International Standard 3856/6

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Paints and varnishes — Determination of "soluble" metal content — Part 6: Determination of total chromium content of the liquid portion of the paint — Flame atomic absorption spectrometric method

Peintures et vernis — Détermination de la teneur en métaux «solubles» — Partie 6: Détermination de la teneur totale en chrome de la fraction liquide de la peinture — Méthode par spectrométrie d'absorption 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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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Paints and varnishes — Determination of "soluble" metal content — Part 6: Determination of total chromium content of the liquid portion of the paint — Flame atomic absorption 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 absorption spectrometric (AAS) method for the determination of the total chromium content of the liquid portion of the paint, prepared in accordance with sub-clause 9.3 of ISO 6713 or other suitable International Standards.

The method is applicable to paints having total chromium contents in the liquid portion in the range of about 0,05 to 5 % (*m*/*m*).

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

2 References

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

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 absorption of the selected spectral line, emitted by a chromium hollow-cathode lamp or chromium discharge lamp, in the region of 357,9 nm.

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 Hydrochloric acid, $c(HCl) = 0,07$ 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.)

4.2 Acetylene, commercial grade, in a steel cylinder.

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

4.4 Chromium, standard stock solution containing 100 mg of Cr per litre.

Either

a) transfer the contents of an ampoule of standard chromium solution containing exactly 0,1 g of Cr into a 1 000 ml one-mark volumetric flask, dilute to the mark with the hydrochloric acid (4.1) and mix well;

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

2) At present at the stage of draft.

or

b) weigh, to the nearest 0,1 mg, 282,9 mg of dry potassium dichromate, dissolve in the hydrochloric acid (4.1) 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 100 µg of Cr.

4.5 Chromium, standard solution containing 10 mg of Cr per litre.

Prepare this solution on the day of use.

Pipette 10 ml of the standard stock solution (4.4) into a 100 ml one-mark volumetric flask, dilute to the mark with the hydrochloric acid (4.1) and mix well.

1 ml of this standard solution contains 10 µg of Cr.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Flame atomic absorption spectrometer, suitable for measurements at a wavelength of 357,9 nm and fitted with a burner fed with dinitrogen monoxide and acetylene.

5.2 Chromium hollow-cathode lamp or **chromium discharge lamp.**

5.3 Burette, of capacity 25 ml, complying with the requirements of ISO 385/1.

5.4 One-mark volumetric flasks, of capacity 100 ml, complying with the requirements of ISO 1042.

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 100 ml one-mark volumetric flasks (5.4), introduce from the burette (5.3), respectively, the volumes of the standard chromium solution (4.5) shown in the following table, dilute each to the mark with the hydrochloric acid (4.1) and mix well.

Standard matching solution No.	Volume of the standard chromium solution (4.5)	Corresponding concentration of Cr in the standard matching solution
	ml	µg/ml
0 *	0	0
1	2	0,2
2	5	0,5
3	10	1
4	15	1,5
5	20	2

* Blank matching solution.

6.1.2 Spectrometric measurements

Install the chromium spectral source (5.2) in the spectrometer (5.1) and optimize the conditions for the determination of chromium. Adjust the instrument in accordance with the manufacturer's instructions and adjust the monochromator to the region of 357,9 nm in order to obtain the maximum absorbance.

Adjust the flow of the acetylene (4.2) and of the dinitrogen monoxide (4.3) according to the characteristics of the aspirator-burner, and ignite the flame. Set the scale expansion, if fitted, so that the standard matching solution No. 5 (see table) gives almost a full-scale deflection.

Aspirate into the flame each of the standard matching solutions (see 6.1.1) in ascending order of concentration, and repeat with the standard matching solution No. 4 to verify that the instrument has achieved stability. Aspirate water through the burner between each measurement, taking care to keep the rate of aspiration uniform.

6.1.3 Calibration graph

Plot a graph having the masses, in micrograms, of Cr contained in 1 ml of the standard matching solutions as abscissae and the corresponding values of the absorbances, reduced by the reading for the blank matching solution, as ordinates.

6.2 Test solutions

Use the solutions obtained by the procedure described in subclause 9.3 of ISO 6713 or other specified or agreed procedures.

6.3 Determination

Measure first the absorbance of the hydrochloric acid (4.1) 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 absorbance of standard matching solution No. 4 (see 6.1.1) in order to verify that the response of the apparatus has not changed. If the absorbance of a test solution is higher than that of the standard matching solution with the highest chromium concentration, dilute the test solution appropriately (dilution factor F) with a known volume of the hydrochloric acid (4.1).

7 Expression of results

7.1 Calculations

Calculate the mass of chromium in the solution (extract) obtained by the method described in sub-clause 9.3 of ISO 6713, using the equation

$$m_2 = \frac{b_1 - b_0}{10^6} \times V_2 \times F$$

where

b_0 is the chromium 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 chromium concentration, in micrograms per millilitre, of the test solution obtained from the calibration graph;

F is the dilution factor referred to in 6.3;

m_2 is the mass, in grams, of chromium 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).

Calculate the chromium content of the liquid portion of the paints, using the equation

$$c_{Cr_2} = \frac{m_2}{m_3} \times 10^2$$

where

c_{Cr_2} is the chromium 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.

NOTE — The total "soluble" chromium content of the liquid paint, consisting of the "soluble" hexavalent chromium content of the pigment portion plus the total chromium content of the liquid portion of the paint and expressed as a percentage by mass of the paint, is given by the sum of the results obtained according to ISO 3856/5 and this part of ISO 3856.

If the test solutions were prepared by methods other than that given in ISO 6713 (see 6.2), it will be necessary to modify the equations for the calculation of chromium content given above.

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 3856/6);

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) the results of the test, expressed as a percentage by mass of the product, i.e. the chromium content of the liquid portion of the paint;

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

g) the date of the test.