

Australian Standard™

Gold and gold bearing alloys

**Part 1: Determination of gold content
(less than 30%)—Gravimetric (fire
assay) method**



This Australian Standard was prepared by Committee CH-010, Analysis of Metals. It was approved on behalf of the Council of Standards Australia on 13 October 2005.
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The following are represented on Committee CH-010:

Amdel
Australasian Institute of Mining and Metallurgy
Australian Aluminium Council
Institute of Materials Engineering Australasia
International Precious Metals Institute
National Association of Testing Authority
The Royal Australian Chemical Institute

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RECONFIRMATION

OF

AS 3515.1—2005

Gold and gold bearing alloys

**Part 1: Determination of gold content (less than 30%)—Gravimetric (fire assay)
method**

RECONFIRMATION NOTICE

Technical Committee CH-010 has reviewed the content of this publication and in accordance with Standards Australia procedures for reconfirmation, it has been determined that the publication is still valid and does not require change.

Certain documents referenced in the publication may have been amended since the original date of publication. Users are advised to ensure that they are using the latest versions of such documents as appropriate, unless advised otherwise in this Reconfirmation Notice.

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Bureau of Steel Manufacturers of Australia
International Copper Association Australia
International Precious Metals Institute
National Association of Testing Authorities Australia

NOTES

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assay) method**

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PREFACE

This Standard was prepared by the Joint Standards Australia/Standards New Zealand Committee CH-010, Analysis of Metals, to supersede AS 3515.1—1996, *Gold and gold bearing alloys, Part 1: Determination of gold content (less than 30%)—Gravimetric method*. After consultation with stakeholders in both countries, Standards Australia and Standards New Zealand decided to develop this Standard as an Australian, rather than an Australian/New Zealand Standard.

The objective of this Standard is to ensure that a gravimetric method for the determination of gold content less than 30% by gravimetric (fire assay) method is achieved.

This revision incorporates information on acceptance of results into a new Section.

This Standard is Part 1 of a series of Standards for the determination of gold content in gold and gold bearing alloys and reference should be made to the other documents in the series:

AS

- 3515 Gold and gold bearing alloys
- 3515.2 Part 2: Determination of gold content (30%–99.5%)—Gravimetric (fire assay) method
- 3515.3 Part 3: Determination of gold content (greater than 99.5%)—Gravimetric (fire assay) method
- 3515.4 Part 4: Determination of gold content (greater than 99.95%)—Inductively coupled plasma—Atomic emission spectrometry

The term ‘informative’ has been used in this Standard to define the application of the appendix to which it applies. An ‘informative’ appendix is for information and guidance only.

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1.3.2 Cupellation

The process by which the precious metals are separated from the lead and other base metals with which they are alloyed. It is also the process whereby the sample, having been wrapped in lead sheet/foil with the necessary additives (i.e. silver or copper or both), is homogenized in the molten state prior to the separation of the gold and silver.

1.3.3 Gold cornet

A cornet that has been parted.

1.3.4 Laboratory sample

A sample as prepared for sending to the laboratory and intended for inspection or testing.

1.3.5 Parting

The separation of gold from other precious metals by means of nitric acid in a chloride-free environment, where the silver forms soluble silver nitrate and the gold remains undissolved.

1.3.6 Prill

The 'button' or 'bead' of precious metal obtained from the cupellation process.

1.3.7 Proof

A reference sample included in every batch of assays in order to monitor the effect of assay conditions.

1.3.8 Surcharge

The sum of the gains and losses of mass of the proofs during the assay process, determined as a mean value.

1.3.9 Test portion

The portion of material taken from the test sample or, if both are the same, from the laboratory sample, on which the test or assay is actually carried out.

1.3.10 Test sample

A sample prepared from the laboratory sample and from which test portions will be taken.

1.4 REAGENTS

WARNING: SOME REAGENTS USED IN THIS METHOD MAY CONSTITUTE A HEALTH AND SAFETY HAZARD AND THEIR HANDLING SHOULD BE IN ACCORDANCE WITH AS/NZS 2243 PARTS ONE AND TWO, AS 2508 AND AS 2830.1.

1.4.1 General requirements

Analytical reagent (AR) grade reagents and Grade 3 water, as defined by ISO 3696, shall be used throughout this method. Nitric acid shall not contain chloride ion at a concentration greater than 1 mg of chloride per litre.

The preparation of reagents shall be carried out in a fume hood.

1.4.2 Nitric acid ρ_{20} 1.42 g/mL

1.4.3 Nitric acid (1:2)

Cautiously add 1 volume of nitric acid (1.4.2) to 2 volumes of water with continuous stirring.

1.4.4 Nitric acid (1:1)

Cautiously add one volume of nitric acid (1.4.2) to 1 volume of water with continuous stirring.

1.4.5 Silver

Containing greater than 99.9% silver and less than 5 mg of gold per kg of silver.

