



Designation: D6721 – 24

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

This standard is issued under the fixed designation D6721; 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 test method covers the determination of total chlorine in coal with a mass fraction range of 22 mg/kg to 1136 mg/kg.

1.2 The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:²

D2013 Practice for Preparing Coal Samples for Analysis

D3173 Test Method for Moisture in the Analysis Sample of Coal and Coke

D3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases

D4621 Guide for Quality Management in an Organization That Samples or Tests Coal and Coke (Withdrawn 2010)³

D7582 Test Methods for Proximate Analysis of Coal and Coke by Macro Thermogravimetric Analysis

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 For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at www.astm.org/contact. 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.

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

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 10.00 mg 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 specimen.

4. Significance and Use

4.1 This test method permits measurements of the total chlorine mass fraction in coal.

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

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