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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 793

CHEMICAL ANALYSIS OF ALUMINIUM AND ITS ALLOYS

PHOTOMETRIC DETERMINATION OF IRON

(Orthophenanthroline method
applicable to iron content between 0.05 and 2.50 %)

1st EDITION

July 1968

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Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The ISO Recommendation R 793, *Chemical analysis of aluminium and its alloys - Photometric determination of iron (Orthophenanthroline method applicable to iron content between 0.05 and 2.50 %)*, was drawn up by Technical Committee ISO/TC 79, *Light metals and their alloys*, the Secretariat of which is held by the Association Française de Normalisation (AFNOR).

Work on this question by the Technical Committee began in 1956 and led, in 1963, to the adoption of a Draft ISO Recommendation.

In June 1966, this Draft ISO Recommendation (No. 966) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

One Member Body opposed the approval of the Draft :

Ireland

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in July 1968, to accept it as an ISO RECOMMENDATION.

CHEMICAL ANALYSIS OF ALUMINIUM AND ITS ALLOYS

PHOTOMETRIC DETERMINATION OF IRON

(Orthophenanthroline method)

applicable to iron content between 0.05 and 2.50 %)

1. SCOPE

- 1.1 This ISO Recommendation describes a photometric method for the determination of iron in aluminium and its alloys.
- 1.2 The method is applicable to the determination of iron content between 0.05 and 2.50 %.
- 1.3 The method does not apply completely to the following special cases for which it should be modified as described in Annex A and 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

- 2.1 Attack with hydrochloric acid.
- 2.2 Reduction of ferric iron to bivalent iron by hydroxylammonium chloride.
- 2.3 Formation in buffered solution between pH 3.5 and pH 4.5 of the orange-red coloured complex, bivalent iron-orthophenanthroline.
- 2.4 Photometric measurement at a wavelength of about 510 nm.*

3. REAGENTS

- 3.1 *Hydrochloric acid*, $d = 1.1$ (approximately 6 N).
Take 500 ml of hydrochloric acid, $d = 1.19$ (approximately 12 N), and make up the volume to 1000 ml with water.
- 3.2 *Sodium hydroxide solution* (5 N)
Dissolve 200 g of sodium hydroxide (NaOH) in a nickel basin with about 400 ml of water. After cooling, transfer the solution to a 1000 ml volumetric flask, rinsing the basin, and make up the volume to 1000 ml. (Store in polythene containers.)
- 3.3 *Hydrochloric acid*, $d = 1.16$ (approximately 10 N)
Dilute 820 ml of hydrochloric acid, $d = 1.19$, with water and, after cooling to 20 °C, make up the volume to 1000 ml. Check the relative density and, if necessary, adjust the strength of the solution.

* 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.

3.4 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 ($\text{NH}_2\text{OH} \cdot \text{HCl}$) in a little water and make up the volume to 1000 ml.

3.4.2 Orthophenanthroline solution

Dissolve 2.5 g of orthophenanthroline monohydrate ($\text{C}_{12}\text{H}_8\text{N}_2 \cdot \text{H}_2\text{O}$), or 3 g of orthophenanthroline hydrochloride monohydrate ($\text{C}_{12}\text{H}_8\text{N}_2 \cdot \text{HCl} \cdot \text{H}_2\text{O}$), in water, warm slightly in order to complete solution, cool and make up the volume to 1000 ml.

3.4.3 Buffer solution

Dissolve 272 g of sodium acetate ($\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$) in about 500 ml of water, filter, add 240 ml of glacial acetic acid (CH_3COOH), $d = 1.05$ (approximately 17.4 N), then make up the volume to 1000 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 four weeks.

3.5 Standard iron solution, 0.2 g per litre (1 ml contains 0.2 mg of iron).

3.5.1 Dissolve 1.4045 g of ferrous ammonium sulphate [$(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$] in a little water and add 20 ml of hydrochloric acid (3.1). Transfer the solution to a 1000 ml volumetric flask and make up the volume to 1000 ml with water.*

Alternatively

3.5.2 Dissolve, by heating, in a 100 ml beaker covered with a watch-glass, 0.2860 g of pure ferric oxide (Fe_2O_3), previously calcined at 600°C , in 30 ml of hydrochloric acid (3.1). After cooling, transfer the solution to a 1000 ml volumetric flask and make up the volume to 1000 ml with water.

3.6 Standard iron solution, 0.01 g per litre (1 ml contains 0.01 mg of iron).

Transfer 50.0 ml of the standard iron solution (3.5) to a 1000 ml volumetric flask, then make up the volume to 1000 ml with water. Prepare the solution just before use.

4. APPARATUS

4.1 Ordinary laboratory equipment

All volumetric apparatus should comply with national standards.

4.2 Electrophotometer or spectrophotometer (wavelength about 510 nm).

5. SAMPLING

5.1 Laboratory sample

See the appropriate national standard on sampling.

5.2 Test sample

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

* 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 standard iron solution (3.5).

6. PROCEDURE

6.1 Test portion

Mass of test portion : 0.5 ± 0.001 g.

6.2 Plotting of the calibration graph

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 standard iron 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 minutes, carry out the photometric measurements at the maximum of the absorption graph (wavelength about 510 nm), having set the instrument to zero optical density against the compensating solution.

Draw a graph plotting, for example, the amount of iron contained in 100 ml as abscissae against the corresponding values of optical density 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 (e.g. 250 ml) covered with a watch-glass, attack the test portion with 15 ml of hydrochloric acid (3.1). Boil for a few minutes.

Dilute to about 25 ml. Boil for 5 minutes. 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, below); 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

6.4.2 Colour reaction. Take the aliquot of main solution shown in the Table above (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 measurement.* After 30 minutes, carry out the photometric measurement under the same conditions as were used for plotting the calibration graph, having set the instrument to zero optical density against distilled water or the blank test solution. (See Note, section 8.)

7. EXPRESSION OF RESULTS

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

The percentage, by mass, of iron is calculated by the following formula :

$$\text{Fe } \% \text{ (m/m)} = \frac{(A - B) \times D}{10 E}$$

where

- A* is the mass of iron, expressed in milligrammes, found in the aliquot taken from the main solution of the test portion,
- B* is the mass of iron, expressed in milligrammes, found in an aliquot of the same volume taken from the blank test solution,
- D* is the ratio of the volume of the main solution of the test portion to the volume of the aliquot taken,
- E* is the mass, expressed in grammes, of the test portion.

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 optical density. The percentage, by mass, of iron is then calculated from the following formula :

$$\text{Fe } \% \text{ (m/m)} = \frac{A \times D}{10 E}$$

where *A*, *D* and *E* have the same meaning as in the formula in section 7.

9. TEST REPORT

Report the following information :

- (a) the reference to the method used;
- (b) the results and the method used to express them;
- (c) any unusual features noted during the determination;
- (d) any operation not laid down in this ISO Recommendation or regarded as optional.