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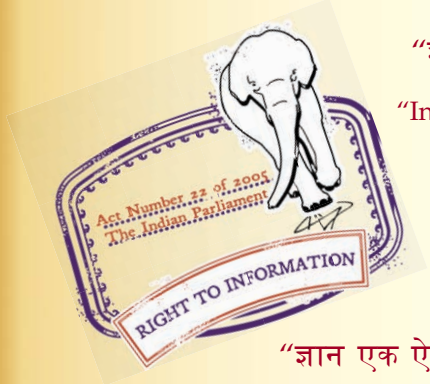

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METHODS OF SAMPLING NON-FERROUS METALS FOR CHEMICAL ANALYSIS

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METHODS OF SAMPLING NON-FERROUS METALS FOR CHEMICAL ANALYSIS

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METHODS OF SAMPLING NON-FERROUS METALS FOR CHEMICAL ANALYSIS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 14 November 1961, after the draft finalized by the Methods of Sampling Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 The difficulty in obtaining a thoroughly representative sample of non-ferrous metals or alloys lies in the fact that the impurities or the alloying elements tend to segregate in the process of solidification. Hence, while sampling non-ferrous metals, due regard should be given to the existing knowledge of the type and extent of segregation likely to occur in the particular item.

0.3 Taking into consideration the views of producers, testing authorities and technologists, the Sectional Committee responsible for the preparation of this standard felt that it should be related to technological and sampling procedures followed in the country in this field. Furthermore, due weightage had to be given to the need for international co-ordination among standards prevailing in different countries of the world in this field. These considerations led the Sectional Committee to derive assistance from the following publications :

B.S. 1499 : 1949 SAMPLING NON-FERROUS METALS. British Standards Institution.

ASTM E 55-48 STANDARD METHOD OF SAMPLING WROUGHT NON-FERROUS METALS AND ALLOYS FOR DETERMINATION OF CHEMICAL COMPOSITION. American Society for Testing Materials.

ASTM E 88-50T TENTATIVE METHOD OF SAMPLING NON-FERROUS METALS AND ALLOYS IN CAST FORM FOR DETERMINATION OF CHEMICAL COMPOSITION. American Society for Testing Materials.

0.4 Metric system has been adopted in India and all quantities and dimensions in this standard have been given in this system.

0.5 In reporting the result of a test or analysis, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with the latest version of IS : 2-1960 Rules for Rounding Off Numerical Values (*Revised*).

0.6 This standard is intended chiefly to cover the technical provisions relating to the sampling of non-ferrous metals and alloys, and it does not include all the necessary provisions of a contract.

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1. SCOPE

1.1 This standard lays down the procedure for preparing samples of non-ferrous metals and alloys, for the determination of chemical composition.

1.2 The procedure laid down in this standard covers the general principles of preparing samples of non-ferrous metals and is not intended to supersede or replace existing specification requirements for sampling of a particular material. The number of samples required to be tested from a lot and the number of pieces to be selected for the preparation of a sample are not considered in this standard since these will vary with the material and will be dealt with in appropriate material specifications.

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definitions shall apply.

2.1 Lot — The quantity of non-ferrous metals and alloys of one type and grade produced under uniform conditions of manufacture and offered for inspection at one time ; a lot may consist of the whole or a part of the quantity ordered for.

2.2 Melt, Cast or Heat — The product of a single furnace charge. Sometimes the furnace contents are tapped into two or more ladles when the product of each ladle may be called a separate cast.

2.3 Sample — The quantity of material, in the form of drillings, sawings, shapings, etc, for chemical analysis.

2.4 Casting — An object, produced by pouring molten metal into specially prepared moulds of desired shape and size depending upon the finished object to be made.

2.5 Cake — A casting intended for rolling into plate or sheet.

2.6 Ingot — An open-mould casting of definite size and shape for remelting.

2.7 Slab — A casting in the form of a bar used for rolling into strip.

2.8 Billet — A solid cylindrical casting used for extrusion into rod, bar, tube, or shape or for hot piercing into tube.

2.9 Wire Bar — A long rectangular cast bar with tapered ends, used for rolling into rod and wire.

2.10 Plate — A rolled product, of rectangular cross section, having a width much greater than thickness which is 3 mm or over.

