Designation: D 5530 – 94 (Reapproved 2003)

Standard Test Method for Total Moisture of Hazardous Waste Fuel by Karl Fischer Titrimetry[1]

This standard is issued under the fixed designation D 5530; 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 by Karl Fischer (KF) titrimetry of total moisture in solid or liquid hazardous waste fuels used by industrial furnaces.

1.2 This test method has been used successfully on numerous samples of hazardous waste fuel composed of solvents, spent oils, paints, and pigments. The expected range of applicability for this test method is between 1.0 and 100 %; however, this evaluation was limited to samples containing approximately 5 to 50 % water.

1.3 The values stated in SI units are to be regarded as the standard.

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:* [2]

D 1193 Specifications for Reagent Water

D 4017 Test Method for Water in Paints and Paint Materials by Karl Fischer Method

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Summary of Test Method

3.1 An amount of solvent (see 7.3) sufficient to immerse the electrode in the titration flask fully (see Note 1) is titrated to dryness as explained in 9.1, and the Karl Fischer reagent factor is determined by titration of measured amounts of water. A weighed portion of the sample is dissolved in KF solvent and titrated with reagent to dryness. If solid material interferes (see 5.3) with the electrode or does not dissolve sufficiently, an extraction using KF solvent is performed prior to introduction into the titration flask. The total moisture in the sample is then determined. The final total moisture percent is an average of two trials for each sample.

3.2 The contents of the tiration flask may be retained and used for additional analyses. The contents of the titration flask will need to be emptied and replaced with new solvent when the capacity of the flask is nearly exhausted or when solid material affects the sensing by the electrode.

4. Significance and Use

4.1 The determination of total moisture is important for assessing the quality of fuels. Water content will affect the heating value of fuels directly and can contribute to instability in the operation of an industrial furnace. Additionally, high water contents can present material handling and storage problems during winter months or in cold environments.

5. Interferences

5.1 A small number of oxidants such as ferric and chromate salts can oxidize iodide and may produce artificially low results.

5.2 Certain reductants oxidized by iodine such as mercaptans, thioacetate, thiosulfate, stannous chloride, sulfides, hydroquinone, and phenylenediamines can consume iodine and may cause artificially high results. Basic materials such as hydroxides, oxides, and inorganic carbonates may cause artificially high results by water-forming reactions.

5.3 Some types of solid material found in waste-derived fuel may interfere with the electrode by blocking its contact with the solvent. Depending on the nature of the solid material, artificially high or low results can occur.

6. Apparatus

6.1 *Karl Fischer Potentiometric Titration Unit*, automated or semi-automated, equipped with a magnetic vessel stirrer. The user must follow the manufacturer's instructions for installation and use.

NOTE 1—The Karl Fischer unit used for developing this test method was equipped with a twin platinum electrode, 25 to 80-mL capacity titration flask, magnetic stirrer, electronic piston burette, adjustable delay interval, LED display, visual and audible endpoint notification.

6.2 *Syringe*, 100-µL capacity, with needle.

[1] This test method is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.06 on Analytical Methods.

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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.