Designation: C 1022 – 02

Standard Test Methods for Chemical and Atomic Absorption Analysis of Uranium-Ore Concentrate[1]

This standard is issued under the fixed designation C 1022; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover procedures for the chemical and atomic absorption analysis of uranium-ore concentrates to determine compliance with the requirements prescribed in Specification C 967.

1.2 The analytical procedures appear in the following order:

	Sections
Uranium by Ferrous Sulfate Reduction—Potassium Dichromate Titrimetry	9
Nitric Acid-Insoluble Uranium	10 to 18
Extractable Organic Material	19 to 26
Arsenic by Diethyldithiocarbamate (Photometric Method)	27 to 36
Carbonate by CO_2 Gravimetry	37 to 43
Fluoride by Ion-Selective Electrode	44 to 51
Halides by Volhard Titration	52 to 59
Moisture by Loss of Weight at 110°C	60 to 66
Phosphorus by Spectrophotometry	67 to 75
Silicon by Gravimetry	76 to 82
Thorium by the Thorin (Photometric) Method	83 to 91
Calcium, Iron, Magnesium, Molybdenum, Titanium, and Vanadium by Atomic Absorption Spectrophotometry	92 to 101
Potassium and Sodium by Atomic Absorption Spectrophotometry	102 to 111
Boron by Spectrophotometry	112 to 121

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Sections 7, 32, and Note 14.

2. Referenced Documents

2.1 *ASTM Standards:*

C 761 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Uranium Hexafluoride[2]

C 859 Terminology Relating to Nuclear Materials[2]

C 967 Specification for Uranium Ore Concentrate[2]

C 1254 Test Method for Uranium in Mineral Acids by X-Ray Fluoresence

C 1267 Test Method for Uranium by Iron (II) Reduction in Phosphoric Acid Followeed by Chromium (IV) Titration in the Presence of Vanadium[2]

C 1347 Practice for Preparation and Dissolution of Uranum Materials for Analysis[2]

D 1193 Specification for Reagent Water[3]

E 60 Practice for Photometric and Spectrophotometric Methods for Chemical Analysis of Metals[4]

3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods, refer to Terminology C 859.

4. Significance and Use

4.1 The test methods in this standard are designed to show whether a given material meets the specifications prescribed in Specification C 967.

4.2 Because of the variability of matrices of uranium-ore concentrate and the lack of suitable reference or calibration materials, the precision and bias of these test methods should be established by each individual laboratory that will use them. The precision and bias statements given for each test method are those reported by various laboratories and can be used as a guideline.

4.3 Instrumental test methods such as X-ray fluorescence and emission spectroscopy can be used for the determination of some impurities where such equipment is available.

5. Interferences

5.1 Interferences are identified in the individual test methods.

5.2 Ore concentrates are of a very variable nature; therefore, all interferences are very difficult to predict. The individual

[1] These test methods are under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and are the direct responsibility of Subcommittee C26.05 on Methods of Test.

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[2] *Annual Book of ASTM Standards,* Vol 12.01.

[3] *Annual Book of ASTM Standards,* Vol 11.01.

[4] *Annual Book of ASTM Standards,* Vol 03.05.

user should verify the applicability of each procedure for specific ore concentrates.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

7. Precautions

7.1 Proper precautions should be taken to prevent inhalation or ingestion of uranium during sample preparation and any subsequent sample analysis.

8. Sampling

8.1 Collect samples in accordance with Specification C 967.

8.2 Special requirements for subsampling are given in the individual test methods.

URANIUM BY FERROUS SULFATE REDUCTION—POTASSIUM DICHROMATE TITRIMETRY

9. Scope

9.1 This test method covers the determination of uranium in uranium-ore concentrates. This test method was discontinued in January 2002 and replaced with Test Method C 1267.

9.2 The uranium content of the sample may also be determined using Test Method C 1254. The user's laboratory must establish and document method performance.

NOTE 1—Dissolution of UOC samples may be achieved using the techniques or combination of techniques described in C 1347 The laboratory must validate the performance of C 1347 using characterized UOC samples. If C 1347 methods are not suitable for UOC sample dissolution, the user may establish and document applicable dissolution methods.

