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Jewellery — Sampling of precious metal alloys for and in jewellery and associated products

Joaillerie, bijouterie — Échantillonnage des alliages de métaux précieux pour la joaillerie, bijouterie et produits associés

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Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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ISO 11596 was prepared by Technical Committee ISO/TC 174, *Jewellery*.

Jewellery — Sampling of precious metal alloys for and in jewellery and associated products

1 Scope

This International Standard specifies a method of sampling precious metal jewellery alloys for the determination of the precious metal content. It is applicable to raw materials, semi-finished products and finished products of the jewellery alloys of precious metals. The purpose of this International Standard is to define all the operations needed to obtain samples intended for the determination of the precious metal content of a particular jewellery alloy. It is intended to be applied when sampling alloys are claimed to be homogeneous.

This International Standard does not cover alloys of precious metals used in industrial products, coins qualified as legal tender, dentistry or decorative coatings on other material. It is not intended to apply to procedures employed for the purposes of production control or for the provision of samples other than for the determination of the precious metal content.

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

2.1
casting
process in which a molten alloy is allowed to solidify in a mould

NOTE The product obtained by such a process is also referred to as a casting.

2.2
casting grain
material in discrete droplet or granular form, only suitable for re-melting

2.3
component parts
findings
products in a form that constitutes components of a finished article

2.4
electroform
article produced by an electrolytic process using a metallic or non-metallic substrate, in which the precious metal coating is sufficiently thick for the article to be used once the substrate is removed

NOTE Electroforms from alloys are often not homogeneous.

2.5
hollow tube method
method by which a tube of precious metal alloy is manufactured using mechanical means on a non-precious metal support that is removed at the end of the manufacturing process

2.6
ingot
cast unwrought product suitable for further manufacture

2.7
jewellery
products made of precious metal or precious metal alloys

NOTE These products are generally intended for ornamentation.

2.8
lot
batch
product, or collection of units of product, from which a sample (or samples) is (are) to be drawn

NOTE Each lot or batch consists of units of product of a single type, grade, class, size and composition, manufactured under essentially the same conditions and visibly presenting the same characteristics.

2.9
lot size
batch size
weight of products, or number of units of product, in a lot or batch

2.10
mixed precious metal jewellery
articles made from two or more separate precious metals, or precious metal alloys

2.11
precious metal
gold, platinum, palladium or silver

2.12
precious metal alloy
material used to form a product with an intended combination of one or more precious metals, usually with one or more other materials, in a homogeneous form

2.13
rod
solid wrought product of uniform cross-section along its own length, supplied in straight form in defined lengths

2.14
sample
quantity of representative material taken from a product, or part of a product

2.15
sampling
defined procedure whereby a part of a substance, material or product is taken to provide a representative sample, or samples, of the whole for analysis

2.16
semi-finished product
product that can be easily used to produce a finished item and/or a component part

2.17
sheet
strip
flat wrought product of exact length and of rectangular cross-section and uniform thickness

NOTE The term “foil” is also sometimes applied to thin sheet.

2.18
solder
alloy used to join metal parts

2.19
test portion
part of the sample used for the determination of the precious metal content

2.20
tube
hollow wrought or cast product of uniform cross-section, with only one enclosed void along its whole length and with uniform wall thickness supplied in straight lengths or in coiled form

2.21
wire
solid wrought product of uniform cross-section along its own length, supplied in coil form, on spools or reels, or as individual lengths

2.22
wrought product
product obtained by hot and/or cold plastic deformation processes, such as extruding, forging, hot rolling, cold rolling or drawing, either exclusively or in combination

3 Tools

3.1 General

The following list of tools shall be used, which satisfy the criterion of not contaminating the sample:

a) power drill mounted in a drilling stand and capable of operating in the range 800 r/min to 1 200 r/min; the stand shall have facilities for holding the material being sampled;

b) high speed twist drill bits, one for each type of alloy;

c) small bench cutter;

d) anvil with hard polished face;

e) anvil hammer with a convex face and a suitable mass;

f) fine-grained grinding paper for cleaning the anvil and hammer after each use;

g) assay shears;

h) sample splitters in polished stainless steel;

i) fine (3 mm to 5 mm) bore quartz or graphite tube with appropriate suction device to extract molten alloy;

j) quartz, graphite or carbon-coated stainless steel ladle with a dip capacity of 5 ml to 10 ml;

NOTE Carbon coating can be achieved using a carbon-rich flame.

k) shallow open mould in material suitable for rapid cooling without contamination;

l) polished laboratory rolling mill;

m) saw;

n) file;

o) scraper consisting of a triangular steel or ceramic rod set in a handle.

