



Standard Practice for Determination of Water Soluble Alkali Content in Coal¹

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1. Scope

1.1 This practice describes a procedure for the determination of water soluble sodium as sodium oxide (Na_2O) and potassium as potassium oxide (K_2O) in the analysis of coal samples prepared in accordance with Practice [D2013](#).

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 This practice is derived in part from the ASME procedure water soluble alkalis.²

1.4 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*³

[D121 Terminology of Coal and Coke](#)

[D1193 Specification for Reagent Water](#)

[D2013 Practice for Preparing Coal Samples for Analysis](#)

[D3173 Test Method for Moisture in the Analysis Sample of Coal and Coke](#)

[D3302 Test Method for Total Moisture in Coal](#)

[D3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases](#)

[D7448 Practice for Establishing the Competence of Laboratories Using ASTM Procedures in the Sampling and Analysis of Coal and Coke](#)

[D7582 Test Methods for Proximate Analysis of Coal and Coke by Macro Thermogravimetric Analysis](#)

[E832 Specification for Laboratory Filter Papers](#)

¹ This test method is under the jurisdiction of ASTM Committee [D05](#) on Coal and Coke and is the direct responsibility of Subcommittee [D05.21](#) on Methods of Analysis.

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² "Coal Fouling and Slagging Parameters." ASME H-86. Editor E.C. Winegartner, 1974.

³ 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. Terminology

3.1 For definitions of terms used in this practice, refer to Terminology [D121](#).

4. Summary of Practice

4.1 Sodium and potassium salts are leached from the sample during a lengthy digestion with water and a wetting agent. The concentrations of sodium and potassium are measured in the filtered liquid by an appropriate spectrometric technique for the elements sodium and potassium.

5. Significance and Use

5.1 Results obtained from the practice are used to serve a number of interests, including the indication of possible boiler tube fouling and possible furnace corrosion problems.

6. Interferences

6.1 Several types of interference effects may contribute to inaccuracies in the determination of sodium and potassium. Follow the manufacturer's operating guide to develop and apply correction factors to compensate for the interferences.

7. Apparatus

7.1 *Analytical balance*, with sensitivity of 0.1 mg.

7.2 *Digestion Apparatus*

7.2.1 *Stirring Hotplate and Bars*, with operating temperature up to 200 °C.

7.2.1.1 Erlenmeyer flask with cover, 250 mL.

7.2.1.2 Refluxing column (optional).

7.2.2 *Hot Block Digestion*

7.2.2.1 *Polycarbonate Bottles*, 250 mL capacity with an O-ring seal and screw cap, capable of withstanding temperatures up to 130 °C, the pressure that is developed during the digestion, and resistant to oxidation. Other types of bottles or vials may be used provided they are capable of withstanding the temperatures and pressures developed during the digestion.

7.3 *Filtering Apparatus*

7.3.1 Filter Paper or 0.45 micron filter. The paper used must be ash-free, quantitative Type II filter paper, as described in Specification [E832](#).

7.3.2 Filter (gravity or vacuum).

7.3.3 Volumetric flask, 250 mL.

7.4 *Spectrometer*—Because of the differences between various makes and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, follow the instructions provided by the manufacturer of the particular instrument. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be investigated and established for each individual analyte line on that particular instrument. All measurements must be within the instrument's linear range in which correction factors are valid. It is the responsibility of the analyst to verify that the instrument configuration and operating conditions used satisfy the analytical requirements of this method and to maintain quality control data confirming instrument performance and analytical results.

8. Reagents and Materials

8.1 *Purity of Reagents*—Use reagent grade chemicals in all tests that conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society,⁴ where such specifications are available.

8.2 *Purity of Water*—Unless otherwise indicated, references to water are understood to mean Type II reagent water as defined in Specification **D1193**.

8.3 *Wetting Agent*—Approximately 1 to 5 mL of isopropyl alcohol, methanol or ethanol may be added to the sample.

8.4 *Calibration Standards*—Standard stock solutions of 1000 mg/L for each element are needed for preparation of dilute calibration standards in the nominal range from <0.1 to 100 mg/L. Prepare calibration standard solutions from 99.99 % (mass/mass) purity metals or salts. Alternatively, use commercially available standard stock solutions specifically prepared for the correct spectroscopy method. Dilute calibration standard solutions are also used initially and periodically to verify the baseline of the calibration has not changed significantly.

8.5 *Blank Solutions*—Two types of blank solutions are required: a calibration blank of water that is used to establish the analytical calibration curve and a method blank which is used to evaluate possible contamination and assess spectral background. The calibration blank is also used initially and periodically to verify the baseline of the calibration has not changed significantly.

8.5.1 *Calibration Blank*—water.

8.5.2 *Method Blank*—Process the method blank through the same digestion procedure as the samples, using the same volumes of water and wetting agent as used in preparing the samples.