1.4.6 Proof gold

Containing at least 99.996% gold with a previously determined accurate value.

1.4.7 Copper metal**1.4.8 Lead foil****1.4.9 Ammonium hydrogen difluoride****1.5 APPARATUS****1.5.1 Magnesite cupels**

The choice of cupel size is governed by the ability of the cupel to absorb lead, and the expected amount of base metal in the test sample. The most commonly used cupel weighs approximately 36 g, and is suitable for up to 7 g of lead. A 54 g cupel is used for test samples containing a high base metal content, and is suitable for up to 14 g of lead.

NOTE: Magnesite cupel blocks of similar lead absorption capacity are also suitable.

1.5.2 Anvil and hammer

Of polished steel.

1.5.3 Analytical balance

Capable of a resolution of 0.01 mg.

1.5.4 Cupellation furnace

Capable of attaining and maintaining a controlled oxidizing atmosphere and a uniform temperature of at least $1100^{\circ}\text{C} \pm 50^{\circ}\text{C}$.

NOTE: A muffle furnace is not a satisfactory cupellation furnace.

1.5.5 Annealing furnace

Capable of maintaining a temperature of $800^{\circ}\text{C} \pm 50^{\circ}\text{C}$.

1.5.6 Rollers

Constructed of polished steel, capable of reducing the prill to a thickness of 0.2 mm to 0.3 mm.

1.5.7 Parting trays (baskets)

Of platinum or fused quartz.

1.5.8 Prill cleaning brush

Having tufts of stiff bristle or nylon.

NOTE: Metal tufts are not satisfactory for this purpose.

SECTION 2 SAMPLE PREPARATION AND PRELIMINARY DETERMINATION

2.1 GENERAL

This Section specifies the preliminary determination undertaken to determine the total precious metal content in order to choose which of the two procedures set out in Sections 3 and 4 is applicable, see Clause 2.4.

2.2 SAMPLE INSPECTION AND PREPARATION

The sample shall be taken and inspected for the presence of discernible inhomogeneities, contaminants and, in the case of a sample prepared by the vacuum tube method, piping (hollow centre). If any of these defects are present, the sample shall be rejected. If not supplied in a form suitable for assay, the sample shall be flattened to render it suitable for cutting.

NOTE: The sample should be taken using one of the options listed in Appendix A.

2.3 PRELIMINARY DETERMINATION OF THE TOTAL PRECIOUS METALS, APPROXIMATE GOLD, SILVER AND TOTAL BASE METALS CONTENT

2.3.1 Test portion preparation

If the total precious metals content, approximate gold content or total base metals content is unknown, a preliminary assay shall be carried out as follows:

- (a) Fold one sheet of approximately 7 g of lead foil (1.4.8) into a cone.
- (b) Weigh a test portion of approximately 500 mg and record the mass (m_1) to 0.1 mg.
- (c) Fold the lead cone tightly around the contents and carefully squeeze it into a compact form.

2.3.2 Cupellation

The cupellation procedure shall be as follows:

- (a) Preheat the cupellation furnace (1.5.4) to $1100^{\circ}\text{C} \pm 50^{\circ}\text{C}$. If nickel is present, the cupellation temperature should be at least 1100°C .
- (b) Place cupels (1.5.1) into the preheated cupellation furnace using one cupel for each test portion and allow the cupels to attain the furnace temperature.

NOTE: Cupels should be arranged so that test portions are evenly distributed from left to right and from the back to the front of the furnace.

- (c) To minimize the temperature loss in the cupellation furnace, quickly place the folded cones onto preheated cupels in order from left to right and from the back to the front.
- (d) Maintain the samples at the appropriate temperature until cupellation is complete, (25 min to 30 min), ensuring that adequate airflow is maintained through the furnace to promote oxidation.
- (e) Remove the cupels from the furnace, maintaining them in the original spatial order, and allow them to cool in a well ventilated area.
- (f) As soon as the material has solidified, loosen each prill from its cupel. Check that the prills are smooth, hard and shiny. If any prills are dull, flat or fragmented (this being due to a high base-metal content), the determination shall be repeated with additional lead.

- (g) Place each cooled prill on its side on the anvil (1.5.2) and hammer it once firmly to dislodge adhering cupel material. Carefully brush off excess magnesite with the prill cleaning brush (1.5.8).
- (h) Weigh the prill and record the mass to 0.1 mg (m_2).

2.3.3 Re-inquarting

The procedure shall be as follows:

- (a) To a folded 4 g lead cone, add the sample prill (2.3.2), 400 mg of silver and approximately 40 mg of copper.
- (b) Fold the lead cones tightly around the contents and carefully squeeze to a compact form.
- (c) Cupel again in accordance with Clause 2.3.2 (a) to (g).