The above list of tools shall not be considered exclusive: other tools that satisfy this criterion may also be used.

3.2 Use of tools

The relative softness of many of these alloys makes it easy for impurities to become embedded in the sample. Therefore, if sampling involves cutting, the tool used shall be sharp and care shall be taken to ensure that samples contain representative proportions of the material being sampled.

All tools, machinery and containers used to provide, store, or transport the samples shall be cleaned before use to prevent any contamination of the sample for analysis.

4 Sample selection

The samples shall be selected in accordance with applicable technical standards or recommendations.

5 Surface preparation before sampling

Dust, oil, grease, etc. shall be removed by a cleaning agent that leaves no residue on drying. Excess cleaning agent shall be removed before sampling.

Chlorinated hydrocarbons or other harmful substances shall not be used.

Any kind of coating shall be removed by an appropriate method (e.g. chemical, mechanical) and any contamination of the precious metal alloy shall be avoided.

6 Sampling methods

6.1 General

The sampling operation shall be carried out in such a way as to produce material capable of acceptable subdivision into equivalent portions.

NOTE 1 Annex A provides guidelines for certain products.

Where products contain more than one alloy composition of a precious metal, cross-contamination shall be avoided. For products that have been assembled by soldering, the solder line shall be avoided.

NOTE 2 Inclusion of solder in the sample is subject to individual national legislation.

6.2 Dip sampling of molten alloy

6.2.1 Samples shall be taken from well-stirred melts using either of the methods described below.

When the melt is protected by a non-metallic layer, this should be excluded from the melt sample, or physically separated from the solidified alloy before selecting the test portions.

6.2.2 Method 1 — A 3 mm to 5 mm diameter quartz or graphite tube shall be used to extract a 7 cm long cylinder of molten metal. After removal of all the quartz, the cylinder of metal shall be flattened to provide a thin strip of alloy from which duplicate test portions shall be cut after discarding the first 2 mm of the two end sections. The required thickness of the strip shall be determined by the rate of attack by acid in the dissolution process.

6.2.3 Method 2 — A ladle made of graphite or carbon-coated steel (for gold and silver) or of quartz (for platinum and palladium), capable of holding about 5 ml, shall be dipped. The liquid sample shall be cooled rapidly either by

a) pouring into water, or

b) casting in a flat mould.

This mould shall not be made of a graphite product when casting platinum or palladium. The granules obtained by quenching in water shall be flattened and heated at 150 °C to 200 °C until dry, and then subdivided by standard techniques (see Clause A.2) to provide the test sample.

6.2.4 The potential for segregation on cooling and for losses before and during solidification should be assessed before accepting such samples as representatives of the solid form.

NOTE When sampling by this technique, precautions are needed to prevent the uptake of oxygen.

6.3 Drilling

A 3 mm to 6 mm twist drill bit shall be operated at about 1 000 r/min without lubricants, and at least half the thickness of the product shall be drilled. Drillings shall be broken, if necessary, and the broken spiral drillings combined with other material from the same hole before selecting the test portions for 6 (or 4 or 2) assays. Material from different holes shall not be combined unless it has a mass of less than 50 % of the specified mass for the standard methods of analysis.

The drills used for this process shall be thoroughly cleaned before use with separate drills for each fineness of alloy. Drill bits shall be replaced as necessary.

6.4 Scraping

If a scraper is used, it shall be maintained sharp and used by experienced hands, so that it is possible to take samples that are sufficiently uniform and representative without damaging the product.

This method shall not be used for electroforms, for any product having a precious metal coating of the substrate or for products obtained using the hollow tube method.

6.5 Cutting

When sampling by cutting, a complete cross-section shall be taken.

6.6 Sawing or filing

A representative area of the cross-section shall be sectioned to produce representative samples. Cleaned saws and files shall be used, and the resultant sawings or filings shall be checked for contamination. Contamination from previous use of the saw or file shall be eliminated by discarding the sawings or filings from the initial strokes.

When finely divided samples are produced by these processes for analysis by cupellation, spurious results may arise due to spattering.

7 Retention of samples

7.1 General

If there is a requirement for retention of samples, proper documented storage shall be carried out.

Sample containers shall be labelled to provide full identification, and shall be clean and secured in order to avoid contamination or loss.

7.2 Bars, ingots, sheet, rod, tube, wire, casting grain and other raw materials

The prepared sample shall weigh at least three times the amount required for an assay in duplicate, where practical.

7.3 Component parts and finished products

The prepared sample shall provide sufficient material for one assay in duplicate.