2.11 Sheet — Material, generally flat, of uniform thickness and over 500 mm width.

2.12 Strip — Material, generally coiled, of uniform specified thickness and under 500 mm width.

2.13 Wire — A solid section, other than strip, supplied in coils or on spools, reels or bucks. Flat wire, including square, however, may also be furnished in straight lengths.

2.14 Supplier — The party supplying the material. The supplier may or may not be the actual manufacturer of the material.

2.15 Purchaser — The party purchasing the material. The term 'purchaser' shall also cover person or persons expressly authorized in writing by the purchaser to act on his behalf for inspection of the material.

3. SAMPLING OF MOLTEN METAL

3.1 During the tapping of the molten metal, from each cast or heat, three heavily chilled ingots shall be prepared out of three spoonfuls of molten metal collected during the beginning, middle and the end of the tapping period taking care to avoid dust and slag. The spoon used shall be of such material which does not contaminate the metal sampled. The convenient size for such ingots is 150 × 50 × 6 mm. The sample shall be prepared from these ingots following the appropriate procedures described under **4.2.1** to **4.2.4.**

3.2 If the metal tends to segregate very much during solidification as an ingot or if the ingot cannot be satisfactorily machined, a granulated sample

or a splash sample shall be prepared as given below :

a) *Granulated Sample* — The three spoonfuls of molten metal collected during the beginning, middle and the end of the tapping period, taking care to avoid dust and slag, shall be poured in a thin stream into cold water, adjusting the diameter of stream to produce granules. A sufficient quantity of the granules so obtained shall be reduced in size by crushing through a jaw crusher or a roll crusher or broken in a mortar. From this a sample shall be prepared as under **5**.

b) *Splash Sample* — The three spoonfuls of molten metal collected during the beginning, middle and the end of the tapping period, taking care to avoid dust and slag, shall be poured separately on to a clean surface so as to produce a thin sheet less than one millimetre thick. The sheet shall be sampled as under **4.2.7** and further processed as under **5**.

4. SAMPLING OF SOLID METAL

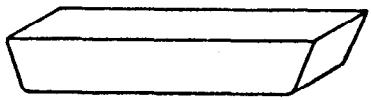
4.1 Wherever high accuracy is desired, and provided there is no risk of change in the composition of the metal/alloy due to remelting, a sufficient quantity of the solid metal should be remelted and sampled as under **3**.

4.2 When solid metals are to be sampled by machining the preferred procedure, to yield a representative sample, is to collect material from the whole cross section of the metal and for this purpose milling or sawing is recommended. In some cases it may be impracticable, uneconomic or under certain circumstances undesirable to take millings or sawings, in which case drillings may be taken.

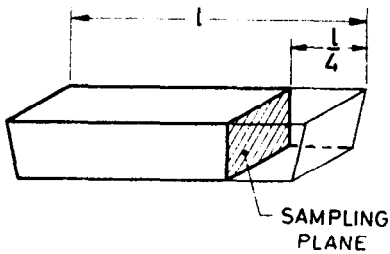
NOTE 1 — The preference for samples representing the whole cross section of a solid mass is dictated by the knowledge that in most materials and most solid forms segregation is greatest, if present at all, across a plane at right angles to the longest axis of the piece. It is important, however, to choose a plane removed from the ends of the piece, particularly if the piece is cast, since there may also be segregation from the end inwards along the major axis. In some cases where the length and thickness of the piece are similar, a sample from a single cross-sectional plane may still give a sample not truly representative of the whole but the best result is likely to be obtained if the plane chosen is distant from one end of the piece by one-quarter of the length of the piece.

NOTE 2 — When agreed between the purchaser and the supplier, the remains of the test pieces subjected to the physical tests may be used conveniently, in place of specially selected samples, for chemical analysis.

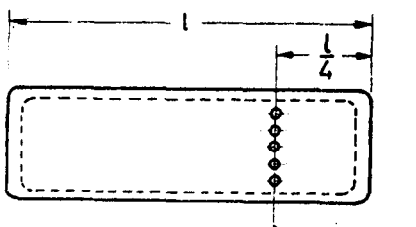
4.2.1 *Un-notched Ingot Without Lugs* (*See Fig.* 1) — Un-notched ingots without lugs (*see* Fig. 1A) shall be sawn or milled across the whole section of the ingot along a plane at right angles to the long axis and halfway between the end of the ingot and the point of maximum shrinkage, that is, at approximately ¼ of the way along the length of the ingot (*see* Fig. 1B) from one end. Alternatively, five holes shall be drilled in the sampling plane as shown in Fig. 1C. The drilling shall be through the total thickness and, if necessary, drilled from both the directions, top and bottom.



