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Standard Guide for Determination of Sodium and Potassium Content For Silver-Cadmium Oxide Contact Materials[1]

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1. Scope

1.1 This guide describes procedures for use in conducting analysis of silver-cadmium oxide materials for sodium and potassium content. The exact methods for the performance of this testing are not stated since many different procedures are equally applicable. This guide points out specific procedures for decreasing the chance of recognized potential errors and specifies methods for increasing the sensitivity of the testing.

1.2 *This standard may involve hazardous materials, operations, and equipment. 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 this standard to become familiar with all hazards including those identified in the appropriate Material Safety Data Sheet (MSDS) for this product/material as provided by the manufacturer, to establish appropriate safety and health practices, and determine the applicability of regulatory limitations prior to use.* For specific precautions see Sections 4 and 6.

1.3 Whoever uses this standard should consult the Material Safety Data Sheet concerning the products involved.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E663 Practice for Flame Atomic Absorption Analysis (Withdrawn 1997)[3]

2.2 *Other Document:*

Operation Guide for Chemical Analysis of Metals and Metal Bearing Ores by Atomic Absorption Spectrophotometry[4]

3. Significance and Use

3.1 This guide is recommended for use in analyzing the sodium and potassium content of silver-cadmium oxide materials and is meant to be used in conjunction with standard methods of atomic absorption spectroscopy. The recommended procedures cite methods for minimizing extraneous sodium and potassium contamination through use of specific handling procedures and shorter sample preparation methods. This guide also recommends procedures that prevent the formation of nonstable and potentially hazardous silver-acetylene compounds.

4. Precautions

4.1 Since silver nitrate reacts with acetylene to form silver acetylide, the guide recommends either alternative flames to air–acetylene or the removal of silver from solution. Silver acetylide is unstable and may explode violently upon being disturbed.

5. Sampling

5.1 Handle the sample so as to avoid contamination by sodium and potassium; avoid contact with hands.

6. Procedures

6.1 First clean the sample in nitric acid and distilled or deionized water to remove extraneous surface contamination. Test powder samples as received. Then store the samples in such a manner as to minimize contamination.

6.2 Use a sample size of approximately 2 g or more for each test.

6.3 Handle the samples with gloves and utensils, never with bare hands.

6.4 The use of TFE-fluorocarbon or other suitable substitutes for glassware is recommended. If glass is used, aged glass

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] 1982 Annual Book of ASTM Standards, Part 12 (see Related Materials Section).

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rather than new glass is recommended. Clean the glassware with ammonia and rinse with deionized water. Avoid the use of detergents.

6.5 Calibrate the instrument and determine the optimum calibration-solution concentration in accordance with Practice E663 and other standards as cited in Section 2.

6.6 Fully dissolve the sample in a nitric acid solution in accordance with standard procedures and concentrations recommended for the atomic absorption procedures and instruments used. Also prepare a reagent blank.

6.7 An air-propane or air-hydrogen flame is recommended rather than air-acetylene in order to prevent the formation of silver acetylide. (**Warning—**Silver acetylide may explode violently if disturbed.) The silver then may be left in solution.

6.8 If an air-acetylene flame is used, a precipitation and settling procedure is recommended over filtrations for removal of silver. A settling time of 24 h is recommended in laboratory ware that will not contribute to contamination followed by careful aspiration of the supernatant solution into the air-acetylene flame. If the choice is made to remove the silver from solution, the possibility of some of the sodium and potassium being removed with the precipitate should be recognized.

7. Report

7.1 Since slight variations in technique can easily cause discrepancies between laboratory results, the exact method used shall be listed. The report statement shall include such items as follows:

TABLE 1 Sodium Analysis

Mean, ppm	σ	
	ppm	%
7	4	57
35	8	23
190	26	14
500	94	19

TABLE 2 Potassium Analysis

Mean, ppm	σ	
	ppm	%
6	11	180
12	4	33

7.1.1 Sample size,
7.1.2 Type of solution containers used,
7.1.3 Whether silver was precipitated, and
7.1.4 Specific type of gas flame used.

7.2 Supplier and user shall reach agreement concerning all variables in technique and procedure before attempting to compare results of specific samples.

8. Precision

8.1 The precision is based on the tests performed by competent operators of a number of different laboratories; see Table 1 and Table 2. All the laboratories were given the basic guidelines as described in this guide.

9. Keywords

9.1 analysis; cadmium oxide; contacts; potassium; silver; silver–cadmium oxide; sodium

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