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Jewellery and precious metals — Sampling of precious metals and precious metal alloys

Joaillerie, bijouterie et métaux précieux — Échantillonnage des métaux précieux et des alliages de métaux précieux

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 174, *Jewellery and precious metals*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

This second edition cancels and replaces the first edition (ISO 11596:2008), which has been technically revised.

The main changes are as follows:

— The content has been reviewed to allow a broader field of application of the standard;

— The scope has been aligned with the ISO standards related to the present document and delimited more clearly;

— Terms and definitions have been reorganized.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Jewellery and precious metals — Sampling of precious metals and precious metal alloys

1 Scope

This document specifies a method of sampling precious metals and precious metal alloys for the determination of their precious metal content and for the assessment of their homogeneity. The document is applicable to raw materials, semi-finished products and finished products and is intended to be used only for the sampling of entirely metallic materials.

NOTE 1 Standards for determination of precious metals contents for different metals are listed in the Bibliography.

NOTE 2 For assaying techniques different from the listed ones other sampling procedures can be required.

NOTE 3 For the purpose of production control or lot inspections the International Standards for the sampling indicated in the Bibliography or corresponding guidelines can be applied in addition.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3954, *Powders for powder metallurgical purposes — Sampling*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1 Sampling

3.1.1
sampling
defined procedure whereby a part of a substance, material or product is taken to provide a representative *sample* (3.1.4), or samples, of the whole for analysis

3.1.2
dip sampling
sampling (3.1.1) method intended to produce primary samples representative for a melt

Note 1 to entry: A secondary sampling is then required in order to produce the samples for analysis.

Note 2 to entry: Depending on the specific sampling technique adopted, dip sampling is also referred to as pin sampling, or as bead (or button) sampling, respectively.

3.1.3
portion
fraction of a lot, item or *sample* (3.1.4) obtained by appropriate sample subdivision techniques in order to represent correctly the properties of the whole to be tested

3.1.4
sample
quantity of representative material taken from a product, or part of a product

Note 1 to entry: In References [7] to [15] of the Bibliography, the term "sample" is used in some instances instead of the term "test portion".

3.1.5
sampling position
geometrically defined position specified by a *sampling* (3.1.1) scheme, from where the sampled material is taken from an item

Note 1 to entry: Depending on the quantity of material to be drawn it may consist in several individual points corresponding to the same geometrical definition.

3.1.6
test portion
part of the *sample* (3.1.4) used for a single determination of the precious metal content

3.1.7
lot
product, or collection of units of product, from which a *sample* (3.1.4) (or samples) is (or are) drawn

Note 1 to entry: Each lot consists of units of product manufactured under essentially the same conditions and visibly presenting the same characteristics, i.e. same type, grade, class, size and composition.

3.1.8
homogeneous
presenting the same physical and chemical characteristics down to the scale of the *sample* (3.1.4) and within the uncertainty of the relevant assaying method

3.2 Precious metals and precious metal articles

3.2.1
precious metal alloy
metal made by combining at least one precious metal as a major element with one or more metallic elements, constituted either by a solid solution of the metal elements or by a mixture of metallic phases at microscopic level

3.2.2
jewellery
jewels made of precious metal or *precious metal alloys* (3.2.1)

3.2.3
mixed precious metal articles
articles made from two or more separate precious metals, or *precious metal alloys* (3.2.1)

3.2.4
solder
alloy used to join metal parts

3.3 Product classes

3.3.1
raw material
unprocessed material used to produce finished products, or intermediate materials which are feedstock for future finished products

3.3.2
crystals
precious metal in form of metallic crystals constituting the typical product of electrochemical refining and used as feedstock for further processing

3.3.3
powder
precious metal in finely dispersed form normally used as feedstock for further processing

3.3.4
casting grain
material in discrete droplet or granular form, only suitable for re-melting

3.3.5
cast product
product obtained by solidification of a molten metal or metal alloy usually poured into a mould

3.3.6
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

3.3.7
semi-finished product
product that can be easily used to produce a finished item and/or a component part

3.3.8
component parts
products in a form that constitutes components of a finished article

3.4 Products and manufacturing methods

3.4.1
ingot
cast unwrought product suitable for further manufacture

3.4.2
bar
cast or minted finished product, or cast material intended to be refined

Note 1 to entry: These finished products are generally intended for investment.

3.4.3
rod
solid wrought semi-finished product of uniform cross-section along its own length, supplied in straight form in defined lengths

3.4.4
sheet
strip
flat wrought product of exact length and of rectangular cross-section and uniform thickness

Note 1 to entry: The term “foil” is also sometimes applied to thin sheet.

