



Designation: E 926 – 94

Standard Practices for Preparing Refuse-Derived Fuel (RDF) Samples for Analyses of Metals¹

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1. Scope

1.1 These practices described herein cover the preparation of milled refuse-derived fuel (RDF) sample for analyses of metals by atomic absorption spectroscopy or inductively coupled plasma spectroscopy (see Appendix X1), or both.

1.2 These practices may be applicable to any waste material from which a laboratory analysis sample can be prepared.

1.3 The following four practices are described in this standard:

1.3.1 *Practice A*—Nitric-Hydrofluoric-Perchloric Acid Digestion.

1.3.2 *Practice B*—Nitric-Sulfuric-Hydrofluoric Acid Digestion.

1.3.3 *Practice C*—Bomb, Acid Digestion Method.

1.3.4 *Practice D*—Oxygen Bomb Combustion Method.

1.4 The four practices will cover the scope of preparing most RDF samples for subsequent analyses of most metals. Many times the nature of the sample and the metal in question will dictate the sample preparation technique. In such cases, modifications of these practices may be made with care as to not alter the integrity of the analyte in question.

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 hazard statements, see Sections 9, 10.6, 14, 19, and 24.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water²

E 711 Test Method for Gross Calorific Value of Refuse-Derived Fuel by the Bomb Calorimeter³

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

¹ These practices are under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.06 on Recovery and Reuse.

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 11.04.

Laboratory Samples for Analysis³

3. Summary of Practice

3.1 The organic matter of the analysis sample is completely destroyed by strong acid digestion and oxidation. The inorganic constituents are solubilized and the resulting solution analyzed for metals by either atomic absorption spectroscopy or inductively coupled plasma spectroscopy.

4. Significance and Use

4.1 These practices are available to producers and users of RDF for preparing RDF samples for subsequent analyses of metals.

5. Apparatus

5.1 *Analytical Balance*, capable of weighing to 0.0001 g.

5.2 *TFE-fluorocarbon Beakers*,⁴ 500-mL capacity.

5.3 *Hot Plate*, capable of heating to at least 510°C.

5.4 *Volumetric Flasks*, 50, 100, and 250-mL.

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in this test. 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.

6.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean at least Type III reagent water conforming to Specification D 1193.

6.3 *Nitric Acid* (HNO₃), concentrated sp gr 1.42.

6.4 *Hydrofluoric Acid* (HF), 48 %.

⁴ Tradename for tetrafluoroethylene (TFE) fluorocarbon polymer.

⁵ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.



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6.5 *Perchloric Acid* (HClO_4), concentrated 70 to 72 %.

6.6 *Hydrochloric Acid* (HCl), concentrated sp gr 1.19.

7. Hazards

7.1 Due to the origins of RDF in municipal waste, common sense dictates that precautions should be observed when conducting tests on 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.

7.2 The hot acidic solutions in these procedures pose a significant potential hazard. Proper laboratory safety practices and equipment should be employed throughout these procedures.

7.3 All wet ashing must be performed in a fume hood. Where perchloric acid is used a perchlorate hood must be used.

8. Sampling⁶

8.1 RDF products are frequently non-homogeneous. For this reason significant care should be exercised to obtain a representative laboratory sample from the RDF lot to be characterized.

8.2 The sampling method for these procedures should be based upon agreement between involved parties.

8.3 The laboratory sample is prepared in accordance with Practice E 829.

PRACTICE A—NITRIC-HYDROFLUORIC-PERCHLORIC ACID DIGESTION

9. Hazards

9.1 Hot acidic solutions in this procedure pose a significant potential hazard. Proper laboratory safety practices and equipment should be employed throughout.

9.2 All wet ashing must be performed in a perchlorate fume hood.

9.3 Perchloric acid is a colorless, hygroscopic, strong oxidizing agent. It can explode in contact with organic materials, or by shock or heat. Controlled redox potential of perchloric acid is imperative when used for oxidation of organic material (see Appendix X2).

10. Procedure

10.1 Weigh accurately approximately 5 g of the analysis RDF sample into a 500-mL TFE-fluorocarbon beaker.

10.2 Add 50 mL of concentrated HNO_3 . Cover the beaker with a loose fitting cover.

10.3 Heat on low heat for several hours. Add HNO_3 as needed. Continue heating until most of the organic matter is decomposed.

NOTE 1—Additions of HNO_3 may be made to hot solutions without fear of violent splattering if the acid is added by dropwise additions, or as a thin stream from a wash bottle down the sides of the beaker.

10.4 Slowly add 10 mL of 48 % HF, cover, and continue digesting the sample over low heat. Frequently rinse the inside

walls of the beaker with deionized water to ensure that all material is in contact and can react with the acid mixtures.

10.5 Continue digesting until the solution becomes clear. Add additional HNO_3 as needed.

NOTE 2—Some samples will require several days for complete digestion to occur.

10.6 In a perchlorate hood add 10 mL of HClO_4 to the digestion mixture, cover, and continue digestion on low heat for several hours. At frequent intervals rinse the inside walls of the beaker with HF. Approximately 20 to 25 mL of additional HF can be added during this digestion (see Appendix X2).

NOTE 3—**Warning:** An excessively high temperature may cause an explosion if carbonaceous material is present. The addition of a cold solution to hot acid solutions may cause splattering.

10.7 Remove the cover, elevate the hot plate temperature, and slowly evaporate to dryness.

10.8 Cool the contents and beaker to room temperature.

10.9 Dissolve the solids in 35 to 50 mL of 10 % V/V solution of HCl using moderate heat. If all material is not soluble, add 20 to 25 mL of HF and digest on low heat for an additional hour. Continue as described in 10.7, 10.8, and 10.9.

10.10 Quantitatively transfer the sample solution to a 100-mL volumetric flask and dilute to mark with 1 % HCl solution.

10.11 This solution will contain those metals as contained in 5 g of sample RDF in 100 mL of solution. Should a more concentrated or a more dilute solution be required, the sample size or the final dilution, or both, can be adjusted accordingly. This adjustment will be a function of the magnitude of concentration of the metal in question and the sensitivity of the method of analysis.

10.12 Prepare a reagent blank in the same manner as described in 10.2-10.10 using the same volumes of acids used for the digestion of the sample.

PRACTICE B—NITRIC-SULFURIC-HYDROFLUORIC ACID DIGESTION

11. Summary of Practice

11.1 The organic matter of the analysis sample is completely destroyed by strong acid digestion and oxidation. The inorganic constituents are solubilized and the resulting solution analyzed for metals by either atomic absorption spectroscopy or inductively coupled plasma spectroscopy.

12. Apparatus

12.1 *Analytical Balance*, capable of weighing to 0.0001 g.

12.2 *Berzelius Beakers*, 600-mL capacity.

12.3 *Hot Plate*, capable of heating to at least 510°C.

12.4 *Volumetric Flasks*, 50, 100, and 250-mL.

13. Reagents and Materials

13.1 *Purity of Reagents*—See 6.1.

13.2 *Purity of Water*—See 6.2.

13.3 *Nitric Acid* (HNO_3), concentrated sp gr 1.42.

13.4 *Sulfuric Acid* (H_2SO_4), concentrated sp gr 1.84.

13.5 *Hydrofluoric Acid* (HF), 48 %.

14. Hazards

14.1 Hot acidic solutions in this procedure pose significant

⁶ ASTM Subcommittee E38.01 is currently in the process of developing procedures for sampling RDF.