



Designation: C 1234 – 98

Standard Practice for Preparation of Oils and Oily Waste Samples by High- Pressure, High-Temperature Digestion for Trace Element Determinations¹

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1. Scope

1.1 This practice describes a high-pressure, high-temperature digestion technique using the high-pressure ashers (HPA) for preparation of oils and oily waste specimens for determination of up to 28 different elements by inductively coupled plasma-atomic emission spectroscopy (ICP-AES), cold-vapor atomic absorption spectroscopy (CVAAS), and graphite furnace atomic absorption spectroscopy (GFAAS), inductively coupled plasma-mass spectrometry (ICPMS), and radiochemical methods. Oily and high-percentage organic waste streams from nuclear and non-nuclear manufacturing processes can be successfully prepared for trace element determinations by ICP-AES, CVAAS, and GFAAS. This practice is applicable to the determination of total trace elements in these mixed wastes. Specimens prepared by this practice can be used to characterize organic mixed waste streams received by hazardous waste treatment incinerators and for total element characterization of the waste streams.

1.2 This practice is applicable only to organic waste streams that contain radioactivity levels that do not require special personnel or environmental protection from radioactivity or other acute hazards.

1.3 A list of elements determined in oily waste streams is found in Table 1.

1.4 This test practice has been used successfully to completely digest a large variety of oils and oily mixed waste streams from nuclear processing facilities. While the practice has been used to report data on up to 28 trace elements, its success should not be expected for all analytes in every specimen. The overall nature of these oily wastes tends to be heterogeneous that can affect the results. Homogeneity of the prepared sample is critical to the precision and quality of the results.

1.5 This practice is designed to be applicable to samples whose preparation practices are not defined, or not suitable, by

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TABLE 1 List of Elements and Applicable Lower Concentration Ranges

Element	Lower Reportable Limit, ^A $\mu\text{g/g}$	Analysis Method
Aluminum	3.3	ICP-AES
Antimony	8.3	ICP-AES or GFAAS
Arsenic	8.3	ICP-AES or GFAAS
Barium	0.17	ICP-AES
Beryllium	0.05	ICP-AES
Boron	0.67	ICP-AES
Cadmium	0.50	ICP-AES or GFAAS
Calcium	0.67	ICP-AES
Chromium	1.7	ICP-AES
Cobalt	0.83	ICP-AES
Copper	0.67	ICP-AES
Iron	0.67	ICP-AES
Lead	8.3	ICP-AES or GFAAS
Lithium	0.67	ICP-AES
Magnesium	0.08	ICP-AES
Manganese	0.17	ICP-AES
Mercury	0.03	CVAAS
Nickel	1.7	ICP-AES
Potassium	100	ICP-AES
Selenium	8.3	ICP-AES or GFAAS
Silver	1.0	ICP-AES
Sodium	3.3	ICP-AES
Strontium	0.07	ICP-AES
Thallium	1.7	GFAAS
Titanium	0.50	ICP-AES
Vanadium	0.83	ICP-AES
Zinc	0.17	ICP-AES
Zirconium	0.83	ICP-AES

^ALower reportable limits are based on a 0.3-g sample diluted to a final volume of 50 mL. These limits should only be used as a guide. Actual values are instrument and sample dependent.

other regulatory procedures or requirements, such as the U.S. Environmental Protection Agency (EPA) SW-846 and EPA-600 4-79-020 documents. This digestion practice is designed to provide a high level of accuracy and precision, but does not replace or override any regulatory requirements for sample preparation.