3.4.5
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

3.4.6
wire
solid wrought product of uniform cross-section along its own length, supplied in coil form, on spools or reels, or as individual lengths

3.4.7
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 1 to entry: Electroforms from alloys are often not homogeneous.

3.4.8
hollow tube method
manufacturing method producing a *tube* (3.4.5) of precious metal alloy by mechanical means on a non-precious metal support that is removed at the end of the manufacturing process

4 Tools

4.1 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 500 r/min to 900 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) hydraulic press with stainless steel plates, to be used instead of d) and e);

g) fine-grained grinding paper for cleaning the anvil and hammer or the plates of the hydraulic press after each use;

h) assay shears;

i) sample splitters in polished stainless steel;

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

k) 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.

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

m) polished laboratory rolling mill;

n) saw;

NOTE Blades made of HSS (High Speed Steel) are appropriate.

o) file;

p) scraper consisting of a triangular steel or ceramic rod set in a handle;

q) equipment for lot or sample splitting as described in ISO 3954.

The above list of tools shall not be considered exclusive: other tools that satisfy the non-contamination criterion may also be used.

4.2 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.

5 Sample selection

Samples of individual items shall be taken from different sampling positions according to appropriate sampling schemes in order to provide information on the homogeneity of the sampled item and shall be analysed separately.

Similarly, powders, crystals and casting grain shall be sampled by appropriate lot splitting techniques to provide representative portions for testing.

NOTE Annex A provides guidelines for selected products.

If appropriate studies testify a sufficient homogeneity for the relevant product type and composition, a sampling scheme comprising a reduced number of sampling positions may be used.

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

6 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).

7 Sampling methods

7.1 General

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

For bars, ingots, sheet, rod, tube, wire, casting grain and other raw materials or semi-finished products each prepared sample shall weigh at least two times the amount required for a full assay in duplicate, where practical.

For component parts and finished products each prepared sample shall provide sufficient material for one full assay in duplicate.

If the sample material from a specific sampling position (e.g. drilling hole) is not sufficient, further material shall be sampled in immediate proximity or from a geometrically equivalent position.

In the case of small items the required minimum weight shall be made up with a sufficient number of pieces.

In the case of mixed precious metal articles cross-contamination shall be avoided. For products that have been assembled by soldering, the solder line shall be avoided.

NOTE Inclusion of solder in the sample can be subjected to national legislations.

WARNING — Use of the tools without appropriate safety measures can result in serious injuries.

7.2 Dip sampling of molten alloy

7.2.1 General

In dip sampling a small quantity of molten metal is drawn from a melt and allowed to cool rapidly in order to minimize segregation effects (primary sampling). The solidified metal is then submitted to a secondary sampling.

The sampling shall be carried out 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 proceeding to the secondary sampling.

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.

7.2.2 Method 1 – Pin sampling

A 3 mm to 5 mm diameter quartz or graphite tube shall be used to extract an approximately 7 cm long cylinder (pin) of molten metal. After cooling and removal of all the quartz or graphite, the cylinder shall be flattened to provide a thin strip of alloy from which the required number of test portions shall be cut after discarding the two end sections. The thickness of the strip shall be adequate for the intended analytical technique.

7.2.3 Method 2 - Bead sampling, button sampling

A ladle made of graphite or sooted 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 into 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, heated at 150 °C to 200 °C until dry, and then subdivided by standard techniques (see A.1.2) to provide the test portions. The small ingot or disk obtained by pouring the metal into a mould shall be sampled by drilling in several spots, and the drillings combined to constitute a single sample.

7.3 Drilling

A 3 mm to 6 mm twist drill bit shall be operated at about 500 r/min to 900 r/min without lubricants or with some drops of ethanol, and at least half the thickness of the product shall be drilled unless otherwise specified. Drillings shall be broken, if necessary, and the broken spiral drillings combined with other material from the same sampling position before selecting the required number of test portions. Drillings from different sampling positions shall not be combined unless unavoidable to provide the required minimum sample weight specified in 7.1.

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

NOTE Depending on the shape and size of the sampled items, the use of ordinary drilling devices can constitute a safety hazard. In such cases the use of CNC tooling or sampling by sawing can constitute a safer alternative.

7.4 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.

7.5 Cutting

When sampling by cutting, a complete cross-section shall be taken as sample whenever possible.

NOTE Annex A provides guidelines for cutting samples from larger items (see A.2.2).

7.6 Sawing or filing

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

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

NOTE Sampling by filing is only appropriate where other sampling techniques do not allow to provide the required samples, e.g. in the case of particularly hard materials.

8 Retention of samples

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.

Annex A
(informative)

Guidelines for the sampling of typical products and items

A.1 Raw material and cast products

A.1.1 Cast bars

Each bar shall be sampled separately, by drilling to a pre-arranged diagonal pattern as shown in Figure A.1. Sampling may also be done by sawing as shown in Figure A.2 or by cutting.

