Designation: E 778 – 87 (Reapproved 2004)

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

This standard is issued under the fixed designation E 778; 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 *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]

D 1193 Specification for Reagent Water

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals

E 200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis

E 790 Test Method for Residual Moisture in Refuse-Derived Fuel Analysis Sample

E 791 Test Method for Calculating Refuse-Derived Fuel Analysis Data from As-Determined to Different Bases

E 829 Practice for Preparing Refuse-Derived Fuel (RDF) Laboratory Samples for Analysis

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *refuse-derived fuel*—solid forms of refuse-derived fuels from which appropriate analytical samples may be prepared are defined as follows in *ASTM STP 832:*[3]

RDF-1—Wastes used as a fuel in as-discarded form with only bulky wastes removed.

RDF-2—Wastes processed to coarse particle size with or without ferrous metal separation.

RDF-3—Combustible waste fraction processed to particle sizes, 95 % passing 2-in. square screening.

RDF-4—Combustible waste fraction processed into powder form, 95 % passing 10-mesh screening.

RDF-5—Combustible waste fraction densified (compressed) into the form of pellets, slugs, cubettes, or briquettes.

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 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.

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[1] These test methods are under the jurisdiction of ASTM Committee D34 on Waste Management and are the direct responsibility of Subcommittee D34.06 on Recovery and Reuse.

Current edition approved Aug. 28, 1987. Published October 1987. Originally published as E 778 – 81. Last previous edition E 778 – 81.

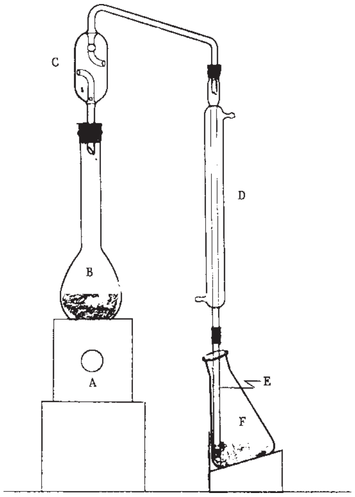
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] *Thesaurus on Resource Recovery Terminology, ASTM STP 832*, ASTM, 1983, p. 72.

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.

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 (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 or a gas burner of comparable capacity. Either type of heater 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.

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 D 1193, 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.

(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 (*c*)).

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

[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".

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 E 200.

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 E 200.

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 E 200, 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 practices include use of gloves when handling RDF; wearing dust masks (NIOSH-approved type), especially while milling RDF samples; conducting tests under a negative pressure hood when possible; and washing hands before eating or smoking.

9.2 The hot acidic and basic solutions in this procedure pose a significant potential hazard. Proper laboratory safety practices and equipment should be employed throughout this procedure.

10. Sampling

10.1 RDF products are frequently nonhomogeneous. For this reason significant care should be exercised to obtain a representative laboratory sample from the RDF lot to be characterized.

10.2 The sampling method for this procedure should be based on agreement between the involved parties.

10.3 The laboratory sample must be air-dried and particle size reduced to pass a 0.5-mm screen as described in Practice E 829. This procedure must be performed carefully to preserve the sample's representative characteristics, other than particle size, while preparing the analysis sample to be used in the procedures.

11. Procedure for Kjeldahl-Gunning Test Method

11.1 *Digestion of Sample*.

11.1.1 After thoroughly mixing the RDF analysis sample to provide the best possible mix of heavy fines with milled fluff, weigh approximately 1 g to the nearest 1 mg of sample into a weighing scoop.

11.1.2 Carefully transfer the sample into a 500 or 800-mL Kjeldahl flask containing 7 to 10 g of K_2SO_4 and 0.6 to 0.8 g of mercury (see Note 3).

11.1.3 Add 30 mL of H_2SO_4(sp gr 1.84) to the mixture by pouring it down the neck of the flask while rotating the flask to wash any sample adhering to the walls into the mixture. Swirl the contents of the flask several times to ensure thorough mixing and wetting of the sample.

11.1.4 Incline the flask at an angle of 45 to 60° on the digestion heater in a fume hood (Note 3). Heat the contents gradually. If frothing or foaming occurs, or both, lower the heat and digest at a lower temperature until the frothing or foaming ceases.

NOTE 3—When fume exhaust ducts or hoods are not available another method must be used to exhaust fumes from the flask, such as aspiration.

11.1.5 Heat the contents to boiling, controlling the heat input in such a manner that the H_2SO_4 vapors condense no more than halfway up the neck of the flask (see Note 1). Continue the digestion until all sample particles are oxidized, as evidenced by a nearly colorless solution, or for at least 2 h after the solution has reached a straw color. The total time of digestion will require 3 to 6 h.

11.1.6 When the digestion is completed and the solution has cooled, a few crystals of $KMnO_4$ may be added to ensure complete oxidation; further heating may be necesary to destroy the excess permanganate and decolorize the solution.

11.2 *Distillation of Digestate* (see Fig. 1).

11.2.1 Dilute the cooled digestion mixture to about 300 mL with water and remove any heat of dilution by cooling the flask under running water or by allowing it to stand until cool.