Annex A
(informative)

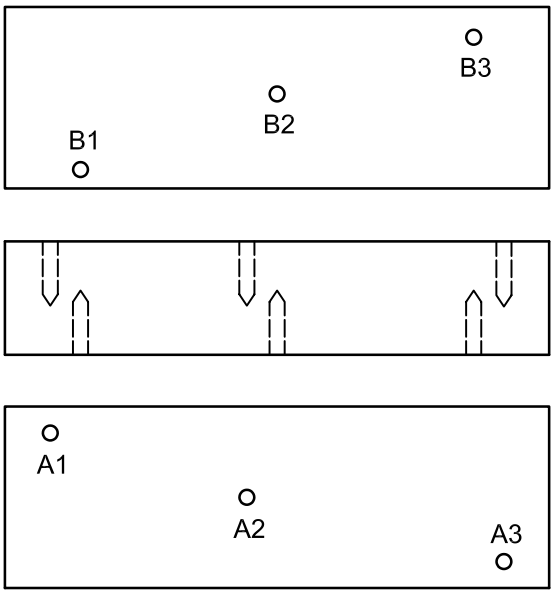
Guidelines for third-party testing for the usual technique of sample selection

A.1 Raw material

A.1.1 Cast ingot form and cast bars

Each ingot or bar shall be sampled separately, by drilling to a pre-arranged pattern with equal numbers of drillings on the top and the bottom. The number of drillings from each side shall normally be three, arranged along a diagonal, as shown in Figure A.1.

When sample combination is necessary, this shall be such as to permit separate results to be obtained for the top and bottom of the ingot or bar. Additional holes on the other diagonals may be required if insufficient material is available (see Figure A.1).



For bars of 2 kg and above, sample at the positions outlined.

For bars of between 0,5 kg and 2 kg, omit samples at positions A2 and B2.

For bars of less than 0,5 kg, sample at positions A1 and B3 only.

Figure A.1 — Drill sampling a bar

A.1.2 Granular form

A representative sample of the whole batch shall be taken by coning and quartering, or by use of other standard subdivision techniques.

Many granular materials have a wide range of grain sizes, with a significant number having a mass equal to the standard sample mass. If this applies, the sample obtained by coning and quartering shall weigh not less than 5 g.

The granules shall then be flattened on the anvil and test portions obtained by cutting similar sectors from at least five grains of comparable size. Three test portions shall be made up from different sizes of flattened grains covering the range of sizes in the sample. The precious metal content of the granular sample shall then be calculated from the results obtained with these test portions and the proportions of the three granule sizes in the sample.

A.2 Semi-finished and finished wrought and cast products

A.2.1 Rod, tube and wire

The wide range of sizes and forms commercially available make identifying a single method for selecting and taking samples difficult. Some wire and tube is produced on a reel or spool, while thicker products are traded as lengths or in coils.

Samples in all instances shall be taken from close to the two ends. Any material with visible flaws or colour variations should be cut off and rejected. Sampling should start at least 2 cm from this last visible defect.

For rod and thick-walled (2 mm) tube, samples shall be taken by drilling transverse holes at each end. If only two samples are taken, the drilling at the two ends should be at right angles to each other. Samples from the two ends shall not be combined. When more than one drilling at an end is needed to provide sufficient sample material, the second and all subsequent drillings shall be at right angles to the preceding one and 1 cm away from it in distance.

Rods of 1 cm section or more may show transverse variations in composition. These shall be drilled at each end and in two directions, at right angles to each other, to a depth not exceeding 50 % of the total. Drillings may be combined only if they have been taken in the same direction and at the same end.

When drilling is inappropriate, the samples shall be cut from each end. When wire is wound on a spool or reel, sufficient material at the ends of the wire shall be readily accessible to enable samples to be selected.

A.2.2 Sheet and strip

Sheet should have a flawless appearance and shall be sampled from the edges at the centres of the ends and the sides, providing four separate samples which are not combined.

Thicker ($>$ 2 mm) sheet may be sampled by drilling, but cutting may be the only practicable method for thin products.

A.3 Component parts (findings)

Where the product is made from a single alloy, involving no solder or joining of parts as confirmed by visual and spot tests, the test portion shall, as far as possible, be one item. If the product consists of alloys of different compositions and colours, samples of each alloy shall be taken.

Where one item would provide less than 50 % of the minimum standard weight of sample stipulated in the method, a number of identical items may be combined to reach this value. In such cases, each item shall contribute in virtually the same manner to form the test portion.

Where one item is more than 200 % of the minimum standard weight of sample stipulated in the method, it may be sectioned to provide, as far as possible, equivalent parts that are approximately equal to the standard weight. One only of these should be selected as a test portion.

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