



**International
Standard**

ISO 6863

**Nuclear fuel technology —
Preparation of spikes for isotope
dilution mass spectrometry (IDMS)**

*Technologie du combustible nucléaire — Préparation de traceurs
pour les analyses par spectrométrie de masse avec dilution
isotopique (IDMS)*

**First edition
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Foreword

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Nuclear fuel technology — Preparation of spikes for isotope dilution mass spectrometry (IDMS)

1 Scope

This document specifies a method which applies to the preparation and validation of the standard materials generally called “large size spikes” with an uncertainty suitable for international nuclear safeguards used for measuring the content of plutonium and/or uranium by isotope dilution mass spectrometry.

This measurement methodology can be applied to input solutions of irradiated Magnox and light water reactor fuels (boiling water reactor or pressurized water reactor); in final products at spent-fuel reprocessing plants; in feed and products of mixed oxide of plutonium and uranium (MOX); and in uranium fuel fabrication.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 8299, *Nuclear fuel technology — Determination of the isotopic and elemental uranium and plutonium concentrations of nuclear materials in nitric acid solutions by thermal-ionization mass spectrometry*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

solution spike

nitric acid solutions with accurately quantified uranium and/or plutonium content and isotopic composition

Note 1 to entry: One of the reference materials for IDMS.

3.2

dried spike

prepared by aliquoting *solution spikes* (3.1) into glass vessels and then drying

Note 1 to entry: One of the reference materials for IDMS.

3.3

large-sized dried spike

LSD spike

dried spike containing both uranium and plutonium in milligram size, were originally developed to analyse high concentrated input solution

Note 1 to entry: One of the reference materials for IDMS.

Note 2 to entry: The LSD spikes can simplify the sample preparation process with only one spiking step to the sample, which also contains uranium and plutonium, and reduce measurement uncertainty by less dilution factor than smaller sized spikes.

4 Principle

Element content measurement using thermal ionization mass spectrometry (TIMS) is made on a sample and mixture of the sample and a spike, consisting of an enriched isotope of the element to be analysed and determine element content by calculating the difference of isotopic composition before and after spike mixture. This method of measuring an element's content is called isotope dilution mass spectrometry (IDMS).

The isotopic compositions of the sample and spike is required to be significantly different. Therefore, it is desirable that spikes are composed of isotopes that are not present, or only minimally present, in the unspiked sample. It is necessary that the isotopic composition and the content of spikes be known or measured accurately and has small uncertainties because it reflects to the uncertainty of the final results, element content.

Chemically-pure compounds of separated plutonium or uranium isotopes are dissolved to prepare stock solutions of spikes, in general terms, in 3 mol/l to 7 mol/l nitric acid to obtain an optimized content based on its design to obtain reliable results. Aliquots of the plutonium and uranium spike stock solutions can be mixed to prepare mixed spikes. Aliquoted spikes are used for IDMS in solution state or after dried.

5 Design of spikes

As the uncertainty to be considered in determining the reliability of nuclear fuel material analysis techniques for safeguards, International Target value (ITV)^[1] is decided for IDMS. It is desirable that spikes are used for IDMS after being optimized for the sample to be analysed. Different target values are set depending on the environment to be measured and spikes used. Spikes shall be designed so that measurement by IDMS can achieve the appropriate ITVs.

There are two types of spikes: solution spikes and dry spikes. Basically, either spike is fine. In general, choose which spike to prepare according to user needs.

5.1 Optimization of spikes

In IDMS, when the isotopic composition in the unknown sample and in the spike are significantly different, the measurement accuracy become higher. On the other hand, the isotopic composition of available certified reference materials for preparing spikes are limited. Therefore, it is important to design the plutonium and/or uranium composition of spikes by evaluating, in advance, the isotopic composition and the amounts of elements of spikes that can be obtained sufficient accuracy. The following sentences describe an example of calculations for optimizing spikes.

