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ISO RECOMMENDATION

R 797

CHEMICAL ANALYSIS OF ALUMINIUM AND ITS ALLOYS

GRAVIMETRIC DETERMINATION OF SILICON

(Silicon content greater than or equal to 0.30 %)

1st EDITION

July 1968

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BRIEF HISTORY

The ISO Recommendation R 797, *Chemical analysis of aluminium and its alloys—Gravimetric determination of silicon (Silicon content greater than or equal to 0.30 %)*, 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. 970) 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	Spain
Austria	India	Sweden
Belgium	Ireland	Switzerland
Brazil	Israel	Turkey
Bulgaria	Italy	U.A.R.
Canada	Japan	United Kingdom
Chile	Netherlands	U.S.A.
Czechoslovakia	Norway	U.S.S.R.
France	Poland	Yugoslavia
Germany	South Africa,	
Hungary	Rep. of	

No Member Body opposed the approval of the Draft.

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.

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CHEMICAL ANALYSIS OF ALUMINIUM AND ITS ALLOYS

GRAVIMETRIC DETERMINATION OF SILICON

(Silicon content greater than or equal to 0.30 %)

1. SCOPE

- 1.1 This ISO Recommendation describes a gravimetric method for the determination of silicon in aluminium and its alloys.
- 1.2 The method is applicable to silicon content greater than or equal to 0.30 %.
- 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) 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

- 2.1 Attack with sodium hydroxide.
- 2.2 Acidification with perchloric acid; dehydration of the silica.
- 2.3 Filtration, drying, calcination and weighing of the silica.
- 2.4 Volatilization of the silica, by means of hydrofluoric acid, and weighing of the residue.
- 2.5 Determination of the silica by difference in mass.

3. REAGENTS

- 3.1 *Sodium hydroxide pellets.* (Store in a plastics container).
- 3.2 *Hydrobromic acid, 48 %, $d =$ approximately 1.49.*
- 3.3 *Hydrochloric acid, $d = 1.01$ (approximately 0.62 N).*
Take 5 ml of hydrochloric acid, $d = 1.19$ (approximately 12 N), and make up the volume to 100 ml with water.
- 3.4 *Hydrofluoric acid, 40 %, $d =$ approximately 1.15.*
- 3.5 *Nitric acid, $d = 1.40$ (approximately 15 N).*
- 3.6 *Perchloric acid, $d = 1.67$ (approximately 11.7 N). **
- 3.7 *Perchloric acid, $d = 1.22$ (approximately 4 N).*
Take 35 ml of perchloric acid, $d = 1.67$, and make up the volume to 100 ml with water.
- 3.8 *Sulphuric acid, $d = 1.48$ (approximately 17.5 N).*
Carefully add 50 ml of sulphuric acid, $d = 1.84$ (approximately 35.6 N), to water, cool and make up the volume to 100 ml.

* Perchloric acid, $d = 1.54$ (approximately 9 N), may also be used. (1000 ml of perchloric acid, $d = 1.67$, is equivalent to 1270 ml of perchloric acid, $d = 1.54$).

3.9 *Bromine water*, saturated solution.

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

Take 17 ml of hydrogen peroxide, 36 %, $d = 1.12$, and make up the volume to 100 ml with water.

3.11 *Sodium hydroxide solution*, $d = 1.05$ (approximately 1.25 N).

Dissolve 50 g of sodium hydroxide (NaOH) in a little water and, after cooling, make up the volume to 1000 ml. (Store in a plastics container).

4. APPARATUS

Ordinary laboratory equipment

All volumetric apparatus should comply with national standards.

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.

6. PROCEDURE

6.1 Test portion

Weigh the test portion with an accuracy of ± 0.001 g, in accordance with the quantities shown in Table 1, below.

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.

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 following Note.

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 (e.g. about 250 ml and of 120 to 150 mm diameter) fitted with a nickel or silver lid, add the appropriate quantity of 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 dropwise 5 to 6 ml of hydrogen peroxide (3.10) and again evaporate to a syrupy consistency. If necessary, repeat the treatment by hydrogen peroxide (3.10).

- 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 (e.g. a porcelain basin) of suitable capacity containing nitric acid (3.5), 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 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 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 minutes). 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 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 hydrochloric acid (3.3) and after 5 or 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 minutes.

Cool, take up in hot water as indicated in clause 6.3.2, filter on a second medium texture filter paper (filter B) and wash as indicated in clause 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, below); calcine moderately at 500 to 600 °C until complete combustion of the filters, then calcine in a muffle furnace at 1100 to 1150 °C for one hour. 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 sulphuric acid (3.8) (see Note 2, below) and 3 to 5 ml of hydrofluoric acid (3.4), evaporate to dryness and then calcine in the muffle furnace at 1000 °C to constant mass. If necessary, repeat the treatment by hydrofluoric acid (3.4).

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

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

$$\text{Si } \% \text{ (m/m)} = \frac{[(M_2 - M_1) - (m_2 - m_1)] \times 0.4675 \times 100}{E}$$

where

- M_2 is the mass, expressed in grammes, of the crucible and its contents, before volatilization of the silica in the test portion;
- M_1 is the mass, expressed in grammes, of the crucible and any residue, after volatilization of the silica in the test portion;
- m_2 is the mass, expressed in grammes, of the crucible and its contents, before volatilization of the silica in the blank test;
- m_1 is the mass, expressed in grammes, of the crucible and any residue, after volatilization of the silica in the blank test;
- E is the mass, expressed in grammes, of the test portion;
- 0.4675 is the conversion factor, silica to silicon.

8. 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.

ANNEX A

Special case of aluminium-silicon alloys (silicon content greater than 3 %) and aluminium-magnesium alloys

A.1 PRINCIPLE OF THE SPECIAL METHOD USED

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 clause 6.3.1 by the following clause :

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

TABLE A.1*

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 section 6, read "Table A.1" in place of "Table 1".

Place the test portion in a nickel or silver basin of suitable capacity (e.g. about 250 ml and of 120 to 150 mm diameter) fitted with a nickel or silver lid, add the appropriate volume of sodium hydroxide solution (3.11) and, as soon as the primary reaction is complete, add the corresponding quantity of 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 dropwise 5 to 6 ml of hydrogen peroxide (3.10) and again evaporate to a syrupy consistency. If necessary, repeat the treatment by hydrogen peroxide (3.10).