NITRIC ACID-INSOLUBLE URANIUM

10. Scope

10.1 This test method covers the determination of that quantity of uranium in uranium-ore concentrate that is not soluble in nitric acid.

11. Summary of Test Method

11.1 A sample of ore concentrate is digested in 10 *M* nitric acid at 95 to 100°C for 1 h. The slurry is filtered and the residue washed with 1 *M* nitric acid until the filtrate gives a negative test for uranium. The washed residue is then dried and ignited at 1000 ± 25°C for 1 h. The uranium content is determined on the ignited residue by spectrophotometry.

12. Interference

12.1 At the specification limit for nitric acid insoluble uranium usually established for uranium-ore concentrates, interference effects are insignificant.

13. Apparatus

13.1 *Digestion Flask*, 500-mL, with side entry tube and attached reservoir.

13.2 *Stirring Apparatus*, with sleeve-type stirrer.

13.3 *Heating Mantle*, 250-W, controlled by a variable transformer.

13.4 *Büchner Funnel*.

13.5 *Porcelain Crucibles*, 40-mL.

13.6 *Muffle Furnace*.

13.7 *Filter Paper*, [6] of medium porosity.

13.8 *Spectrophotometer*, with 1-cm cells that are in accordance with Practice E 60.

14. Reagents

14.1 *Nitric Acid* (10 *M*)—Dilute 62.5 mL of HNO_3 (sp gr 1.42) to 100 mL with distilled water.

14.2 *Nitric Acid* (1 *M*)—Dilute 62.5 mL of HNO_3 (sp gr 1.42) to 1 L with distilled water.

14.3 *Sodium Hydroxide* (100 g/L)—Dissolve 10 g of NaOH in 100 mL of water.

14.4 *Hydrogen Peroxide* (H_2O_2, 30 %).

14.5 *Hydrochloric Acid* (HCl, sp gr 1.19).

14.6 *Hydrofluoric Acid* (HF, 48 %).

14.7 *Sulfuric Acid* (9 *M*)—Add 500 mL H_2SO_4 (sp gr 1.84) to 500 mL of iced water with constant stirring. Cool and dilute to 1 L with water.

15. Procedure

15.1 Weigh a 50.0 ± 0.1-g sample directly into the digestion flask.

15.2 Place the flask in the heating mantle and adjust the support ring so that the joints of the flask and sleeve stirrer are engaged, and the stirrer blades turn freely but just clear the bottom of the flask.

15.3 Transfer 95 mL of 10 *M* nitric acid to a 250-mL beaker and heat between 95 to 100°C.

15.4 Slowly transfer the heated nitric acid solution to the digestion flask through the entry side tube with the stirrer turning.

NOTE 2—The stirrer is started before the acid is added to prevent material from sticking to the flask.

15.5 Align a thermometer in such a manner that the mercury chamber of the thermometer is immersed in the stirring slurry, but adequately clears the turning stirrer blades.

[5] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

[6] Whatman brand No. 40 or its equivalent has been found suitable.

15.6 Quickly bring the sample to 97°C and digest between 95 to 100°C for 1 h while stirring. (Measure the 1-h digestion time after the temperature of the slurry has reached 97°C.)

15.7 Turn off the variable transformer, but allow the stirrer to continue turning.

15.8 Remove the thermometer and carfully rinse with water all slurry that adheres to it.

15.9 Wipe the immersed portion of the thermometer with one fourth of a circle of filter paper and transfer the paper to a prepared Büchner funnel fitted with a filter paper.

15.10 Add 10 mL of paper pulp to the slurry and continue stirring for about 5 min.

15.11 Turn off the stirrer, then lower the flask and mantle.

15.12 Carefully wash the slurry that adheres to the stirrer shaft and blades into the flask with water.

15.13 Wipe the shaft and blades with one fourth of a circle of filter paper and transfer the filter paper to the Büchner funnel.

15.14 Filter the slurry through the Büchner funnel and wash contents of the flask into the funnel.

15.15 Wash the residue with 1 *M* nitric acid until a 10-mL portion of the filtrate shows no detectable yellow color when made basic with sodium hydroxide and after a few drops of H_2O_2 (30 %) have been added as a color developer.

