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**Nuclear fuel technology — Chemical separation and purification of uranium and plutonium in nitric acid solutions for isotopic and isotopic dilution analysis by solvent extraction chromatography —**

**Part 2:  
Samples containing plutonium and uranium in the nanogram range and below**

*Technologie du combustible nucléaire — Séparation et purification chimiques de l'uranium et du plutonium dans les solutions d'acide nitrique par extraction chromatographique par solvant pour les mesures isotopiques et les analyses par dilution isotopique —*

*Partie 2: Échantillons ayant des teneurs en plutonium et en uranium de l'ordre du nanogramme et inférieures*





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# Contents

|   | Page      |
|---|-----------|
| Foreword .....  | iv        |
| <b>1 Scope</b> .....  | <b>1</b>  |
| <b>2 Principle of the method</b> .....  | <b>1</b>  |
| <b>3 Apparatus</b> .....  | <b>2</b>  |
| <b>4 Reagents</b> .....   | <b>2</b>  |
| <b>5 Procedure (see <a href="#">Figure 1</a>)</b> .....   | <b>3</b>  |
| <b>6 Characteristics of the separation</b> .....  | <b>5</b>  |
| <b>7 Quality control</b> .....  | <b>5</b>  |
| <b>8 Interferences</b> .....  | <b>6</b>  |
| <b>Annex A (normative) Packing and conditioning of the chromatographic mini-columns as used in the ISO 15366-2 procedure, loaded with an inert polyethylene support coated with tri-<i>n</i>-octyl-phosphine-oxide (TOPO)</b> ..... | <b>7</b>  |
| <b>Bibliography</b> .....   | <b>10</b> |

## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2. [www.iso.org/directives](http://www.iso.org/directives)

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#)

The committee responsible for this document is ISO/TC 85, *Nuclear Energy*, Subcommittee SC 5, *Nuclear Fuel Technology*.

This first edition, together with the first edition of ISO 15366-1, cancels and replaces the first edition of ISO 15366:1999, which has been technically revised.

ISO 15366 consists of the following parts, under the general title *Chemical separation and purification of uranium and plutonium in nitric acid solutions for isotopic and isotopic dilution analysis by solvent extraction chromatography* —

- *Part 1: Sample containing plutonium in the microgram range and uranium in the milligram range*
- *Part 2: Sample containing plutonium and uranium amounts in the nanogram range and below*

# Nuclear fuel technology — Chemical separation and purification of uranium and plutonium in nitric acid solutions for isotopic and isotopic dilution analysis by solvent extraction chromatography —

## Part 2: Samples containing plutonium and uranium in the nanogram range and below

### 1 Scope

This part of ISO 15366 describes procedures to chemically separate and purify uranium and plutonium in dissolved solutions of irradiated light water reactor fuels and in samples of high active liquid waste of spent fuel reprocessing plants, prior to their isotopic analysis by e.g. mass spectrometric method (see ISO 8299[1]) or alpha spectrometry (see ISO 11483[2]). This part of ISO 15366 describes a slightly different separation technique from ISO 15366-1, based on the same chemistry, using smaller columns, different support material and special purification steps, applicable to samples containing plutonium and uranium amounts in the nanogram range and below. The detection limits were found to be 500 pg plutonium and 500 pg uranium. [Annex A](#) describes the preparation of the columns and the column support materials.

In comparison with ISO 15366-1, as uranium and plutonium amounts are lowest, additional purification on an anion exchange resin is performed.

### 2 Principle of the method

The chemical separation of small plutonium and uranium amounts (>500 pg) is also based on a column extraction chromatography using tri-*n*-octylphosphine-oxide (TOPO) as extractant.[3][4] The necessary valency adjustment prior to the separation is done with iron(II) sulfate and sodium nitrite. The extraction process is performed in disposable mini-columns loaded with a polyethylene or silica gel powder coated with the TOPO. Plutonium(IV) and uranium(VI) in 3 mol·l<sup>-1</sup> nitric acid medium are selectively extracted into the TOPO while americium, the fission products and other interfering elements are not retained. Plutonium is eluted after reduction to the trivalent state with ascorbic acid[5]; uranium is eluted by an ammonium carbamate solution and finally purified from ammonium salts by an ion exchange separation[6] or fuming by 14 mol·l<sup>-1</sup> nitric acid.