The following [Formula \(1\)](#) can be obtained by partially differentiating the theoretical equation of IDMS with sample to spike mass ratio, p ^[2].

$$\begin{aligned} \left[\frac{\sigma(p)}{p} \right]^2 &= \frac{1}{p^2} \cdot \frac{(1+p)^2 (1+\gamma KR_T)^2}{(R_S - R_T)^2 (1+\gamma KR_S)^2} \cdot \varepsilon^2 R_S^2 + \frac{(1+p)^2 (1+\gamma KR_S)^2}{(R_S - R_T)^2 (1+\gamma KR_T)^2} \cdot \varepsilon^2 R_T^2 \\ &+ \frac{1}{p^2} \cdot \frac{[pR_T (1+\gamma KR_S) + R_S (1+\gamma KR_T)]^2 [p(1+\gamma KR_S) + (1+\gamma KR_T)]^2}{(R_S - R_T)^2 (1+\gamma KR_T)^2 (1+\gamma KR_S)^2} \cdot \varepsilon^2 \\ &+ \frac{\gamma^2 (R_T - R_S)^2}{(1+\gamma KR_T)^2 (1+\gamma KR_S)^2} \cdot \sigma_K^2 \end{aligned} \quad (1)$$

where

- $\sigma(p)$ is the standard deviation of p ;
- R_S is the isotopic ratio of sample;
- R_T is the isotopic ratio of spike;
- γ is absolute mass ratio of second to first major isotope;
- K is mass discrimination factor;
- ε is standard deviation of for isotopic ratio measurement.

By replace p to sample to spike number of atoms ratio, q and putting γ equal to unity. If K equal to 1, [Formula \(1\)](#) is described as following [Formula \(2\)](#):

$$\left[\frac{\sigma(q)}{q} \right]^2 = \frac{1}{q^2} \cdot \frac{(1+q)^2 (1+R_T)^2}{(R_S - R_T)^2 (1+R_S)^2} \cdot \varepsilon^2 R_S^2 + \frac{(1+q)^2 (1+R_S)^2}{(R_S - R_T)^2 (1+R_T)^2} \cdot \varepsilon^2 R_T^2 + \frac{1}{q^2} \cdot \frac{[qR_T(1+R_S) + R_S(1+R_T)]^2 [q(1+R_S) + (1+R_T)]^2}{(R_S - R_T)^2 (1+R_T)^2 (1+R_S)^2} \cdot \varepsilon^2 \quad (2)$$

Where, the value " $[\sigma(q)/q]^2/\varepsilon^2$ " means the size of the error depending on the mixing ratio of spike and sample, and from this, the amount of spike to sample and/or isotopic ratio is calculated and able to be optimized.

5.2 Uncertainty of spikes

The uncertainty of spikes can be combined from the Certified Reference Material (CRM), analytical balance, and also from the equipment and tracer used for instrumental analysis for the purpose of characterization or standardization. These individual uncertainties should be combined and should be given as expanded uncertainty when the coverage factor $k = 2$. The uncertainty can be calculated in accordance to the ISO/IEC Guide 98-3^[3].

6 Reference materials and reagents

The reagents listed below are prepared from analytical grade reagents unless it is specified otherwise.

6.1 Reference materials

Examples of reference material for preparing spike are shown below.

6.1.1 Uranium reference materials

- a) natural uranium metal CRM of purity with an elemental content certified to $\pm 0,05\%$ ($k = 2$) or better, such as NBL-CRM-112A (e.g. NBS-960D), EC-101, CETAMA-MU2.
- b) other uranium metal, powder or pellet CRM of purity with an elemental content certified to $\pm 0,05\%$ ($k = 2$) or better, such as NBL-CRM-116A (HEU metal), CRM-125A (UO₂ pellet) and CRM-129 (U₃O₈ powder). CRM solution can also be available as a source. Abundance of the major isotope of uranium shall be greater than or equal to 80 %.

