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Aluminium and aluminium alloys — Determination of iron — Orthophenanthroline photometric method

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up has the right to be represented on that Committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, International Standard ISO 793 replaces ISO Recommendation R 793-1968 drawn up by Technical Committee ISO/TC 79, *Light materials and their alloys*.

The Member Bodies of the following countries approved the Recommendation :

- Argentina
- Austria
- Belgium
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- Chile
- Czechoslovakia
- Egypt. Arab Rep. of
- France
- Germany
- Hungary
- Korea, Rep. of
- India
- Israel
- Italy
- Japan
- Netherlands
- Norway
- Poland
- South Africa, Rep. of
- Spain
- Sweden
- Switzerland
- Turkey
- United Kingdom
- U.S.A.
- U.S.S.R.
- Yugoslavia

The Member Body of the following country expressed disapproval of the Recommendation on technical grounds :

Ireland

Printed in Switzerland

Aluminium and aluminium alloys — Determination of iron — Orthophenanthroline photometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a photometric method for the determination of iron in aluminium and aluminium alloys.

The method is applicable to the determination of iron content between 0,05 and 2,50 %.

The method does not apply completely to the following special cases for which it should be modified as described in Annex A or Annex B :

a) unalloyed aluminium, aluminium-silicon alloys and any other aluminium alloy that is not easily attacked with hydrochloric acid (see Annex A);

b) alloys containing copper (copper content over 5 %), zinc (zinc content over 4 %), nickel (nickel content over 2 %), or alloys with a proportional combination of these elements more than 5 % total (see Annex B).

2 PRINCIPLE

Attack with hydrochloric acid. Reduction of iron(III) to iron(II) by hydroxylammonium chloride.

Formation in buffered solution between pH 3,5 and pH 4,5 of the orange-red coloured complex, bivalent iron-orthophenanthroline.

Photometric measurement at a wavelength of about 510 nm.[1]

3 REAGENTS

During the analysis use only distilled water or water of equivalent purity.

3.1 Hydrochloric acid, ρ 1,1 g/ml, approximately 6 N solution.

Take 500 ml of hydrochloric acid (ρ 1,19 g/ml), approximately 12 N, and make up the volume to 1 000 ml with water.

3.2 Hydrochloric acid, ρ 1,16 g/ml, approximately 10 N solution.

Dilute 820 ml of hydrochloric acid (ρ 1,19 g/ml) with water and, after cooling to 20 °C, make up the volume to 1 000 ml. Check the relative density and, if necessary, adjust the strength of the solution.

3.3 Sodium hydroxide, 5 N solution.

Dissolve 200 g of sodium hydroxide (NaOH) in a nickel basin with about 400 ml of water. After cooling, transfer the solution to a 1 000 ml volumetric flask, rinsing the basin, and make up the volume to 1 000 ml. (Store in polythene containers.)

3.4 Mixed reagent

Mix the following solutions in the ratio 1 : 1 : 3 by volume :

3.4.1 Hydroxylammonium chloride solution

Dissolve 10 g of hydroxylammonium chloride ($NH_2OH.HCl$) in a little water and make up the volume to 1 000 ml.

3.4.2 Orthophenanthroline solution

Dissolve 2,5 g of orthophenanthroline monohydrate ($C_{12}H_8N_2.H_2O$), or 3 g of orthophenanthroline hydrochloride monohydrate ($C_{12}H_8N_2.HCl.H_2O$), in water, warm slightly in order to complete solution, cool and make up the volume to 1 000 ml.

3.4.3 Buffer solution

Dissolve 272 g of sodium acetate ($CH_3COONa.3H_2O$) in about 500 ml of water, filter, add 240 ml of glacial acetic acid (CH_3COOH), (ρ 1,05 g/ml), approximately 17,4 N, then make up the volume to 1 000 ml with water.

The mixed reagent should be stored in a dark coloured glass container; it should not be used after storage for more than 4 weeks.