In order to ensure a favourable kinetics of chemical reactions the (gravity) column flow rates should not exceed 0,4 ml·min<sup>-1</sup>.

Parallel measurement of blank and/or control sample is recommended to verify the analysis.

Blanks are run in parallel with the samples to verify the absence of significant external cross-contamination or cross-contamination between samples.

Control samples prepared from certified or analysed materials are also prepared and separated along with the sample to verify that suitable valency adjustment, isotopic equilibration and separation efficiency are achieved.

The whole process is carried out under clean conditions preferably in a laminar flow fume hood.

### 3 Apparatus

**3.1 Biological shielding**, e.g. shielded glove box or fume cupboard, preferably a laminar flow fume cupboard.

**3.2 Disposable polypropylene columns and frits** (see [Figure A.1](#)). Type: micro-column. The packing and conditioning of the columns are described in [Annex A](#). The chromatographic columns shall be disposed of in the radioactive waste after use.

**3.3 Two hot plates.**

**3.4 Standard laboratory equipment** (flasks and beakers, pipettes, glassware, stands and supports for columns, sample vials, fraction tubes, etc.). It is recommended to leach all equipment, which will be in contact with samples, eluates or eluents with nitric acid ([4.1](#)) and distilled water before use. After drying, store it in plastic bags in a clean area.

**3.5 PTFE vials**, of dimensions: volume 15 ml, diameter 28 mm, height 37 mm. With screw caps and metal jackets.

These vials may be used at least 10 times, after a careful cleaning with hot nitric acid, followed by a rinsing with demineralized or de-ionized water.

### 4 Reagents

Use only reagents of recognized suprapure grade or equivalent. All aqueous solutions shall be prepared with distilled water (resistivity 1 M $\Omega$ ·m to 10 M $\Omega$ ·m).

**4.1 Concentrated nitric acid and nitric acid solutions**,  $c(\text{HNO}_3) = 6 \text{ mol}\cdot\text{l}^{-1}$  and  $3 \text{ mol}\cdot\text{l}^{-1}$ .

**4.2 Formic acid solution**,  $c(\text{HCOOH}) = 1 \text{ mol}\cdot\text{l}^{-1}$ .

**4.3 Ammonium carbamate** [CAS No. 1111-78-0] **solution**,  $c(\text{NH}_4\text{CO}_2\text{NH}_2) = 0,7 \text{ mol}\cdot\text{l}^{-1}$ .

**4.4 Polyethylene powder**<sup>1)</sup> GUR X 117/PE-UHMW Ultra-high-molecular grain size 25  $\mu\text{m}$  to 95  $\mu\text{m}$ , pore size  $\leq 5 \mu\text{m}$  (see [A.2.5](#)).

**4.5 L(+)-Ascorbic acid solution**  $c(\text{C}_6\text{H}_8\text{O}_6) = 10^{-5} \text{ mol}\cdot\text{l}^{-1}$  or  $c(\text{C}_6\text{H}_8\text{O}_6) = 10^{-3} \text{ mol}\cdot\text{l}^{-1}$  in formic acid solution ([4.2](#))

**4.6 Tri-*n*-octylphosphine-oxide**,  $c(\text{TOPO}) = 0,2 \text{ mol}\cdot\text{l}^{-1}$  solution in cyclohexane.

**WARNING — This reagent is flammable and should always be handled in a well-ventilated place and never in the vicinity of a naked flame.**

**4.7 Iron(II) sulfate solution**,  $c(\text{FeSO}_4) = 0,1 \text{ mol}\cdot\text{l}^{-1}$  or  $c(\text{FeSO}_4) = 0,8 \text{ mol}\cdot\text{l}^{-1}$  in water. To be prepared freshly for each working session.

**4.8 Sodium nitrite solution**,  $c(\text{NaNO}_2) = 1 \text{ mol}\cdot\text{l}^{-1}$  or  $c(\text{NaNO}_2) = 5 \text{ mol}\cdot\text{l}^{-1}$  in water. To be prepared freshly for each working session.