11.2.2 Accurately pipet 20.0 mL of 0.2 *N* H_2SO_4 into a 250 or 300-mL Erlenmeyer flask. Add 6 drops of methyl red or bromcresol green indicator solution.

11.2.3 Attach the glass connecting tube to the discharge end of the condenser, using a short piece of rubber tubing as a seal.

11.2.4 Incline the Erlenmeyer flask at a suitable angle and insert this tube so that the end is immersed well below the surface of the acid solution (see Fig. 1).

11.2.5 Add 1 to 2 g of granular zinc to the digestion mixture in the Kjeldahl flask (two or three small pieces, if mossy zinc is used), and slowly add 100 mL of alkali solution so that it forms a distinct layer under the acid solution. This may be accomplished by inclining the flask at an angle of 45 to 60° and pouring the alkali solution slowly down the neck of the flask. Failure to maintain discrete layers during the operation may lead to a fairly fast exothermic reaction and loss of ammonia.

11.2.6 Quickly connect the flask to the distilling condenser through the Kjeldahl connecting bulb and swirl the contents to promote thorough mixing.

NOTE 4—All connections must be air-tight so no loss of ammonia will be experienced.

11.2.7 Bring the contents of the Kjeldahl flask to a boil carefully in order to avoid bumping or foaming, or both, and distill the ammonia over into the acid solution in the Erlenmeyer flask.

11.2.8 Continue the distillation at a maximum rate of approximately 350 mL/h until 100 to 150 mL of distillate have been collected.

11.2.9 Discontinue the boiling, and remove the glass connecting tube from the condenser and Erlenmeyer flask. Rinse the tube with water, collecting the washings in the Erlenmeyer flask.

11.2.10 Titrate the excess acid in the Erlenmeyer flask to a methyl red or bromcresol green end point, using 0.1 to 0.2 *N* NaOH solution as the titrant.

11.3 *Blank Determination*—Run a blank determination in the same manner as described in 11.1 and 11.2, using approximately 1 g of sucrose (weighed to the nearest 1 mg) as the sample material.

NOTE 5—A blank determination must be made with every series of analyses performed. The blank determination serves two principal functions:

(1) Since the principle of the method is ascertaining the amount of standardized acid being consumed in a reaction and a back-titration is necessary, the blank is a check on the concentration of the standard solutions.

(2) The blank serves as a correction for nitrogen from sources other than the sample.

12. Calculation

12.1 Calculate the percentage of nitrogen in the analysis sample as follows:

$$\text{Nitrogen, \%} = \frac{(B - A)\, N \times 0.014}{C} \times 100 \qquad (1)$$

where:

A = millilitres of NaOH solution required for titration of the sample,
B = millilitres of NaOH solution required for titration of the blank,
N = normality of the NaOH solution,
C = grams of sample used, and
0.014 = milliequivalent weight of nitrogen.

13. Procedure for Acid-Titration Test Method

13.1 *Digestion of Sample*.

13.1.1 Digest the sample as described in 11.1.

13.2 *Distillation of Digestate* (see Fig. 1).

13.2.1 Dilute the cooled digestion mixture to about 300 mL with water and remove any heat of dilution by cooling the flask under running water or by allowing it to stand until cool.

13.2.2 Add 20 mL of H_3BO_3 solution into a 250 or 300-mL Erlenmeyer flask and add 6 drops of mixed indicator solution.

13.2.3 Set up the distillation apparatus and distill as described in 11.2.3-11.2.9.

13.2.4 Titrate the ammonia collected in the Erlenmeyer flask containing the H_3BO_3 to the mixed indicator end point using 0.2 *N* H_2SO_4 as the titrant.

13.3 *Blank*—Run a blank determination in the same manner as described in 11.1 and 11.2, using approximately 1 g (weighed to the nearest 1 mg) of sucrose as the sample material (Note 5 (*2*)).

14. Calculation

14.1 Calculate the percent of nitrogen in the analysis sample as follows:

$$\text{Nitrogen, \%} = \frac{(A - B)\, N \times 0.014}{C} \times 100 \qquad (2)$$

where:

A = millilitres of H_2SO_4 required for titration of the sample,
B = millilitres of H_2SO_4 required for titration of the blank,
N = normality of H_2SO_4,
C = grams of sample used, and
0.014 = milliequivalent weight of nitrogen.

15. Report

15.1 The results of the nitrogen analysis may be reported on any number of bases, differing from each other in the manner by which moisture is treated.

15.2 The numerical moisture value established by Test Method E 790, shall be used for converting nitrogen data from the as-determined basis to the dry basis as in Test Method E 791.

16. Precision and Bias [6]

16.1 *Precision*:

16.1.1 The standard deviations of individual determinations, in percent absolute, are as follows:

Typical Average Value, %	Within-Laboratory, %	Between-Laboratories, %
0.6	0.04	0.05

16.1.2 These precision estimates are based on an interlaboratory study conducted in accordance with Practice E 180.

16.2 *Bias*—The bias of this test method can not be determined due to the lack of a recognized standard reference material.

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[6] Supporting data are available on loan from ASTM Headquarters. Request RR:E38-1000.

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