6.1.2 Plutonium reference materials

Plutonium metal CRM with an elemental content certified to $\pm 0,05\%$ ($k = 2$) or better, such as NBL-CRM-126 or 126A, EC-201 or CETAMA-MP2. Abundance of the major isotope of plutonium shall be greater than or equal to 80 %.

6.1.3 Secondary standard materials

For both plutonium and uranium, secondary standard materials in solution or in solid and traceable to CRMs or verified by means of a laboratory intercomparison can also be used. The values should be certified to $\pm 0,05\%$ ($k = 2$) or better.

6.2 Other chemical reagents

The reagents listed below are in analytical grade and other reagents could also be used if results are confirmed to be equivalent.

6.2.1 Nitric acid solutions, $c(\text{HNO}_3) = 1 \text{ mol/l}$, 3 mol/l , 4 mol/l , 7 mol/l to 8 mol/l and other.

6.2.2 Hydrochloric acid solutions, $c(\text{HCl}) = 0,1 \text{ mol/l}$, 3 mol/l , 4 mol/l , 12 mol/l and other are applicable for dissolution and etching for uranium metal and/or plutonium metal.

6.2.3 Hydrofluoric acid, $c(\text{HF}) = 27 \text{ mol/l}$. HF can mix with nitric acid solution before use.

WARNING — Hydrofluoric acid is strongly corrosive. Safety precautions are necessary.

6.2.4 Potassium carbonate solution, $c(\text{K}_2\text{CO}_3) = 1,45 \text{ mol/l}$.

6.2.5 Water, complying with at least grade 1 in accordance with ISO 3696.

6.2.6 Ethanol.

6.2.7 Acetone.

7 Apparatus

7.1 Apparatus for mass spectrometry, shall be in accordance with ISO 8299.

7.2 Glove boxes, for handling uranium and plutonium safely.

7.3 Analytical balance, capable of accurately weighing items to the nearest $0,1 \text{ mg}$ in a sealed cell or glove box. If the mass is 100 g or more, an accuracy of 1 mg is sufficient. Evaluate uncertainty based on ISO/IEC Guide 98-3^[3] before use.

7.4 Ionizer, to remove static electricity of vessel, is recommended to be installed in analytical balance room, a shielded cell or a glove box.

7.5 Pipet, volume adjustable with disposable pipette tips, installed in a shielded cell or a glove box.

7.6 Hot plate, capable of heating to at least $150 \text{ }^\circ\text{C}$ in a glove box to concentrate solutions or dissolving the standard reference materials. Parallel use with vapour condensing system is recommended. Heating block to have enough reflux in the vial is also recommended.

7.7 Dry-cell battery, capacity of at least $6 \text{ A}\cdot\text{h}$ for plutonium metal electro polishing^[4].

7.8 Platinum wire and crucible, for plutonium metal electro polishing^[4].

7.9 Stock bottle, is used as container for storing solution during aliquot.

7.10 Common laboratory ware, consisting of disposable plastic containers, flasks, watch glass, beakers, vials, and others.

NOTE It is recommended to clean glassware before use.

8 Preparation of spikes

There are several types of spikes applicable to perform IDMS. These are the major spikes used for analysing plutonium and/or uranium in the samples.

8.1 Large-sized dried (LSD) spikes

The general method of preparing LSD spikes is as follows:

- a) Uranium (6.1.1) and plutonium (6.1.2) CRMs are dissolved to prepare respective stock solutions.

NOTE Secondary reference materials (6.1.3) which are traceable to CRM can also be used as starting material of LSD spikes. Guidance for the preparation of secondary reference materials can be found in ASTM C1128^[5].

- b) Mixing stock solutions to be desired uranium content and plutonium content and preparing the mixed stock solution.

NOTE Uranium to plutonium ratio can be optimized according to 5.1.

NOTE As an alternative to methods a) to b), mixed stock solutions can also be prepared by adding one reference to the other stock solution and dissolving.

- c) Taking aliquots from mixed stock solution to vials and drying them.

The details of each step are explained in the following subparagraphs.