**4.9 Aqua regia**, mixture 3:1 (per volume) of concentrated hydrochloric acid and concentrated nitric acid.

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1) Silica gel may also be used. See ISO 15366-1.

**4.10 Concentrated hydrochloric acid and hydrochloric acid solution**  $c(\text{HCl}) = 6 \text{ mol}\cdot\text{l}^{-1}$ 

**4.11 Anion exchange resin AG MP1**, slurry in distilled water. Fill the mini-columns (3.2) with about 0,5 ml of the slurry. Condition the columns with two times 1 ml of distilled water and with three times 1 ml concentrated hydrochloric acid (4.10) immediately before use.

**5 Procedure (see Figure 1)**

NOTE This procedure is an example. If equivalent results could be expected, other conditions than these described in Clause 5, can be applied for sample preparation.

**5.1** The sample should contain 500 pg to 1 µg plutonium and 500 pg to 100 µg uranium in a volume of 0,5 ml of  $3 \text{ mol}\cdot\text{l}^{-1}$  nitric acid solution (4.1). Whenever starting with dried samples, apply the following dissolution procedure.

- a) Add 0,5 ml of  $6 \text{ mol}\cdot\text{l}^{-1}$  nitric acid solution (4.1) to the dry samples and evaporate slowly on the hot plate, keeping the temperature slightly below the boiling point to avoid any splashing and bubbling until nitrate salts crystallize.
- b) Remove the sample vessels from the hot plate and redissolve the salts by adding 0,5 ml of  $3 \text{ mol}\cdot\text{l}^{-1}$  nitric acid solution (4.1), while still warm ( $40 \text{ }^\circ\text{C}$  to  $60 \text{ }^\circ\text{C}$ ). Shake the vessels for a few seconds.

**5.2** Perform a redox valency cycle to ensure that all plutonium isotopes are in the tetravalent state before starting the separation, as follows.

- a) Add 50 µl of Iron(II) sulfate solution (4.7) to the sample.
- b) Mix and wait for 5 min for a complete reduction of plutonium(VI) or plutonium(IV) to plutonium(III).
- c) Add 50 µl sodium nitrite solution (4.8) to reoxidize plutonium to the tetravalent state and add further 100 µl of  $6 \text{ mol}\cdot\text{l}^{-1}$  nitric acid solution (4.1) to reach an acid concentration of  $3 \text{ mol}\cdot\text{l}^{-1}$ . Mix again and wait for at least 5 min.

**5.3** Transfer half of the pretreated sample on to the column, wait approximately 1 min, add the rest of the sample and let it flow through. This favours the retention of plutonium and uranium in the very upper layers of the column.

**5.4** Rinse the sample vial with 0,5 ml of  $3 \text{ mol}\cdot\text{l}^{-1}$  nitric acid solution (4.1) and transfer the solution to the column.

**5.5** Wash out the fission products, including americium, from the column using  $3 \text{ mol}\cdot\text{l}^{-1}$  nitric acid solution (4.1) in four successive aliquots of 1 ml.

**5.6** Condition the column for the plutonium elution by adding twice 0,5 ml of  $1 \text{ mol}\cdot\text{l}^{-1}$  formic acid solution (4.2). Discard waste collected until this time.

**5.7** Elute the plutonium from the column with the ascorbic acid solution (4.5) with five successive aliquots of 1 ml. Place the vials containing the collected plutonium fractions on one of the hot plates.

**5.8** Wash out the “tail” of the plutonium with twice 1 ml of the ascorbic acid solution (4.5) in formic acid solution in one aliquot and discard the plutonium “tail” washings to the waste.

**5.9** Condition the column for the elution of the uranium fraction by adding two aliquots each of 1 ml distilled or de-ionized water. Discard the water washings to the waste.

**5.10** Elute the uranium with 4 ml ammonium carbamate solution (4.3). Place the vials containing the collected uranium fractions on the other hot plate.

**5.11** Let the plutonium and uranium fractions evaporate gently to dryness on the hot plates at 90 °C.

**5.12** Remove the fractions from the hot plates, add 0,25 ml of concentrated nitric acid (4.1) and evaporate again to dryness. Repeat this step once. Proceed with the uranium fractions with 5.17 if necessary.