1.6 This practice uses hazardous materials, operations, and equipment at high pressure (90–110 bars, 89–108 atm, or 1305–1595 lb/in.²) and high temperatures, up to 320°C, and therefore poses significant hazards if not operated properly. *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility*

TABLE 2 HPA Temperature Program for Oils and Oily Waste Samples

Phase	Starting Temperature, °C	Time, min	Ending Temperature, °C
1	100	30	125
2	125	60	300
3	300	60	300
4	25	30	25
5	0	0	0

of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific hazard statements are given in Sections 10 and 11.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods³

2.2 US EPA Standards:

Methods for Chemical Analysis of Water and Wastes, 600/4-79-020, 1983⁴

Test Methods for Evaluating Solid Waste, SW-846, 3rd Ed., 1986⁴

3. Terminology

3.1 Definitions:

3.1.1 *heating block*—aluminum block used to hold samples inside the HPA autoclave/pressure chamber.

3.1.2 *HPA-TC controller*—computer interface between HPA autoclave and an IBM-compatible computer.

3.1.3 *pressure chamber*—chamber within the HPA autoclave where heating block filled with samples is placed. The chamber is designed to hold pressures up to 200 bar (197 atm or 2900 lb/in.²) and temperatures up to 320°C.

3.1.4 *safety lid vent stack*—top plate and cylinder that covers the autoclave pressure chamber.

3.1.5 *sample vessel*—sample container, constructed of quartz or glassy carbon, designed for use in the HPA.

3.1.6 *temperature program*—software program which controls the temperature ramping of the HPA during the run. The program used for preparation of oil samples is shown in Table 2.

4. Summary of the Practice

4.1 Oil or oily waste specimens are digested in nitric and hydrochloric acids using HPA high-pressure, high-temperature equipment. Prepared specimens of 0.2 to 0.7 g will provide enough solution for analysis by CVAAS, ICP-AES, and GFAAS for up to 28 elements.

5. Significance and Use

5.1 This practice is useful for preparation of difficult-to-digest, primarily oils and oily wastes, specimens for trace element determinations of up to 28 elements by atomic

absorption or plasma emission techniques. Specimen preparation by high-pressure ashing is primarily applicable to specimens whose preparation by EPA SW-846 protocols is either not applicable or not defined. This sample preparation practice is applicable for the trace element characterization of mixed oily wastes for use by waste treatment facilities such as incinerators or waste stabilization facilities.

6. Interferences

6.1 Preparation of samples for trace element determinations is subject to matrix and chemical interferences. Although the HPA practice is designed to totally digest most matrices, there are some matrix types that are not applicable to this practice, for example, highly reactive substances (explosives), extremely flammable materials, and some silicone-based lubricants.

7. Apparatus

7.1 *HPA High Pressure Asher*⁵—High-pressure, high-temperature autoclave under computer control allowing complete digestion of difficult specimens using mineral acids. The system includes:

7.1.1 Compatible computer with hard drive and with one open RS-232C serial interface.

7.1.2 HPA-TC temperature controller—this device provides the interface between the HPA autoclave and the computer by means of RS-232C serial interface.

7.1.3 HPA autoclave unit.

7.1.4 HPA sample vessels, appropriate size and construction for specimen type and mineral acid used.

7.2 *Ultrasonic Homogenizer*—Specimen homogenizer using ultrasonic disruption tip to homogenize specimens that cannot be mixed by hand.

8. Reagents and Materials

8.1 *Purity of Reagents*—Chemicals used in the preparation of spiking standards must be of ultra purity grade. Chemicals and reagents used in the preparation practice must conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁶ Other grades may be used, provided it is first ascertained that the reagent is of sufficient purity to permit its use without lessening the accuracy of the determination.

8.2 *Reagent Water*—References to water shall be understood to mean reagent water as defined by Type 1 of Specification D 1193.

8.3 *Nitric Acid (sp gr 1.42)*—Ultra pure concentrated nitric acid (HNO₃).

8.4 *Hydrochloric Acid (sp gr 1.19)*—Ultra pure concentrated hydrochloric acid (HCl).

⁵ High-pressure ashing equipment manufactured by Anton Paar K. G., Graz, Austria has been found to be satisfactory for this procedure.

⁶ "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 "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

² Annual Book of ASTM Standards, Vol 11.01.

³ Annual Book of ASTM Standards, Vol 14.02.

⁴ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.