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Aluminium and aluminium alloys — Determination of silicon — Gravimetric method

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FOREWORD

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The Member Bodies of the following countries approved the Recommendation :

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No Member Body expressed disapproval of the Recommendation.

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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.)

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[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 sample[1]

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 ± 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.

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[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 :

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 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".

ANNEX B

SPECIAL CASE OF ALUMINIUM ALLOYS CONTAINING TIN OR ANTIMONY

B.1 PRINCIPLE

Tin and antimony, which interfere in the perchloric acid medium because of hydrolysis, are volatilized from the perchloric acid solution as bromides.

B.2 MODIFICATIONS TO THE GENERAL METHOD

B.2.1 *Replace the beginning of 6.3.1 by the following :*

According to the assumed content of silicon, tin and/or antimony, take the size of test portion shown in table 3, and use the corresponding quantities of the reagents.

Place the test portion in a nickel or silver basin ... [*From this point, 6.3.1 remains unchanged.*]

B.2.2 *Replace 6.3.2 by the following :*

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 perchloric acid (3.6) and water in the quantities shown in table 3 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).

Boil for a few minutes. Then, in a fume cupboard, add cautiously, in small portions, the quantities of the bromine water (3.9) and the hydrobromic acid (3.2) shown in table 3 in relation to the size of test portion and its assumed content of tin and/or antimony. Concentrate the solution by raising the temperature gradually until copious white perchloric acid fumes are emitted. Continue with the evaporation and emission of fumes for 5 min; then cover the basin with a watch-glass and continue heating for about 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).

TABLE 3*

Assumed silicon content	Assumed tin and/or antimony content	Mass of test portion	Mass of sodium hydroxide (3.1) + Volume of water	Volume of perchloric acid (3.6) + Volume of water	Volume of hydrobromic acid (3.2)	Volume of bromine water (3.9)
%	%	g	g + ml	ml + ml	ml	ml
0,30 to 1	< 6	5	15 + 30	160 + 60	30	20
	6 to 20	5	15 + 30	160 + 60	70	20
over 1 to 3	< 6	2	8 + 15	80 + 30	15	10
	6 to 20	2	8 + 15	80 + 30	30	10
over 3 to 7	$\leqslant 6$	1	6 + 10**	80 + 30	10	10
over 7	$\leqslant 1$	0,5 to 1	10 + 20**	80 + 30	10	10

* Throughout clause 6, read "table 3" in place of "table 1".

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