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Standard Practice for Preparation of Disk Specimens of Steel and Iron for Spectrochemical Analysis by Remelting¹

This standard is issued under the fixed designation E1010; 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 describes the preparation of disk specimens of steel and iron by melting chunks, chips, drillings, turnings, wire, or powder briquets with an electric arc in an argon atmosphere. Solidification of the specimen takes place in the crucible in an argon atmosphere. The disk obtained is suitable for quantitative spectrochemical analysis.

1.2

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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 and health practices, and determine the applicability of regulatory limitations prior to use. Specific precautionary warning statements are given in 6.2.1, and Section 8, and 10.1.2.1.

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

3. Terminology

3.1 For definitions of terms used in this procedure, refer to Terminology E135.

4. Summary of Practice

4.1 The sample of steel or iron is placed in a water-cooled copper crucible. The furnace is flushed with argon at a controlled rate of flow. An arc is struck between the electrode and the sample material and is maintained until the melting is complete. The molten specimen is allowed to solidify in the crucible in an argon atmosphere. After solidification, the specimen is removed from the crucible and prepared for spectrochemical analysis.

4.2 Partial losses of some elements may be experienced during the preparation melting of the disk specimen. This procedure, if carefully followed, will provide consistent losses. Elemental losses can be determined by correlating the analysis of the charge material with the spectrochemical analysis of the remelted specimen.

5. Significance and Use

5.1 Most spectrochemical instruments employed for analyzing steel and iron require a solid specimen with a flat surface large enough for analytical excitation and measurement procedures. This practice describes a procedure for converting unusual types of steel and iron samples to satisfactory spectrochemical specimens.

6. Apparatus

6.1 Melting Furnace,³ consisting of a chamber that contains the following:

6.1.1 Crucible, of copper and water-cooled, in which samples of steel or iron are melted, then solidified to form specimens for spectrochemical analysis.

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 E01.01 on Iron, Steel, and Ferrous Alloys.

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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 Melting furnaces, manufactured by Hankison Corp., Cannonsburg, PA 15317 and Zeebac Inc., Berea, OH 44017, have been found suitable for this purpose.

³ Melting furnaces, manufactured by Cianflone Scientific, 228 RIDC Park West Drive, Pittsburgh, PA 15275, http://www.cianflone.com, have been found suitable for this purpose.

6.1.2 *Electrode Holder*, water-cooled and of negative polarity, that can be moved up and down easily, and may have provisions for circular motion and adjusting the arc gap to a fixed spacing.

6.1.3 *Viewing Window*, ~~composed~~ composed of dark welding-type glass with an inner-protective glass that is impervious to heat and splatter from the molten metal.

6.2 *D-C Electric Power Generator*, to supply electric current and voltage equivalent to that required for electric arc welding. It may be a rotating d-c generator or a static rectifier with provisions to adjust the current in the 0 A to 600 A range.

6.2.1 ~~Caution—A~~ Warning—A safety interlock shall be provided to prevent electrical shocks to the operator when the melting furnace is open.

6.3 *Vacuum Pump*, with free air capacity of 50 L/min and vacuum of 350 μm , minimum.

7. Materials

7.1 *Inert Gas*, argon of at least 99.96 % purity.

7.2 *Electrode*, thoriated tungsten or high-purity graphite.

8. Safety Precautions ~~Hazards~~

8.1 Operating personnel should adhere to the manufacturer's operating recommendations to avoid electrical shock and physical harm ~~due to~~ from light and heat. See 6.2.1 and 10.1.2.1 ~~for specific precautions.~~ for specific warnings.

9. Preparation of Samples

9.1 Remove grease from samples and dry before melting. Remove other surface contaminants by suitable methods. For consistent melting, fine powders, chips, drillings, turnings, or wire may be compacted in a briquetting press with 35-mm die at a pressure of 2800 kgf/mm².

10. Preparation of Specimens

10.1 Place 40 g to 50 g of sample in the crucible. Close the furnace. The melting of the sample and solidification of the specimen may vary slightly depending on the design of the furnace and the type of metal being ~~prepared.~~ melted. Two suggested procedures are as follows:

10.1.1 *Procedure A*—The following steps are programmed automatically after pressing the start button: (1) ~~Flushing~~ flushing of the crucible with argon for 30 s, (2) ~~Igniting~~ igniting the arc, (3) ~~Melting~~ melting with the arc for 20 s to 45 s, (4) ~~Reduction~~ reduction of arc current from 500 A to 250 A, (5) ~~Cooling~~ cooling the specimen in the crucible in inert gas for approximately 2 min, and (6) ~~Indication~~ indication by light and buzzer that the melt cycle is completed. When the program is completed, open the furnace and remove the hot specimen with magnet or forceps.

10.1.2 *Procedure B*—Evacuate the crucible to a pressure of approximately 350 μm of mercury. Flush the furnace with argon and evacuate. Reflush and evacuate a third time. Shut off the vacuum pump and flush the furnace with argon. Turn on the power supply and lower the electrode until an arc is struck to the sample material (Note 1). Adjust the power supply current to 500 A. Raise or lower the electrode or move it in a circular motion to provide uniform melting and melt any particles that cling to the inside of the chamber. Melt for approximately 1 min, then turn off the power supply and raise the electrode. Allow the specimen to solidify in the crucible in the argon atmosphere for approximately 2 min. Open the furnace and remove the specimen by tilting chamber. Catch the hot specimen in a suitable container.

10.1.2.1 ~~Caution—When~~ Warning—When melting fine powders, use an initial current of 100 A until the powders appear to be well fused. Raise the current to 300 A and complete the melting. This prevents loss of sample ~~due to~~ because of splattering of the powder when the arc is first struck.

NOTE 1—If the determination of carbon in the specimen is required, use a thoriated-tungsten electrode. If the determination of tungsten or thorium is required, use a graphite electrode.

11. Precision and Bias

11.1 Precision:⁴

11.1.1 Tables 1-3 show the percent standard deviations and the percent relative standard deviations among disks of various melted ferrous metals analyzed with both ~~optical~~ atomic emission spectrometers and X-ray fluorescence spectrometers. The precision data are included to serve as a guide for the precision obtainable from melted specimens prepared as described in this practice. The data were calculated in accordance with Practice E876.

11.1.2 The relative standard deviations among melted specimens can be quite large. The large deviations are due to element losses or enrichment during melting which can be minimized by good melting technique, particularly for carbon, sulfur, and copper. Cleaning the crucible between melts can reduce contamination errors, especially when widely differing materials are melted. The physical appearance of the melted specimens will sometimes be an indication of the homogeneity.

⁴Supporting data for this practice, obtained by cooperative testing, are included in Research Report E02-1018, which is filed at ASTM Headquarters.

⁴Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: E02-1018.