



Designation: ~~E1306—17~~ E1306 – 22

Standard Practice for Preparation of Metal and Alloy Samples for ~~Chemical Analysis by Electric Arc Remelting~~ Melting for Spectrochemical Analysis¹

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, wires, sponge 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~~ safety, health, and health 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[E876 Practice for Use of Statistics in the Evaluation of Spectrometric Data \(Withdrawn 2003\)](#)³

[E1010 Practice for Preparation of Disk Specimens of Steel and Iron by Remelting for Spectrochemical Analysis \(Withdrawn 2022\)](#)³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

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.

4. Summary of Practice

4.1 Various forms are melted into a button approximately $\pm 32 \text{ mm} \frac{1}{4} \text{ inches} (1.25 \text{ in.})$ in diameter and approximately $\frac{1}{4} \text{ inches} 6 \text{ mm} (0.25 \text{ in.})$ 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.

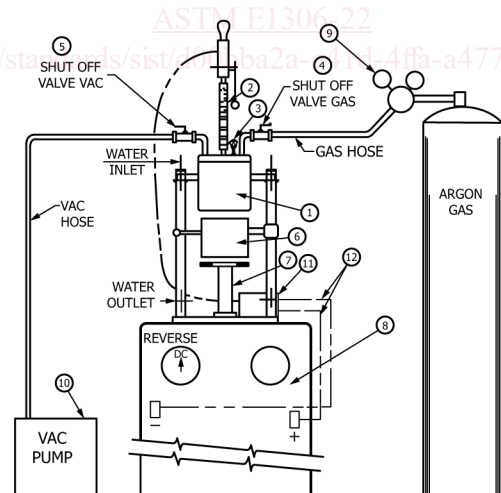
5.2 This practice has been used extensively for the preparation of zirconium, zirconium alloy, titanium, and titanium alloy materials, and is applicable to other reactive, refractory, ferrous and nonferrous alloys, such as cobalt, cobalt alloys, niobium, nickel, nickel alloys, cast irons, steels, stainless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

6. Interferences

6.1 The user should carefully consider the impact of using ~~remelted~~ melted samples for analysis as ~~remelted samples~~ these may be subject to selective volatilization or segregation of any 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, plus carbon, which may be added hydrogen. Carbon content may increase if a graphite anode is used. A tungsten anode may be substituted if carbon pickup contamination is a concern. Tungsten contamination may occur if this electrode is used. Copper contamination also concern; however, tungsten contamination may occur. Copper contamination may be introduced from the melting crucible.

7. Apparatus

7.1 ~~Electric Arc Remelt Furnace~~ Melting Furnace⁴—This section describes the various components of an electric arc ~~remelt~~ melting furnace. Refer to **Fig. 1** to see how each component is arranged. for component arrangement. The number assigned



Legend:

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

FIG. 1 Schematic of Electric Arc RemeltMelting Furnace

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

to each component in the 7.1.1 following description through 7.8 corresponds to the number in Fig. 1 the schematic. A safety interlock shall be provided to prevent electrical shock when the melting furnace is open.

7.1.1 *Water-Cooled Upper Housing (1)*, ~~(4)~~, approximately 6 inches 152 mm (6 in.) in diameter and 6 1/4 inches 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)*, ~~(6)~~, approximately 5 3/4 inches 146 mm (5.75 in.) in diameter and 5 1/2 inches 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 1.5 m³/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 (optional) settings. If a foot switch is not available, provided, the low to high power and the high to low power transition can be performed with the rheostat designed for adjusting the current.

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

8. Reagents and Materials

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

8.2 *Anode*, graphite or tungsten, 1/2 inches 13 mm (0.5 in.) in diameter and 264 mm 10 3/8 inches (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 ~~off~~ during melting ~~have been~~ 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**.

11.1.1 Attach the cooling water to the apparatus and adjust the flow rate per manufacturer's ~~recommendations~~ instructions.

11.1.2 Attach the argon supply and adjust the two-stage regulator output per manufacturer's ~~recommendations~~ instructions.