**5.13** Redissolve the plutonium fractions in 0,5 ml of 3 mol·l<sup>-1</sup> nitric acid solution (4.1) and transfer the solutions to the PTFE vials (3.5).

**5.14** Rinse the vials of the plutonium fraction with 0,5 ml of 3 mol·l<sup>-1</sup> nitric acid solution (4.1) and transfer the solutions to the corresponding PTFE vials.

**5.15** Place all PTFE vials with the plutonium fractions on the hot plate, preferably in metal jackets for an even heating. Evaporate the solutions gently to dryness at 90 °C avoiding any bubbling and splashing.

**5.16** After cooling down to room temperature, secure the cap of the PTFE vials with the plutonium fractions and forward them for mass spectrometric measurement. The mass spectrometer operator will redissolve the residues in a minimum (2 µl and less) of 3 mol·l<sup>-1</sup> nitric acid solution immediately before loading the solutions on to the filaments of the mass spectrometer. Performing this redissolution step by means of a microscope is recommended.

**5.17** Redissolve all uranium fractions with 250 µl aqua regia (4.9) and evaporate to dryness.

**5.18** Redissolve all uranium fractions with 250 µl concentrated hydrochloric acid (4.10) and evaporate to dryness.

**5.19** Redissolve all uranium fractions with 1 ml concentrated hydrochloric acid (4.10) and transfer slowly all solutions to the conditioned anion exchange resin columns (4.11).

**5.20** Rinse all uranium fraction vials with 1 ml concentrated hydrochloric acid (4.10) and transfer slowly all solutions to the corresponding columns.

**5.21** After the solutions (5.20) have completely passed the columns, wash each column with 0,5 ml of hydrochloride acid solution 6 mol·l<sup>-1</sup> (4.1).

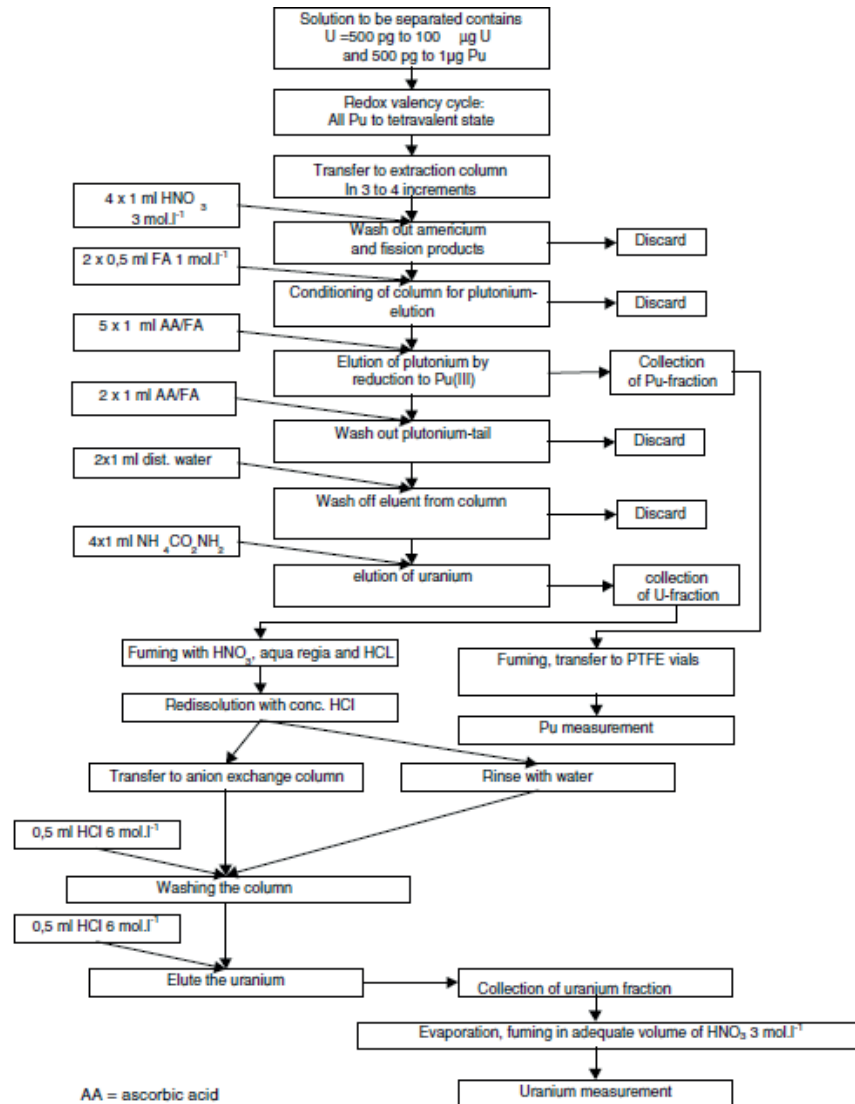
**5.22** Elute the uranium into PTFE vials (3.5) with four successive aliquots of 1 ml distilled water.