8.1.1 Preparation of uranium stock solution

Open a unit of a uranium certified reference material such as those mentioned in 6.1.1 and use it in accordance with the certificate. If not specified in the certificate, or if reagents specified in the certificate are not available, follow the steps below.

Secondary reference materials can also be used to prepare stock solution such as those mentioned in 6.1.3 and use it in accordance with the certificate.

- a) Rinse the uranium metal with water (6.2.5) and etch it in 7 mol/l to 8 mol/l nitric acid solution (6.2.1) or 3 mol/l hydrochloric acid solution (6.2.2) until the surface of the metal takes a uniform and bright metallic shine. Heat at around 125 °C on hot plate if necessary.
- b) Rinse rapidly with water (6.2.5), then with ethanol (6.2.6) or acetone (6.2.7) and dry quickly in air or in vacuumed desiccator at room temperature.
- c) Transfer the uranium metal into a tared vessel for dissolution. Conical flask is recommended.
- d) Immediately measure the gross mass of the vessel to 0,1 mg and calculate the mass, m_1 , of the uranium metal collected in the vessel.
- e) Add sufficient 7 mol/l to 8 mol/l nitric acid solution (6.2.1) to cover the metal into the vessel.
- f) Cap the vessel with a reflux head or a watch glass and start a gentle dissolution on hot plate.
- g) As the dissolution ceases, it is recommended to add 7 mol/l to 8 mol/l nitric acid solution (6.2.1) in small portions to maintain a gentle reaction.
- h) When the dissolution process is completed, wash the reflux head or the watch glass with 7 mol/l to 8 mol/l nitric acid solution (6.2.1) and collect all liquid into the vessel.

- i) Wait until the solution reaches thermal equilibrium.
- j) Dilute with water (6.2.5) and/or nitric acid solution (6.2.1) to obtain the desired volume of solution.
- k) Cap the vessel tightly and shake it to homogenize the uranium solution.
- l) Measure the gross mass of the vessel with cap to 0,1 mg and calculate the net mass of the uranium solution, m_2 . The mass fraction of uranium, ω_U , in milligrams per gram, in the stock solution is calculated using Formula (3):

$$\omega_U = \frac{m_1}{m_2} \cdot P_S \quad (3)$$

where, P_S is the purity, expressed as a mass fraction of uranium element (grams of uranium per gram of material), of the certified reference material reported on the certificate.

- m) Until the stock solution will be used, cap the vessel and leave to stand on a stable place.

8.1.2 Preparation of plutonium stock solution

Open a unit of a plutonium certified reference material such as those mentioned in 6.1.2 and use it in accordance with the certificate. If not specified in the certificate, or if reagents specified in the certificate is not available, follow the steps below.

Secondary reference materials such as those mentioned in 6.1.3 can also be used to prepare stock solution and use it in accordance with the certificate.

8.1.2.1 From the reference materials distributed in units of certified masses

- a) Record its mass, m'_1 , in milligrams.
- b) Transfer the plutonium metal into a tared and dry vessel. Conical flask is recommended.
- c) Immediately measure the gross mass of the vessel to 0,1 mg and calculate the mass, m''_1 , in milligrams, of the plutonium metal collected in the vessel; compare it to the certified value, m'_1 , to confirm the identity of the unit. If there is a discrepancy between m'_1 and m''_1 , evaluate the discrepancy before proceeding further.
- d) Cover the plutonium metal with 0,1 mol/l hydrochloric acid solution (6.2.2).
- e) Rinse or leach the vial or the ampoule several times with 1 ml aliquots of 0,1 mol/l hydrochloric acid solution (6.2.2) and collect the rinse into the vessel.
- f) Add 12 mol/l hydrochloric acid solution (6.2.2) and adjust the acid concentration to 4 mol/l.
- g) Cap the flask with a reflux head or a watch glass and start a gentle dissolution on hot plate.
- h) When the dissolution process is completed, wash the reflux head or the watch glass with 4 mol/l hydrochloric acid solution (6.2.2) and collect all liquid into the vessel.
- i) Wait until the solution reaches thermal equilibrium.
- j) Dilute with water (6.2.5) and/or nitric acid solution (6.2.1) to obtain the desired volume of solution.
- k) Cap the vessel tightly and shake it to homogenize the plutonium solution.
- l) Measure the gross mass of the vessel with cap to 0,1 mg and calculate the net mass of plutonium solution, m'_2 , in gram. The mass fraction of plutonium, ω_{Pu} , in milligrams per gram, in the stock solution is calculated using Formula (4).

