, thereby removing source instability.

The samples are introduced in liquid form in 0.3 M nitric acid at concentration around 200 ng.g^{-1} using a conventional introduction system, including a nebulizer associated to a nebulization chamber. Alternatively, a de-solvating nebulizer may be employed. Detector calibrations, included in the software of the instrument, are performed each day. Each analysis is preceded by a washout of around 5 to 10 minutes. All sample measurements must be associated with the measurement of a reference material in order to take into account the mass -dependent fractionation ("mass bias effect") inherent to ICPMS techniques. For each sample or standard, measurement times were around 10 minutes.

The technique is also used for U and Pu isotopic ratios and for the determination of U and Pu assay using isotopic dilution (ID-MC ICPMS) in the same way as thermal ionization mass spectrometry (TIMS).

Factors affecting the method performance

A major factor affecting method performance is isobaric interference including ^{238}U - ^{238}Pu and ^{241}Pu - ^{241}Am . Before measurements, except for pure U solutions, uranium and plutonium must be purified, in the majority of cases by liquid chromatography, in order to avoid isobaric interferences or matrix effects.

Concerning instrumental performance, due to mass bias effect, isotope ratios must be corrected using certified isotopic reference materials. In nuclear samples the correction is performed using sample standard bracketing (SSB) technique. Each sample is bracketed by certified isotopic standards and the average mass bias correction factor, calculated on these two standards, is applied to the sample. This correction is the major source of uncertainty on isotope ratio measurements, since it includes the uncertainty on the reference material used. All other sources of uncertainties including gain calibration, blank corrections, and counting statistics must also be taken into account.

Source of top-down value and ITV applicability

The ITVs were evaluated using a top-down approach. Evaluation was based on historical paired measurement data obtained from the Agency's measurements database. Data evaluation methodology included the following steps: data quality check, outlier detection and removal, application of the Analysis of Variance with Random Effects for the differences in paired data, calculation of random $u(r)$ and short-term systematic $u(s)$ uncertainties.

The ITVs are applicable for measurements taken: for safeguards purposes, in safeguarded facilities, with measurement procedure and instrument specification equivalent to the one described in the “Short description” and the “Factors affecting the method performance”.
Source of bottom-up value (only for prospective ITV not based on historical data)
N/A
Method references
[1] Walder A.J.et Freedman P.A. (1992) Isotopic ratio measurement using a double focusing magnetic sector mass analyzer with an inductively coupled plasma as an ion source. JAAS, 7, pp. 571-575.

Prepared by: Helen Isnard

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Method/measurement system (including software): HKED / KEDG / XRF**Application:** Measurement of Uranium and Plutonium in Solutions

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)

K-Edge Densitometry (KEDG), X Ray Fluorescence (XRF), and their combination, Hybrid K-Edge (HKED), are techniques used to determine uranium and plutonium mass fractions or concentrations in dissolver, product, secondary flow, and waste solutions related to fuel reprocessing. A single measurement system, comprising of two High-Purity Germanium (HPGe) detectors, an X ray generator, shielding, collimators, data acquisition electronics, and computer, is typically deployed and capable of all three methods [1]. All three methods are rapid, non-destructive assay techniques, though they are typically listed among destructive assay techniques due to the low uncertainty of KEDG (< 1%). Samples are typically assayed with three (3) replicate measurements, each for a time of 1000 to 3000 s.

The KEDG method measures the X ray transmission discontinuity across K-edge energies for an absolute elemental concentration determination in highly concentrated uranium and plutonium solutions (> 50 g U per liter or > 50 g Pu per liter).

The XRF method determines the absolute elemental concentrations in pure or mixed solutions of uranium and plutonium in the range of 1 to 50 g per liter by quantifying the fluorescent X rays emitted due to excitation by the generator X rays.