11.1.3 Attach the vacuum pump.

11.1.4 Attach the dc electric welder to the apparatus at the control panel.

11.1.5 Attach the graphite anode to the manipulator assembly.

11.2 *Preparation of Anode*—The lifetime of the anode can be extended significantly by dipping it into the molten metal. When this procedure is used, it is imperative that there be a separate anode for each type of metal or alloy to prevent cross contamination of the samples.

12. Procedure

12.1 Turn on the water valve and the master power switch. Adjust the current at the control box so that the low power setting will be 400 A and the high power setting will be ~~800 A~~ 800 A. A fixed power between ~~400 A and 800 A~~ 400 A and 800 A can also be used.

Caution—When melting fine powders, use an initial current of 100 A until the powder appears to be well fused. Raise the current to 300 A and complete the melting. This will help prevent loss of sample due to splattering of the powder when the arc is first struck.

12.2 Clean the melting crucible with a wire brush before each melt.

12.3 *Charging the Crucible:*

12.3.1 Weigh sufficient sample to fill the crucible. Material density and the form and size of the chips or turnings will determine the ~~weight~~ mass of the sample that can be placed into the crucible. Typically 40 g to 50 g is sufficient. Ensure the entire sample is contained within the crucible and that none is above the top edge. Carefully pack the sample into the crucible with the tamping rod. ~~Powdered metals may need to be compacted before melting. Use special care when compacting powdered metals.~~

~~Note 1—It will take approximately 40 g of zirconium and zirconium alloy materials to fill the crucible.~~

12.3.1.1 It should be determined experimentally the exact quantity of material to charge in the crucible, and the correct current and ~~the correct time~~ required to produce a ~~sample~~button. The maximum melt time should also be determined to avoid damaging furnace components.

12.3.2 Rotate the lower body into position. ~~Raise the~~To close the furnace, raise the crucible with the jack until the O-ring is firmly seated against the upper ~~body to close the furnace~~body.

12.4 *Flushing the Crucible:*

12.4.1 Turn on the vacuum pump ~~on~~ and open the vacuum valve. Evacuate the chamber until the anode manipulator assembly has been pulled down to its lowest position.

NOTE 1—The pump will become quiet when a partial pressure of approximately 300 millitorr is reached.

12.4.2 Close the vacuum valve and open the argon valve until the anode manipulator assembly has returned to its fully extended position.

12.4.3 Repeat the evacuation and argon flushing procedure if desired.

12.4.4 Leave the argon valve open after the last flush to maintain a positive pressure during the melting process.

12.4.5 Turn off the vacuum pump or close vacuum shut off valve.

12.5 *Melting the Sample:*

12.5.1 Place both hands on the anode manipulator assembly. Energize the welder. Lower the manipulator assembly until the arc is initiated. Continue to lower the manipulator assembly, without touching the sample, until the charge is completely melted.

12.5.2 After maximum power is attained, hold melt for approximately ~~30 s~~30 s. Keep the anode close to the molten pool without touching it. The current can be controlled by moving the anode closer or further from the molten pool.

12.5.2.1 To coat the anode with metal as suggested in ~~10.211.2~~, with the power still on high, thrust the anode down quickly through the molten pool of metal until it just touches the crucible bottom. Then bring it up quickly to clear the metal.

12.5.3 After approximately ~~30 s~~30 s minimum at maximum power, de-energize welder and guide the manipulator assembly upward to its raised position.

12.5.4 To prevent oxidation and maintain a bright shiny surface, allow the button to sufficiently cool for at least ~~60 s~~60 s before opening the chamber.

12.6 *Removal of the Sample:*

12.6.1 Close the argon valve if desired. Open the chamber by releasing the pressure on the jack.

12.6.2 Invert the lower body to remove the button. Allow it to cool to room temperature before handling.

12.6.3 Examine the ~~sample~~crucible to ensure that all material has been fully melted. If not, ~~invert~~replace the ~~sample~~button and remelt.

12.7 *Preparation of the Sample for Analysis:*

12.7.1 Machine the surface of the button flat and smooth, suitable for analysis.

12.7.2 The button may also be chipped to provide specimens for test methods requiring solutions or chips.