15.16 Wash the residue several times with water after a negative test is obtained.

15.17 Draw air through the filter until the residue and filter pad are dry.

15.18 Scrape the residue and paper into a preignited (1000°C) tared 40-mL crucible, place on a hot plate and slowly char off the organic material.

15.19 Ignite the residue for 1 h at 1000°C in a muffle furnace.

15.20 Cool the crucible in a desiccator and weigh.

15.21 Calculate the percentage of solids in accordance with 17.1.

NOTE 3—If the percentage of solids (insoluble residue) is greater than 0.1 %, grind and mix the residue and determine the total milligrams of uranium in the residue by the photometric procedure in 16.1-16.10.

16. Photometric Procedure for Uranium

16.1 Transfer the ground, blended residue from 15.20 to a 100-mL beaker.

16.2 Add 10 mL of water and 10 mL of HCl (sp gr 1.19), cover, and boil for 10 min.

16.3 Add 5 mL of HNO_3 (sp gr 1.42) and boil until fuming of NO_2 ceases. Remove cover glass.

16.4 Add 5 mL of 9*M* H_2SO_4 and 2 mL of HF (48 %), then heat to dryness on the hotplate. Bake to fume off remaining H_2SO_4 and cool.

16.5 Wash down sides of beaker with water and add 5 mL of HNO_3.

16.6 Cover with a watchglass and digest for approximately 10 min near the boiling point.

16.7 Quantitatively transfer the solution to a 250-mL volumetric flask. Add 25 mL of NaOH solution and a few drops of H_2O_2. Make up to mark with water and mix.

NOTE 4—The solution must be basic for yellow sodium peruranate color to develop.

16.8 Measure the absorbance of the solution in a spectrophotometer at 425 nm in a 1-cm cell using a blank as reference. The blank is prepared by diluting 25 mL of NaOH, plus a few drops of H_2O_2, to 250 mL with water.

16.9 Prepare a calibration curve covering the range from 0 to 50 mg of uranium from aliquots of a standard uranium solution. Proceed as in 16.5-16.8. Plot the milligrams of uranium against absorbance readings.

16.10 Determine the total milligrams of uranium in the sample solution from the calibration curve.

NOTE 5—If the sample solution falls outside the calibration range, dilute a portion with the reference-blank solution and read again.

17. Calculation

17.1 Calculate the percentage of insoluble residue, *R*, present as follows:

$$R = \frac{R_w \times 100}{S_w} \quad (1)$$

where:
R_w = weight of residue (see 15.20), g, and
S_w = weight of samples, g.

17.2 If the insoluble residue exceeds 0.1 %, calculate the percentage of nitric acid-insoluble uranium, U_N, and present as follows:

$$U_N = \frac{U}{S_w \times 10} \quad (2)$$

where:
U = uranium content calculated in 16.10, mg, and
S_w = weight of sample, g.

17.3 Calculate the percentage of nitric acid-insoluble uranium, U_u, on a uranium basis as follows:

$$U_u = \frac{U_N \times 100}{U_s} \quad (3)$$

where:
U_N = nitric acid-insoluble residue present (see 17.2), %, and
U_s = uranium in sample, %.

18. Precision and Bias

18.1 *Precision*—A relative standard deviation for this test method has been reported as 10 % at the 0.2 % HNO_3 insoluble uranium level (see 4.2).

18.2 *Bias*—For information on the bias of this test method see 4.2.

EXTRACTABLE ORGANIC MATERIAL

19. Scope

19.1 This test method is used to determine the extractable organic material in uranium-ore concentrates. It is recognized that certain water-soluble organic materials, such as flocculating agents, are not measured by this test method.

20. Summary of Test Method

20.1 This test method consists of a dual extraction using trichlorofluoromethane on the solid uranium-ore concentrate sample and chloroform on a subsequent nitric acid solution of the sample. Each of the extractants is evaporated to measure the amount of organic material extracted.

21. Interferences

21.1 At the specification limit for extractable organic material established for uranium-ore concentrations, and within the scope of this test method, interferences are insignificant.

