Designation: C 1110 – 03

Standard Practice for Sample Preparation for X-Ray Emission Spectrometric Analysis of Uranium in Ores Using the Glass Fusion or Pressed Powder Method[1]

This standard is issued under the fixed designation C 1110; 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 This practice covers the preparation of uranium ore samples to be analyzed by X-ray emission. Two separate techniques, the glass fusion method or the pressed powder method, may be used.

1.2 The values stated in SI (metric) units are to be regarded as the standard.

1.3 *This standard does not purport to address all of the safety problems, 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.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals[2]

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials[2]

3. Terminology

3.1 *Definitions*—Refer to Terminology E 135 for terms used in this practice.

4. Summary of Practice

4.1 Two types of specimen preparation methods are offered. These are the glass fusion technique and the pressed powder technique. The glass fusion specimens are fused and cast into disks in a platimum-gold alloy crucible in the presence of an oxidizing agent at a preset temperature and time. The pressed powder specimens are ground in a mill to a specified particle size and briquetted into a solid specimen possessing a flat homogeneous surface to be analyzed.

5. Significance and Use

5.1 This practice is useful for the preparation of specimens of ore bodies for the analysis of uranium by X-ray emission. Two separate preparation techniques are described.

6. Apparatus

6.1 *Platimum-Gold Crucible*, 95 % platinum 5 % gold, with flat 32-mm diameter bottom.

6.2 *Muffle Furnace*, 1200°C capacity with controller of ±10°C resolution, or fusion apparatus with similar capabilities.

NOTE 1—A fusion apparatus may be substituted for the muffle furnace throughout this practice. If such is used, follow the manufacturer's operating instructions.

6.3 *Rotary Swing Mill*, with tungsten or boron carbide vials.

6.4 *Pellet Die*, 32-mm diameter.

6.5 *Hydraulic Press*, 25-ton capacity.

6.6 *Crucible*, porcelain, Coors, 30-mL capacity.

6.7 *Refractory Brick*, 60 to 70 % alumina firebrick.

6.8 *Analytical Balance*, accurate to ±0.001 g.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[3] 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 (see Practices E 50).

7.2 *Lithium Tetraborate*—($Li_2B_4O_7$), anhydrous, spectrographic grade.

7.3 *Oxidizing Agent*, ammonium nitrate (NH_4NO_3).

7.4 *Ethanol*, anhydrous.

[1] This practice is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Test.

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

[3] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

7.5 *Binding Agent*, for example, pelletizing grade graphite powder, cellulose powder, "Somar Mix," or equivalent.

7.6 *Boric Acid*, 99.9 % pure.

8. Preparation of Standards and Specimens

8.1 *Glass Fusion Method*:

8.1.1 *Ignition Loss of Lithium Tetraborate*:

NOTE 2—Commercially availabe fused and ground lithium tetraborate may be used, and only the 105°C drying step is required.

8.1.1.1 Heat a 95 % platinum/5 % gold crucible in a muffle furnace at 1100°C for at least 10 min and cool to room temperature in a dessicator.

8.1.1.2 Weigh the cooled crucible on an analytical balance to ±0.001 g (W_1).

8.1.1.3 Add to the tared crucible approximately 5 g of lithium tetraborate weighed to the nearest ±0.001 g (W_2).

8.1.1.4 Heat the crucible plus lithium tetraborate in the muffle furnace at 1100°C for 15 min.

8.1.1.5 Remove the crucible from the furnace, cool, and store in a desiccator.

8.1.1.6 Weigh the crucible plus the ignited lithium tetraborate on an analytical balance to ±0.001 g (W_3).

8.1.1.7 Calculate the ignition factor in accordance with Section 9.

8.1.1.8 Remove the lithium tetraborate glass by inverting the crucible and tapping lightly on the bottom. Save this pellet to use as a blank in future calibrations.