1A INGOT



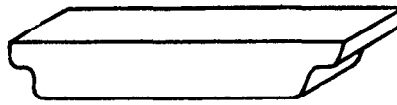
1B PLANE POSITION FOR SAWING OR MILLING



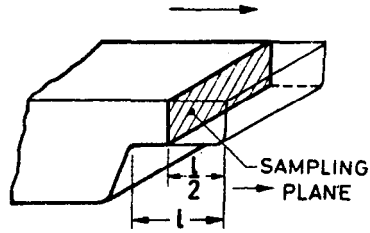
1C PLANE POSITION FOR DRILLING

FIG. 1 SAMPLING OF UN-NOTCHED INGOTS WITHOUT LUGS

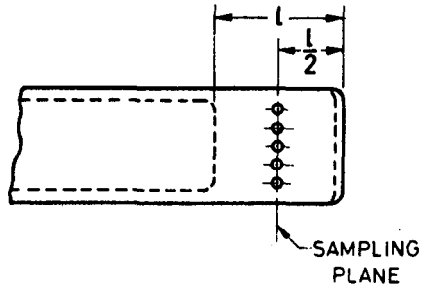
4.2.2 *Un-notched Ingot With Lugs* (*See Fig.* 2)—Lugs which are not more than 25 mm in thickness cool sufficiently rapidly on solidification and will be free from serious segregation. For sampling un-notched ingots with lugs (*see* Fig. 2A) one half of a lug shall be detached by sawing at right angles to the longer axis of the ingot and milled across the sawn faces of the piece (*see* Fig. 2B). Alternatively, five holes shall be drilled through the lug along the sampling plane (*see* Fig. 2C).



2A INGOT



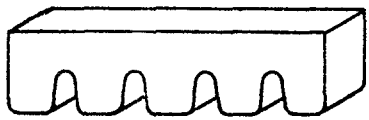
2B PLANE POSITION FOR SAWING OR MILLING



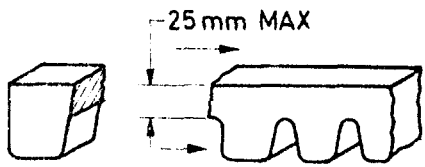
2C PLANE POSITION FOR DRILLING

FIG. 2 SAMPLING OF UN-NOTCHED INGOTS WITH LUGS

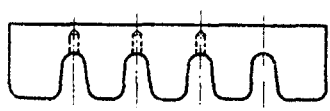
4.2.3 *Notched Ingots (See Fig. 3)*—In notched ingots of the normal type (*see* Fig. 3A), the metal immediately above the notch cools rapidly and may be considered to be free from serious segregation. For sampling, the ingot shall be broken or sawn across the centre of the notch and milled across the broken or sawn faces. Milling shall be stopped when the depth of the ingot faces exposed, is 25 mm (*see* Fig. 3B). Alternatively, holes shall be drilled through the thickness of the chilled section immediately above the notches as shown in Fig. 3C. If this section is very thin or the individual sections are very small and drilling becomes impracticable, the ingot shall be sampled by milling. Ingots with a single notch and in certain cases large ingots with two or three notches may be treated as if each section was an *un-notched ingot without lug.*



3A INGOT



3B PLANE POSITION FOR SAWING OR MILLING



3C PLANE POSITION FOR DRILLING

FIG. 3 SAMPLING OF NOTCHED INGOTS

4.2.4 *Ingots of Other Types (Shapes), Castings and Cakes* — In sampling ingots of types other than those referred to under **4.2.1**, **4.2.2** and **4.2.3** and in sampling of castings and cakes, due regard should be given to the existing knowledge of the type and extent of segregation likely to occur in the particular item. Machinable items shall be milled or sawn at suitable points to collect the sample ; alternatively, they shall be sampled by drilling five holes at suitable points.