The drillings from the upper part of hole B, roughly until a depth corresponding to the one of holes A and C, shall be discarded.

Additional holes on the other diagonals can be required if insufficient material is available.

Dimensions in millimetres

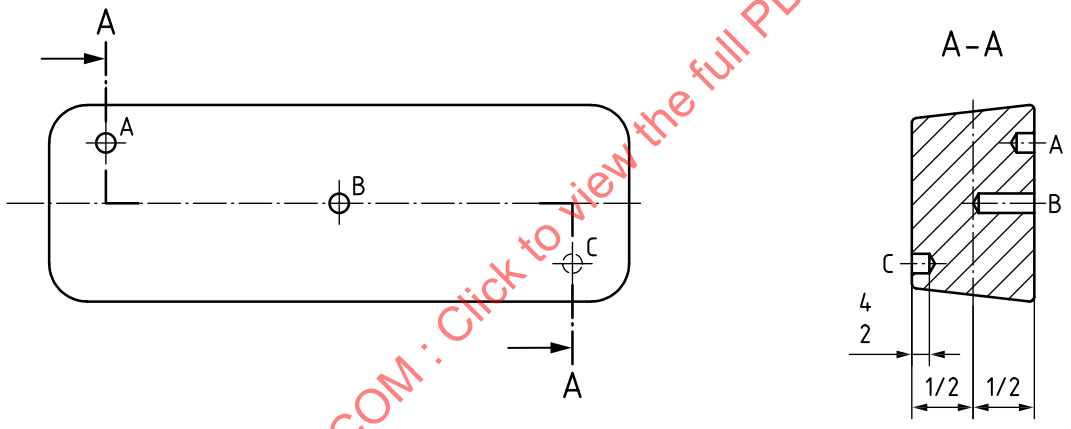

Figure A.1 — Sampling of cast bars by drilling

Dimensions in millimetres

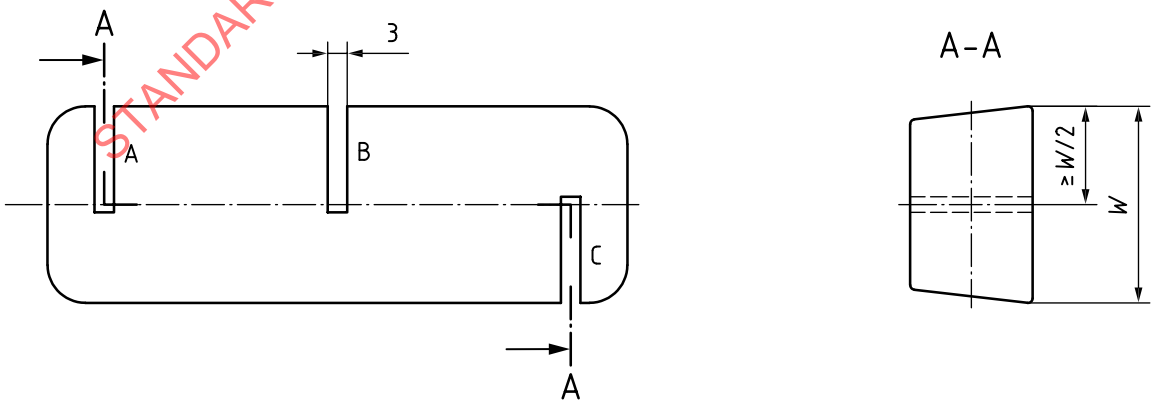

Figure A.2 — Sampling of cast bars by sawing

For thin cast bars (indicatively up to 15 mm to20 mm thickness) sample at position A.

For thick cast bars (indicatively from 30 mm to 40 mm thickness where heterogeneity can be presumed, otherwise from 60 mm to 80 mm) sample at positions A, B, C. If significant heterogeneity can be presumed, sampling by sawing should be preferred.

For bars of intermediate thickness sample at positions A and B.

For bars cast in vertical clamshell moulds the sampling scheme A.1 shall be replaced by the sampling scheme A.3.