22. Apparatus

22.1 *Soxhlet Extraction Apparatus*—The trichlorofluoromethane extraction is done in a Soxhlet extraction apparatus. Construct as follows (see Fig. 1):

22.1.1 Modify a medium Soxhlet extraction tube so that the sidearm siphon is about 2 cm high, therefore, reducing the volume of solvent needed. Insert a 3 to 4-cm long, 25-mm outside diameter glass tube upright into the extraction tube in such a manner that an extraction thimble may be placed on it.

22.1.2 Connect a 250-mL Florence flask, that has a 24/40 ground-glass joint on the lower end to the top of the extraction tube. A250-mL heating mantle connected to a 7.5-A variable transformer shall be used to heat this.

22.1.3 Connect a Friedrichs condenser, that has a 45/50 ground-glass joint on the lower end, to the top of the extraction tube. Turn this side of the condenser upward, and fuse the outer member of a 24/40 ground-glass joint to it.

22.1.4 Connect a Graham condenser, that has a 24/40 ground-glass joint on the lower end, to the modified sidearm of the Friedrichs condenser. Unless the relative humidity is low, insulate the Graham condenser to prevent the condensation of water on the outside surface that might seep through the joint to the Friedrichs condenser. Foam insulation 1 cm thick may be used for this purpose. The Graham condenser is cooled with cold water from a water bath cooler, and may be required when trichlorofluoromethane is used for the extraction.

22.2 *Heat gun* (hot-air electric dryer), may be used to evaporate the solvent in procedure 24.6 or 24.15.

22.3 *Extraction Thimbles*.

22.4 *Filter Paper*. [7]

22.5 *Phase Separator Paper*. [8]

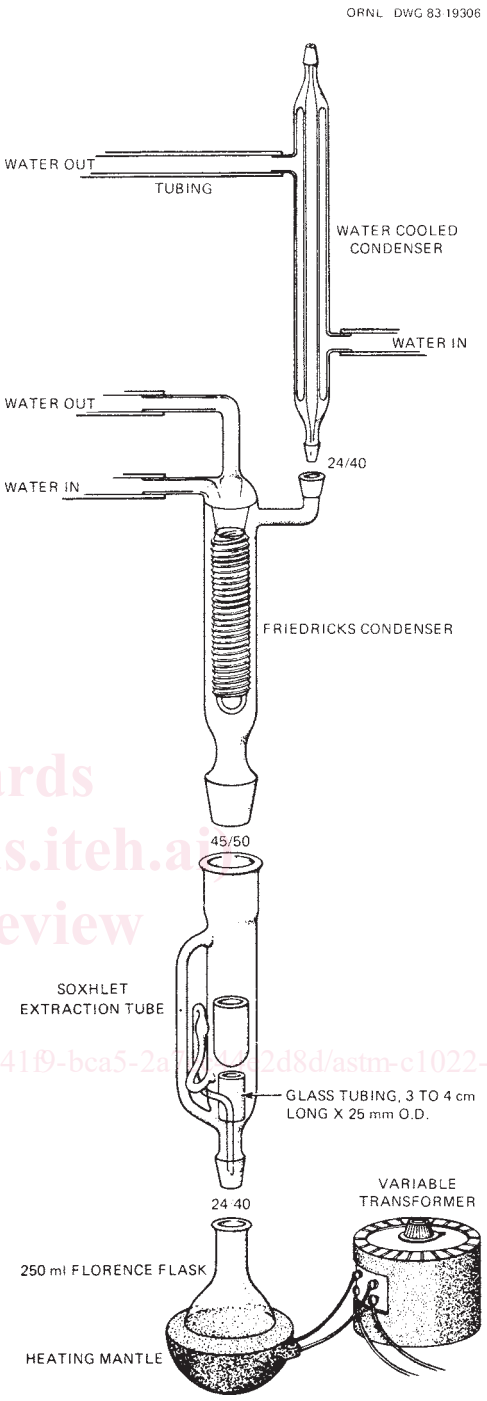

FIG. 1 Freon Extraction Unit

23. Reagents

23.1 *Trichlorofluoromethane* (or 1,1,2 trichlorotrifluoroethane)—Whenever a new supply is used, it should be checked for nonvolatile residue. Evaporate 100.0 mL just to dryness in a weighted platinum dish, cool to room temperature, and reweigh the dish. If there is any residue, either make the appropriate blank correction or distill the solvent before use to remove the nonvolatile impurities.