4.2.5 *Slabs* — Where the size of the slab permits, it shall be sawn through the cross section at ¼ of the length from one end ; alternatively, one of the sawn faces shall be milled. Where this is impracticable, say for large slabs, at least five holes shall be drilled to half the depth of the slab and at regular intervals along one of the diagonals of the major faces from both the directions, top and bottom.

4.2.6 *Billets and Wire Bars* — Billets and wire bars shall be sawn through the cross section in a plane perpendicular to the longest axis and approximately $\frac{1}{4}$ of the length from one end. Alternatively, they shall be milled right across one of the sawn faces.

4.2.7 *Plates, Sheets or Strips* — Clippings, millings or sawings representing the whole cross section along a plane at right angles to the length in the case of strips and at a convenient angle to the direction of rolling, normally 45° or less, in the case of sheets shall be taken. Alternatively, sheets or strips may be folded once or more by bringing the ends together and closing the bends ; the inside sheared edges may then be milled or flat surface shall be drilled; special precautions are needed for coated materials.

4.2.8 *Wires* — Clippings shall be taken from both ends of the coil.

4.2.9 *Metal Scrap* — Usually metal scrap will consist of old castings, sheets, plates, old tubes, wires, bars, etc. For sampling, pieces representing each type of material in the same proportion as in the scrap shall be selected. Proportionate quantity of drillings, sawings, millings or clippings taken from these selected pieces shall be charged into a pre-heated crucible and melted as rapidly as possible without overheating. The use of a flux, which might alter the composition by reacting with the melt, shall be avoided.

NOTE — When testing for the non-ferrous metal contents in metal scraps mixed with ferrous impurities, the ferrous portion shall be removed by use of strong magnet and separately accounted for in the analysis.

5. PREPARATION OF SAMPLE AND STORAGE

5.1 Drillings, millings, sawings, clippings, etc., shall be carefully examined to remove any foreign material. Non-magnetic metals or alloys shall be carefully treated with a strong magnet to remove any particles of iron introduced in the process of drilling, milling, etc. They shall be mixed well and reduced to the requisite quantity to constitute a sample. The weight of the sample shall comply with the sampling requirements of Indian Standard specification covering that material but shall not be less than 100 g. The sample shall be divided into three approximately equal parts, each of which shall be placed in a package and sealed for distribution — one each to the supplier, the purchaser and a referee.

5.2 Samples that are to be stored over long periods or that are oxidized readily or otherwise altered in composition under varying atmospheric conditions or that may become seriously contaminated in contact with paper or cardboard shall be kept in wide-mouthed and well stoppered glass bottles. In other cases tight leakproof paper sample envelopes or cardboard cartons shall be used to store the sample.

6. GENERAL PRECAUTIONS

6.1 The ingot, slab, sheet, etc, to be sampled shall be clean and free from scale, dirt, oil, grease, etc. If necessary, they shall be cleaned using suitable solvent, and dried. Scale and dirt may be removed by suitable mechanical or chemical treatment ; if chemical methods of cleaning are used, such operations should not alter the metal surface in any way. When testing for proportion of metal contained, as in the case of scraps, these provisions do not apply as a general rule.

6.2 The saw, drill, cutter or other tools used shall be capable of producing small and uniform particles. They shall be thoroughly cleaned before use. Depth of cut, speed of cutting, etc., shall be so regulated that excessive heating and consequent oxidation of the sample shall be avoided. Brittle metal and alloys shall be pulverized in a suitable percussion mortar.

6.3 No lubricant shall be used for drilling, sawing, etc., except in cases where experience shows that the use of lubricants does not vitiate sampling and lubrication is necessary to avoid excessive oxidation of the sample, to reduce power requirements, or to save labour and time. In such cases the use and choice of a satisfactory lubricant shall be agreed upon between the supplier and the purchaser. Lubricants selected shall not alter or change the composition of the materials during contact. The lubricants shall be completely removed as soon as possible after drilling or milling operation by the use of suitable solvents, and dried.

6.4 Drillings, sawings, or clippings of non-magnetic non-ferrous alloys shall be carefully treated with a strong magnet to remove any particles of iron introduced in the operation. Contamination of this kind should be avoided as far as possible by the use of suitable cutting tools. In the case of some metals, an acid dip may be necessary if low iron is critical.

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