9. Analysis Sample

9.1 Prepare the analysis sample in accordance with Practice **D2013** for coal by pulverizing the material to pass a 250 μm (No. 60) U.S.A. standard sieve. Determine moisture in accordance with Test Method **D3173**, **D3302**, or **D7582** to permit calculations to other than as-determined bases.

10. **Procedure**

10.1 The solutions and proportions described below are the typical samples as represented by American coals. Therefore, stronger or weaker dilutions may be required to establish suitable concentrations for those elements of varying percents outside the range of the typical sample. Analysts must determine the sensitivity and linear range of calibration of their own equipment and choose concentration ranges for standards compatible with the samples and instrument specific to their own work.

10.2 *Sample Digestion*

10.2.1 *Method A, Hot Plate*—Accurately weigh 5 g of sample to the nearest 0.1 mg into a 250 mL Erlenmeyer flask. The sample may be wetted with 1 to 5 mL of wetting agent. Add 100 mL of water to the flask. Place the flask with sample onto the hot plate and cover. Allow to digest just under boiling for at least 16 h. Do not allow the sample to go to dryness (a refluxing column may be used to prevent this). Allow to cool to room temperature. Filter by either gravity filtration or vacuum filtration into a 250 mL volumetric flask. Once filtration is complete, dilute the filtrate to volume (250 mL) with water and mix by inversion.

10.2.2 *Method B, Hot Block*—Accurately weigh 1 g of sample to the nearest 0.1 mg into a weighing dish and quantitatively transfer to a polycarbonate bottle. The sample may be wetted with 1 to 5 mL of wetting agent. Add 25 mL of water to the tube. Place the sample on the hot block and digest at 120 ± 5 °C for 16 h. Do not allow the sample to go to dryness. Allow to cool to room temperature. Filter by either gravity filtration or vacuum filtration into a 50 mL centrifuge tube. Once filtration is complete, dilute the filtrate to volume (50 mL) with water and mix by inversion.

10.3 *Determination of Sodium and Potassium in the Test Solution*—Measure the concentrations of sodium and potassium in the test solution (filtrate from 10.2.1 or 10.2.2) according to the selected spectrometric method.

11. Instrument Operation

11.1 Consult the manufacturer's instructions for the chosen spectrometer. The present method assumes that good operating procedures are followed. Design differences among instruments and different selected analytical wavelengths for individual spectrometers make it impractical to list detailed conditions.

11.2 *Calibration Procedure*—Calibrate the instrument according to the procedure recommended by the manufacturer using a calibration blank and calibration standards.

11.3 *Initial Calibration Verification*—Before analyzing test samples, analyze the method blank and verify the proper calibration of the instrument by analyzing a reference material. Results for the reference material must be within the stated uncertainty limits or the calibration procedure must be repeated.

⁴ *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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

11.4 *Periodic Calibration Verification and Recalibration*—In accordance with Guide **D7448**, analyze a control sample on a periodic basis. Results obtained for the control sample must be within 10 % of the stated value or all results obtained since the last successful control check for that element must be rejected and the calibration procedure repeated.

12. Report

12.1 For reporting analyses to other than as-determined basis, refer to Practice **D3180**.

12.2 For instruments that do not have calculation functions provided by the manufacturer for converting the concentration amount of the analyte as analyzed to the appropriate oxide, the following calculations can be used:

12.2.1

$$\%Na_2O \text{ (as Det)} = \frac{(A - B)VC}{D} \times 1.348 \times \frac{(1 \text{ g Na})}{(1000 \text{ mg Na})} \times 100 \% \quad (1)$$

where:

- A* = as measured concentration of Na, mg/L,
- B* = as measured concentration of blank, mg/L,
- V* = volume of solution, L
- C* = dilution factor, if applied, and
- D* = sample mass, g (as determined basis).

Note:

$$\frac{(MW \text{ Na}_2\text{O})}{2(AW \text{ Na})} = 1.348$$

where:

- MW* = molecular weight, and
- AW* = atomic weight.

12.2.2

$$\%K_2O \text{ (as Det)} = \frac{(A - B)VC}{D} \times 1.205 \times \frac{(1 \text{ g K})}{(1000 \text{ mg K})} \times 100 \% \quad (2)$$

where:

- A* = as measured concentration of K, mg/L,
- B* = as measured concentration of blank, mg/L,
- V* = volume of solution, L
- C* = dilution factor, if applied, and
- D* = sample mass, g (as determined).

Note:

$$\frac{(MW \text{ K}_2\text{O})}{2(AW \text{ K})} = 1.205$$

where:

- MW* = molecular weight, and
- AW* = atomic weight.

13. Keywords

13.1 alkali; potassium oxide; sodium oxide; water soluble

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