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Aluminium and aluminium alloys — Determination of silicon — Gravimetric 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 797 replaces ISO Recommendation R 797-1968 drawn up by Technical Committee ISO/TC 79, *Light metals and their alloys*.

The Member Bodies of the following countries approved the Recommendation :

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

No Member Body expressed disapproval of the Recommendation.

Aluminium and aluminium alloys – Determination of silicon – Gravimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a gravimetric method for the determination of silicon in aluminium and aluminium alloys.

The method is applicable to the determination of silicon content greater than or equal to 0,30 %.

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) aluminium-silicon alloys (silicon content greater than 3 %) and aluminium-magnesium alloys (see Annex A);
- b) aluminium alloys containing tin or antimony (see Annex B).

2 PRINCIPLE

Attack with sodium hydroxide. Acidification with perchloric acid; dehydration of the silica. Filtration, drying, calcination and weighing of the silica.

Volatilization of the silica, by means of hydrofluoric acid, and weighing of the residue. Determination of the silica by difference in mass.

3 REAGENTS

3.1 Sodium hydroxide pellets. (Store in a plastics container.)

3.2 Hydrobromic acid, ρ approximately 1,49 g/ml, 48 % (V/V) solution.

3.3 Hydrochloric acid, ρ 1,01 g/ml, approximately 0,62 N solution.

Take 5 ml of hydrochloric acid (ρ 1,19 g/ml), approximately 12 N, and make the volume to 100 ml with water.

3.4 Hydrofluoric acid, ρ approximately 1,14 g/ml, 40 % (m/m) solution.

3.5 Nitric acid, ρ 1,40 g/ml, approximately 15 N solution.

3.6 Perchloric acid, ρ 1,67 g/ml, approximately 11,7 N solution.¹⁾

3.7 Perchloric acid, ρ 1,22 g/ml, approximately 4 N solution.

Take 35 ml of perchloric acid (3.6) and make up the volume to 100 ml with water.

3.8 Sulphuric acid, ρ 1,48 g/ml, approximately 17,5 N solution.

Carefully add 50 ml of sulphuric acid (ρ 1,84 g/ml), approximately 35,6 N, to water, cool and make up the volume to 100 ml.

3.9 Bromine water, saturated solution.

3.10 Hydrogen peroxide, 6 % (V/V) (about 20 volumes).

Take 17 ml of hydrogen peroxide, 36 % (V/V), ρ 1,12 g/ml, and make up the volume to 100 ml with water.

3.11 Sodium hydroxide, ρ 1,05 g/ml, approximately 1,25 N solution.

Dissolve 50 g of sodium hydroxide (NaOH) in a little water and, after cooling, make up the volume to 1 000 ml. (Store in a plastics container.)

1) Perchloric acid (ρ 1,54 g/ml) approximately 9 N, may also be used. 1 000 ml of perchloric acid (ρ 1,67 g/ml), is equivalent to 1 270 ml of perchloric acid (ρ 1,54 g/ml).

4 APPARATUS

Ordinary laboratory equipment.

5 SAMPLING

5.1 Laboratory sample¹⁾

5.2 Test sample

Chips not more than 1 mm thick obtained by drilling or milling.

6 PROCEDURE

6.1 Test portion

Weigh the test portion with an accuracy of $\pm 0,001$ g, in accordance with the quantities shown in table 1.

For the hypereutectic aluminium-silicon alloys, the masses of coarse and fine particles in the test portion should be taken in the same proportion as in the test sample.

6.2 Blank test

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

6.3 Determination

6.3.1 Attack of the test portion

In accordance with the assumed silicon content, take the size of test portion shown in table 1 and use the corresponding quantities of the reagents.

NOTE — It is possible to use the normal single-stage attack method as described in the procedure, even for the analysis of aluminium-silicon alloys (silicon content greater than 3 %) and aluminium-magnesium alloys.

Place the test portion in a nickel or silver basin of suitable capacity (for example about 250 ml and of 120 to 150 mm diameter) fitted with a nickel or silver lid, add the appropriate quantity of the sodium hydroxide (3.1) (see table 1), then carefully add, in small portions, the corresponding volume of water.

When the attack is complete, rinse the cover and the wall of the basin with the smallest possible quantity of hot water, place the covered basin on a hot-plate (or sand bath) and evaporate the solution to a syrupy consistency, avoiding spattering.

After cooling, add, drop by drop, 5 to 6 ml of the hydrogen peroxide (3.10) and again evaporate to a syrupy consistency. If necessary, repeat the treatment by hydrogen peroxide.

TABLE 1

Assumed silicon content	Mass of test portion	Mass of sodium hydroxide (3.1)	+	Volume of water	Volume of nitric acid (3.5)	Volume of perchloric acid (3.6)	+	Volume of water
%	g	g + ml			ml	ml + ml		
0,30 to 1	5	15 + 30			5	110 + 60		
over 1 to 3	2	8 + 15			5	60 + 30		
over 3 to 7	1	6 + 10*			5	45 + 20		
over 7	0,5 to 1	10 + 20*			5	60 + 30		

* Alternatively, for the two-stage attack method, see Annex A and the Note in 6.3.1.

1) The sampling of aluminium and aluminium alloys will form the subject of a future International Standard.

6.3.2 Dehydration of the silica

Add 100 ml of hot water, washing the wall of the basin, boil to dissolve the salts, cool and transfer the contents of the basin into a vessel (for example a porcelain basin) of suitable capacity containing the nitric acid (3.5), the perchloric acid (3.6) and water in the quantities shown in table 1 for the corresponding size of test portion.