2.3.4 Parting

The procedure for parting shall be as follows:

- (a) Hammer each flattened prill into an oval sheet, ensuring that a thin edge is produced to facilitate rolling. Anneal each sheet briefly at $800^{\circ}\text{C} \pm 50^{\circ}\text{C}$ in a furnace (1.5.5).
- (b) Pass each sheet through rollers (1.5.6) reducing the roller clearance with each pass until the resultant sheets are between 0.2 mm and 0.3 mm in thickness.
- (c) Anneal each strip as in Step (a).
- (d) Crimp over the end and carefully roll each strip into a compact cornet. Sufficient clearance should be allowed between each fold to allow acid penetration of the cornet.
- (e) Place the cornet into a 150 mL beaker and add 50 mL nitric acid 1:2 (1.4.3) to complete the parting.
- (f) Gently boil until the fumes oxides of nitrogen cease to evolve then decant the acid waste.
- (g) Wash until acid-free and transfer the residue to a ceramic crucible with water.
- (h) Decant the water and dry the residue on a hotplate.
- (i) Anneal the gold residue at a temperature of $800^{\circ}\text{C} \pm 50^{\circ}\text{C}$ in a furnace (1.5.5).
- (j) Allow the gold residue to cool to room temperature.
- (k) Weigh the gold (m_3), and record the mass to 0.1 mg.

2.3.5 Calculations

Calculate the total precious metals content from the following equation:

$$\text{Total precious metal (\%)} = \frac{m_2}{m_1} \times 100 \quad \dots 2.3(1)$$

where

m_2 = mass of prill determined in Step 2.3.2(k), in milligrams

m_1 = mass of test portion used in Step 2.3.1(b), in milligrams

Calculate the approximate gold content of the test portion from the following equation:

$$\text{Approximate gold content (\%)} = \frac{m_3}{m_1} \times 100 \quad \dots 2.3(2)$$

where

m_3 = mass of gold determined in Step 2.3.4(k) in milligrams

m_1 = mass of test portion used in Step 2.3.1(b) in milligrams

Calculate the approximate silver content, as a percentage, by subtracting the approximate gold content (calculated from Equation 2.3(2)) from the total precious metals content (calculated from Equation 2.3(1)).

Calculate the total base metals content, as a percentage, by subtracting the total precious metals content from 100.

2.4 CRITERIA FOR SELECTION OF DETERMINATION METHOD

Where the total precious metals content is less than 40%, Section 3 shall be used, otherwise Section 4 shall be used.

SECTION 3 TOTAL PRECIOUS METALS CONTENT LESS THAN 40 %

3.1 SCOPE OF SECTION

This Section sets out a method for the determination of gold in gold bearing alloys containing less than 30% gold and with a total precious metals content less than 40%.

3.2 PRINCIPLE

Test samples that contain less than 40% total precious metals are treated with dilute nitric acid to remove silver, base metals and other interfering elements. The gold residue is annealed, weighed, transferred to a lead cone, cupelled, parted and weighed.

3.3 NUMBER OF DETERMINATIONS

A minimum of two determinations shall be carried out on each test sample.

3.4 PROOF TESTS

A minimum of two proof tests shall be carried out with each batch of test portions, and shall be subjected to the same procedure as the test portions. The proofs shall contain a mass of proof gold (1.4.6) equivalent to and within $\pm 5\%$ of the mass of gold in the test portions.

3.5 PREPARATION OF TEST PORTION AND PROOF

The procedure shall be as follows:

- (a) Weigh and record to 0.01 mg a test portion (m_4) from each test sample, such that:
 - (i) Mass of the recovered gold is in the range 150 mg to 500 mg.
 - (ii) The maximum weight of the test portion is 75 g.
- (b) Transfer the test portion to a beaker.

NOTE: If tin is present, to complex it add 2 g of ammonium hydrogen difluoride (1.4.9) and dissolve in a minimum volume of water.
- (c) Cautiously add sufficient 1:2 nitric acid (1.4.3) to the test portion, the reaction may be quite vigorous depending on the base metal content.
- (d) Cover and heat the beaker until reaction ceases.
- (e) Allow the beaker to cool and dilute the contents with water to approximately 80% of the beaker nominal capacity.
- (f) Mix thoroughly, allow contents to settle and separate the gold by decanting. The gold residue should be washed with water.

NOTE: Filtration may be necessary in case of presence of fine particles.
- (g) Quantitatively transfer the gold to a ceramic crucible, dry and anneal in a furnace (1.5.5) at $800^\circ\text{C} \pm 50^\circ\text{C}$.
- (h) Remove from the furnace and allow to cool in a breeze free environment.
- (i) Weigh and record the mass of the gold residue (m_5) to 0.01 mg.

NOTE: This weight is used to determine the mass of—

 - (a) silver to be added prior to cupellation; and
 - (b) proof gold to be cupelled.