**5.23** Place all PTFE vials with the uranium fractions on the other hot plate, preferably in metal jackets for an even heating. Evaporate the solutions gently to dryness at 90 °C avoiding any bubbling and splashing.

**5.24** Remove the uranium fractions from the hot plates, add 0,25 ml of concentrated nitric acid (4.1) and evaporate again to dryness in the same way as in 5.23. Repeat this step once.

**5.25** After cooling down to room temperature secure the cap of the PTFE vials with the uranium fractions and forward them for mass spectrometric measurement. The mass spectrometer operator will redissolve the residues in a minimum (2 µl and less) of 3 mol·l<sup>-1</sup> nitric acid solution immediately before loading the solutions on to the mass spectrometer filaments. Performing this redissolution step by means of a microscope is recommended.





## Key

AA ascorbic acid

FA formic acid

Figure 1 — U and Pu separation scheme

## 6 Characteristics of the separation

The element recovery is 80 % for U and 60 % for Pu found after separations of the lowest element concentrations (500 pg). In higher concentrations, the Pu recovery reaches 80 % and more.

## 7 Quality control

**7.1 Blanks:** blank samples are spiked with known amounts of highly enriched tracers, such as  $^{242}\text{Pu}$  and  $^{233}\text{U}$  and processed in the same way as, and in parallel with the actual samples. The amounts of plutonium and uranium coming from the blank are determined by isotope dilution mass spectrometry according to ISO 8299.[1] The results of these blank level determinations should be checked for their actual value and their fluctuation (in terms of repeatability and mid-term reproducibility). The observed fluctuation should be commensurate with the assessment of the uncertainties of the method. Otherwise an uncontrolled source of contamination shall be suspected, which requires beforehand remedial environmental measures.

The actual value of the blank level depends on the available laboratory environment. It is recommended to correct the sample results for blank, if the determined blank level is statistically significant above zero.

**7.2** Control samples: solutions of plutonium and uranium nitrate of known isotopic and elemental composition, similar to the composition of the actual samples, are used to monitor the accuracy of the isotopic and elemental assays by alpha and mass spectrometry following the separation. Samples of such solutions are processed and measured in the same way as, and in parallel with the actual samples. The results of such control measurements are preferably followed with statistical control charts.

## 8 Interferences

**8.1** Extractable elements: according to Reference [3], only Ti(IV), Zr(IV), Hf(IV), Ge(IV), Sn(IV), Tc(VIII), Eu(III), Ce(IV), Th(IV), Np(IV) and Np(V) may be extracted from nitric acid, 3 mol·l<sup>-1</sup>, along with uranium and plutonium.

**8.2** Interferences in mass spectrometry measurements according to ISO 8299[1]: elements yielding ions with mass 233, 234, 235, 236 and 238 cause interference in the mass spectrometric analysis of uranium if they have not been removed, or if they have been introduced as impurities during the chemical treatment; potassium for example will emit hexa-atomic ions of mass 234.

Elements yielding ions with mass 238 (particularly <sup>238</sup>U), 239, 240, 241 and 242 cause interference in the mass spectrometric analysis of plutonium if they have not been completely removed during chemical treatment.

