International Standard

ISO **3856/3**

Paints and varnishes — Determination of "soluble" metal content —
Part 3 : Determination of barium content — Flame atomic emission spectroscopic method

Peintures et vernis — Détermination de la teneur en métaux «solubles» — Partie 3 : Détermination de la teneur en baryum — Méthode par spectroscopie d'émission atomique dans la flamme

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Foreword

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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.

International Standard ISO 3856/3 was developed by Technical Committee ISO/TC 35, *Paints and varnishes*, and was circulated to the member bodies in April 1978.

It has been approved by the member bodies of the following countries :

Australia	Iran	Romania
Austria	Ireland	South Africa, Rep. of
Brazil	Israel	Sweden
Bulgaria	Italy	Switzerland
Canada	Kenya	Turkey
Egypt, Arab Rep. of	Korea, Rep. of	United Kingdom
France	Mexico	USSR
Germany, F. R.	Norway	
India	Poland	

No member body expressed disapproval of the document.

Paints and varnishes — Determination of "soluble" metal content —

Part 3 : Determination of barium content — Flame atomic emission spectroscopic method

0 Introduction

This document is a part of ISO 3856, *Paints and varnishes — Determination of "soluble" metal content*.

1 Scope and field of application

This part of ISO 3856 specifies a flame atomic emission spectroscopic (AES) method for the determination of the barium content of the test solutions prepared according to ISO 6713 or other suitable International Standards.[1]

The method is applicable to paints having "soluble" metal contents in the range of about 0,05 to 5 % (m/m).

Other methods can be used by agreement between the interested parties but, in case of dispute, this method should be used.

2 References

ISO/R 385, *Burettes*.

ISO 648, *Laboratory glassware — One-mark pipettes*.

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*.

ISO 6713, *Paints and varnishes — Preparation of acid extracts from liquid paints*.[1]

3 Principle

Measurement of the radiation emitted by barium in a dinitrogen monoxide/acetylene flame at a wavelength of 553,5 nm. The ionization of barium atoms in the flame is suppressed by addition of potassium chloride.

4 Reagents and materials

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Potassium chloride, 50 g/l solution.

4.2 Hydrochloric acid, 0,07 mol/l solution.

Use the identical hydrochloric acid solution as used for the preparation of the test solutions.

4.3 Dinitrogen monoxide gas, in a steel cylinder.

4.4 Acetylene, in a steel cylinder.

4.5 Barium, standard solution corresponding to 1 g of Ba per litre.

Either

a) transfer the contents of an ampoule of standard barium solution containing exactly 1 g of Ba into a 1 000 ml one-mark volumetric flask, dilute to the mark with the hydrochloric acid solution (4.2), and mix well;

or

b) weigh, to the nearest 1 mg, 1,779 g of barium chloride dihydrate ($BaCl_2.2H_2O$), dissolve in the hydrochloric acid solution (4.2) in a 1 000 ml one-mark volumetric flask, dilute to the mark with the same hydrochloric acid solution, and mix well.

1 ml of this standard solution contains 1 mg of Ba.

4.6 Barium, standard solution corresponding to 20 mg of Ba per litre.

Pipette 20 ml of the standard barium solution (4.5) into a 1 000 ml one-mark volumetric flask, dilute to the mark with the hydrochloric acid solution (4.2) and mix well.

Prepare this solution on the day of use.

1 ml of this standard solution contains 20 μg of Ba.

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[1] The preparation of acid extracts from dried films and powder coatings will form the subject of future International Standards.

5 Apparatus

For this test, the laboratory glassware shall be barium-free.

Ordinary laboratory apparatus and

5.1 Flame atomic emission spectrometer, fitted with a burner fed with dinitrogen monoxide and acetylene.

5.2 Pipettes, of a suitable volume, complying with the requirements of ISO 648.

5.3 Burettes, of capacity 10 and 50 ml, complying with the requirements of ISO/R 385.

5.4 One-mark volumetric flasks, of capacity 50 ml, complying with the requirements of ISO 1042.

If available :

5.5 Recording apparatus.

A compensating recorder is recommended.

6 Procedure

6.1 Preparation of the calibration graph

6.1.1 Preparation of the standard matching solutions

Introduce from a burette (5.3) into a series of six 50 ml one-mark volumetric flasks (5.4), the volumes of the standard barium solution (4.6) shown in the following table, add 5 ml of potassium chloride solution (4.1), dilute each to the mark with the hydrochloric acid solution (4.2) and mix well.

Prepare these solutions on the day of use.

Standard matching solution No.	Standard barium solution (4.6)	Corresponding concentration of Ba in the standard matching solution
	ml	μg/ml
0*	0	0
1	2	0,8
2	5	2,0
3	10	4,0
4	20	8,0
5	40	16,0

* Blank test on reagents for calibration graph.

6.1.2 Spectroscopic measurements

Measure the emission of the standard matching solutions in the spectrometer (5.1), using the operating conditions specified by the manufacturer of the instrument.

In order to determine and correct for the background emission due to calcium, carry out a measurement at a suitable point close to the barium line or record the emission over a wavelength range from 553,0 to 554,0 nm.

6.1.3 Plotting of the graph

Plot a graph having the masses, in micrograms, of Ba contained in 1 ml of the standard matching solutions as abscissae and the corresponding values of the emission, corrected for background emission, as ordinates.

6.2 Test solutions

6.2.1 Pigments and extenders

Pipette a suitable volume (volume V_3) of the solution obtained by the procedure specified in sub-clause 7.2.1 of ISO 6713 into a 50 ml one-mark volumetric flask (5.4) so that the barium concentration of the test solution will be within the calibration range. Add 5 ml of the potassium chloride solution (4.1), dilute to the mark with the hydrochloric acid solution (4.2) and mix well.