The HKED method determines simultaneously uranium and plutonium in solutions where the uranium has higher concentrations, as in the case of dissolver solutions. The XRF portion of HKED quantifies the uranium to plutonium (U/Pu) ratio, and the KEDG portion measures the uranium concentration. The combination of two methods determines the absolute plutonium concentration. Typical uranium concentrations and U/Pu ratios are in the ranges of 150 to 250 g U per liter and 80:1 to 150:1, respectively.

HKED systems are installed primarily in on-site analytical laboratories associated with spent fuel reprocessing facilities.

Factors affecting the method performance

The main factors impacting measurement precision and bias of all three methods include the following [1]: counting statistics, X ray generator stability, detector and associated electronics stability, sample homogeneity, sample matrix, sample isotopic abundances, sample container dimensions, sample positioning, and sample temperature.

Some of these factors, such as sample temperature and sample isotopic abundances, are corrected for directly in the analysis software. X ray generator stability and sample positioning are typically monitored to be within certain control criteria for every assay or on a periodic basis. Detector electronics stability is also monitored and corrected for by using an in-situ energy calibration check source for each measurement. The radioactive decay of certain check sources, such as Cd-109, can also affect background evaluation in some KEDG algorithms over time. Its decay rate at the time of installation and sample measurement should be considered during the measurement evaluation. Sample container dimensions are typically measured for each container or monitored by a random sampling of a batch of containers.

Some factors, such as homogeneity and matrix effects, are specific to certain sample types and methods. For instance, high concentration Pu samples measured by KEDG should be performed as quickly as possible due to bubble formation and changing concentration from radiolysis effects. Samples containing suspended particles such as high active liquid waste need homogenization and correction factors, as the XRF calibrations are typically made with clean solutions. The XRF calibrations are also typically made with pure uranium or pure plutonium solutions, and mixed samples may need correction factors to account for attenuations of both the interrogating and fluorescent X rays. Similarly, hybrid-XRF calibrations are typically made with mixed solutions containing no fission products. When present in dissolver solutions,

fission products can change the background subtraction analysis and induce additional fluorescence not present in the calibration measurements. Corrective measures such as subtracting an additional passive spectrum measurement without the generator X rays or performing improved background and peak fitting algorithms may be employed.

For uranium assay using KEDG and HKED for the typical three replicate measurements (each of counting time between 1000 s and 3000 s), the ITV values are only valid for concentration levels above 150 g U per liter. When using the HKED technique for dissolved MOX pellets and powders, the uncertainties are typically higher than the ITV values. This is due to lower U/Pu ratios and lower uranium concentrations compared to dissolver solutions. Elemental concentrations below 1 g per liter can be measured by XRF, though with longer counting times and uncertainties larger than the ITV values.

Source of top-down value and ITV applicability

The ITVs were evaluated using a top-down approach. Evaluation was based on historical paired measurement data obtained from the Agency's measurements database. Data evaluation methodology included the following steps: data quality check, outlier detection and removal, application of the Analysis of Variance with Random Effects for the differences in paired data, calculation of random $u(r)$ and short-term systematic $u(s)$ uncertainties.

The ITVs are applicable for measurements taken: for safeguards purposes, in safeguarded facilities, with measurement procedure and instrument specification equivalent to the one described in the "Short description" and the "Factors affecting the method performance".

Source of bottom-up value (only for prospective ITV not based on historical data)

N/A

Method references

[1] H. Ottmar and H. Eberle, "The Hybrid K-Edge/K-XRF Densitometer: Principles – Design –Performance," Report KfK 4590, Karlsruhe, 1991.

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Method/measurement system (including software): The measurements are performed with the Total Evaporation-Thermal Ionization Mass Spectrometry method described for Table 5a and 6a using Multi-Collector Thermal Ionization Mass spectrometer.

Application: Measurement of plutonium and uranium mass fraction in solution samples and in dissolved solid samples (e.g., metals, alloys, oxide powders, oxide pellets, mixed oxides)

Short Description of the method/measurement system and typical measurement conditions (shielding, measurement time, etc.)