In addition to the isobaric interferences, another class of interfering elements can alter the fractionation patterns in mass spectrometric analyses. For example, thorium, zirconium, hafnium, rare earth metals, aluminium, and titanium can increase the temperature required to volatilize and ionize uranium and plutonium. Iron, vanadium, copper and alkali metals can lower the temperature at which volatilization of uranium and plutonium occurs. Among these, only Th, Zr, Hf and Ti may be retained by TOPO from nitric acid, 3 mol·l<sup>-1</sup>.

**8.3** Interferences in alpha spectrometry measurements according to ISO 11483[2]: the nuclide <sup>241</sup>Am[5] emits alpha particles of energies very close to the energies of the radiation emitted by the nuclide <sup>238</sup>Pu. Its presence yields positive biases in the determination of the <sup>238</sup>Pu abundance by alpha spectrometry. In the case of a five-year-old plutonium sample, containing 1 % of the <sup>238</sup>Pu isotope, 10 % of the <sup>241</sup>Pu isotope and 2,7 % of the <sup>241</sup>Am isotope, the relative bias is equal to 0,06 % immediately after separation, if the americium decontamination factor is 10<sup>3</sup>. An additional bias of 0,027 % accumulates every day following the separation.

The efficiency of the <sup>241</sup>Am-decontamination should be checked regularly by gamma spectrometry or by repeating the alpha spectrometry after submitting the plutonium fraction to a second separation. The alpha spectrometry should be done within one week after the separation is completed.

Nuclides emitting alpha particles of energies between 4,9 MeV and 5,6 MeV can interfere in the alpha spectrometric analysis if they are not separated during the purification of plutonium.

Any chemical compound, which is not completely eliminated during the chemical separation or during the preparation of the source, decreases the quality of the source and the resolution of the alpha spectra. Even if these compounds are not radioactive, the errors in the processing of the spectra become significant when the half height width of the peaks become greater than 25 keV. Silicon compounds which might leach out of the column shall be removed by prewashing the column just before use as described in [A.3](#) and [A.5](#).

## Annex A (normative)

### Packing and conditioning of the chromatographic mini-columns as used in the ISO 15366-2 procedure, loaded with an inert polyethylene support coated with tri-*n*-octyl-phosphine-oxide (TOPO)

#### A.1 General

This annex is an integral part of the ISO 15366-2 procedure and describes the procedure for the preparation of the chromatographic columns.

The goal is to obtain an inert support uniformly coated with tri-*n*-octyl-phosphine-oxide and its homogeneous packing into the column, ensuring that the flow of the effluents is reproducible and uniform during the separation process.

#### A.2 Apparatus

The column shall be prepared in a clean area where contamination by actinides can be excluded.

**A.2.1 Columns and frits** (see [Figure A.1](#)). Type: micro-column.

**A.2.2 Dispensing spoon** (PVC custom-made, to measure 150 mg to 160 mg of the TOPO/polyethylene powder).

**A.2.3 Rubber bulb**, with 4 mm tip.

**A.2.4 PVC rod**, 4 mm in diameter, 150 mm length, to push the frit into the column.

**A.2.5 Polyethylene powder**, GUR X 117/PE-UHMW Ultra-high molecular.

#### A.3 Coating the polyethylene support with TOPO

The polyethylene powder is coated with TOPO as follows to obtain a dry and loose powder.

**A.3.1** First clean the polyethylene in the following manner:

Weigh 40 g polyethylene ([4.4](#)) into a beaker and wash with about 100 ml of nitric acid solution  $3 \text{ mol}\cdot\text{l}^{-1}$  ([4.1](#)), swirling and mixing the slurry each 10 min for at least half an hour. Allow the slurry to set and pour it into a Buchner filter. Then wash the polyethylene in a similar way with distilled water at least 10 times in order to obtain an acid free support. Dry the wet polyethylene on a big watch-glass for at least 40 height hours at 25 °C mixing regularly. Allow the powder to cool down before further use.

**A.3.2** Weigh 30 g of the washed and dried polyethylene into a 150 ml discardable beaker.

## ISO 15366-2:2014(E)

**A.3.3** Place the beaker on the balance in a well ventilated area and add swiftly 45 g of 0,2 mol·l<sup>-1</sup> solution of TOPO in cyclohexane.

Using a volumetric cylinder to measure the required amounts of TOPO-solution is not commended, as the cyclohexane would evaporate so fast during its transfer that an uncontrolled amounts of TOPO would crystallize on the inner walls of the cylinder.