23.2 *Nitric Acid* (1 + 1)—Mix equal volumes of concentrated (sp gr 1.42) reagent grade HNO_3 and distilled water.

23.3 *Chloroform*—Whenever a new supply of chloroform is to be used, it should be checked for nonvolatile residue as described in 23.1.

24. Procedure

24.1 Weigh 50.0 g of well-mixed, undried uranium-concentrate sample and transfer to an extraction thimble while tapping the thimble on a table top to compact and level the sample.

24.2 Place a plug of glass wool in the thimble above the sample. Support the thimble on the glass tube in the Soxhlet

[7] Whatman brand size 33 by 94 mm has been found suitable.

[8] Whatman IPS has been found suitable.

extraction tube so that when solvent condenses on the lower tip of the Friedrichs condenser, it will drop into the thimble.

24.3 Connect the extraction tube to the bottom of the Friedrichs condenser that is in series with the Graham condenser. Turn on the tap water coolant to the condensers.

NOTE 6—Tap water may be used in cooling both condensers if the amount of reagent lost during the refluxing (see 24.5) is not greater than 10 % of the volume added in 24.4. If the tap water is too warm, then the Graham condenser must be cooled by the refrigerated water cooler, or an ice-cooled condenser may be used in place of the Graham condenser.

24.4 Add a piece of sintered glass or several glass boiling beads and then 120 to 125 mL trichlorofluoromethane to the 250-mL Florence flask. Attach the flask to the Soxhlet extraction tube.

NOTE 7—If the humidity is high and results for extractable organic material on recent lots from the same producer have been less than 0.05 %, 1,1,2 trichlorofluoroethane may be used in place of trichlorofluoromethane to reduce any difficulty caused by the condensation of moisture.

24.5 Place the heating mantle below the Florence flask, connect to the variable transformer set at 55 to 60 V, and allow the reagent to reflux rapidly for 3 ½to 4 h.

24.6 Pour the refluxed reagent into a weighed (W_1 in grams) platinum dish, and evaporate in a hood. An infrared lamp or hot air stream from a heat gun may be used.

NOTE 8—Exercise care in this evaporation. If a heat source is used, adjust the rate of heat input and velocity of air across the dish so that no sample will be mechanically lost. If a heat gun is used, the amount and temperature of the air directed against the sample are especially critical because the high rate of evaporation is likely to lower the temperature of the solution to the point where water will condense in the dish.

24.7 Allow the dish to come to room temperature while tilting and rotating it to spread the last few drops of solvent uniformly over the bottom.

NOTE 9—Do not allow the temperature of the dish to go below the dewpoint.

24.8 Weigh in open air at intervals on an analytical balance, recording the weight of the dish 5 min after the rate of loss has decreased to 0.5 mg/min.

NOTE 10—This weight is in grams as W_2.

24.9 Add a plastic-covered magnetic stirring bar and 100 mL of (1 + 1) nitric acid to a 400-mL beaker.

24.10 While magnetically stirring the acid, cautiously add the extracted sample from the extraction thimble. Stir until the sample is dissolved or until it is apparent that practically no more sample will dissolve.

24.11 Cool to about room temperature and transfer to a 500-mL separatory funnel. Add 100.0 mL of chloroform, stopper tightly, and shake as vigorously as possible for 60 s.

24.12 Allow the phases to separate.

NOTE 11—If emulsions form, transfer to centrifuge tubes and centrifuge to separate the phases.

24.13 Drain off the lower phase. If the lower phase is the chloroform layer, filter through a phase-separator filter paper into a graduated cylinder or narrow-neck flask. If the lower phase is the aqueous phase, drain and discard. Then filter the upper phase through a phase-separator filter paper into a graduated cylinder or narrow-neck flask.