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[1] Copper, which may interfere if present in appreciable amounts, is largely eliminated in the attack. Of the elements normally present in aluminium and its alloys, some do not interfere, while others form colourless soluble complexes with orthophenanthroline, which do not absorb at the wavelength at which the photometric measurement is performed.

3.5 **Iron,** 0,2 g/l standard solution (1 ml contains 0,2 mg of iron).

3.5.1 Dissolve 1,404 5 g of ferrous ammonium sulphate $[(NH_4)_2Fe(SO_4)_2.6H_2O]$ in a little water and add 20 ml of the hydrochloric acid (3.1). Transfer the solution to a 1 000 ml volumetric flask and make up the volume to 1 000 ml with water.[1]

Alternatively

3.5.2 Dissolve, by heating in a 100 ml beaker covered with a watch-glass, 0,286 0 g of pure ferric oxide (Fe_2O_3), previously calcined at 600 °C, in 30 ml of the hydrochloric acid (3.1). After cooling, transfer the solution to a 1 000 ml volumetric flask and make up the volume to 1 000 ml with water.

3.6 **Iron,** 0,01 g/l standard solution (1 ml contains 0,01 mg of iron).

Transfer 50,0 ml of the iron standard solution (3.5) to a 1 000 ml volumetric flask, then make up the volume to 1 000 ml with water. Prepare the solution just before use.

4 APPARATUS

4.1 Ordinary laboratory equipment

4.2 Spectrophotometer, or

4.3 Photoelectric absorptiometer.

5 SAMPLING

5.1 Laboratory sample[2]

5.2 Test sample

Chips not more than 1 mm thick shall be obtained from the laboratory sample by drilling or milling. By means of a magnet separate any accidental iron contamination.

6 PROCEDURE

6.1 Test portion

Mass of test portion : 0,5 ± 0,001 g.

6.2 Preparation of the calibration curve

Introduce into a series of eight 100 ml volumetric flasks 0 (compensating solution) – 2,50 – 5,0 – 10,0 – 15,0 – 20,0 – 30,0 – and 40,0 ml respectively of the iron standard solution (3.6) containing 0,01 mg of iron per millilitre.

Dilute each solution to about 50 ml, add 25 ml of the mixed reagent (3.4), make up the volume to 100 ml and mix. After 30 min, carry out the photometric measurements at the maximum of the absorption curve (wavelength about 510 nm), after having adjusted the instrument to zero absorbance against the compensating solution.

Plot a graph having, for example, the amount of iron contained in 100 ml as abscissae, against the corresponding values of absorbance as ordinates.

6.3 Blank test

Carry out a blank test in parallel with the determination, following the same procedure and using the same quantities of all reagents.

6.4 Determination

6.4.1 *Attack of the test portion and preparation of the main solution*

In a beaker of suitable capacity (for example 250 ml) covered with a watch-glass, attack the test portion with 15 ml of the hydrochloric acid (3.1). Boil for a few minutes.

Dilute to about 25 ml. Boil for 5 min. Dilute to about 50 ml. Filter, wash and collect the filtrate in a 250 ml or 500 ml volumetric flask, depending on the assumed iron content (see table); cool, make up to volume and mix.

TABLE

Assumed iron content	**Volume of main solution**	**Volume of aliquot to be taken**
%	ml	ml
0,05 to 0,2	250,0	50,0
0,2 to 0,8	500,0	50,0
0,8 to 1,5	500,0	25,0
1,5 to 2	500,0	20,0
2 to 2,50	500,0	10,0

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[1] If the effective strength of the ferrous ammonium sulphate is not known, measure it by titration with potassium dichromate and correct accordingly the mass to be taken for the iron standard solution (3.5).

[2] The sampling of aluminium and aluminium alloys will form the subject of a future International Standard.

6.4.2 *Colour reaction*

Take the aliquot of main solution shown in the table (making allowance for the characteristics of the instrument available for the photometric measurement), transfer it to a 100 ml volumetric flask, dilute if necessary to about 50 ml, add 25 ml of the mixed reagent (3.4), make up the volume to 100 ml and mix.