6.2.2 Liquid portion of the paint

Pipette a suitable volume (volume V_4) of the solution obtained by the procedure specified in clause 8 of ISO 6713 into a 50 ml one-mark volumetric flask (5.4) so that the barium concentration of the test solution will be within the calibration range. Add 5 ml of the potassium chloride solution (4.1), dilute to the mark with the hydrochloric acid solution (4.2) and mix well.

6.2.3 Other test solutions

Pipette a suitable volume of the solution obtained by the specified or agreed procedure into a 50 ml one-mark volumetric flask (5.4) so that the barium concentration of the test solution will be within the calibration range. Add 5 ml of the potassium chloride solution (4.1), dilute to the mark with the hydrochloric acid solution (4.2) and mix well.

6.3 Determination

Measure the emission of each test solution (6.2) three times in the apparatus after having adjusted it as specified in 6.1.2. Measure first the emission of the hydrochloric acid solution (4.2), then that of the test solution and afterwards that of the hydrochloric acid solution again. Finally, re-determine the emissions of the standard matching solutions (6.1.1) in order to verify that the adjustment of the apparatus has not changed. If the emission of a test solution is higher than that of the standard matching solution with the highest barium concentration, dilute the test solution appropriately with a known volume of the hydrochloric acid solution (4.2).

7 Expression of results

7.1 Calculations

7.1.1 Pigments and extenders

The mass of "soluble barium" in the hydrochloric acid extract, obtained by the method specified in sub-clause 7.2.1 of ISO 6713, is given by the equation

$$m_0 = \frac{a_1 - a_0}{10^6} \times \frac{V_1}{V_3} \times 50 \times F_1$$

$$= (a_1 - a_0) \times \frac{V_1}{V_3} \times F_1 \times 5 \times 10^{-5}$$

where

a_0 is the barium concentration, in micrograms per millilitre, of the blank test solution prepared by the method specified in sub-clause 7.3 of ISO 6713;

a_1 is the barium concentration, in micrograms per millilitre, of the test solution obtained from the calibration graph;

F_1 is the dilution factor referred to in 6.3;

m_0 is the mass, in grams, of "soluble" barium in the hydrochloric acid extract;

V_1 is the volume, in millilitres, of the hydrochloric acid solution plus ethanol used for the extraction specified in sub-clause 7.2.1 of ISO 6713 (assumed to be 77 ml);

V_3 is the volume, in millilitres, of the aliquot portion of the hydrochloric acid plus ethanol extract taken for the test (6.2.1).

The "soluble" barium content of the pigment and extender portion of the paint is given by the equation

$$c_{Ba_1} = m_0 \times \frac{10^2}{m_1} \times \frac{P}{10^2}$$

$$= \frac{m_0 \times P}{m_1}$$

where

c_{Ba_1} is the "soluble" barium content, of the pigment and extender portion of the paint, expressed as a percentage by mass of the paint;

m_1 is the mass, in grams, of the test portion taken to prepare the solution specified in sub-clause 7.2.1 of ISO 6713;

P is the pigment and extender content of the paint, expressed as a percentage by mass, obtained by the appropriate method specified in clause 6 of ISO 6713.

7.1.2 Liquid portion of the paint

The mass of barium in the solution (extract), obtained by the method specified in clause 8 of ISO 6713, is given by the equation

$$m_2 = \frac{b_1 - b_0}{10^6} \times \frac{V_2}{V_4} \times 50 \times F_2$$

$$= (b_1 - b_0) \times \frac{V_2}{V_4} \times F_2 \times 5 \times 10^{-5}$$

where

b_0 is the barium concentration, in micrograms per millilitre, of the blank test solution prepared by the method specified in sub-clause 6.5 of ISO 6713;

b_1 is the barium concentration, in micrograms per millilitre, of the test solution obtained from the calibration graph;

F_2 is the dilution factor referred to in 6.3;

m_2 is the mass, in grams, of barium in the solution obtained according to clause 8 of ISO 6713;

V_2 is the volume, in millilitres, of the solution (100 ml) obtained by the method specified in clause 8 of ISO 6713 ($= 100$ ml);

V_4 is the volume, in millilitres, of the aliquot portion of the solution taken for the test.

The barium content of the liquid portion of the paint is given by the equation

$$c_{Ba_2} = \frac{m_2}{m_3} \times 10^2$$

where

c_{Ba_2} is the barium content, of the liquid portion of the paint, expressed as a percentage by mass of the paint;

m_3 is the total mass, in grams, of paint comprising a "set" as specified in sub-clause 6.4 of ISO 6713.

7.1.3 Paint

The total "soluble" barium content of the paint is given by the sum of the results obtained according to 7.1.1 and 7.1.2, thus

$$c_{Ba_3} = c_{Ba_1} + c_{Ba_2}$$

where c_{Ba_3} is the total "soluble" barium content of the paint, expressed as a percentage by mass.

7.1.4 Other test solutions

If the test solutions were prepared by methods other than that given in ISO 6713 (see 6.2.3), it will be necessary to modify the

equations for the calculation of barium content given in 7.1.1 and 7.1.2.

7.2 Precision

No precision data are currently available.

8 Test report

The test report shall include at least the following information :

a) the type and identification of the product tested;

b) a reference to this International Standard (ISO 3856/3) or to a corresponding national standard;

c) the method for the separation of the solid portion of the product under test according to ISO 6713, clause 6 (method A, B or C);

d) the solvent or the solvent mixture used for the extraction;

e) any deviation, by agreement or otherwise, from the test procedure specified;

f) the results of the tests, each expressed as a percentage by mass of the product, i.e. the "soluble" barium content of the pigment and extender, the barium content of the liquid portion of the paint and the total "soluble" barium content of the paint;

g) the date of the test.

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