24.14 Transfer 50.0 mL of the filtered chloroform into an ignited (900°C) platinum dish.

24.15 Place the platinum dish in a hood and evaporate until about 1 mL of chloroform remains. This evaporation may be done as described in 24.6.

24.16 Allow the dish to cool to room temperature while tilting and rotating it to spread the last few drops uniformly over the bottom.

24.17 Weigh in open air on a recording balance or at intervals on an analytical balance, recording the weight of the dish 5 min after the rate of weight loss has decreased to 0.5 mg/min.

NOTE 12—This weight is in grams as W_3.

24.18 Ignite the platinum dish at 900°C for a minimum of 30 min, cool to room temperature, and weigh.

NOTE 13—This weight is in grams as W_4.

25. Calculation

25.1 Calculate the percentage of extractable organic material, O_m, as follows:

$$O_m = \frac{100\,[(W_2 - W_1) + 2\,(W_3 - W_4)]}{S_w} \quad (4)$$

where:

W_2 = weight of platinum dish in 24.8, g,
W_1 = weight of platinum dish in 24.6, g,
W_3 = weight of platinum dish in 24.17, g,
W_4 = weight of platinum dish in 24.18, g, and
S_w = weight of sample.

26. Precision and Bias

26.1 *Precision*—A relative standard deviation for this test method has been reported as 18 % at the 0.1 % extractable organic level (see 4.2).

26.2 *Bias*—For information on the bias of this test method see 4.2.

ARSENIC BY DIETHYLDITHIOCARBAMATE (PHOTOMETRIC) METHOD

27. Scope

27.1 This test method covers the determination of arsenic in uranium-ore concentrate.

27.2 Sample aliquots containing up to 25 µg of arsenic may be analyzed by this test method.

28. Summary of Test Method

28.1 Arsenic compounds are reduced to gaseous arsine by hydrogen generated by zinc in an acid medium. The resulting mixture of gases is passed through a scrubber containing borosilicate wool impregnated with lead acetate solution, and then into an absorption tube containing a solution of silver diethyldithiocarbamate dissolved in pyridine. Arsine reacts with this reagent to form a soluble red substance having maximum absorbance at 540 nm. The absorbance of the

solution is measured spectrophotometrically and the arsenic determined by reference to a calibration curve prepared from standards.

29. Interferences

29.1 Although many samples are relatively free of interferences, cobalt, nickel, mercury, silver, platinum, copper, chromium, and molybdenum may interfere with the evolution of arsine and with the recovery of arsenic. The presence of any or all of these metals in a sample being analyzed must be considered as a potential source of interference and the analyst must be aware of the extent of actual interferences if any.

29.2 Hydrogen sulfide and other sulfides interfere, but commonly encountered quantities are effectively removed by the lead-acetate scrubber.

29.3 Antimony interferes by forming stibine which distills along with the arsine. Stibine reacts with the color-forming reagent to form a red compound having maximum absorbance at 510 nm. The absorbance for antimony at 540 nm is about 8 % of that of arsenic for equal concentrations.

30. Apparatus

30.1 *Arsine Generator, Scrubber, and Absorber*. [9]

30.2 *Spectrophotometer*, with 1-cm cells in accordance with Practice E 60.

31. Reagents

31.1 *Arsenic Solution, Standard I* (1 mg As/mL)—Dissolve 1.320 g of arsenic trioxide (As_2O_3) (**Warning**, Note 14) dried for at least 1 h at 110°C, in 10 mL of 10 *M* sodium hydroxide (NaOH) solution and dilute to 1 L with water. This solution is stable.

NOTE 14—**Warning:** Arsenic trioxide is extremely toxic. Avoid ingestion.

31.2 *Arsenic Solution, Standard II* (10 µg As/mL)—Dilute 5 mL of arsenic standard solution I to 500 mL with water in a volumetric flask.

31.3 *Arsenic Solution, Standard III* (1 µg As/mL)—Dilute 10 mL of arsenic standard solution II to 100 mL with water in a volumetric flask. Prepare freshly before each use.

31.4 *Hydrochloric Acid* (HCl, sp gr 1.19)—Use analytical-grade acid with an arsenic content no greater than 0.01 µg/mL.