$$\omega_{\text{Pu}} = \frac{m'_1}{m_2} \cdot P'_s \quad (4)$$

where, P'_s is the purity, expressed as mass fraction of plutonium element (grams of plutonium per gram of material).

m) Until the stock solution is used, should cap the flask and leave to stand on a stable place.

8.1.2.2 From the plutonium metal reference material which requires surface treatment

- a) Connect one platinum wire to plutonium metal and the other platinum wire to a platinum crucible.
- b) Put 1,45 mol/l potassium carbonate solution (6.2.4) in a platinum crucible, contact plutonium metal to 1,45 mol/l potassium carbonate solution (6.2.4) and connect two platinum wires to dry-cell battery.
- c) Add current 6 A·h for 2 min to 3 min if the sample is in 1 g to 2 g size.
- d) Remove the plutonium metal when it turned shiny.
- e) Rinse rapidly with water (6.2.5), then with ethanol (6.2.6) or acetone (6.2.7) and dry quickly in air or in vacuumed desiccator at room temperature.
- f) Transfer the plutonium metal into a tared and dry vessel. Conical flask is recommended.
- g) Immediately measure the gross mass of the vessel to 0,1 mg and calculate the mass, m'_1 , in milligrams, of the plutonium metal collected in the vessel.
- h) Add sufficient 7 mol/l to 8 mol/l nitric acid solution (6.2.1) and 1 drop or 2 drops of hydrofluoric acid (6.2.3) to cover the metal into the vessel.
- i) Cap the flask with a reflux head or a watch glass and start a gentle dissolution on hot plate.
- j) If there are still particles of the plutonium metal on bottom, add 7 mol/l to 8 mol/l nitric acid solution (6.2.1) and 1 drop or 2 drops of hydrofluoric acid again.
- k) When the dissolution process is complete, wash the reflux head or the watch glass with 7 mol/l to 8 mol/l nitric acid solution (6.2.1) and collect all liquid into the vessel.
- l) Wait until the solution reaches thermal equilibrium.
- m) Dilute with 1 mol/l, 3 mol/l, 4 mol/l, or 7 mol/l to 8 mol/l nitric acid solution (6.2.1) to obtain the desired volume of solution.
- n) Follow same procedure for 8.1.2.1 k) to m).

8.1.3 Preparation of mixed stock solution

The plutonium and uranium contents of mixed stock solution are calculated using the certified values, the masses of reference materials taken and the measured mass of the solution, including consideration of the traces of uranium contained in the plutonium certified reference material. The isotopic composition of the plutonium spike is taken from the certificate of the certified reference material.

The ^{235}U isotopic abundance and the isotope ratio $^{235}\text{U}/^{238}\text{U}$ are calculated using the certified values and the masses of reference materials taken, including consideration of the traces of uranium contained in the plutonium certified reference material. Continued in growth of uranium isotopes from plutonium alpha decay should also be considered if it significantly effects to the result.

If the remaining the stock solution need to store after use, wipe the inside of the cap and around the head of the vessel, then close the cap and measure the gross mass of the vessel.

8.1.3.1 Mixing with solutions

- a) Measure the gross mass of vessels containing stock solutions of uranium (8.1.1) and plutonium (8.1.2) to 0,1 mg.
- b) Compare the gross mass with final gross mass of vessels (8.1.1 l) and (8.1.2.1 l)) and calculate evaporation factor.

