AS 1050.33—1988
Reconfirmed 2016

Australian Standard®

METHODS FOR THE ANALYSIS OF IRON AND STEEL

Part 33—DETERMINATION OF NICKEL CONTENT—FLAME ATOMIC ABSORPTION SPECTROMETRIC METHOD

This Australian Standard was prepared by Committee CH/10, Analysis of Metals. It was approved on behalf of the Council of the Standards Association of Australia on 21 January 1988 and published on 5 April 1988.

The following interests are represented on Committee CH/10:

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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
Confederation of Australian Industry
Copper Producers Association of Australia
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Additional interests participating in preparation of Standard:

BHP Steel International, Rod and Bar Division
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RECONFIRMATION

OF
AS 1050.33—1988
Methods for the analysis of iron and steel
Part 33: Determination of nickel content—Flame atomic absorption spectrometric method

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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.

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International Copper Association Australia
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NOTES

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Part 33—DETERMINATION OF NICKEL CONTENT—FLAME ATOMIC ABSORPTION SPECTROMETRIC METHOD

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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 the AS 1050 series of Standards. The method is technically identical to ISO 4940 (1985).

ASKI-11 (1963), *Nickel in iron and steel and permanent magnet alloys—Titrimetric method* will be withdrawn on the publication of this Standard.

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Australian Standard

METHODS FOR THE ANALYSIS OF IRON AND STEEL

PART 33: DETERMINATION OF NICKEL CONTENT— FLAME ATOMIC ABSORPTION SPECTROMETRIC METHOD

1 SCOPE. This Standard specifies a method for the determination of nickel in steel and cast iron by means of flame atomic absorption spectrometry.

2 APPLICATION. The method is applicable to nickel contents in the range 0.002 to 0.5 percent *(m/m)*.

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

AS
1213 Iron and steel — Methods of sampling

2134 Recommended practice for chemical analysis by atomic absorption spectrometry Part 1: Flame atomic absorption spectrometry (AS 2134.1)

2164 One-mark volumetric flasks

ISO
5725 Precision of test methods — Determination of repeatability and reproducibility by inter-laboratory tests

4 PRINCIPLE. The sample is dissolved in a hydrochloric acid-nitric acid mixture followed by evaporation to fuming with perchloric acid. The nickel content of the solution is determined by flame atomic absorption spectrometry.

5 REAGENTS.

5.1 General requirements. During the analysis only reagents of recognized analytical reagent grade, and distilled water or water of equivalent purity, shall be used.

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 three parts by volume of hydrochloric acid (ϱ_{20} 1.18 g/mL), one part by volume of nitric acid (ϱ_{20} 1.42 g/mL), and two parts by volume of water. Prepare this mixture immediately before use.

5.3.2 *Nitric acid-perchloric acid mixture.* Mix 100 mL of nitric acid (ϱ_{20} 1.42 g/mL) with 800 mL of perchloric acid (ϱ_{20} 1.54 g/mL). Dilute to 1 litre and mix.

NOTE: Perchloric acid (ϱ_{25} 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_{25} 1.67 g/mL).

5.4 Standard solutions.

5.4.1 *Stock nickel solution (1 mL ≡ 1 mg Ni).* Weigh, to the nearest 0.0001 g, 0.5000 g of high purity nickel (≥ 99.9 percent (*m/m*)), and dissolve in 25 mL of nitric acid (ϱ_{20} 1.42 g/mL diluted 1 + 1). Boil to remove nitrous fumes. Cool and transfer the solution to a 500 mL one-mark volumetric flask, dilute to the mark and mix.

5.4.2 *Standard nickel solution (1 mL ≡ 40 μg Ni).* Transfer 10.0 mL of stock solution (5.4.1) into a 250 mL one-mark volumetric flask, dilute to the mark and mix. Prepare this standard solution immediately before use.

6 APPARATUS.

6.1 Glassware. Grade A volumetric glassware complying with AS 2164 shall be used throughout. All glassware shall first be washed in hydrochloric acid (ϱ_{20} 1.18 g/mL, diluted 1 + 1), then in water. The quantity of nickel present in the beakers and flasks can be checked by measuring the absorption of the distilled water introduced into the glassware after the acid wash.

6.2 Atomic absorption spectrometer.

6.2.1 *General.* The instrument and practice shall comply with AS 2134.1.

Ensure that the spray system and drainage system are washed free from perchloric acid after use.

6.2.2 *Operating parameters.* Suitable operating parameters for this test are as follows:

Wavelength ..... 352.5 nm
Flame ..... A slightly lean air/acetylene flame (5 cm or 10 cm slot burner).

NOTE: With some instruments it is not possible to obtain sufficient sensitivity at the wavelength of 352.5 nm for low concentrations of nickel near the bottom end of the application range and in such cases the alternative wavelength of 232.0 nm should be used.

At the wavelength of 352.5 nm, the signal-to-noise ratio is higher than at a wavelength of 232.0 nm. In general, use of the 352.5 nm line will give better reproducibility.

6.2.3 *Performance criteria.* For all instruments, including those with automatic standardization and direct concentration readout, the following performance criteria shall be met prior to the test:

(a) *Minimum sensitivity.* The absorbance of the calibration solution of highest nickel content (see Clause 9.1) shall not be less than 0.4.

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