- (j) For each proof and for the gold residue from each test portion, fold one 4 g sheet of lead foil (1.4.8) into a cone.
- (k) Weigh and record an amount of proof gold (m_6) (see Clause 3.4) equivalent in mass to the gold residue (m_5) and transfer to separate lead cones.
- (l) To each cone containing the proof and to each cone containing the gold residue from each test portion, add 40 mg of copper (1.4.7) and sufficient silver (1.4.5) to give a silver to gold ratio of 2.5:1.

NOTES:

- 1 The mass of silver to be added to the test portion is therefore, $m_5 \times 2.5$ and for the proof, $m_6 \times 2.5$.
 - 2 A small amount of copper added to a sample containing no copper acts as a deoxidant, and helps prevent cracking and splitting of the cornet edges when it is being hammered and rolled.
- (m) Fold the cones tightly around the contents and carefully squeeze to a compact form.

3.6 CUPELLATION

The cupellation procedure shall be as follows:

- (a) Preheat the cupellation furnace (1.5.4) to $1100^{\circ}\text{C} \pm 50^{\circ}\text{C}$.
- (b) Place cupels (1.5.1) into the preheated cupellation furnace using one cupel for each test portion and proof and allow the cupels to attain the furnace temperature.
NOTE: Cupels should be arranged so that test portions and proofs are evenly distributed from left to right and from the back to the front of the furnace.
- (c) To minimize the temperature loss in the cupellation furnace, quickly place the folded cones onto preheated cupels in order from left to right and from the back to the front.
- (d) Maintain the samples at the cupellation temperature until cupellation is complete, (25 – 30 min) ensuring that adequate airflow is maintained through the furnace to promote oxidation.
- (e) Remove the cupels from the furnace, maintaining them in the original spatial order, and allow them to cool in a breeze-free environment. As soon as the material has solidified, loosen each prill from its cupel.
- (f) Check that the prills are smooth, hard and shiny. Reject any that are dull, flat or fragmented.
- (g) Place each cooled prill on its side on the anvil (1.5.2), and hammer it once, firmly, to dislodge adhering cupel material. Carefully brush off the excess magnesite with a prill cleaning brush (1.5.8).

3.7 PARTING

The procedure for parting shall be as follows:

- (a) Hammer each flattened prill into an oval sheet, ensuring that a thin edge is produced to facilitate rolling. Anneal each sheet briefly at $800^{\circ}\text{C} \pm 50^{\circ}\text{C}$ in a furnace (1.5.5).
- (b) Pass each sheet through rollers (1.5.6) reducing the roller clearance with each pass until the resultant sheets are between 0.2 mm and 0.3 mm in thickness.
- (c) Anneal each strip as in Step (a).
- (d) Crimp over the end and carefully roll each strip into a compact cornet. Sufficient clearance should be allowed between each fold to allow acid penetration of the cornet.

- (e) Preheat to approximately 90°C a quantity of the following solutions sufficient to immerse the parting tray (1.5.7).
 - (i) Sufficient nitric acid 1:2 (1.4.3) as will enable the complete submersion of the parting tray (1.5.7) for Step (f).
 - (ii) Sufficient nitric acid 1:1 (1.4.4) as will enable the complete submersion of the parting tray (1.5.7) for Step (g).
 - (iii) Sufficient water for Step (h).
- (f) Carefully place each cornet into separate thimbles of a parting tray. Submerge the parting tray in hot nitric acid (Step (e)(i)). Allow the parting reaction to subside and boil gently until fumes of oxides of nitrogen have ceased.
- (g) Transfer the parting tray to a second vessel containing hot nitric acid (Step (e)(ii)) and boil gently for 30 min.
- (h) Transfer the parting tray to a container of hot water (Step (e)(iii)) and wash in three changes of hot water by agitating the tray.
- (i) Remove excess water from the parting tray by draining and carefully dry it on a hotplate. Anneal the cornets at a temperature of approximately 800°C ±50°C in a furnace (1.5.5) for 5 min.
- (j) Cool and allow the parting tray to come to equilibrium with the balance room temperature.
- (k) Weigh the gold cornet from each test portion (m_7) and each proof (m_8) and record to 0.01 mg.

3.8 CALCULATIONS

3.8.1 Change in mass of proof

The change in mass for each proof shall be calculated from the following equation:

$$\Delta m = m_8 - m_6 \quad \dots 3.8(1)$$

where

Δm = loss or gain in mass for the proof, in milligrams

m_8 = mass of the proof cornet from Step 3.7(k), in milligrams

m_6 = mass of proof gold from Step 3.5(k), in milligrams

3.8.2 Determination of surcharge

The surcharge shall be determined as follows:

- (a) If the range of the Δm values for the proofs is 0.15 mg, or greater, the determination shall be repeated.
- (b) If the range of the Δm values is less than 0.15 mg, then the surcharge \bar{m} shall be the average change in mass of the proof (Δm).

