Designation: E1306 – 22

Standard Practice for Preparation of Metal and Alloy Samples by Electric Arc Melting for Spectrochemical Analysis[1]

This standard is issued under the fixed designation E1306; 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 This practice covers the preparation of solid samples of reactive and refractory metals and alloys by electric arc remelting. The samples for melting may be in the form of drillings, chunks, chips, turnings, wire, sponge, powder briquettes, and powdered metals.

1.1.1 This practice is also suitable for preparation of solid samples of other metals, such as cast irons, steels, stainless steels, tool steels, nickel, nickel alloys, cobalt, and cobalt alloys.

1.2 *Units*—The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

1.3 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 9.

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E876 Practice for Use of Statistics in the Evaluation of Spectrometric Data (Withdrawn 2003)[3]

E1010 Practice for Preparation of Disk Specimens of Steel and Iron by Remelting for Spectrochemical Analysis (Withdrawn 2022)[3]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminology E135.

4. Summary of Practice

4.1 Various forms are melted into a button approximately 32 mm (1.25 in.) in diameter and approximately 6 mm (0.25 in.) thick using an electric arc furnace. The action of the arc creates agitation and mixing of the molten metal which produces a homogeneous sample.

5. Significance and Use

5.1 This sampling practice is useful for converting material taken from ingots or other solid materials into a homogeneous solid sample suitable for direct excitation on a spark atomic emission or X-ray fluorescence spectrometer. The resultant button may itself be chipped to provide specimens for test methods requiring solutions or chips.

5.2 This practice has been used extensively for the preparation of zirconium, zirconium alloy, titanium, and titanium alloy materials, and is applicable to other reactive, refractory, ferrous and nonferrous alloys, such as cobalt, cobalt alloys, niobium, nickel, nickel alloys, cast irons, steels, stainless steels, tantalum, tool steels, and tungsten.

6. Interferences

6.1 The user should carefully consider the impact of using melted samples for analysis as these may be subject to selective volatilization or segregation of various elements. Elements known to volatilize are bismuth, cadmium, chlorine, lead, magnesium, sodium, tellurium, thallium, uranium, and zinc. Other elements that may change in content are the interstitial gases, oxygen, nitrogen, and hydrogen. Carbon content may increase if a graphite anode is used. A tungsten anode may be

[1] This practice is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.20 on Fundamental Practices.

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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.

substituted if carbon contamination is a concern; however, tungsten contamination may occur. Copper contamination may be introduced from the melting crucible.

7. Apparatus

7.1 *Electric Arc Melting Furnace*[4]—This section describes the various components of an electric arc melting furnace. Refer to Fig. 1 for component arrangement. The number assigned to each component in 7.1.1 through 7.8 corresponds to the number in Fig. 1. A safety interlock shall be provided to prevent electrical shock when the melting furnace is open.

7.1.1 *Water-Cooled Upper Housing (1),* approximately 152 mm (6 in.) in diameter and 159 mm (6.25 in.) in height, and having a smooth, flat sealing surface.

7.1.1.1 This upper housing may be equipped with a viewing window composed of dark welding-type glass with an inner protective glass that is impervious to heat and splatter from the molten sample.

7.1.2 *Rubber Boot*—(*2*), shall cover the anode manipulator assembly to prevent electrical shock.

7.1.2.1 The anode manipulator assembly (or electrode holder) can typically be moved up and down and in a circular motion to facilitate spacing between the anode and the sample to enable the arc to effectively melt the sample. See 12.5.

7.1.3 The top of the housing shall be fabricated from an electrical and thermal insulating material, such as Bakelite, and shall support the following items:

7.1.3.1 *Relief Valve*—(*3*), to relieve excessive pressure during the melting process.

7.1.3.2 *Inlet Fitting*—(*4*), for argon.

7.1.3.3 *Outlet Fitting*—(*5*), for connecting to a vacuum pump.

7.1.4 *Water-Cooled Lower Housing (6),* approximately 146 mm (5.75 in.) in diameter and 140 mm (5.5 in.) in height containing the copper melting crucible. Its upper surface shall be fitted with a neoprene O-ring to seal against the upper anode housing.

7.1.4.1 The lower housing shall be capable of being inverted for removal of the button after it has cooled.

7.1.5 *Jack*—(*7*), to raise the lower housing against the upper anode housing, compressing the O-ring and sealing the crucible chamber.

7.2 *DC Electric Welder*—(*8*), to provide an arc current of 400 A to 800 A.

7.3 *Pressure Regulator*—(*9*), two-stage, for argon gas.

7.4 *Vacuum Pump*—(*10*), having an initial pumping rate of 50 L⁄min or more.

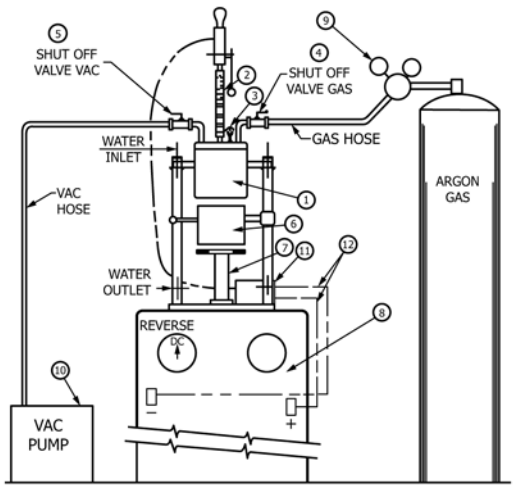
7.5 *Wire Brushes,* to clean the crucible.

7.6 *Tamping Rod,* suitable for packing the sample into the crucible.

7.7 *Foot Switch, Optional,* to provide low and high power settings. If a foot switch is not provided, the low to high and high to low power transition can be performed with a rheostat designed for adjusting the current.

7.8 *Control Panel (11),* containing the master power switch and rheostat for adjusting the current.

[4] Melting furnaces, manufactured by Cianflone Scientific, 135 Industry Drive, Pittsburgh, PA 15275, www.cianflone.com, have been found suitable for this purpose.



Legend:

(1) Anode Housing
(2) Rubber Boot
(3) Relief Valve
(4) Inlet Fittings for Argon
(5) Outlet Fitting for Vacuum
(6) Crucible Housing
(7) Jack
(8) DC Electrical Welder
(9) Pressure Regulator
(10) Vacuum Pump
(11) Control Panel
(12) Power Cable

FIG. 1 Schematic of Electric Arc Melting Furnace

8. Reagents and Materials

8.1 *Argon Gas,* 99.99 % purity, supplied from a gas or liquid tank.

8.2 *Anode,* graphite or tungsten, approximately 13 mm (0.5 in.) in diameter and 64 mm (2.5 in.) in length with a tapered tip.

9. Hazards

9.1 Wear safety glasses with side shields, or full face shield.

9.2 Wear insulated gloves when changing hot electrodes and handling hot buttons.

9.3 An electrical shock hazard exists if the rubber boot is removed from around the anode manipulator assembly at the top of the furnace.

9.4 If fumes evolved during melting are determined to be hazardous, then an exhaust vent should be installed over the furnace.

9.5 Follow the manufacturer's instructions to avoid electrical shock and harm from light and heat.

10. Sample Preparation

10.1 Remove any organic contamination and dry thoroughly before melting. Compacting fine powders, chips, drillings, turnings, or wire into a briquette may provide more consistent melting.

11. Preparation of Apparatus

11.1 *Initial Setup*—Refer to Fig. 1.