Carefully wash the basin and the cover with hot water, then with a small quantity of the perchloric acid (3.7), about 10 ml in all, removing any particles adhering to the basin and to the cover (using, for example, a glass rod fitted with a ribbed thimble of para rubber). Rinse lastly with hot water and add the washings to the main solution.

Stir with a glass rod and, if the solution has a brown colour owing to the presence of manganese dioxide, add a few drops of the hydrogen peroxide (3.10). Cover the basin with a watch-glass and evaporate to the release of copious white fumes of perchloric acid until the solution begins to crystallize (duration of white perchloric acid fumes about 15 to 20 min). Allow to cool, then take up in hot water and make up the volume of the solution to about 200 ml for a test portion of 0,5 to 1 g, 400 ml for a test portion of 2 g, and about 600 ml for a test portion of 5 g.

Stir carefully with a glass rod, heat in order to bring the salts into complete solution, and dissolve any manganese dioxide that may have separated by adding a few drops of the hydrogen peroxide (3.10).

6.3.3 Filtration and washing

Bring the particles of silica into suspension by stirring with a glass rod, then filter through a medium texture filter paper of known ash content (filter A), collecting the filtrate and the successive washings in a beaker. Wash the filter with a hot solution of the hydrochloric acid (3.3) and after 5 to 6 washings rinse with boiling water until the salts are completely eliminated.

NOTE — Washing should be complete because any perchloric acid retained by the silica might lead to spattering during calcination.

6.3.4 Recovery of silica from the filtrate

Transfer the filtrate and the washings into the vessel previously used, evaporate to the release of copious white fumes of perchloric acid and continue the evaporation and the fuming for 15 to 20 min.

Cool, take up in hot water as indicated in 6.3.2, filter on a second medium texture filter paper (filter B) and wash as indicated in 6.3.3. Recover any silica adhering to the wall of the basin.

6.3.5 Calcination and volatilization of the silica

Place the two filters A and B and their contents together in a platinum crucible (see note 1); calcine moderately at 500 to 600 °C until complete combustion of the filters, then

calcine in a muffle furnace at 1 100 to 1 150 °C for 1 h. Cool in a desiccator containing either activated alumina or silica gel and weigh; then calcine again at the same temperature to constant mass. To the residue add 1 to 2 ml of the sulphuric acid (3.8) (see note 2) and 3 to 5 ml of the hydrofluoric acid (3.4), evaporate to dryness and then calcine in the muffle furnace at 1 000 °C to constant mass.

If necessary, repeat the treatment by hydrofluoric acid.

NOTES

1 The use of a tared platinum crucible is advised, in order to permit a check of both the quantity of residue and any variation of the mass of the crucible during calcination.

2 The excess of sulphuric acid shown is necessary to avoid even partial volatilization of any titanium and zirconium present.

7 EXPRESSION OF RESULTS

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

$$\text{Si \% (m/m)} = \frac{[(m_1 - m_2) - (m_3 - m_4)] \times 0,467\ 5 \times 100}{m_0}$$

where

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

m_1 is the mass, in grams, of the crucible and its contents, before volatilization of the silica in the test portion;

m_2 is the mass, in grams, of the crucible and any residue, after volatilization of the silica in the test portion;

m_3 is the mass, in grams, of the crucible and its contents, before volatilization of the silica in the blank test;

m_4 is the mass, in grams, of the crucible and any residue, after volatilization of the silica in the blank test;

0,467 5 is the conversion factor, silica to silicon.

8 TEST REPORT

The test report shall include the following particulars :

- the reference of the method used;
- the results and the method of expression used;
- any unusual features noted during the determination;
- any operation not included in this International Standard, or regarded as optional.

ANNEX A

SPECIAL CASE OF ALUMINIUM-SILICON ALLOYS (SILICON CONTENT GREATER THAN 3 %) AND ALUMINIUM-MAGNESIUM ALLOYS

A.1 PRINCIPLE

The attack of the test portion is carried out in two stages, firstly using the quantity of sodium hydroxide, in dilute solution, necessary to dissolve the aluminium, and subsequently adding the quantity of sodium hydroxide pellets necessary to complete the attack of the test portion and the formation of sodium silicate.

A.2 MODIFICATION TO THE GENERAL METHOD

Replace 6.3.1 by the following :

In accordance with the assumed silicon content, take the size of test portion shown in table 2 and use the corresponding quantities of the reagents.

Place the test portion in a nickel or silver basin of suitable capacity (for example about 250 ml and of 120 to 150 mm diameter) fitted with a nickel or silver lid, add the appropriate volume of the sodium hydroxide solution (3.11) and, as soon as the primary reaction is complete, add the corresponding quantity of the sodium hydroxide pellets (3.1). When the attack is complete, rinse the cover and wall of the basin with the smallest possible amount of hot water, place the covered basin on a hot-plate (or sand bath), heat moderately, then carefully evaporate to a syrupy consistency, avoiding spattering.

After cooling, add, drop by drop, 5 to 6 ml of the hydrogen peroxide (3.10) and again evaporate to a syrupy consistency. If necessary, repeat the treatment by hydrogen peroxide.

TABLE 2*

Assumed silicon content	Mass of test portion	Volume of sodium hydroxide solution (3.11)	Mass of sodium hydroxide pellets (3.1)	Total sodium hydroxide used	Volume of nitric acid (3.5)	Volume of perchloric acid (3.6)	+ Volume of water
%	g	ml	g	g	ml	ml + ml	
3 to 7	1	30	4,5	6	5	45 + 20	
over 7	0,5 to 1	30	8,5	10	5	60 + 30	

* Throughout clause 6, read "table 2" in place of "table 1".