3.8.3 Adjusted mass of the gold cornet

The adjusted mass of the gold cornet shall be calculated from the following equation:

$$m_a = m_7 - \bar{m} \quad \dots 3.8(2)$$

where

m_a = adjusted mass of the gold cornet, in milligrams

m_7 = mass of the gold cornet from each test portion, from Step 3.7(k), in milligrams

\bar{m} = surcharge, in milligrams from 3.8.2(b)

3.8.4 Gold content

The gold content of each test portion shall be calculated from the following equation:

$$\text{Gold content (\%)} = \frac{m_a}{m_4} \times 100 \quad \dots 3.8(3)$$

where

m_a = adjusted mass of the gold cornet, from Clause 3.8.3, in milligrams

m_4 = mass of the test portion, from Step 3.5(a), in milligrams

The gold content of the test sample is the mean of the duplicate determinations. For precision and acceptance of results, see Section 5.

SECTION 4 TOTAL PRECIOUS METALS CONTENT IS 40% OR GREATER

4.1 SCOPE OF SECTION

This Section sets out a method for the determination of gold in alloys containing less than 30% gold but more than 40% total precious metals.

4.2 PRINCIPLE

The test portions are weighed and gold is added such that after parting, a gold cornet is produced. The weight of added gold is then subtracted from the gold cornet to give the amount of gold in the test portion.

4.3 NUMBER OF DETERMINATIONS

Two determinations shall be carried out on each test sample.

4.4 PROOF TESTS

A minimum of two proof tests shall be carried out with each batch of test portions, and shall be subjected to the same procedure as the test portions. The proofs shall contain a mass of proof gold (1.4.6) equivalent to and within $\pm 5\%$ of the mass of gold in the test portion plus the added gold. Copper (1.4.7) or other base metals, if known, shall be added in quantities equivalent to the estimated total base metals content of the test sample. If there is no base metal content in the sample, 40 mg of copper should be added to the proof.

4.5 PREPARATION OF THE TEST PORTION AND PROOF

The method shall be as follows:

- (a) Fold a sheet of lead foil (1.4.8) into a cone for each test portion and proof.
NOTE: The amount of lead foil required varies. The greater the base metal content, the greater the amount of lead foil to be used. Not less than 4 g or more than 14 g of lead foil should be used.
- (b) Weigh in duplicate, record the mass to 0.01 mg and transfer the following to the appropriate lead cone:
 - (i) 500 mg ± 5 mg of each test portion (m_9) and the appropriate mass of proof gold (1.4.6) (m_{10}) such that the resulting gold cornet weighs in the range 150 mg to 500 mg.
 - (ii) The appropriate amount of proof gold (m_{11}) (see Clause 4.4).
- (c) To each cone containing a test portion, add sufficient silver if necessary (1.4.5) to give a silver to gold ratio of 2.5:1, and if the test sample does not contain obvious copper, add 40 mg of copper (1.4.7).
- (d) To each cone containing a proof, add metals as specified in Clause 4.4 and add silver to give a silver to gold ratio of 2.5:1.
NOTE: A small amount of copper added to a sample containing no copper acts as a deoxidant and helps prevent cracking and splitting of the cornet edges when it is being hammered and rolled.
- (e) Fold the cones tightly around the contents and carefully squeeze to a compact form.

4.6 CUPELLATION

The cupellation procedure shall be as follows:

- (a) Preheat the cupellation furnace (1.5.4) to $1100^{\circ}\text{C} \pm 50^{\circ}\text{C}$. If nickel is present, the cupellation temperature shall be at least 1100°C .
- (b) Place cupels (1.5.1) into the preheated cupellation furnace using one cupel for each test portion and proof and allow the cupels to attain the furnace temperature.
NOTE: Cupels should be arranged so that test portions and proofs are evenly distributed from left to right and from the back to the front of the furnace.
- (c) To minimize the temperature loss in the cupellation furnace, quickly place the folded cones onto preheated cupels in order from left to right and from the back to the front.
- (d) Maintain the samples at the cupellation temperature until cupellation is complete, (25–30 min) ensuring that adequate airflow is maintained through the furnace to promote oxidation.
- (e) Remove the cupels from the furnace, maintaining them in the original spatial order, and allow them to cool in a breeze-free environment. As soon as the material has solidified, loosen each prill from its cupel.
- (f) Check that the prills are smooth, hard and shiny. Reject any that are dull, flat or fragmented.
- (g) Place each cooled prill on its side on the anvil (1.5.2), and hammer it once, firmly, to dislodge adhering cupel material. Carefully brush off the excess magnesite with a prill cleaning brush (1.5.8).

