International Standard ISO 3856/3

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

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

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

Second edition — 1984-11-01

UDC 667.6 : 543.423 : 546.431 **Ref. No. ISO 3856/3-1984 (E)**

Descriptors: paints, varnishes, printing inks, pigments, chemical analysis, determination of content, soluble matter, barium, atomic emission spectrometry.

Price based on 4 pages

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 3856/3 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*.

ISO 3856/3 was first published in 1980. This second edition cancels and replaces the first edition, of which it constitutes a thorough revision.

Printed in Switzerland

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

0 Introduction

This International Standard 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 describes a flame atomic emission spectrometric (AES) method for the determination of the barium content of the test solutions prepared according to ISO 6713 or other suitable International Standards.

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

Other methods can be used by agreement between the interested parties but this method is the referee method in cases of dispute.

2 References

ISO 385/1, *Laboratory glassware — Burettes — Part 1: General requirements.* [1]

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

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

ISO 3696, *Water for laboratory use — Specifications.* [2]

ISO 6713, *Paints and varnishes — Preparation of acid extracts from paints in liquid or powder form.*

3 Principle

Aspiration of the test solution into a dinitrogen monoxide/acetylene flame. Measurement of the radiation emitted by barium 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 water of at least grade 3 purity according to ISO 3696.

4.1 Potassium chloride, 50 g/l solution.

4.2 Hydrochloric acid, $c(\mathrm{HCl}) = 0{,}07$ mol/l.

Use the hydrochloric acid, identical to that used for the preparation of the test solutions in accordance with ISO 6713. (See 6.2.)

4.3 Dinitrogen monoxide, commercial grade, in a steel cylinder.

4.4 Acetylene, commercial grade, in a steel cylinder.

---

[1] At present at the stage of draft. (Partial revision of ISO/R 385-1964.)

[2] At present at the stage of draft.

4.5 Barium, standard stock solution containing 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 (4.2) and mix well;

or

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

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

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

Prepare this solution on the day of use.

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

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

5 Apparatus

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

Ordinary laboratory apparatus and

5.1 Flame atomic emission spectrometer, suitable for measurements at a wavelength of 553,5 nm and fitted with a burner fed with dinitrogen monoxide and acetylene.

5.2 Pipettes, of a suitable volume (see 6.2), complying with the requirements of ISO 648.

5.3 Burettes, of capacity 10 and 50 ml, complying with the requirements of ISO 385/1.

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

Prepare these solutions on the day of use.

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

Standard matching solution No.	Volume of the 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
3	10	4
4	20	8
5	40	16

* Blank matching solution.

6.1.2 Spectrometric 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 constant background emission due to the presence of 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 Calibration 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 Pigment portion of the liquid paint and paint in powder form

Pipette a suitable volume (volume V_3) of each of the solutions obtained by the procedure described in sub-clause 8.2.3 of ISO 6713 into 50 ml one-mark volumetric flasks (5.4) so that the barium concentration of each 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 (4.2) and mix well.

6.2.2 Liquid portion of the paint

Pipette a suitable volume (volume V_4) of each of the solutions obtained by the procedure described in sub-clause 9.3 of ISO 6713 into 50 ml one-mark volumetric flasks (5.4) so that the barium concentration of each 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 (4.2) and mix well.

6.2.3 Other test solutions

Pipette a suitable volume of each of the solutions obtained by the specified or agreed procedure into 50 ml one-mark volumetric flasks (5.4) so that the barium concentration of each 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 (4.2) and mix well.

6.3 Determination

Measure first the emission of the hydrochloric acid (4.2) in the spectrometer (5.1) after having adjusted it as described in 6.1.2. Then measure the absorbance of each test solution (6.2) three times and, afterwards, that of the hydrochloric acid again. Finally, re-determine the emission of the standard matching solution No. 4 (6.1.1) in order to verify that the response 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 (dilution factor F) with a known volume of the hydrochloric acid (4.2).

7 Expression of results

7.1 Calculations

7.1.1 Pigment portion of the liquid paint

Calculate the mass of "soluble" barium in the hydrochloric acid extract, obtained by the method described in sub-clause 8.2.3 of ISO 6713, using 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 described in sub-clause 8.4 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 plus ethanol used for the extraction described in sub-clause 8.2.3 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).

Calculate the "soluble" barium content of the pigment portion of the paint, using the equation

$$c_{\mathrm{Ba}_1} = m_0 \times \frac{10^2}{m_1} \times \frac{P}{10^2} = \frac{m_0 \times P}{m_1}$$

where

c_{Ba_1} is the "soluble" barium content, of the pigment 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 described in sub-clause 8.2.3 of ISO 6713;

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

7.1.2 Liquid portion of the paint

Calculate the mass of barium in the solution (extract), obtained by the method described in sub-clause 9.3 of ISO 6713, using 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 described 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 liquid portion of the paint;

V_2 is the volume, in millilitres, of the solution obtained by the method described in sub-clause 9.3 of ISO 6713 (= 100 ml);

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

Calculate the barium content of the liquid portion of the paint, using the equation

$$c_{\mathrm{Ba}_2} = \frac{m_2}{m_3} \times 10^2$$

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 described in sub-clause 6.4 of ISO 6713.

7.1.3 Liquid paint

Calculate the total "soluble" barium content of the liquid paint as the sum of the results obtained according to 7.1.1 and 7.1.2, thus

$$c_{\mathrm{Ba}_3} = c_{\mathrm{Ba}_1} + c_{\mathrm{Ba}_2}$$

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

7.1.4 Paint in powder form

The total "soluble" barium content of the paint in powder form is obtained by appropriate modification of the calculations given in 7.1.1.

7.1.5 Other test solutions

If the test solutions were prepared by methods other than those 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 contain at least the following information:

a) the type and identification of the product tested;

b) a reference to this International Standard (ISO 3856/3);

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), where appropriate[1];

d) the solvent or the solvent mixture used for the extraction, where appropriate[1];

e) the results of the test, expressed as a percentage by mass of the product: either

- the "soluble" barium content of the pigment portion of the paint, the barium content of the liquid portion of the paint and the total "soluble" barium content of the liquid paint, or
- the total "soluble" barium content of the paint in powder form;

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

g) the date of the test.

---

[1] Not required for paints in powder form (see clause 7 of ISO 6713).