Designation: C 997 – 83 (Reapproved 1993)ε1

Standard Test Methods for Chemical and Instrumental Analysis of Nuclear-Grade Sodium and Cover Gas[1]

This standard is issued under the fixed designation C 997; 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.

ε1 NOTE—Section 264, Keywords, was added editorially in April 1993.

1. Scope

1.1 These test methods provide instructions for performing chemical, radiochemical, and instrumental analyses of sodium metal and for determining impurities in cover gas.

1.2 The analytical procedures appear in the following order:

2. Referenced Documents

2.1 *ASTM Standards:*

A 370 Test Methods and Definitions for Mechanical Testing of Steel Products[2]

C 859 Terminology Relating to Nuclear Materials[3]

D 1193 Specification for Reagent Water[4]

E 146 Methods for Chemical Analysis of Zirconium and Zirconium Alloys[5]

3. Significance and Use

3.1 Sodium metal is used as a coolant (heat-transfer medium) in nuclear reactors, particularly in fast breeder reactors. An inert gas (argon, nitrogen, or helium) is used to cover sodium within a reactor and during transfer and shipping operations to protect it from oxygen and water. To be suitable for use, the metal and gas must meet specified criteria for purity as determined by analysis.

3.2 During reactor operation, chemical and radiochemical impurities resulting from corrosion and neutron activation must be maintained within specification levels established for the reactor system. The sodium and cover gas must be analyzed periodically to monitor buildup of those impurities.

3.3 These methods are applicable to the analysis of sodium and cover gas for the above purposes.

4. Reagents

4.1 *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.[6,7] 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.

5. Safety Precautions

5.1 Sodium is a reactive metal. It reacts vigorously with water and alcohol to form hydrogen, which is easily ignited

[1] These test methods are under the jurisdiction of ASTM Committee C-26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Test Methods.

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[2] *Annual Book of ASTM Standards*, Vol 01.03.

[3] *Annual Book of ASTM Standards*, Vol 12.01.

[4] *Annual Book of ASTM Standards*, Vol 11.01.

[5] Discontinued; see 1991 *Annual Book of ASTM Standards*, Vol 03.05.

[6] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, D.C. 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."

[7] Met-L-X is a tradename for a NaCl-based powder.

and which can cause an explosion. Take care when dissolving a sodium sample, and it is recommended to use a safety shield and fume hood. The proper type of fire extinguisher shall be readily available, and locally established safety precautions for handling sodium shall be followed.

5.2 Radioactive sodium must be handled in fume hoods or other protective facilities, depending upon the degree of radiation exposure involved. Locally established radiation protection and monitoring regulations shall be followed.

BYPASS SAMPLING

6. Scope

6.1 This method is required to obtain a sample for the determination of carbon by the oxyacidic flux method. In addition, it may be used for those procedures in which the sodium is dissolved directly out of the container, whether the solvent is water, alcohol, or mercury.

7. Summary of Method

7.1 A sodium sample is collected in a container that, through extended treatment in flowing sodium, has been cleaned and equilibrated isothermally with the bulk sodium.

8. Apparatus

8.1 *Sampling Vessel*, may be a section of seamless metal tubing; for example, stainless-steel tubing having an inside diameter of >⅜ in. (>9.5 mm) and an internal finish of 32 µin. AA (0.81 mµ) or better, or a vessel as shown in Fig. 1. The vessel in Fig. 1 consists of two matching sections clamped together. Its main body, that has an inside diameter of 0.855 in. (21.7 mm), tapers at each end to an inside diameter of 0.279 in. (7.09 mm). Vessels may be made of either nickel or stainless steel. Attachment of the vessel to a system is done by coupling consistent with locally approved safety practices. Provisions must be available to heat the vessel and maintain its temperature as required.

9. Reagents and Materials

9.1 *Methanol*, redistilled using a quartz or borosilicate glass still and stored in polyethylene bottles. Ethanol may be substituted for methanol.

9.2 *Nitric acid*, diluted 1-part nitric acid with 5-parts distilled water.

9.3 *Water*, distilled and passed through a high-quality mixed-bed ion exchange column and stored in a polyethylene bottle.

10. Precautions

10.1 An important safety consideration in sampling is the mode of connection of the sampler to the system. Three modes of connection for the bypass sampler are by welding, by Swagelok fittings, and by Conoseal fittings. In general, experience has shown that fewer sodium leaks are experienced when connections are welded. This is especially true at temperatures above approximately 400°C (750°F). At low pressures, Swagelok and Conoseal fittings can be used successfully at temperatures moderately above 400°C (750°F). The fittings must be installed, maintained, and monitored in accordance with locally approved safety practices.

11. Procedure

11.1 Rinse the sampling vessel successively with 1 + 5 nitric acid, water, and methanol. Dry, cap, and store until used.

11.2 Attach the sampling vessel to the system in a manner consistent with local safety practices.

11.3 Check the system as follows:

11.3.1 Check the sampling system for leaks according to locally approved operating and safety practices. Use helium-leak testing whenever possible. In that case, a helium-leak rate of $<1 \times 10^{-7}$ $cm^3 \cdot atm/s$ ($<1 \times 10^{-8}$ $m^3 \cdot Pa/s$) through the connectors or welds shall be attained. For systems that can tolerate introduction of small amounts of gas, this step may be replaced by 11.3.2.

11.3.2 Purge the vessel with an inert gas. Connect one fitting to a sampling port while continuing the purge. Discontinue the purge and immediately connect the second fitting to the other sampling port. Check the sampling system for leaks, in accordance with locally approved operating and safety practices. Use helium-leak testing whenever possible. In that case, a leak rate of $<1 \times 10^{-7}$ $cm^3 \cdot atm/s$ ($<1 \times 10^{-8}$ $m^3 \cdot Pa/s$) through the connectors or welds should be attained.

11.4 Heat the entire sampling-vessel system to a temperature greater than 100°C (212°F), taking care to heat progressively from either the solid-liquid or solid-gas interface toward the control valves. Raise the temperature of the entire system to approximately 150°C (300°F).

11.5 Establish sodium flow by opening the outlet and inlet valves in the proper sequence. If step 11.3.2 has been used, the sequence of opening first the outlet and then the inlet valve is desirable because this sequence relieves the gas pressure in the vessel to the outlet line.

11.6 Adjust the sodium-flow rate, if necessary. A minimum flow rate of 0.1 g/m (6.3×10^{-6} m^3/s) should be maintained.

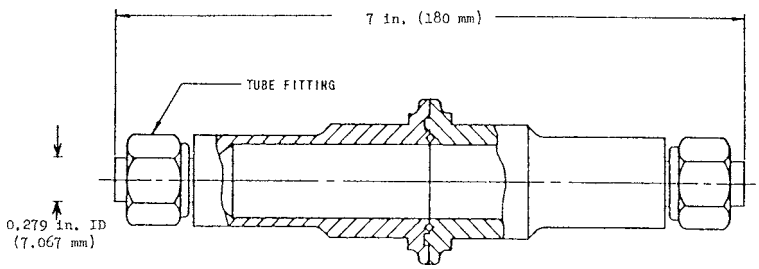

FIG. 1 Typical Sample Vessel