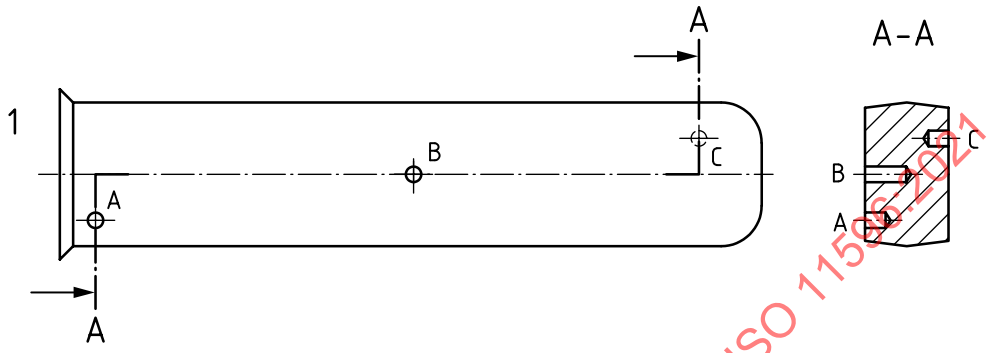

Figure A.3 — Sampling of bars cast in vertical clamshell moulds by drilling

Specific, insoluble elements like Osmium/Iridium and Ruthenium in Gold tend to concentrate in specific zones of the cast bars (Os/Ir at the centre and at the bottom face, Ru at the centre and at the upper face). When this presence is suspected additional samples from the possibly affected zones should be taken in order to provide additional analytical confidence.

A.1.2 Casting grain

The required number of samples shall be taken by appropriate subdivision techniques, e.g. by use of a rotary sample divider, a riffle sampler or by coning and quartering. Lot portioning can be performed by analogy as described in ISO 3954. Grab sampling should be avoided.

For each lot weighing up to 2 kg at least two samples, and for each lot weighing more than 2 kg at least three samples shall be drawn and analysed separately.

NOTE Granular materials can display a wide range of grain sizes. In this case it is advisable to separate the material into at least three different grain size classes before the sampling, to record their mass portion and to draw subsequently separate samples for each collected class.

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.

A.1.3 Powders and crystals

For metallic powders and crystals, whose lot homogeneity can be significantly affected by their production processes, similar considerations concerning subdivision techniques and particle size related requirements as indicated for the metals in casting grain in A.1.2 apply.

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

A.2.1 Cast ingots, 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 circular 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 (A1, A2 in Figure A.4). 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 (1, 2) and in two directions at right angles to each other (A, B) as shown in Figure A.4, 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.

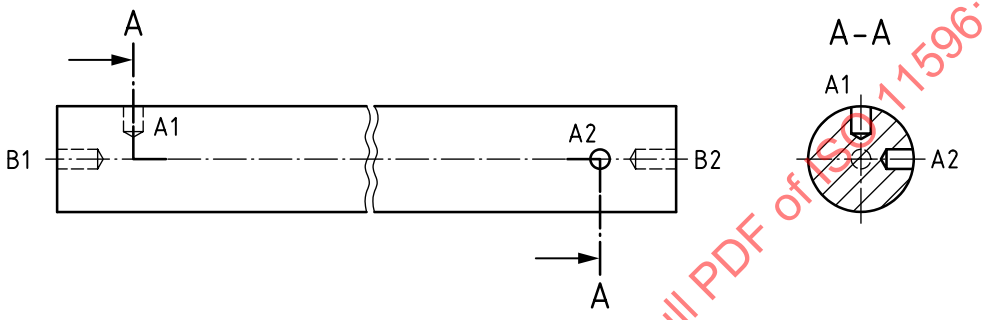

Figure A.4 — Sampling of a thick circular rod (d > 1 cm)

For cast ingots and rectangular rods similar considerations apply; for alloys deemed to be homogeneous, the samples should be drawn according to Figure A.5 for material cast in vertical clamshell moulds and to Figure A.6 for materials produced by continuous casting.

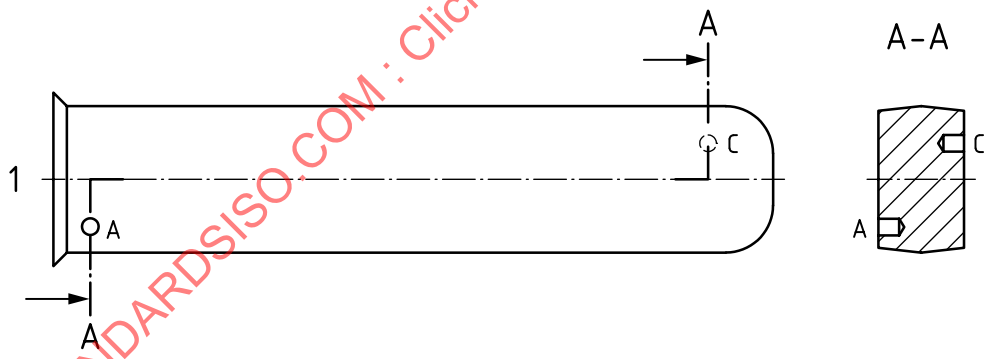

Figure A.5 — Sampling scheme for homogeneous vertical cast ingots or rectangular rods