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Requirements for representative sampling of uranyl nitrate solutions for the determination of uranium concentration

Exigences pour obtenir un échantillonnage représentatif de solutions de nitrate d'uranyle en vue de déterminer la concentration d'uranium

Reference number
ISO 11598:1995(E)

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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Requirements for representative sampling of uranyl nitrate solutions for the determination of uranium concentration

1 Scope

This International Standard specifies essential precautions to be taken to ensure that installed tank mixing and sampling systems produce samples from a batch of pure uranyl nitrate solution which are representative, and that the samples remain representative until used.

When the requirements given in this International Standard are applied, the difference between the uranium concentration of the samples, taken from the batch, and the average concentration of uranium in the solution is less than 0,2 %.

2 General

Because uranium processing plants vary in design, process and equipment, uniform procedures for sampling uranyl nitrate storage tanks cannot be established. However, certain guidelines may be applied to all sampling systems.

Representative samples are a fundamental necessity for accurate measurement of uranyl nitrate solutions. Some chemical and physical properties of uranyl nitrate solutions and their containers make representative sampling difficult or impossible, for example:

a) Solutions may be stored in tanks with a critically safe and therefore complex geometry. Prolonged mixing times are then required to achieve mixing of the total volume.

b) High density and viscosity, particularly at the high uranium concentrations, make prolonged mixing times necessary to achieve a homogeneous tank content.

c) The possible presence of a second phase, either crystallized uranium, organic liquids or solids, impedes representative sampling.

Many of these problems can be avoided or controlled by effective engineering at the design stage. Nevertheless, a mixing and sampling procedure shall be established for each tank and representativeness of samples shall be experimentally verified. After any modification of the equipment, these procedures shall be validated again.

The samples taken shall be handled and stored in such a way that their properties at the time of chemical analysis can reliably be related to those properties at the time of sampling. Changes can occur in the samples themselves, for example uranyl nitrate solutions can crystallize (depending on uranium concentration, free acidity and temperature). Handling and storage procedures shall be designed to minimize any changes of sample properties between sampling and analysis, or at least to allow for corrections when changes are unavoidable.

3 Tank mixing and sampling

The following steps should be applied to reduce systematic and random sampling errors to a tolerably low level.

3.1 The uranyl nitrate solution should be thoroughly homogenized in accordance with a fixed and verified mixing procedure, to ensure that the solution is isotopically, chemically and physically uniform.

Procedures should be verified for each tank. Documentation of mixing and sampling studies should be maintained on file as long as the system is in use, in accordance with the ISO 9000 series.

3.2 The uranyl nitrate solution should be free of organic phase and/or solids. If an organic phase and/or solids are visibly present, the solution should be filtered or centrifuged before sampling.

3.3 The uranium concentration should preferably not exceed 450 g/l. The free nitric acid concentration should preferably not exceed 0,2 mol/l.

3.4 Sampling pipes should be short with small cross-sections to reduce the likelihood of concentration changes due to evaporation.

3.5 During mixing, the sampling pipes should be flushed with the solution to be sampled.

3.6 In case the mixing is by air sparging, the sparging should be discontinued during sampling.

3.7 Sample containers should be completely clean and dry.

3.8 If possible, several samples should be taken from different parts of the tank and their representativity verified by density measurements, for example with a vibrating capillary densitometer, in accordance with ISO 11597:1995, *Verification of samples of uranyl or plutonium nitrate solutions by density measurements*. The density of these samples should not differ by more than 0,000 5 g/cm^3.

3.9 The recommended individual sample size is between 5 ml and 100 ml. The time of sampling shall be noted for each sample.

3.10 The total volume or mass of solution in the tank should be determined at the time of sampling, with an accuracy comparable to that of the analytical uranium determination.

4 Sample handling and storage

The following procedures should be applied to ensure that the sample concentration at the time of analysis can be related to the sample concentration at the time of sampling.

4.1 Samples shall be clearly marked to ensure unequivocal identification.

4.2 Samples should be stored in dark glass bottles, closed by stoppers with a proven tightness, to minimize evaporation losses and photo-induced reactions. The air volume above the liquid should be kept to a minimum.

4.3 Sample bottles should be handled and stored at ambient temperature in an upright position. Wetting the bottle neck shall be avoided.

4.4 Sample bottles shall not be left open for long periods. They should be closed immediately after filling or removal of an aliquot.

4.5 If samples are to be stored for a long period, or if they are taken in plastics bottles, then the following weight correction procedure should be applied:

a) Use sample bottles without a removable label and with a stopper having a stable tare weight (better than ± 0,001 g under storage conditions).

b) Briefly open the stopper to equalize the air pressure in the bottle with the ambient pressure, and weigh the empty bottle with the stopper; record the weight as W_1.

c) Weigh the bottle with stopper plus the sample; record the weight as W_2.

d) Do not stick an additional label on the bottle; handle and store it in such a way that it remains clean, dry and dust-free. Check the tightness of the stopper regularly.

e) Prior to analysis, briefly open the stopper and reweigh the bottle with stopper plus the sample; record the weight as W_3.

f) Before taking aliquots of the sample for analysis, verify that no crystal or solid deposits have formed, stopper the sample bottle tightly and shake it to homogenize the solution in order to assure that sample aliquots will be representative of the sample.

g) Redetermine the gross weight of the bottle with stopper plus the sample after aliquoting; record the new gross weight.

h) Calculate the concentration of analyte Y at the time of sampling from the measured concentration X using the equation:

$$Y = \frac{W_3 - W_1}{W_2 - W_1} X$$

where

W_1, W_2 and W_3 are as above.

NOTE 1 Results based on this procedure become less reliable if the weight change during storage exceeds 1 % of the sample weight.

4.6 Studies should be performed to assess the validity of sample results after storage of the samples. The studies should be well documented and the documentation should be maintained in a file in compliance with the ISO 9000 series (Quality management and quality assurance standards). Allowance should be made in the studies for various periods of storage and various concentrations.

5 Conclusion

Because measurement system details differ, mixing and sampling procedures should be verified for each tank before use. Any changes in equipment or operation should be evaluated to verify the applicability of existing procedures.

Similarly, storage conditions vary from laboratory to laboratory and from time to time. The suitability of the recommended storage conditions shall be verified before use.

Care shall be taken that changes in container materials, sample handling and storage procedures or laboratory procedures for the determination of uranium concentration or isotopic composition do not introduce new sources of error. The applicability of existing procedures shall therefore be evaluated whenever changes occur.

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