4.7 PARTING

The parting shall be as follows:

- (a) Hammer each flattened prill into an oval sheet, ensuring that a thin edge is produced to facilitate rolling. Anneal each sheet briefly at $800^{\circ}\text{C} \pm 50^{\circ}\text{C}$ in a furnace (1.5.5).
- (b) Pass each sheet through rollers reducing the roller clearance with each pass, until the resultant sheets are between 0.2 mm and 0.3 mm.
- (c) Anneal each strip as in Step (a).
- (d) Crimp over the end and carefully roll each strip into a compact cornet. Sufficient clearance should be allowed between each fold to allow acid penetration of the cornet.
- (e) Preheat to approximately 90°C a quantity of the following solutions sufficient to immerse the parting tray (1.5.7):
 - (i) Sufficient nitric acid 1:2 (1.4.3) as will enable the complete submersion of the parting tray (1.5.7) for Step (f).
 - (ii) Sufficient nitric acid 1:1 (1.4.4) as will enable the complete submersion of the parting tray (1.5.7) for Step (g).
 - (iii) Sufficient water for Step (h).
- (f) Carefully place each cornet into separate thimbles of a parting tray. Submerge the parting tray in hot nitric acid (Step(e)(i)). Allow the parting reaction to subside and gently boil until fumes of oxides of nitrogen have ceased.
- (g) Transfer the parting tray to a second vessel containing hot nitric acid (Step (e)(ii)) and boil gently for 30 min.

- (h) Transfer the parting tray to a container of hot water (Step (e)(iii)) and wash in three changes of hot water by agitating the tray.
- (i) Remove excess water from the parting tray by draining and carefully dry it on a hotplate. Anneal the cornets at $800^{\circ}\text{C} \pm 50^{\circ}\text{C}$ in a furnace (1.5.5) for 5 min.
- (j) Cool and allow the parting tray to come to equilibrium with the balance room temperature.
- (k) Weigh the gold cornet from each test portion (m_{12}) and each proof (m_{13}) and record to 0.01 mg.

4.8 CALCULATIONS

4.8.1 Change in mass of proof

The change in mass of each proof shall be calculated from the following equation:

$$\Delta m = m_{13} - m_{11} \quad \dots 4.8(1)$$

where

Δm = loss or gain in mass of proof, in milligrams

m_{13} = mass of the proof cornet (see Step 4.7(k)), in milligrams

m_{11} = mass of proof gold (see Step 4.5(b)(ii)), in milligrams

4.8.2 Determination of surcharge

The surcharge shall be determined as follows:

- (a) If the range of the Δm values for the proofs is 0.15 mg, or greater, the determination shall be repeated.
- (b) If the range of the Δm values is less than 0.15 mg, then the surcharge \bar{m} shall be the average change in mass of the proof (Δm).

4.8.3 Adjusted mass of the gold cornet

The adjusted mass of the gold cornet shall be calculated from the following equation:

$$m_a = m_{12} - (\bar{m} + m_{10}) \quad \dots 4.8(2)$$

where

m_a = adjusted mass of the gold cornet

m_{12} = mass of gold cornet from each test portion, from Step 4.7(k), in milligrams

\bar{m} = mass of surcharge, from Clause 4.8.2(b), in milligrams

m_{10} = mass of gold added, in Step 4.5(b)(i), in milligrams

4.8.4 Gold content

The gold content of each test portion shall be calculated from the following equation:

$$\text{Gold content (\%)} = \frac{m_a}{m_g} \times 100 \quad \dots 4.8(3)$$

where

m_a = adjusted mass of the gold cornet, from Clause 4.8.3, in milligrams

m_g = mass of test portion from Step 4.5(b)(i), in milligrams

The gold content of the test sample is the mean of duplicate determinations. For precision and acceptance of results, see Section 5.

SECTION 5 PRECISION OF ACCEPTANCE OF RESULTS

5.1 PRECISION

The planned trial was carried out in accordance with AS 2850 by seven analysts, each from a different laboratory. Four tests were carried out by each analyst on each of five samples. From the results obtained, the 95% confidence limits have been calculated (see Table 1).

TABLE 1
PRECISION DATA FOR GOLD DETERMINATIONS

Sample No.	Gold content %	Repeatability <i>r</i>	Reproducibility <i>R</i>
1	1.066 1	0.008 0	0.013 2
2	6.967 2	0.023 8	0.039 9
3	17.526 8	0.052 0	0.055 0
4	24.959 6	0.055 5	0.053 8
5	27.599 4	0.039 6	0.049 4

5.2 ACCEPTANCE OF RESULTS

5.2.1 Reference materials

If the value obtained for the certified reference materials does not agree to within $0.7R$ of its recognized value, the procedure shall be repeated with the original and a different reference material.

5.2.2 Test results

Where duplicate determinations have been carried out, the difference between the results should not exceed the value of r and the value for the reference material should agree to within $0.7R$ of its recognized value. If this is not the case, the determination shall be repeated.

5.3 TEST REPORT

The test report shall contain the following information:

- (a) Identification and description of sample.
- (b) Date on which the sample was collected.
- (c) Date on which the sample was analysed.
- (d) The gold content of the test sample, expressed as a percentage by mass of the sample, to two decimal places.
- (e) Any unusual observations made during the course of the test which may have had an effect on the results.
- (f) Reference to this Standard, i.e. AS 3515.1.