31.5 *Lead Acetate Solution* (100 g/L)—Dissolve 10 g of lead acetate ($Pb(C_2H_3O_2)_2 \cdot 3H_2O$) in water and dilute to 100 mL. Store reagent in a tightly stoppered container.

31.6 *Nitric Acid* (1 + 1)—Add 250 mL of nitric acid (HNO_3, sp gr 1.42) to 250 mL of water and mix.

31.7 *Perchloric Acid*—($HClO_4$, 62 %), use analytical-grade acid with an arsenic content no greater than 0.01 µg/mL.

31.8 *Potassium Iodide Solution* (150 g/L)—Dissolve 15 g of KI in water and dilute to 100 mL. Store in an amber bottle.

31.9 *Silver Diethyldithiocarbamate - Pyridine Solution* (5 g/L)—Dissolve 0.5 g of silver diethyldithiocarbamate in 100 mL of pyridine. This solution is stable for at least several months when stored in an amber bottle.

31.10 *Sodium Hydroxide Solution* (420 g/L)—Dissolve 42 g of NaOH pellet in water and dilute to 100 mL.

31.11 *Stannous Chloride - Hydrochloric Acid Solution* (400 g/L)—Dissolve 40 g of arsenic-free $SnCl_2 \cdot 2H_2O$ in 100 mL of HCl (sp gr 1.19). Add a few small pieces of mossy tin.

31.12 *Zinc* (*Granular*, 20 mesh)—Arsenic content must not exceed 1 µg of arsenic per gram of zinc.

31.13 *Sulfuric Acid* (H_2SO_4, sp gr 1.84)—Use analytical-grade acid with an arsenic content no greater than 0.01 µg/mL.

32. Precautions

32.1 Take proper precautions to prevent inhalation or ingestion of arsine, arsenic, or pyridine during the analysis. Furthermore, do not allow the pyridine to contact the skin.

33. Calibration

33.1 Clean all glassware before use by rinsing first with hot (1 + 1) HNO_3, then with water. Also, rinse the absorbers with acetone and air dry.

33.2 Prepare a blank and a series of standards by pipetting appropriate volumes of arsenic standard solution III (**Caution**, Note 14) into arsine-generating flasks. The standards should cover the range from 0 to 25 µg of arsenic.

33.3 Dilute each standard and blank to approximately 25 mL.

33.4 Add successively to each flask, with thorough mixing after each addition, 5 mL of HCl (sp gr 1.19), 2 mL of KI solution, and 8 drops of $SnCl_2$ solution. Let stand about 15 min to allow the arsenic to completely reduce to the tirvalent state.

33.5 Place in each scrubber a plug of borosilicate wool that has been impregnated with lead acetate solution. Assemble the generator, scrubber, and absorber, making certain that all parts fit and are correctly adjusted. Add 3.00 mL of silver diethyldithiocarbamate - pyridine solution to each absorber, then add glass beads to the absorbers until the liquid just covers them.

33.6 Disconnect each generator, add 3 g of zinc, and reconnect immediately.

33.7 Allow 30 min for complete evolution of arsine. Warm the generator flasks for a few minutes to make sure all arsine is released.

33.8 Pour the solutions from the absorbers directly into clean spectrophotometer cells and, within 30 min, measure the absorbance of each at 540 nm using water in the reference cell.

33.9 Prepare a calibration curve by plotting micrograms of arsenic against the corresponding absorbance of each standard after correcting for the blank.

34. Procedure

34.1 Clean all glassware by rinsing with hot (1 + 1) HNO_3 before use.

34.2 Weigh 1 g of uranium-ore concentrate into a 250-mL Erlenmeyer flask. Add 12 mL of H_2SO_4, 5 mL of $HClO_4$, and 5 mL (1 + 1) HNO_3. Boil the sample on a hot plate until the fumes of the H_2SO_4 just begin to come off. Cool, transfer to a 100-mL volumetric flask, and dilute to volume with water.

34.3 Pipet an aliquot of sample containing less than 25 µg of arsenic (25 mL maximum) from the volumetric flask into a generating flask and dilute to approximately 25 mL.

[9] An adequate system can be obtained from laboratory supply houses.