Isotope dilution mass spectrometry^[1] (IDMS) has been applied as a reliable analytical technique for accountancy measurements of uranium and plutonium contents in dissolved fuel when high accuracy is needed. Reference materials well characterized for their isotopic composition and their isotope content of uranium and plutonium called large-sized dried spikes (LSD) are used. Such spikes preferentially contain a high enrichment of an isotope that is not the main isotope of the element being measured in the sample. Typically, highly enriched ²³⁵U and ²³⁹Pu and certified reference metals with low uncertainty are used as starting materials for the preparation of LSD spikes. In addition, to avoid dilution steps for dissolver solutions in reprocessing plants these spikes contain milligram rather than microgram quantities of nuclear material in the dried form, often for stability and integrity covered with an organic material^[2]. Test samples are added to LSD spike vials^[3,4,5], dissolved and mixed to assume complete equilibration of spike with the analyte. Aliquots of the final solution are diluted and subjected to chemical separation^[6] and finally measured by Total Evaporation Thermal Ionization Mass Spectrometry TE-TIMS^[7]. With the isotopic composition of the test sample, the isotopic concentrations of the LSD spikes, weights of the sample and spike weight, the mass fraction of uranium and plutonium in the sample can be calculated^[3, 4, 5].

The key measurement steps and their purposes are outlined below:

- Test samples are typically dissolver solutions but also dissolved solid samples can be used.
- One aliquot has to be used to characterize the isotopic composition of the test sample.
- Another aliquot of the test sample is added to LSD spike vial by weighing. The mixture is heated for several hours to be assure that the LSD spike is dissolved, and aliquoted for further analysis.
- Uranium-plutonium mixtures of pure sample and sample-spike mixtures have to be separated and isolated, to perform U and Pu measurement by TE-TIMS. Therefore, the test samples are loaded onto evaporation filaments (Re or W) and to measure isotope amount ratios of U and Pu. For details see narrative of TE-TIMS and^[7].
- The mass fraction of U or/and Pu in the sample can be calculated using the isotopic composition of the test sample, the isotopic composition of the mixture and the isotopic composition and mass of the LSD spike and the test sample^[1].
- For quality assurance purposes suitable certified or well characterized laboratory reference materials are used as quality control samples.

