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International Standard

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Steel and iron — Determination of chromium content — Potentiometric or visual titration method

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Aciers et fontes — Dosage du chrome — Méthode par titrage potentiométrique ou visuel

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4937 was prepared by Technical Committee ISO/TC 17, Steel.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Steel and iron — Determination of chromium content — Potentiometric or visual titration method

1 Scope and field of application

This International Standard specifies a method for the determination of chromium in steel and iron by potentiometric or visual titration.

The method is applicable to chromium contents between 0,25 and 35 % (m/m).

If vanadium is present, the visual titration is applicable only to test portions containing less than 3 mg of vanadium.

2 References

ISO 377, Wrought steel — Selection and preparation of samples and test pieces.

ISO 385/1, Laboratory glassware — Burettes — Part 1: General requirements.

ISO 648, Laboratory glassware - One-mark pipettes.

ISO 1042, Laboratory glassware — One-mark volumetric flasks.

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

3 Principle

Dissolution of a test portion with appropriate acids.

Oxidation of chromium in an acid medium to chromium(VI) by ammonium peroxydisulfate in the presence of silver sulfate. Reduction of manganese(VII) by hydrochloric acid.

Reduction of chromium(VI) by ammonium iron(II) sulfate standard solution.

In the case of potentiometric detection, determination of the equivalence point by measurement of the potential variation when the ammonium iron(II) sulfate standard solution is being added.

In the case of visual detection, titration of the excess ammonium iron(II) sulfate by potassium permanganate standard solution which also acts as the indicator.

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity, free from oxidizing or reducing activity.

- 4.1 Urea.
- **4.2** Perchloric acid, ϱ approximately 1,67 g/ml.
- **4.3** Hydrofluoric acid, ϱ approximately 1,15 g/ml.
- **4.4** Orthophosphoric acid, ϱ approximately 1,70 g/ml.
- **4.5** Nitric acid, ϱ approximately 1,40 g/ml.
- **4.6** Hydrochloric acid, ϱ approximately 1,19 g/ml, diluted 1 + 1.
- **4.7** Hydrochloric acid, ϱ approximately 1,19 g/ml, diluted 1 + 10.
- **4.8** Sulfuric acid, ϱ approximately 1,84 g/ml, diluted 1 + 1.
- **4.9** Sulfuric acid, ϱ approximately 1,84 g/ml, diluted 1 + 5.
- **4.10** Sulfuric acid, ϱ approximately 1,84 g/ml, diluted 1 + 19.
- 4.11 Silver sulfate, 5 g/l solution.
- **4.12** Ammonium peroxydisulfate $[(NH_4)_2S_2O_8]$, 500 g/l solution.

Prepare this solution immediately before use.

- **4.13** Manganese sulfate [MnSO₄·H₂O], 4 g/l solution.
- 4.14 Manganese sulfate [MnSO₄·H₂O], 100 g/l solution.
- 4.15 Potassium permanganate, 5 g/l solution.
- 4.16 Sodium nitrite, 3 g/l solution.

Prepare this solution immediately before use.

4.17 Sulfamic acid (NH₂SO₃H), 100 g/l solution.

This solution remains stable for one week only.