Designation: ~~E778 – 08~~ E778 – 15

Standard Test Methods for Nitrogen in ~~the Analysis Sample of~~ Refuse-Derived Fuel Analysis Samples[1]

This standard is issued under the fixed designation E778; 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 These test methods cover the determination of total Kjeldahl nitrogen in prepared analysis samples of solid forms of refuse-derived fuel (RDF). The procedures measure free ammonia or ammonia formed from the conversion of organic nitrogenous compounds such as amino acids and proteins. However, the procedures may not convert the nitrogenous compounds of some wastes to ammonia. Examples of such compounds that may not be measured are nitro compounds, hydrozones, oxines, nitrates, semicarbazones, pyridines, and some refractory tertiary amines.

1.2 Two alternatives are described for the final determination of the ammonia, the Kjeldahl-Gunning Test Method and the Acid-Titration Test Method.

1.3 The analytical data from these test methods are to be reported as part of the ultimate analysis where ultimate analysis is requested.

1.4 These test methods may be applicable to any waste material from which a laboratory analysis sample can be prepared.

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

1.6 *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.* For specific precautionary statements see 8.4.1 and Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

D5681 Terminology for Waste and Waste Management

E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[3]

E200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis

E790 Test Method for Residual Moisture in a Refuse-Derived Fuel Analysis Sample

E791 Test Method for Calculating Refuse-Derived Fuel Analysis Data from As-Determined to Different Bases

E829 Practice for Preparing Refuse-Derived Fuel (RDF) Laboratory Samples for Analysis (Withdrawn 2002)[3]

3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology D5681.

4. Summary of Test Methods

4.1 The determination of nitrogen is made by either the Kjeldahl-Gunning Test Method (Section 11) or the Acid-Titration Test Method (Section 12). In both these methods the nitrogen in the sample is converted into ammonium salts by destructive digestion

[1] These test methods are under the jurisdiction of ASTM Committee D34 on Waste Management and are the direct responsibility of Subcommittee D34.03 on Treatment, Recovery and Reuse.

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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.

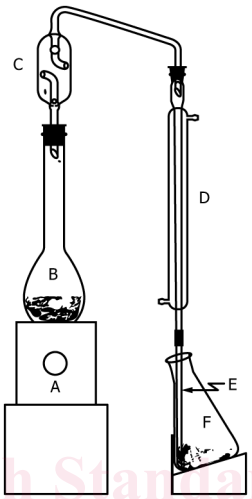
[3] The last approved version of this historical standard is referenced on www.astm.org.

of the sample with a hot, catalyzed mixture of concentrated sulfuric acid and potassium sulfate. The salts are subsequently decomposed in a hot alkaline solution from which the ammonia is recovered by distillation, and finally determined by alkalimetric or acidimetric titration.

5. Significance and Use

5.1 The standard sample is available to producers and users of RDF as a method for determining the weight percent of nitrogen in the analysis ~~sample.~~samples.

5.2 Nitrogen is part of the ultimate analysis and can be used for calculation of combustion parameters.



A = electric heater
B = Kjeldahl digestion flask
C = Kjeldahl connecting bulb
D = condenser
E = connecting tube
F = receiving flask

FIG. 1 Kjeldahl Distillation Apparatus

6. Interferences and Limitations

6.1 Because of the nature of RDF, nitrogenous compounds may be present which will not readily be converted to ammonia by this test method (see 1.1). Modifications to the digestion of the waste may enhance the conversion of these nitrogenous compounds to the ammonium salts.[4]

7. Apparatus

7.1 *Digestion Unit*—An electrical heater of approximately 500-W minimum ~~rating~~ rating, microwave digester, or a gas burner of comparable capacity. ~~Either type of heater~~ These digestion units shall be provided with adequate means of control to maintain digestion rates as described in 11.1 (Note 1). Commercially made, multiple-unit digestion racks provided with fume exhaust ducts may be used.

NOTE 1—If commercially made electrical heaters are used, auxiliary voltage control equipment, such as an autotransformer, may be needed to maintain the specified rates of digestion and distillation.

7.2 *Distillation Unit* (Fig. 1)—An electrical heater or gas burner as described in 7.1. Either type shall be provided with adequate means of control to maintain rates as described in 11.2. Commercially made, multiple-unit distillation racks provided with water-cooled glass or block tin condensers may be used.

7.3 *Condenser,* glass, water-cooled, having a minimum jacket length of 500 mm.

7.4 *Kjeldahl Digestion Flask,* of heat-resistant glass, having a capacity of 500 or 800 mL. Borosilicate glass has been found satisfactory for this purpose.

7.5 *Kjeldahl Connecting Bulb,* cylindrical type, 45 mm in diameter by 100 mm long, or larger, with curved inlet and outlet tubes.

