Steel — Determination of calcium content by flame atomic absorption spectrometry —

Part 2:
Determination of total calcium content

1 Scope

This part of ISO 10697 specifies a flame atomic absorption spectrometric method for the determination of the total calcium content in steel.

The method is applicable to calcium contents between 0,000 5 % (*m/m*) and 0,005 % (*m/m*).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 10697. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 10697 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 377-2:1989, *Selection and preparation of samples and test pieces of wrought steels — Part 2: Samples for the determination of the chemical composition.*

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements.*

ISO 648:1977, *Laboratory glassware — One-mark pipettes.*

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

3 Principle

Dissolution of a test portion in hydrochloric and nitric acids.

Fusion of the acid-insoluble residue with sodium carbonate.

Addition of potassium chloride/lanthanum nitrate solution as a spectrochemical buffer.

Spraying of the solution into a dinitrogen monoxide-acetylene flame.

Spectrometric measurement of the atomic absorption of the 422,7 nm spectral line emitted by a calcium hollow cathode lamp.

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade having very low calcium contents and only grade 2 water as specified in ISO 3696.

4.1 Pure iron, containing less than 0,000 1 % (*m/m*) calcium.

4.2 Sodium carbonate, anhydrous, super pure.

4.3 Suitable solvent, for example acetone or dichloromethane.

4.4 Hydrochloric acid, ρ about 1,19 g/ml.

4.5 Hydrochloric acid, ρ about 1,19 g/ml, diluted 1 + 1.

4.6 Hydrochloric acid, ρ about 1,19 g/ml, diluted 1 + 2.

4.7 Nitric acid, ρ about 1,40 g/ml.

4.8 Perchloric acid, ρ about 1,54 g/ml.

4.9 Hydrofluoric acid, ρ about 1,15 g/ml.

4.10 Potassium chloride/lanthanum nitrate, solution.

Dissolve 95,34 g of potassium chloride (KCl) and 62,35 g of lanthanum nitrate hexahydrate [$La(NO_3)_3.6H_2O$] in water, dilute to 1 000 ml and mix.

4.11 Calcium, standard solutions.

4.11.1 Stock solution, corresponding to 100 mg of Ca per litre.

Dry several grams of calcium carbonate [purity $\geqslant$ 99,5 % (*m/m*)] in an air oven et 100 °C for at least 1 h and cool to room temperature in a desiccator. Weigh 0,249 7 g of the dried product into a 400 ml beaker, cover with a watch-glass and add 5 ml of hydrochloric acid (4.5) to dissolve. Cool and transfer the solution to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this stock solution contains 100 µg of Ca.

4.11.2 Standard solution, corresponding to 10 mg of Ca per litre.

Transfer 10,0 ml of the stock solution (4.11.1) into a 100 ml one-mark volumetric flask and add 5 ml of hydrochloric acid (ρ about 1,19 g/ml, diluted 1 + 10). Dilute to the mark with water and mix.

Prepare this standard solution immediately before use.

1 ml of this standard solution contains 10 µg of Ca.

5 Apparatus

All volumetric glassware shall be class A, in accordance with ISO 385-1, ISO 648 or ISO 1042, as appropriate.

Ordinary laboratory apparatus, and

5.1 Filter media, 0,22 µm pore size, 47 mm diameter cellulose-acetate filter.

5.2 Suction apparatus, i.e. a clean empty flask placed between the suction device and filtering flask to prevent any back suction and contamination.

5.3 Platinum crucible, of capacity 25 ml.

5.4 Muffle furnace, capable of being maintained at between 500 °C and 800 °C.

5.5 Atomic absorption spectrometer, equipped with a calcium hollow cathode lamp and supplied with dinitrogen monoxide and acetylene sufficiently pure to give a steady clear fuel-lean flame, free from water and oil, and free from calcium.

The atomic absorption spectrometer used will be satisfactory if, after optimization according to 7.3.6, the limit of detection and characteristic concentration are in reasonable agreement with the values given by the manufacturer and if it meets the precision criteria given in 5.5.1 to 5.5.3.

It is also desirable that the instrument should conform to the additional performance requirements given in 5.5.4.

5.5.1 Minimum precision (see A.1)

Calculate the standard deviation of 10 measurements of the absorbance of the most concentrated calibration solution. The standard deviation shall not exceed 1,5 % of the mean absorbance.

Calculate the standard deviation of 10 measurements of the absorbance of the least concentrated calibration solution (excluding the zero member). The standard deviation shall not exceed 0,5 % of the mean absorbance of the most concentrated calibration solution.

5.5.2 Limit of detection (see A.2)

This is defined as twice the standard deviation of 10 measurements of the absorbance of a solution containing the appropriate element at a concentration level selected to give an absorbance just above that of the zero member.