Designation: E 1306 – 94 (Reapproved 1998)$^{\epsilon 1}$

Standard Practice for Preparation of Metal and Alloy Samples by Electric Arc Remelting for the Determination of Chemical Composition[1]

This standard is issued under the fixed designation E 1306; 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.

ϵ[1] NOTE—Footnote 1 was corrected in October 1998.

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 chips, turnings, wires, and sponge. Powdered metals need to be compacted before melting.

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

1.2 *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.* Specific hazard statements are given in Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials[2]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminology E 135.

4. Summary of Practice

4.1 Metal chips, turnings, or wires are melted into a button approximately 1¼ in. in diameter and approximately ¼-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 chips, turnings, and wires taken from ingots or other solid materials into a homogeneous solid sample suitable for direct excitation on an optical emission or X-ray fluorescence spectrometer. The resultant button may itself be chipped to provide samples for plasma emission, atomic absorption, and wet chemical analysis.

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, columbium (niobium), nickel, nickel alloys, stainless steels, tantalum, tool steels, and tungsten.

6. Interferences

6.1 Test samples of known composition shall be used to determine if there is any selective volatilization or segregation of the impurity elements. Elements known to volatilize are bismuth, cadium, 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 due to the graphite anode. A tungsten anode may be substituted if carbon pickup is a concern. Copper contamination also may be introduced from the melting crucible.

7. Apparatus

7.1 *Electric Arc Remelt Furnace*—An apparatus suitable for this practice is shown schematically in Fig. 1.[3] It shall be equipped as follows:

7.1.1 *Water-Cooled Upper Housing* (*1*), approximately 6-in. diameter and 6¼ in. high, and having a smooth, flat sealing surface.

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

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.

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

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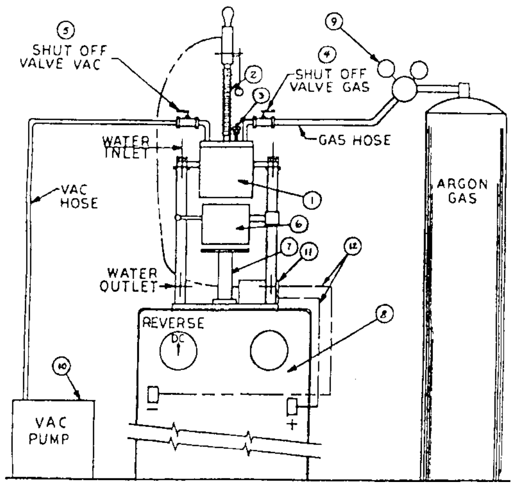
[2] *Annual Book of ASTM Standards*, Vol 03.05.

[3] A commercially available unit, including expendable parts, manufactured by Cianflone Scientific Instruments Corp., Pittsburgh, PA, has been found satisfactory.



Legend:

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

FIG. 1 Schematic of Electric Arc Remelt Furnace

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

7.1.4 *Water-Cooled Lower Housing* (*6*), approximately 5¾-in. diameter and 5½ in. high 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 *Hydraulic Jack* (*7*), to raise the lower housing against the upper anode housing, compressing the O-ring and sealing the crucible chamber.

7.2 *D-C Electric Welder* (*8*), to provide an arc current of 400 to 600 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.

7.5 *Wire Brushes*, to clean the crucible.

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

7.7 *Foot Switch*, to provide low and high power settings (optional). If a foot switch is not available, 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*), containing the master power switch and rheostat for adjusting the current.

8. Reagents and Materials

8.1 *Argon*, 99.99 %, gas or liquid.

8.2 *Anode*, graphite, ½-in. diameter and 2½ in. long 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 An exhaust vent shall be installed over the furnace to remove any harmful fumes that may be given off during the melting cycle.

10. Preparation of Apparatus

10.1 *Initial Setup*—Refer to Fig. 1.

10.1.1 Attach the cooling water to the apparatus and adjust the flowrate to ¾ L/min.

10.1.2 Attach the argon supply and adjust the two-stage regulator output to 6 psi (41 kPa).

10.1.3 Attach the vacuum pump.

10.1.4 Attach the d-c electric welder to the apparatus at the control panel.

10.1.5 Attach the graphite anode to the manipulator assembly.

10.2 *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.

11. Procedure

11.1 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.

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

11.3 *Charging the Crucible*:

11.3.1 Weigh sufficient sample to fill the crucible. Material density and the form and size of the chips or turnings will determine the weight of the sample which can be placed into the crucible. Be certain that all of the sample is contained within the crucible and none is above the top edge. Carefully pack the sample into the crucible with the tamping rod.

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

NOTE 2—It shall be determined experimentally the exact quantity of material to charge the crucible and the correct current and time to produce a sample.

11.3.2 Rotate the lower body into position. Raise the crucible with the hydraulic jack until the O-ring is firmly seated against the upper body to close the furnace.

11.4 *Flushing the Crucible*:

11.4.1 Turn 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 3—The pump will become quiet when a partial pressure of approximately 300 millitorr is reached.

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

11.4.3 Repeat the evacuation and argon flushing procedure two more times.

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

11.4.5 Turn off the vacuum pump.

11.5 *Melting the Sample*:

11.5.1 Place both hands on the anode manipulator assembly. Depress and hold the foot switch in the low-power position. 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.

11.5.2 Without interrupting the circuit, increase to high power with the foot switch. Hold for 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.

NOTE 4—To coat the anode with metal as suggested in 10.2, 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.

11.5.3 After 30 s at high power, release the foot switch and guide the manipulator assembly upward to its raised position.

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

11.6 *Removal of the Sample*:

11.6.1 Open the chamber by releasing the pressure on the hydraulic jack.

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

11.7 *Preparation of the Sample for Analysis*:

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

11.7.2 The button may also be chipped to provide specimens for plasma emission, atomic absorption, and chemical analyses.

12. Keywords

12.1 arc melting; button melting; melting; remelting; sample preparation