



Standard Test Method for Determination of Chlorine in Coal by Oxidative Hydrolysis Microcoulometry¹

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1. Scope

1.1 This test method covers the determination of total chlorine in coal.

2. Referenced Documents

2.1 ASTM Standards:

D 2013 Method of Preparing Coal Samples for Analysis²
D 3173 Test Method for Moisture in the Analysis Sample of Coal and Coke²

D 3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases²

D 4621 Guide for Accountability and Quality Control in the Coal Analysis Laboratory²

D 5142 Test Methods for Proximate Analysis of the Analysis Samples of Coal and Coke by Instrumental Procedures²

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications³

2.2 Other Standards

ISO 5725-6:1994 Accuracy of measurement methods and results-Part 6: Use in practice of accuracy values⁴

3. Summary of Test Method

3.1 A 5.00 to 40.00 mg sample of coal is combusted with tungsten accelerator in a humidified oxygen gas flow, at 900°C. Halogens are oxidized and converted to hydrogenated halides, which are flushed into a titration cell where they accumulate. Chlorine is converted to hydrochloric acid. Once the chloride is captured in the electrolyte of the titration cell, it can be quantitatively determined by microcoulometry, where chloride ions react with silver ions present in the electrolyte. The silver ion thus consumed is coulometrically replaced and the total electrical work needed to replace it is proportional to the chloride in the test sample.

4. Significance and Use

4.1 This test method permits measurements of the chlorine content of coals.

5. Interferences

5.1 Bromides and iodides, if present are calculated as chloride. However, fluorides are not detected by this test method.

6. Apparatus

6.1 *Hydrolysis Furnace*, which can maintain a minimum temperature of 900°C.

6.2 *Hydrolysis Tube*, made of quartz and constructed such that when the sample is combusted in the presence of tungsten accelerator and humidified oxygen, the byproducts of combustion are swept into a humidified hydrolysis zone. The inlet end shall allow for the introduction and advancement of the sample boat into the heated zone. The inlet shall have a side arm for the introduction of the humidified oxygen gas. The hydrolysis tube must be of ample volume, and have a heated zone with quartz wool so that complete hydrolysis of the halogens is ensured.

6.3 *Titration Cell*, containing a reference electrode, a working electrode, and a silver sensor electrode, a magnetic stirrer as well as an inlet from the hydrolysis tube.

6.4 *Microcoulometer*, capable of measuring the potential of the sensing-reference electrode pair, comparing this potential with a bias potential, and amplifying the difference to the working electrode pair to generate current. The microcoulometer output voltage should be proportional to the generating current.

6.5 *Controller*, with connections for the reference, working, and sensor electrodes, for setting operating parameters and for data integration.

6.6 *Hydration Tube*, containing water, positioned before the gas inlet on the side arm of the combustion tube, through which oxygen gas bubbles to provide a hydrated gas flow.

6.7 *Dehydration Tube*, positioned at the end of the hydrolysis tube so that effluent gases are bubbled through a 95 % sulfuric acid solution. Water vapor is subsequently trapped while other gases flow into the titration cell.

¹ This test method is under the jurisdiction of ASTM Committee D05 on Coal and Coke and is the direct responsibility of Subcommittee D05.29 on Major Elements in Ash and Trace Elements of Coal.

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² *Annual Book of ASTM Standards*, Vol 05.06.

³ *Annual Book of ASTM Standards*, Vol 14.02.

⁴ Available from International Organization for Standardization 1 Rue de Varembe, Case Postale 56, CH-1211, Geneva 20, Switzerland