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Indian Standard

METHODS FOR SELECTION AND PREPARATION OF SAMPLES FOR SPECTROGRAPHIC ANALYSIS OF ZINC AND ZINC ALLOY INGOTS

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METHODS FOR SELECTION AND PREPARATION OF SAMPLES FOR SPECTROGRAPHIC ANALYSIS OF ZINC AND ZINC ALLOY INGOTS

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(*Continued on page* 7)

Indian Standard

METHODS FOR SELECTION AND PREPARATION OF SAMPLES FOR SPECTROGRAPHIC ANALYSIS OF ZINC AND ZINC ALLOY INGOTS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 28 May 1978, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 For spectrographic analysis of zinc and zinc base alloys, representative ingots have to be selected and samples prepared by either drilling or sawing slices. Need was, therefore, felt for a standard which should describe such methods and also give procedure for melting and casting the sample in a form suitable for spectrographic analysis.

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard specifies methods for selection and preparation of samples for spectrographic analysis of zinc and zinc alloy ingots.

2. SELECTION OF INGOTS

2.1 General — The samples shall be selected from batches, each batch being composed of ingots of the same composition as specified in IS : 209-1966† or IS : 713-1966‡.

2.2 Pure Zinc Ingots — Unless otherwise agreed upon, each consignment may be divided into a series of batches, provided they do not contain less than 25 tonnes. Any consignment of less than 25 tonnes shall be regarded as a single batch.

*Rules for rounding off numerical values (*revised*).
†Specification for zinc (*second revision*).
‡Specification for zinc base alloy ingots for die casting (*first revision*).

2.3 Zinc Alloy Ingots — Unless otherwise agreed upon, each consignment may be divided into a series of batches provided they do not contain less than 5 tonnes. Any consignment of less than 5 tonnes shall be regarded as a single batch.

2.4 Procedure

2.4.1 *Pure Zinc Ingots* — From each batch of ingots, select at random, one ingot from every 100 for Grades Zn 99.99 and Zn 99.95 and one ingot from every 50 for Grade Zn 98. The number of ingots selected shall not be less than 5.

2.4.2 *Zinc Alloy Ingots* — From each batch of ingots, select at random, one ingot from every 50. The number of ingots selected shall be not less than 5.

NOTE — When the consignment is made up of less than 5 ingots, all shall be used for preparing the samples.

2.4.3 Clean the surface of each ingot selected to remove all dirt, oil, grease, etc. If necessary, they shall be cleaned using a suitable solvent and dried.

3. PROCEDURE FOR DRILLING, PELLETIZATION AND RECASTING

3.1 Sampling by Drilling — Arrange the selected ingots flat, side by side, upside down with reference to the position occupied in the ingot mould, in groups of a maximum of 5 ingots. Ensure that the casting marks are arranged in the same way for each ingot. In each group, draw a diagonal across the rectangle thus formed.

3.1.1 With the aid of a tungsten carbide drill of approximately 15 mm diameter and without the use of a lubricant, drill each ingot right through at three points on the diagonal at distance from the long side of the ingot of one-third and two-thirds of the length of the short side as shown in Fig. 1.

NOTE 1 — When the exact position of the point to be drilled coincides with a notch in the ingot, choose another point as close as possible. The drilling obtained should be of thickness between 0·2 to 0·5 mm. Collect all the drillings and break them up to approximately 1 cm size. Drillings should be treated with magnet to remove fine iron particles, if any.

NOTE 2 — In the case of batches less than 5 tonnes, a sufficient number of drillings may be carried out for the mass of the sample to amount to at least 1 kg.

3.2 Preparation of Pelletization

3.2.1 Homogenize the sample by mixing, as completely as possible, all the drillings from the ingots from a single batch.

3.2.2 Take a sample having a mass of at least 1 kg by coning and quartering.

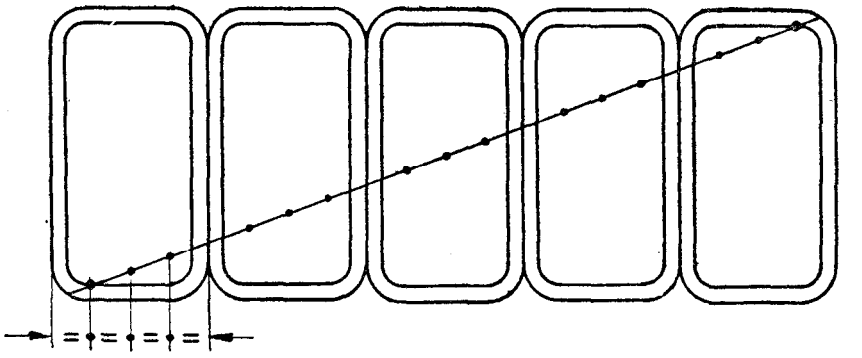

FIG. 1 ARRANGEMENT OF INGOTS FOR SAMPLING BY DRILLING

3.2.3 Divide the sample into two portions, one weighing approximately 750 g, and the other approximately 250 g.

3.2.4 Place the 250-g portion in a suitable container. Close, label and seal the container. This portion is intended for check or arbitration analysis by other standardized methods if required by either the supplier or the purchaser.

3.2.5 Compress the 750-g portion with the aid of a suitable press to form a small number of compact blocks. This operation shall be carried out within 24 hours after the drilling.