The evaporation factor, k , is calculated by dividing the net mass after storage (before use), M' , by the net mass before storage, M , as given by Formula (5):

$$k = \frac{M'}{M} \quad (5)$$

As shown in Formula (6), by multiplying the evaporation factor by the content of the solution before storage, C , the content after storage (before use), C' , is obtained.

$$C' = k \cdot C \quad (6)$$

- c) Confirm vessels capped tightly and shake them to homogenize solutions.
- d) Open the cap of the vessel containing the uranium stock solution and take aliquots to vials for validation of the uranium stock solution.
- e) Transfer the uranium stock solution from the vessel to a tared vessel and measure the gross mass with cap to 0,1 mg or to the lowest digit of the balance.
- f) Open the cap of the vessel containing the plutonium stock solution and take aliquots to vials for validation of the plutonium stock solution.
- g) Open the cap of the vessel containing the uranium solution, transfer the plutonium stock solution from the vessel to tared vessel and measure the gross mass with cap to 0,1 mg or to the lowest digit of the balance.

NOTE Order of transferring uranium solution and plutonium solution to vessel can reverse.

- h) Until the mixed stock solution will be used, should cap the vessel tightly and leave to stand on a stable place.

8.1.3.2 Adding uranium reference materials to the plutonium stock solution

- a) Measure the gross mass of vessels containing stock solutions of plutonium (8.1.2) to 0,1 mg.
- b) Compare the gross mass with final gross mass of the vessels (8.1.2.1 l)) and calculate evaporation factor according to Formula (5).
- c) Add uranium reference materials (6.1.1 or 6.1.3) to the vessel containing plutonium stock solution and dissolve.

NOTE1 If the uranium reference material is a metal, it can have been treated (e.g. 8.1.1 a) to b)) before adding.

NOTE2 The mass of the uranium reference material is measured before adding it to the vessel.

- d) After dissolution, measure the gross mass of the vessel with cap to 0,1 mg or to the lowest digit of the balance.
- e) Until the mixed stock solution is used, should keep under mass control by capping the flask and leave to stand on a stable place.

8.1.4 Taking mixed solution aliquots

During taking aliquots, the stock bottle for storing the solution preferably has a structure that prevents evaporation as much as possible in order to prevent changes in the uranium and plutonium content. Take

aliquot 1 ml to 2 ml from mixed stock solution into 10 ml vials (or suitable size for user) by hand or by machine. The amount of aliquot can be varied as necessary. Taking mixed solution aliquots is carried out as follows;

- a) Weigh initial gross of the vessel containing the mixed stock solution to 0,1 mg or to the lowest digit of the balance.
- b) Compare the gross with final gross of the vessels ([8.1.3.1 g](#)) or [8.1.3.2 d](#)) and calculate evaporation factor according to [Formula \(5\)](#).
- c) Confirm the vessel cap tightly and shake it homogenize the mixed stock solution.
- d) Transfer the mixed stock solution to a stock bottle which is used during taking aliquot.
- e) Take 1 ml to 2 ml aliquots from the stock solution to tared 10 ml vials and measure the gross mass to 0,1 mg.

8.1.5 Drying

The aliquots are carefully heated to dryness in a manner that avoids any loss of material as aerosols and leads to the production of a film of dried spike adhering firmly to the bottom of the vial. Keep several vials in solution state for validation of mixed stock solution.

- a) Reflux aliquot in vials on hot plate (<135 °C) until it changes to glassy condition on bottom of vials. It is also recommended to move vials to flat top hot plate when the spike solution is reduced to confirm its glassy condition.
- b) Close the vial with a chemically stable rubber cap immediately after removing from hot plate.
- c) Cool to room temperature.
- d) Investigate the condition within few days and if spike materials seem to be crystalized, re-dissolve with nitric acid and repeat from a) to prevent flaking off from the bottom.

