



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 spectroscopic 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 spectroscopie 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.

International Standard ISO 3856/6 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	India	Norway
Austria	Iran	Poland
Brazil	Ireland	Romania
Bulgaria	Israel	South Africa, Rep. of
Canada	Italy	Sweden
Egypt, Arab Rep. of	Kenya	Switzerland
France	Korea, Rep. of	Turkey
Germany, F. R.	Mexico	USSR

The member body of the following country expressed disapproval of the document on technical grounds :

United Kingdom

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Part 6 : Determination of total chromium content of the liquid portion of the paint — Flame atomic absorption 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 absorption spectroscopic (AAS) method for the determination of the total chromium content of the liquid portion of the paint, prepared according to clause 8 of 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 AAS method should be used.

2 References

ISO/R 385, *Burettes*.

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

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

3 Principle

Aspiration of the test solution into a dinitrogen monoxide/acetylene flame. Measurement of the absorption of the selected spectral line (357,9 nm) emitted by a chromium hollow-cathode lamp.

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 Hydrochloric acid, 0,07 mol/l solution.

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

4.2 Acetylene, in a steel cylinder.

4.3 Dinitrogen monoxide, in a steel cylinder.

4.4 Chromium, standard solution corresponding to 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 solution (4.1), and mix well;

or

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

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

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

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

Prepare this solution on the day of use.

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

5 Apparatus

Ordinary laboratory apparatus and

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

5.2 Chromium hollow-cathode lamp.

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

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

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

Prepare these solutions on the day of use.

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

* Blank test on reagents for calibration graph.

6.1.2 Spectroscopic measurements

Install the chromium hollow-cathode lamp (5.2) in the spectrometer (5.1) and leave the apparatus switched on for the time necessary to achieve stability. Adjust the lamp current, the attenuation and the slit, to suit the characteristics of the apparatus. Adjust the wavelength in the region of 357,9 nm in order to obtain the maximum absorbance. Adjust the pressures

of the acetylene (4.2) and of the dinitrogen monoxide (4.3) according to the characteristics of the aspirator-burner. Aspirate the series of standard matching solutions (6.1.1) into the flame and measure the absorbance for each. Aspirate water through the burner after each measurement. Take care to keep the rate of aspiration constant throughout the preparation of the calibration graph.

6.1.3 Plotting of the 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 value for the blank solution, as ordinates.

6.2 Test solution

Use the solution obtained by the procedure specified in clause 8 of ISO 6713 or other specified or agreed procedures.

6.3 Determination

Measure the absorbance of the test solution (6.2) three times in the apparatus after having adjusted it as specified in 6.1.2. Measure first the absorbance of the hydrochloric acid solution (4.1), then that of the test solution and afterwards that of the hydrochloric acid solution again. Finally, re-determine the absorbances of the standard matching solutions (6.1.1) in order to verify that the adjustment of the apparatus has not changed. If the absorbance of the test solution is higher than that of the standard matching solution with the highest chromium concentration, dilute the test solution appropriately with a known volume of the hydrochloric acid solution (4.1).

7 Expression of results

7.1 Calculations

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

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

where

b_0 is the chromium 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 chromium 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 chromium in the solution obtained according to clause 8 of ISO 6713;

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

The chromium content of the liquid portion of the paint, is given by 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 specified 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 and extender 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 solution was prepared by a method 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 include at least the following information :

- a) the type and identification of the product tested;
- b) a reference to this International Standard (ISO 3856/6) or to a corresponding national standard;
- c) the method for the separation of the solid portion of the product under test according to clause 6 of ISO 6713 (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 result of the test, expressed as a percentage by mass of the paint, i.e. the chromium content of the liquid portion of the paint;
- g) the date of the test.

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