Factors affecting the method performance
The primary factors impacting IDMS measurement precision and bias are: - Accurate measurement of mass quantity values when preparing test samples for measurement; - Precise determination of the isotopic composition of the test samples; - Use of well certified reference material or well characterized laboratory reference material (LSD); - Precise determination of the isotopic composition of the mixture; - Ensuring measurement control using traceable, uranium and plutonium quality-control reference materials; As direct use of the dissolver samples is possible, dilution steps can be avoided prior to mix it with the spike material, which results in lower uncertainties compared to the use of small spikes.
Source of top-down value and ITV applicability
The ITVs were evaluated using a top-down approach. Evaluation was based on historical paired measurement data obtained from the Agency's measurements database. Data evaluation methodology included the following steps: data quality check, outlier detection and removal, application of the Analysis of Variance with Random Effects for the differences in paired data, calculation of random $u(r)$ and short-term systematic $u(s)$ uncertainties. The ITVs are applicable for measurements taken: for safeguards purposes, in safeguarded facilities, with measurement procedure and instrument specification equivalent to the one described in the "Short description" and the "Factors affecting the method performance".
Source of bottom-up value (only for prospective ITV not based on historical data)
N/A
Method references
[1] Quemet, Alexandre; Hubert, Amélie; Gourgiotis, Alkiviadis; Sanchez Hernandez, Ana Maria; Crozet, Marielle; Bailly, Guillaume; Dobney, Andrew; Duhamel, Georges ; Hiess, Joe; Repinc, Urška; Mialle, Sébastien; Boulet, Béatrice; Escoubé, Raphaëlle; Bouvier-Capely, C.; Pointurier, Fabien; Picart, Sébastien, An Isotope Dilution Mass spectrometry overview: tips and applications for the measurement of radionuclides. Journal of Analytical Atomic Spectrometry - JA-CRV-01-2024-000029.R1 [2] Toth K, Jakopič R, Bujak R, Bauwens J, Hennessy C, Kehoe F, Jakobsson U, Richter S, Aregbe Y, Preparation and Certification of Large-Size Dried (LSD) Spike: IRMM-1027v, EUR 30759 EN, 2021. http://dx.doi.org/10.2760/59260 [3] Rožle Jakopič, Kalman Toth, Jeroen Bauwens, Renata Buják, Carmel Hennessy, Frances Kehoe, Ulf Jakobsson, Stephan Richter, Yetunde Aregbe, 30 years of IRMM-1027 reference materials for fissile material accountancy and control: development, production and characterization. Journal of Radioanalytical and Nuclear Chemistry 330:333–345, 2021. [4] P. van Belle, E. Zuleger and L. Duinslaeger, An improved self-checking weighing practice and a method for contamination detection for use in IDMS nuclear material assay, Proceedings of the 49th INMM Annual Meeting, Tennessee, p 683-691, 2008. [5] Jakopič, R., Aregbe, Y., Richter, S., Zuleger, E., Mialle, S., Balsley, S. D., Repinc, U., Hiess, J.; <i>Verification Measurements of the IRMM-1027 and the IAEA Large-Sized Dried (LSD) Spikes</i>; J Radioanalytical and Nuclear Chemistry 311:1781-1791, 2017. [6] Morgenstern, A., Apostolidis, C., Carlos-Marquez, R., Mayer, K., Molinet, R.; <i>Single-column extraction chromatographic separation of U, Pu, Np and Am</i>; Radiochimica, vol. 90, p. 81, 2002.

[7] M. Romkowski, S. Franzini, L. Koch, Mass-Spectrometric analysis of sub-nanocurie samples of Uranium and Plutonium, in: Proceedings of the 8th Annual ESARDA Symposium, 1987, pp. 111-113.

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Method/measurement system (including software): The measurements are performed with the Total Evaporation -Thermal Ionization Mass Spectrometry method described for Table 5a and 6a.

Application: Measurement of plutonium and uranium mass fraction in solution samples and in dissolved solid samples (e.g., metals, alloys, oxide powders, oxide pellets, mixed oxides)

Short Description of the method/measurement system and typical measurement conditions

Isotope Dilution mass spectrometry using Thermal Ionization Mass Spectrometry (ID-TIMS) has been widely applied to analysis of safeguards nuclear material samples due to its high achievable precision and accuracy for isotope ratio measurements of uranium and plutonium. In most cases, sample dissolution procedures need to be applied to prepare a diluted test solution of a concentration, that is suitable for further analysis ^[1,2]. Isotope amount ratio measurement often needs to be performed on separate sample aliquots to determine the isotopic composition of the sample and chose the suitable spike isotope to use for isotope dilution analysis. As the mass fraction of the element (concentration of U or Pu) in the sample is unknown, aliquots of prepared sample solution are taken to be mixed with weighed amount of spike solution, that has well characterized isotope abundances and spike isotope concentration. As spike materials used often contain mg- to µg-amounts of elements, the naming of small size spikes was introduced. After the sample-spike mixture (blend) is equilibrated, losses of analyte do not affect the accuracy of the procedure. Therefore, blends can be subjected to extended sample preparation steps, to remove the matrix and isolate analytes of interests ^[3,4]. Analyte isolation steps for sample and blend aliquots are often mandatory to assure that Total Evaporation (TE) TIMS measurements remain largely unaffected by the sample matrix.