8.1.6 Stabilization of LSD spike

Coating material, such as cellulose acetate butyrate (CAB) or carboxymethyl cellulose (CMC), can be added to ensure its stability for longer period^{[6][7]}. If coating materials are added, clarify the method of dissolving the additive and confirm that there is no influence on the content analysis result by IDMS.

8.2 Uranium spikes

8.2.1 Uranium liquid spikes

- a) Prepare the stock solution of uranium liquid spike following the same procedure for [8.1.1](#).
- b) Measure the gross mass of the vessel containing the stock solution of uranium liquid spike ([8.1.1](#)) to 0,1 mg.
- c) Compare the gross with final gross of the vessel ([8.1.1 l](#)) and calculate evaporation factor according to [Formula \(5\)](#).
- d) Confirm that the vessel is capped tightly and shake it to homogenize solutions.
- e) Open the cap of the vessel containing the uranium liquid spike stock solution and take aliquots to vials for validation of the uranium liquid spike stock solution.
- f) Weigh tare of ampoules or glass vessels, that can be sealed, to 0,1 mg.
- g) Take aliquot to ampoules or glass vessels from uranium liquid spike stock solution and measure the gross mass of them to 0,1 mg.
- h) Seal ampoules or glass vessels.

- i) Weigh the ampoules or vessels. Record the mass for stability checks based on mass control.

8.2.2 Uranium dried spikes

- a) Take the spike solution, which is prepared in [8.2.1](#), aliquots to vials after diluting if necessary.
- b) Reflux aliquot in vials on hot plate (<135 °C) until it changes to glassy condition on bottom of vials. It is also recommended to move vials to flat top hot plate when the spike solution is reduced to confirm its glassy condition.
- c) Close vials with a chemically stable rubber cap immediately after removing it from the hot plate.
- d) Cool to room temperature.

8.3 Plutonium spikes

8.3.1 Plutonium liquid spikes

- a) Prepare the stock solution of plutonium liquid spike following the same procedure for [8.1.2](#).
- b) Measure the gross mass of the vessel containing stock solutions of plutonium liquid spike ([8.1.2](#)) to 0,1 mg.
- c) Compare the gross with final gross of the vessel ([8.1.2.1 l](#)) and calculate evaporation factor according to [Formula \(5\)](#).
- d) Confirm that the vessel is capped tightly and shake it to homogenize solutions.
- e) Open the cap of the vessel containing the plutonium liquid spike stock solution and take aliquots to vials for validation of the plutonium liquid spike stock solution.
- f) Weigh tare of ampoules or glass vessels, that can be sealed, to 0,1 mg.
- g) Take aliquot to ampoules or glass vessels from plutonium liquid spike stock solution and measure the gross mass of them to 0,1 mg.
- h) Seal ampoules or glass vessels.
- i) Weigh the ampoules or vessels. Record the mass for stability checks based on mass control.

8.3.2 Plutonium dried spikes

- a) Take the spike solution, which is prepared in [8.3.1](#), aliquots to vials after diluting if necessary.
- b) Reflux aliquot in vials on hot plate (<135 °C) until it changes to glassy condition on bottom of vials. It is also recommended to move vials to flat top hot plate when the spike solution is reduced to confirm its glassy condition.
- c) Close the vial with a chemically stable rubber cap immediately after removing it from the hot plate.
- d) Cool to room temperature.

8.4 Uranium – plutonium mixed spikes

When both the plutonium and uranium contents are to be analysed routinely, it is convenient to use a solution spike which contains both plutonium and uranium. The proportion of the solution spikes should be designed following [5.1](#) for that can be obtained sufficient accuracy. This mix solution is prepared as follows;

- a) Prepare uranium stock solution and sample for validation following the same procedure as [8.2.1 a\) to e\)](#).
- b) Prepare plutonium stock solution and sample for validation following the same procedure as [8.3.1 a\) to e\)](#).
- c) Weigh tare of ampoules or glass vessels, that can be sealed, to 0,1 mg.