7.6 *Receiving Flasks*—Erlenmeyer flask having a capacity of 250 or 300 mL.

7.7 *Connecting Tube*—Glass tubing approximately 10 mm in outside diameter by 200 mm in length.

[4] Kolthoff, I. M., and Stenger, V. A., *Volumetric Analysis II*, Intersciences Publishers, Inc., New York, NY, pp. 173–176.

7.8 *Pure Gum Rubber Tubing.*

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water, Type II, conforming to Specification D1193, prepared by the passage through an ion-exchange column containing a strongly acid cation resin in the hydrogen form.

8.3 *Potassium Sulfate* (K_2SO_4), crystals.

NOTE 2—Other satisfactory and permissible catalysts for the digestion, together with the quantities of K_2SO_4 required in their use, are as follows:
~~*(1)* Five grams of a mixture containing 32 parts by weight of K_2SO_4, 5 parts by weight of mercuric sulfate ($HgSO_4$), and 1 part by weight of selenium.~~
(1) Five grams of a mixture containing 32 parts by weight of K_2SO_4, 5 parts by weight of mercuric sulfate ($HgSO_4$), and 1 part by weight of selenium.
(2) Three-tenths gram of mercuric selenite ($HgSeO_3$) with 7 to 10 g of K_2SO_4.
(3) Three-tenths gram of cupric selenite dihydrate ($CuSeO_2 \cdot 2H_2O$) with 7 to 10 g of K_2SO_4. When this mixture is used, the addition of a sulfide to the alkali solution is not necessary.
~~*(2)* Three-tenths gram of mercuric selenite ($HgSeO_3$) with 7 to 10 g of K_2SO_4.~~
~~*(3)* Three-tenths gram of cupric selenite dihydrate ($CuSeO_2 \cdot 2H_2O$) with 7 to 10 g of K_2SO_4. When this mixture is used, the addition of a sulfide to the alkali solution is not necessary.~~

8.4 *Mercury,* metal (see Note 2).

8.4.1 **Warning**—Appropriate safety precautions should be used when handling and disposing of mercury and selenium compounds.

8.5 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4).

8.6 *Potassium Permanganate* ($KMnO_4$), crystals.

8.7 *Zinc,* mossy or granular.

8.8 *Alkali Solution*—Dissolve 8.0 g of potassium sulfide (K_2S) and 500 g of sodium hydroxide (NaOH) in water and dilute to 1 L. The use of appropriate amounts of sodium sulfide (Na_2S) or potassium hydroxide (KOH) may be substituted for the above, if desired (Note 2 (*e3*)).

8.9 *Ethyl Alcohol* (95 %)—Ethyl alcohol conforming to Formula No. 30 or 2A of the U.S. Bureau of Internal Revenue. Methyl alcohol may be substituted.

8.10 *Sucrose*—National Bureau of Standards primary standard grade.

8.11 *Reagents Required for Kjeldahl-Gunning Test Method:*

8.11.1 *Methyl Red Indicator Solution* (0.4 to 1 g/L)—Dissolve 0.04 to 0.1 g of methyl red in 50 mL of 95 % ethyl alcohol or methyl alcohol and add 50 mL of water. Bromcresol green indicator solution of equal concentration may be used.

8.11.2 *Sodium Hydroxide, Standard Solution* (0.1 to 0.2*N*)—Prepare and standardize a 0.1 to 0.2 *N* sodium hydroxide (NaOH) solution against a primary standard, as described in Practice E200.

8.11.3 *Sulfuric Acid, Standard Solution* (0.2*N*)—Prepare and standardize a 0.2 *N* sulfuric acid (H_2SO_4) solution as described in Practice E200.

8.12 *Reagents Required Only for Acid-Titration Test Method:*

8.12.1 *Boric Acid Solution* (50 g/L)—Dissolve 5 g of boric acid (H_3BO_3) in 100 mL of boiling water. Allow to cool to room temperature before use.

8.12.2 *Mixed Indicator Solution*—Prepare a solution containing 0.125 % methyl red and 0.083 % methylene blue in 95 % ethyl alcohol or in methyl alcohol. Prepare fresh solution at bimonthly intervals.

8.12.3 *Sulfuric Acid, Standard Solution* (0.1 to 0.2*N*)—Prepare and standardize a 0.1 to 0.2 *N* sulfuric acid (H_2SO_4) solution. Hydrochloric acid (HCl) of similar concentration, as described in Practice E200, may be substituted.

9. Precautions

9.1 Due to the origins of RDF in municipal waste, ~~common sense dictates that~~ precautions should be observed when conducting tests on the samples. Recommended ~~hygienic~~safety practices include use of suitable gloves when handling RDF; wearing dust

[5] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Analar Standards for Laboratory U.K. Chemicals," BDH Ltd., Poole, Dorset, and the "United States Pharmacopoeia".