The key measurement steps and their purposes are outlined below:

- Weighed amount of the sample is dissolved to prepare a homogenous solution for further analysis.
- Aliquots are taken to be able to determine the isotopic composition of the sample.
- Weighed amount of either a commercially available certified spike material containing enriched spike isotope with suitable analyte concentration, or a well-characterized working reference material are added to separate aliquots of the test sample. Spikes are chosen to preferably contain high abundance of an isotope that is not present in a sample. Most commonly liquid spikes contain ²³⁵U, ²³³U and ²⁴⁰Pu, ²⁴²Pu (or even ²⁴⁴Pu) isotopes in high abundance, and are commonly applied in IDMS analysis of nuclear materials using small size spikes.
- Complete equilibration of the sample-spike mixture.
- Both un-spiked and spiked aliquots have to be chromatographically separated, to collect the U and Pu analytes separately and remove ²⁴¹Am ^[3,4]. For TE-TIMS measurements ^[5,6], small fractions of processed sample aliquots are loaded onto evaporation filaments (Re or W) for isotopic amount ratio measurements.
- Using the isotopic composition of the test sample, blend and the certified value of the spike, the amount of test sample and the amount of the spike added to the test sample, the mass fraction of the element can be calculated.
- Suitable certified or well characterized laboratory reference materials are used as quality control samples, for measurement, processes control and contamination control.

Factors affecting the method performance
The main sources impacting ID-TIMS method precision and bias are: - Weighing uncertainties (buoyancy, electrostatic charge, moisture content, stability of balances used) - Isotope ratio measurements of the sample and the blend (interferences, mass discrimination effects, contamination, abundance sensitivity) - Incomplete digestion, incomplete equilibration of sample-spike mixture, presence of interferences influencing mass spectrometry measurements or incomplete isolation of the analyte causing isobaric interferences, contamination effects. - Uncertainties of certified reference materials or well characterized laboratory reference materials, used as small-size spikes or measurement standards.
Source of top-down value and ITV applicability
The ITVs were evaluated using a top-down approach. Evaluation was based on historical paired measurement data obtained from the Agency's measurements database. Data evaluation methodology included the following steps: data quality check, outlier detection and removal, application of the Analysis of Variance with Random Effects for the differences in paired data, calculation of random $u(r)$ and short-term systematic $u(s)$ uncertainties. The ITVs are applicable for measurements taken: for safeguards purposes, in safeguarded facilities, with measurement procedure and instrument specification equivalent to the one described in the "Short description" and the "Factors affecting the method performance".
Source of bottom-up value (only for prospective ITV not based on historical data)
N/A
Method references
[1] De Bièvre P, (1993) Isotope dilution mass spectrometry as a primary method of analysis. Anal Proc 30:328–333. [2] Sánchez Hernández AM, Andor C, Buda R, Commin L, d'Amati F, Horta Domenech J, Morel S, Nicholl A, Nourry C, Suurkuusk G, Vargas Zuñiga M, Wojnowski D, Zuleger E (2019) Optimising the use of reference materials for destructive analysis in nuclear safeguards. In: Proceedings of 41st ESARDA symposium, 188-199, 10.2760/159550. [3] Morgenstern A, Apostolidis C, Carlos-Marquez R, Mayer K, Molinet R, (2002) Single-column extraction chromatographic separation of U, Pu, Np and Am; Radiochimica, vol. 90, p. 81. [4] DMRC/CETAMA/NT/DO/2020/11/A (2020) Méthode CETAMA 397: Purification par chromatographie de U et de Pu en amont des analyses par Spectrométrie de Masse », Sous-groupe « Méthode » du GT03 « Analyses des actinides », diffusée le 28/07/2020. [5] Romkowski M, Franzini S, Koch L, (1987) Mass-Spectrometric analysis of subnanocurie samples of Uranium and Plutonium, in: Proceedings of the 8th Annual ESARDA Symposium, pp. 111-113, London. [6] C1672–17: Standard test method for determination of uranium and plutonium isotopic composition or concentration by total evaporation method using a thermal ionization mass spectrometer, ASTM International.

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