**A.3.4** Stir steadily and quickly with a plastic spoon taking care that the polyethylene is uniformly wetted and coated with the TOPO-solution.

**A.3.5** Transfer the mixture to a large watch-glass or Petri dish, spread it evenly with the spoon and allow the material to dry. The cyclohexane evaporates rapidly leaving a dry polyethylene coated with TOPO. Leave uncovered in a well-ventilated room for at least 40 height hours at room temperature to reach complete dryness.

Never dry above room temperature. Drying at higher temperatures will produce crusty clumps and the TOPO will tend to flake away from the surface of the polyethylene particles.

**A.3.6** Store the powder in wide neck plastic bottles and use whenever needed.

### A.4 Packing the columns

The chromatographic columns are prepared, as follows, only for a single use and discarded to the radioactive waste after the separation.

**A.4.1** Sieve the TOPO/polyethylene powder through a stainless steel sieve with 0,25 mm pores to ensure that no clumps of material are added to the column.

**A.4.2** Place a plastic funnel on the column

**A.4.3** Fill the special dispensing plastic spoon with sieved TOPO/polyethylene powder up to the rim and pour it through the funnel into the column. Remove the funnel from the column.

**A.4.4** Hit the tip of the column gently on the table to allow the powder to settle uniformly.

**A.4.5** Fill as many columns as needed, following steps [A.4.2](#) to [A.4.4](#).

**A.4.6** Take a 30 µm frit and insert it in the column. Push the frit carefully with the PVC rod with a swift and straight movement until it presses gently on the TOPO/polyethylene filling. Take care that the frit sits horizontally on the TOPO/polyethylene surface (see [Figure A.1](#)). In the case that it is tilted, remove it, place a new frit and repeat the operation.

**A.4.7** Store the column in a sealed plastic bag for later use and condition it as described below before use.

### A.5 Conditioning the columns

Condition the columns immediately before use in a clean area excluding contamination by actinides, and outside of the analytical glove box to avoid unnecessary production of radioactive waste, as follows:

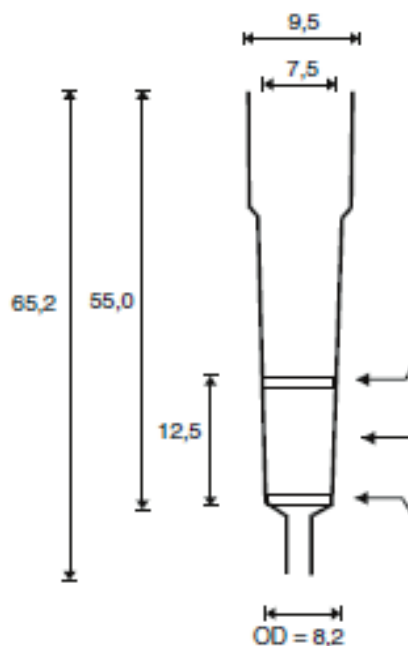
**A.5.1** Bring the desired number of columns into a clean area excluding the risk of contamination by actinides.

**A.5.2** Remove the columns from their storage bag and place them on a stand above a flat-bottomed dish.

**A.5.3** Add 1 ml of nitric acid solution  $3 \text{ mol}\cdot\text{l}^{-1}$  to the column and press the solution gently with a rubber bulb through the TOPO/polyethylene until liquid drops appear at the column tip. The diluted acid will now flow by gravity unattended until the reservoir is empty. Add later twice 1 ml of nitric acid solution  $3 \text{ mol}\cdot\text{l}^{-1}$ .

**A.5.4** When the reagent stops flowing and the reservoir is empty, transfer the column into the glove box and proceed with the separation.

Dimensions in millimetres



#### Key

- 1 reservoir, volume 1,5 ml
- 2 polyethylene frit,  $30 \mu\text{m}$  porosity
- 3 TOPO/polyethylene powder or ion exchange resin, mass about 150 mg to 160 mg, volume 0,4 ml
- 4 polyethylene frit,  $30 \mu\text{m}$  porosity
- 5 column tip

**Figure A.1 — Chromatographic column for separations**

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