6.4.3 *Photometric measurements*

After 30 min, carry out the photometric measurements under the same conditions as were used for plotting the calibration curve, after having adjusted the instrument to zero absorbance against distilled water or the blank test solution. (See note, clause 8.)

7 EXPRESSION OF RESULTS

By means of the calibration graph, determine the masses of iron, in milligrams, corresponding to the values of the photometric measurements of the solution of the test portion and of the blank test solution.

Calculate the iron content, as a percentage by mass, by the formula

$$\text{Fe } \% \ (m/m) = \frac{m_1 - m_2 \times R}{10\, m_0}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass of iron, in milligrams, found in the aliquot taken from the main solution of the test portion;

m_2 is the mass of iron, in milligrams, found in an aliquot of the same volume taken from the blank test solution;

R is the ratio of the volume of the main solution of the test portion to the volume of the aliquot taken.

8 NOTE

If the reagents used for the analysis are very pure, the aliquot taken from the blank test solution, after adding the mixed reagent (3.4) and after making up the volume to 100 ml, has very little colour. In this case it is recommended that this solution should be used for setting the instrument to zero absorbance.

Then calculate the iron content, as a percentage by mass, by the formula

$$\text{Fe } \% \ (m/m) = \frac{m_1 \times R}{10\, m_0}$$

where m_1, R and m_0 have the same meaning as in the formula in clause 7.

9 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard, or regarded as optional.

ANNEX A

SPECIAL CASE OF UNALLOYED ALUMINIUM, ALUMINIUM-SILICON ALLOYS AND ANY OTHER ALUMINIUM ALLOY THAT IS NOT EASILY ATTACKED WITH HYDROCHLORIC ACID

MODIFICATIONS TO THE GENERAL METHOD

Replace 6.4.1 by the following :

Introduce the test portion into a platinum vessel (beaker or dish) of about 100 ml capacity, carefully add 20 ml of the sodium hydroxide solution (3.3) and cover the vessel with a watch-glass.

Warm slightly and, after the test portion has dissolved, rinse the watch-glass and walls of the vessel with a little water; then boil the solution gently for 2 to 3 min.

In the case of alloys having a high silicon content (silicon content greater than 4 %), keep the vessel for about 20 min — or longer if necessary — at a temperature a little below the boiling point of the solution, in order to complete the oxidation of the silicon, taking care to add water to compensate for losses by evaporation. Then boil for 2 to 3 min. Rinse the watch-glass and the vessel with water, dilute the solution to about 25 ml, add 20 ml of the hydrochloric acid (3.2), mix and boil gently for 10 min. Allow to cool, filter any residue through a medium texture filter, then wash the residue and the filter with water, collecting the filtrate and the washings in a 250 or 500 ml volumetric flask according to the iron content (see table). After cooling to room temperature, make up to volume and mix.

ANNEX B

SPECIAL CASE OF ALLOYS CONTAINING COPPER (CONTENT OVER 5 %), ZINC (CONTENT OVER 4 %), OR NICKEL (CONTENT OVER 2 %), OR ALLOYS WITH A PROPORTIONAL COMBINATION OF THESE ELEMENTS MORE THAN 5 % TOTAL

B.1 PRINCIPLE

Copper, zinc and nickel interfere if present in appreciable quantities in the solution. These interferences, within the pH limits adopted, are eliminated by adding an excess of the orthophenanthroline solution (3.4.2) which results in the formation of colourless soluble complexes which do not absorb at the wavelength at which iron is determined (a large part of the copper is eliminated during the attack).

B.2 MODIFICATIONS TO THE GENERAL METHOD

Replace 6.4.2 by the following :

Take the aliquot of the main solution shown in the table (making allowance for the characteristics of the instrument available for the photometric measurement), transfer it to a 100 ml volumetric flask, dilute if necessary to about 50 ml, add 25 ml of the mixed reagent (3.4) and 10 ml of the orthophenanthroline (3.4.2), make up the volume to 100 ml and mix.

Add an excess of 10 ml of the orthophenanthroline solution (3.4.2) when plotting the calibration graph.

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