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METHODS FOR THE ANALYSIS OF COPPER SULPHIDE CONCENTRATES
Part 2—DETERMINATION OF GOLD (FIRE ASSAY METHOD)

STANDARDS ASSOCIATION OF AUSTRALIA
Incorporated by Royal Charter

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The following interests are represented on Committee MN/5:

Australasian Institute of Mining and Metallurgy

Australian Lead Development Association

Australian Mineral Development Laboratories

Australian Mining Industry Council

CSIRO, Institute of Energy and Earth Resources

The Royal Australian Chemical Institute

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AUSTRALIAN STANDARD

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Part 2
DETERMINATION OF GOLD (FIRE ASSAY METHOD)

AS 2917.2—1986

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PREFACE

This standard was prepared by the Association's Committee on Copper, Lead and Zinc Ores and Concentrates under the direction of the Minerals Standards Board as part of its program of standardizing methods for the determination of elements of commercial interest in such materials.

Fire assay collection remains an important method for the determination of precious metals in non-ferrous ores and concentrates.

It was considered important that a standard method for determination of gold, with data on precision, be prepared. Apart from their commercial importance such methods can also be used as a basis for comparison with inferential techniques of atomic absorption and plasma emission spectroscopy.

Test work has shown that the traditional method of incorporating a gravimetric silver determination by difference into the determination of gold and silver by fire assay is unreliable because of the variability of cupellation losses and absorption of base metals in the precious metal bead.

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Australian Standard

METHODS FOR THE ANALYSIS OF COPPER SULPHIDE CONCENTRATES

PART 2—DETERMINATION OF GOLD (FIRE ASSAY METHOD)

1 SCOPE. This standard sets out a fire assay method for the determination of gold content in copper sulphide concentrates.

2 APPLICATION. The method is applicable to the determination of gold in copper concentrates containing 15 percent to 60 percent copper, mainly in the form of chalcopyrite and related sulphide minerals.

The method is applicable to gold contents from 0.5 *g/t* to 300 *g/t*.

3 REFERENCED DOCUMENTS. The following standards are referred to in this standard:

AS 1152 Test Sieves

AS 2134 Code of Practice for the Chemical Analysis of Materials by Flame Atomic Absorption Spectroscopy

AS 2162 Code of Practice for the Use of Volumetric Glassware

AS 2816 Copper, Lead and Zinc Sulphide Concentrates—Determination of Hygroscopic Moisture in the Analysis Sample

AS 2850 Chemical Analysis—Interlaboratory Test Programs—Guide to the Planning and Conduct—For Determining the Precision of Analytical Method(s)

AS 2862 Copper, Lead and Zinc Sulphide Concentrates—Sampling
Part 1—Sampling from Moving Streams*
Part 2—Sampling from Stationary Situations*
Part 3—Preparation of Samples

4 PRINCIPLE. Fire assaying for determination of gold comprises a series of steps to separate firstly, the precious metals from most of the associated base metals, followed by separation of the gold from silver and other metals preconcentrated into a precious metal alloy.

The stages which comprise the determination are as follows:

(a) Crucible fusion of the samples mixed with a litharge-based flux which, under reducing conditions, collects precious metals in a metallic lead button.

(b) Cupellation, an oxidising fusion in which base metals present in the lead button are substantially separated from precious metals. Cupellation produces a bead largely comprising a silver-gold alloy with small quantities of other metals.

(c) Parting of precious metal alloy with nitric acid in an environment free of chloride to prevent gold losses due to dissolution.

(d) Retreatment of all residues to maximize recovery of gold. Silver is used as a collector for residual gold followed by flame atomic absorption spectrometric determination of the gold.

5 REAGENTS.

5.1 General. All reagents shall be of a recognized analytical reagent grade. Distilled or deionized water shall be used throughout.

5.2 Reagents.

5.2.1 *Sodium carbonate, anhydrous.*

5.2.2 *Litharge.* Assay reagent grade with gold content sufficiently low to conform to the total blank constraint of Appendix B.

5.2.3 *Silica.* Precipitated grade.

5.2.4 *Potassium nitrate or sodium nitrate.*
NOTE: If sodium nitrate is used the masses specified for potassium nitrate will have to be modified.

5.2.5 *Flour.*

5.2.6 *Borax.* Fused anhydrous sodium tetraborate (borax glass powder).

5.2.7 *Nitric acid* (ϱ_{20} 1420 kg/m^3). Chloride content less than 0.5 μg/mL.

5.2.8 *Dilute nitric acid* (170 mL/L). To 830 mL of water carefully add, with stirring, 170 mL of nitric acid (5.2.7). Cool and dilute to 1 L.

5.2.9 *Dilute nitric acid* (700 mL/L). To 300 mL of water carefully add, with stirring, 700 mL of nitric acid (5.2.7).

5.2.10 *Silver.* 99.999 percent minimum purity silver.

5.2.11 *Hydrochloric acid.* (ϱ_{20} 1160 kg/m^3 to 1180 kg/m^3).

5.2.12 *Aqua regia solution.* To 50 mL of nitric acid (5.2.7) carefully add 150 mL of hydrochloric acid (ϱ_{20} 1160 kg/m^3). Prepare freshly before use.

6 APPARATUS.

6.1 Conventional fire assaying equipment.

6.1.1 *Assay crucible furnace.* An assay crucible furnace with a maximum required operating temperature of 1200 °C.

6.1.2 *Muffle furnace.* A muffle furnace with a maximum required operating temperature of 1100 °C. Temperature indication, automatic temperature control and controlled air flow are preferable.

* In course of preparation.