8.1.1.9 Clean the platinum crucible.

8.1.1.10 Determine the loss on ignition for samples by following 8.1.1.1 through 8.1.1.7, except ignite 2.0 g of sample in a porcelain crucible.

NOTE 3—Moist specimens should be previously dried at 105°C for 1 h, and the loss on drying should be recorded.

8.1.2 *Specimen Fusion*:

NOTE 4—Numerous ores contain sulfide minerals which at high temperatures will attack the platinum/gold crucible. For specimens that contain sulfide minerals, pre-oxidize a 2-g specimen plus 1.000 g of ammonium nitrate in a porcelain crucible for 15 min at 1100°C. Cool the porcelain crucible, and then use the pre-oxidized material as the specimen and proceed with 8.1.2.1. Ores that contain high concentrations of alkali elements may attack porcelain crucibles; therefore, graphite crucibles are recommended for this pre-oxidation step.

8.1.2.1 Weigh 2.000 g of the specimen into a tared porcelain crucible and ignite at 1100°C for 15 min. Cool and reweigh the crucible and specimen.

8.1.2.2 Weigh 5.000 g of loss-free equivalent lithium tetraborate (determined from igniting the lithium tetraborate), 1.000 g of ignited specimen, and 0.5000 g of ammonium nitrate, all to the nearest 0.001 g.

8.1.2.3 Transfer all three components into a 95 % platinum/5 % gold crucible and briefly mix.

8.1.2.4 Place the crucible and contents in a muffle furnace at 1100°C and fuse for 5 min.

8.1.2.5 Remove the crucible from the furnace with long crucible tongs and swirl the molten material to ensure complete solution and eliminate bubbles.

8.1.2.6 Replace the crucible in the furnace for another 5 min.

8.1.2.7 Repeat 8.1.2.5 and 8.1.2.6 until all of the specimen is dissolved.

8.1.2.8 Remove the crucible from the furnace, place on a refractory brick, and allow it to cool to room temperature.

8.1.2.9 When the crucible is cool, invert and lightly tap it to free the glass disk.

8.1.2.10 Clean the crucible.

8.2 *Pressed Powder Method*:

8.2.1 Grind a representative portion of the specimen weighing at least 5 g to 44 µm (325 mesh).[4] Mix the resulting powdered specimen if necessary.

NOTE 5—Grinding aids may improve grinding time and efficiency.[5]

8.2.2 Press 5 g of the ground specimen at 25 tons for 30 s. Use a suitable pelletizing agent, if necessary.

8.2.3 Clean the mill and die to avoid contaminating the next specimen. Brush any remaining powder residue from the mill and wipe out remaining traces with a paper towel wetted with a suitable solvent such as ethanol. Clean the die assembly in a similar manner.

8.3 Prepare all calibration standards using the same method as used for specimen preparation.

9. Calculation

9.1 *Ignition Factor*— Calculate the ignition factor as follows:

$$IF = (W_3 - W_1)/(W_2 - W_1) \quad (1)$$

where:

IF = ignition factor,
W_1 = weight of crucible, g,
W_2 = weight of crucible and fusion powder before ignition, g, and
W_3 = weight of crucible and fusion powder after ignition, g.

10. Precision and Bias

10.1 No statement is made about the precision or the bias of this practice since this practice addresses only preparation for specimen analysis, and therefore no data are generated.

11. Keywords

11.1 glass fusion; pressed powders; sample preparation; uranium ores; x-ray fluorescence

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[4] Wheeler, B., "Particle Size Effects in X-ray Fluorescence Analysis," *Advances in X-ray Analysis, Proceedings of the 32nd Annual Conference on Application of X-ray Analysis*, Vol 27, Denver, CO, Aug 1984.

[5] Berstein, F., "Particle Size and Mineralogical Effects in Mining Applications," *11th Annual Conference on Application of X-ray Analysis*, Denver Research Institute, University of Denver, Denver, CO, 1962.