APPENDIX A
METHODS OF SAMPLING DORE BULLION
(Informative)

A1 SCOPE

This Appendix provides guidance and information on five methods of sampling dore bullion, as follows:

- (a) Vacuum tube sampling.
- (b) Dip sampling.
- (c) Sampling by clipping.
- (d) Sampling by sawing.
- (e) Sampling by drilling.

A2 CRITERIA FOR SELECTION OF A METHOD

The methods are presented in descending order of preferred use; whenever circumstances permit, either a vacuum tube sampling method or dip sampling method should be used.

The two most preferred methods involve melting of the bullion; the other three methods are used where melting is not permitted or impractical, e.g. where only a small amount of material is available for assay.

NOTE: The presence of iron, lead, antimony or arsenic may cause difficulties in obtaining a homogeneous sample.

A3 VACUUM TUBE SAMPLING

A3.1 Principle

An evacuated, sealed glass tube, having a thin-walled bulb section near one end is plunged into a crucible of molten metal. The bulb melts upon contact with the molten metal, which is then drawn into the tube. After cooling, the sample is removed from the tube and inspected to ensure that it is satisfactory for assay.

A3.2 Procedure

The procedure for vacuum tube sampling is as follows:

- (a) Stir the molten bullion that is to be sampled with a carbon rod for 1 min.
- (b) Immediately plunge the vacuum tube into the melt at an angle of 45°, remove it and quench it in water.
- (c) Remove the tube from the water and shatter the glass to free the sample.
- (d) Remove both ends of the sample and return them to the melt.
- (e) Inspect the surface of the sample for inclusions.
- (f) If inclusions are present, try to remove them or reject the sample if they cannot be removed.
- (g) Cut the dried sample into sections, and inspect them for the presence of 'piping', i.e. a hollow centre. If piping is present, reject the sample.

A4 DIP SAMPLING

A4.1 Principle

A graphite ladle or a (preheated) dipping iron is inserted into a crucible of molten metal, and then withdrawn to remove the sample.

A4.2 Sampling apparatus

The recommended sampling apparatus comprises one of the following:

- (a) *Graphite ladle*—Of capacity 3 mL to 5 mL.
- (b) *Dipping iron*—Constructed of iron and comprising a bent rod to which is attached either a two-dip sampler or a four-dip sampler.

A typical dipping iron is shown in Figure A1; the dimensions are not critical and may be varied to suit individual circumstances. A typical sampler is made from a 10 mm or 12 mm rod and has recesses made with a 10 mm or 12 mm drill bit to a depth of approximately 8 mm. It is essential that, before its first use, the sampler be 'blued' (i.e. heat treated).

Before each use it needs to be either coated with lampblack or held over a smoky flame. Further, it always needs to be preheated to approximately 200°C when being used for sampling.

A4.3 Procedure

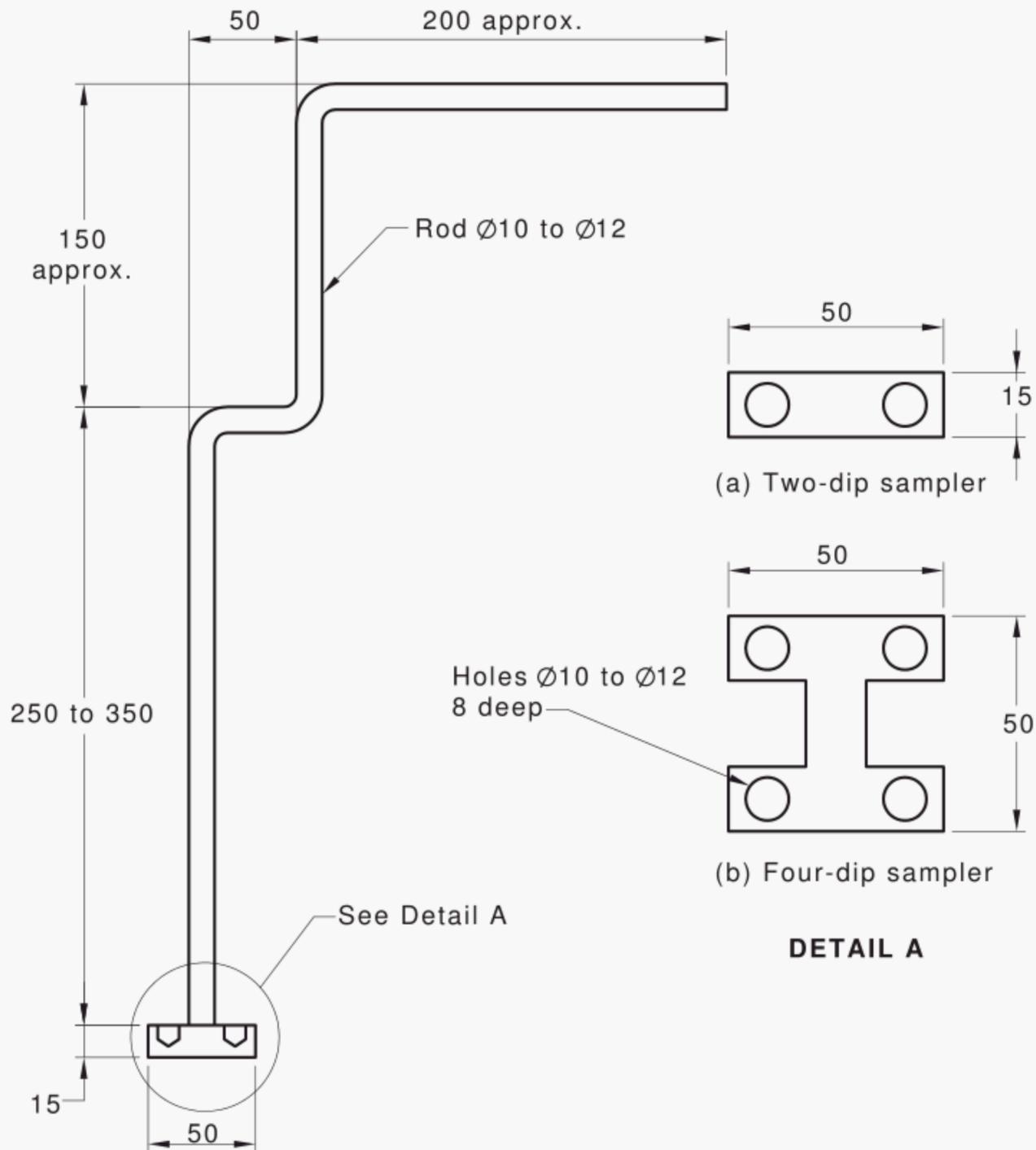
The recommended procedure for dip sampling is as follows:

- (a) Stir the molten bullion that is to be sampled for 1 min with a carbon rod.
- (b) Immediately plunge the sampler into the melt, and withdraw it.
NOTE: The dipping iron will provide either two or four samples and the ladle one sample.
- (c) When using a graphite ladle, either keep the exposed surface of the sample under a reducing flame until it has solidified, or granulate the sample by pouring it into water.
- (d) Remove the samples.
- (e) Inspect the surface of each sample for inclusions.
- (f) Where the samples have been produced by a dipping iron, remove any oxide coating by abrading with a wire brush.
- (g) If inclusions are present, try to remove them. Reject any sample from which they cannot be removed.

A5 SAMPLING BY CLIPPING

The recommended procedure for sampling ingots by clipping is as follows:

- (a) Remove any surface oxide from the ingot using a wire brush.
- (b) Using a guillotine or chisel, cut samples from opposite ends of the ingot.
NOTE: To prevent contamination of samples, the cutting implement should be cleaned before each sample is collected.



DIMENSIONS IN MILLIMETRES

FIGURE A1 TYPICAL DIP SAMPLER

A6 SAMPLING BY SAWING

The recommended procedure for sampling ingots by sawing is as follows:

- (a) Remove any surface oxide from the ingot using a wire brush.
- (b) Using a metal-cutting saw blade in a horizontal attitude, make a cut of 3 mm deep, one quarter of the distance from one end of the ingot.
NOTE: To prevent contamination of samples, the saw should be cleaned before each sample is collected.
- (c) Discard the sawings (i.e. set them aside with other scrap gold).
- (d) Extend the cut to a depth equal to two-thirds of the depth of the ingot, collect the sawings onto a clean sheet of glazed paper and mix them.
- (e) Repeat Steps (a) to (d) on the reverse surface of the ingot, but cut the sample from the opposite end of the ingot to that used in Step (b) and collect the sawings onto a separate sheet of glazed paper.
- (f) Keep the two samples for independent assay.

A7 SAMPLING BY DRILLING

The recommended procedure for sampling ingots by drilling is as follows:

- (a) Remove any surface oxide from the ingot using a wire brush.
- (b) Using a drill bit (approximately 6 mm diameter), drill two holes through the upper surface, each to a depth of 3 mm and at a distance one-quarter of the length of the ingot, and inward from two diagonally opposite corners.
- (c) Discard the drillings (i.e. set them aside with other scrap gold).
- (d) Redrill each hole to a depth equal to two-thirds of the depth of the ingot, empty the drillings onto a clean sheet of glazed paper and mix them.

NOTES:

- 1 To prevent contamination of the samples, the drill bit should be cleaned before use and before taking each set of two samples.
 - 2 Minimal pressure should be applied to the drill so as to produce thin drilling chips that can be cut into smaller pieces without further treatment.
- (e) Repeat Steps (a) to (d) on the reverse side of the ingot, but use the corners on the opposite diagonal to that used in Step (b), and empty the drillings onto a separate sheet of glazed paper.
 - (f) Keep the two samples for independent assay.

NOTES

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