3.3 Melting and Casting

3.3.1 Melting may be carried out in a clean refractory crucible using a furnace heated by fuel oil, gas or electrical resistance or induction. The temperature of the melt shall be between 425 and 450°C.

3.3.2 Melt in the shortest possible time ensuring thorough mixing and cast immediately at least three samples in appropriate moulds of cast iron or steel pre-heated to about 100°C in a form suitable for spectrographic analysis.

3.3.3 Remove the cast sample from the moulds after cooling, fettle if necessary, and mark the samples. One of these is given to each interested party and one may be kept for the check or arbitration analysis in case this is required.

3.3.4 For analysis by dc arc method using oxide sample, take 5 to 10 g of drillings from one of the samples obtained under **3.3.2**. Dissolve 2 g of these drillings in concentrated nitric acid (r.d. = 1·42) in a silica crucible,

To this, add appropriate amounts of internal standards in solution form. Dry the solution on hot plate without boiling. Ignite the dried residue at 450°C for about 30 minutes in a muffle furnace. Cool the converted oxide in a dessicator and grind thoroughly in an agate mortar to fine powder.

4. PROCEDURE FOR SAWING SLICES, MELTING AND CASTING

4.1 Sampling by Sawing Slices

4.1.1 Arrange the selected ingots flat, side by side, upside down with reference to the position occupied in the ingot mould, in groups of a maximum of ten ingots. Ensure that the casting marks are arranged in the same way for each of the ingots.

4.1.2 In each group, draw a diagonal across the rectangle thus formed (*see* Fig. 1).

4.1.3 Prepare a slice of metal by sawing through each ingot following the line of this diagonal. The saw shall first be cleaned free of paint and any adherent metal particles; the sawing shall be carried out without lubricant and without heating the metal to the point of oxidation. The thickness of the slice shall be such that the total mass is at least 1 kg.

4.2 Melting and Casting

4.2.1 Melting of slices, broken up appropriately, may be carried out in a clean refractory crucible using a furnace heated by fuel oil, gas or electrical resistance or induction. The temperature of the melt shall be between 425 and 450°C

4.2.2 Melt in the shortest possible time ensuring thorough mixing and cast immediately at least three samples in appropriate moulds of cast iron or steel pre-heated to about 100°C, in a form suitable for spectrographic analysis.

4.2.3 Remove the cast sample from the moulds after cooling; fettle if necessary, and mark the samples. One of these is given to each interested party and one may be kept for check or arbitration analysis, if required.

4.2.4 For analysis by dc arc method, proceed as in **3.3.4**. Samples obtained by sawing are, however, not recommended for estimation of iron.

4.3 Breaking up of the Samples (4.2) with a View to Possible Analysis by Other Standardized Method — The sampling of chips for analysis by another standard method as specified in relevant Indian Standards may be carried out by milling, turning or drilling to obtain a homogeneous and representative sample of mass not less than 50 g. In each case, a tungsten carbide-tipped tool shall be used.

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METHODS OF CHEMICAL ANALYSIS AND SPECTROGRAPHIC ANALYSIS OF STEELS AND NON-FERROUS METALS

IS:

228 Methods of Chemical Analysis of Steels:

- (Part I)-1972 Determination of carbon by volumetric method (for carbon $\geqslant$0·1 percent) (*second revision*)
- (Part II)-1972 Determination of manganese in plain carbon and low alloy steels (Arsenitic method) (*second revision*)
- (Part III)-1972 Determination of phosphorus by Alkalimetric method (*second revision*)
- (Part IV)-1974 Determination of carbon by gravimetric method (for carbon $\geqslant$0·1 percent) (*second revision*)
- (Part V)-1974 Determination of nickel by dimethylglyoxime (gravimetric) method (for nickel $\geqslant$0·05 percent) (*second revision*)
- (Part VI)-1974 Determination of chromium by persulphate oxidation method (for chromium $\geqslant$0·5 percent) (*second revision*)
- (Part VII)-1974 Determination of molybdenum by alphabenzoinoxime method (for molybdenum above 1 percent) (*second revision*)
- (Part VIII)-1975 Determination of silicon by gravimetric method (for silicon $\geqslant$0·1 percent (*second revision*)
- (Part IX)-1975 Determination of sulphur in plain carbon steels by evolution method (*second revision*)
- (Part X)-1976 Determination of molybdenum by thiocyanate (photometric) method in low and high alloy steels (for molybdenum up to 1 percent) (*second revision*)
- (Part XI)-1976 Determination of silicon by photometric method in carbon steels and low alloy steels (for silicon 0·01 to 0·05 percent) (*second revision*)
- (Part XII)-1976 Determination of manganese by periodate (photometric) method in low and high alloy steels (for manganese up to 2 percent) (*second revision*)

1546-1960 Method for determination of arsenic in iron and steel

6226 (Part I)-1971 Recommendations for apparatus for chemical analysis of metals; Part I Apparatus for determination of carbon by direct combustion

2599-1963 Spectrographic analysis of high purity zinc and zinc base alloys for die casting

6010-1971 Recommended practice for photographic processing in spectrochemical analysis

7072-1973 Glossary of terms relating to Emission spectroscopy

7658-1975 Spectrographic analysis of aluminium