Australian Standard®

METHODS FOR THE ANALYSIS OF IRON AND STEEL

Part 19—DETERMINATION OF NICKEL CONTENT—SPECTROPHOTOMETRIC METHOD

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The following interests are represented on Committee CH/10:

Aluminium Development Council
Australasian Institute of Mining and Metallurgy
Australian Lead Development Association
Australian Mineral Development Laboratories
Australian Tin Information Centre
Australian Zinc Development Association
Bureau of Steel Manufacturers of Australia
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Copper Technical Data Centre
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Electricity Supply Association of Australia
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BHP Steel International, Rod and Bar Division
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PREFACE

This Standard was prepared by the Association's Committee on the Analysis of Metals under the direction of the Chemical Standards Board as a further part of AS 1050. The method is technically identical with ISO 4939 (1984) and supersedes AS K1, *Methods for the sampling and analysis of iron and steel*, Part 19—1963, *Determination of nickel present in small amounts in carbon and low alloy steels (photometric method).*

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METHODS FOR THE ANALYSIS OF IRON AND STEEL

PART 19: DETERMINATION OF NICKEL CONTENT—SPECTROPHOTOMETRIC METHOD

1 SCOPE. This Standard sets out a dimethylglyoxime spectrophotometric method for the determination of the nickel content of iron and steel.

2 APPLICATION. The method is applicable to the determination of nickel contents between 0.10 percent and 4 percent (*m*/*m*) in iron and steel provided that the following elements are not present in amounts greater than that specified:

cobalt	10 percent
copper	1 percent
manganese	2 percent

3 REFERENCED DOCUMENTS. The following Standards are referred to in this Standard:

AS	
1213	Iron and steel—Methods of sampling
2164	One-mark volumetric flasks
2166	One-mark pipettes
2167	Straight pipettes
CK 19	Code of recommended practice for the chemical analysis of materials by ultraviolet visible spectrophotometry
ISO	
5725	Precision of test methods—Determination of repeatability and reproducibility by interlaboratory tests

4 PRINCIPLE. Dissolution of a test portion in hydrochloric, nitric and perchloric acids. Formation of a coloured complex of nickel(III) with dimethylglyoxime in ammoniacal solution containing iodine and potassium iodide.

Spectrophotometric measurement at a wavelength of about 535 nm.

5 REAGENTS.

5.1 General. Use only reagents of recognized analytical grade and distilled water or water of equivalent purity.

5.2 High purity iron. Iron containing >99.5 percent Fe and <500 μg/g Ni.

5.3 Solutions.

5.3.1 *Hydrochloric acid—nitric acid mixture.* Mix 2 volumes of hydrochloric acid (ϱ_{20} 1.18 g/mL), 1 volume of nitric acid (ϱ_{20} 1.42 g/mL) and 2 volumes of water.

5.3.2 *Perchloric acid* (ϱ_{20} *1.54 g/mL*).

NOTE: Perchloric acid (ϱ_{20} 1.67 g/mL) may also be used. 100 mL of perchloric acid (ϱ_{20} 1.54 g/mL) is equivalent to 79 mL of perchloric acid (ϱ_{20} 1.67 g/mL).

5.3.3 *Ammonium citrate solution.* Dissolve 250 g of citric acid monohydrate ($C_6H_8O_7.H_2O$) in 250 mL of ammonia solution (ϱ_{20} 0.91 g/mL), cool, dilute to 1 L and mix.

5.3.4 *Iodine solution.* Dissolve 25 g of potassium iodide and 12.7 g of iodine in the minimum volume of water. Dilute to 1 L and mix.

5.3.5 *Dimethylglyoxime solution.* Dissolve 1 g of dimethylglyoxime in 500 mL of ammonia solution (ϱ_{20} 0.91 g/mL), dilute to 1 L and mix.

5.3.6 *Ammonia solution* (*1 + 1*). Add 100 mL of ammonia solution (ϱ_{20} 0.91 g/mL) to 100 mL of water.

5.3.7 *Standard nickel solution* (*1mL ≡ 0.5 mg Ni*). Weigh, to the nearest 0.0001 g, 0.5000 g of high purity nickel and dissolve in 20 mL of nitric acid (ϱ_{20} 1.42 g/mL diluted 2 + 3). Boil to remove fumes and cool. Transfer quantitatively to a 1 L one-mark volumetric flask, dilute to the mark and mix.

6 APPARATUS.

6.1 Volumetric glassware. Grade A volumetric glassware complying with AS 2164, AS 2166 and AS 2167 shall be used.

6.2 Spectrophotometer. A spectrophotometer capable of measuring absorbance with a spectral slit width of less than 5 nm at 535 nm shall be used. The wavelength measurements shall be accurate to ±2 nm or less. In the absorbance range 0.05 to 0.85 absorbance measurements shall be repeatable to ±0.003 absorbance or better. Spectrophotometric practice shall be carried out in accordance with AS CK19.

6.3 Spectrophotometric cells. Plain type cells shall be used.

7 SAMPLING. Samples shall be taken by the procedures specified in AS 1213.

NOTE: Difficulties associated with segregation in some steels may be minimized by taking proportional masses of both coarse and fine particles present in the test sample.

8 PROCEDURE.

8.1 Number of determinations. This method is written for a single determination only and may be used in that form. Where replicate determinations are required, procedures for acceptance of the results so obtained are given in Clause 11.

8.2 Blank test. Carry out a blank test in parallel with the determination, substituting 0.5 g of high purity iron (5.2) for the sample and using the same procedure as described in Clause 8.4.