International Standard ISO 3856/5

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Paints and varnishes — Determination of "soluble" metal content — Part 5 : Determination of hexavalent chromium content of the pigment and extender portion of the paint — Diphenylcarbazide spectrophotometric method

Peintures et vernis — Détermination de la teneur en métaux «solubles» — Partie 5 : Détermination du chrome hexavalent contenu dans la partie de pigment et de matière de charge de la peinture — Méthode spectrophotométrique à la diphénylcarbazide

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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 3856/5 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*

* on the grounds that parts 5 and 6 of ISO 3856 should be combined.

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Paints and varnishes — Determination of "soluble" metal content — Part 5 : Determination of hexavalent chromium content of the pigment and extender portion of the paint — Diphenylcarbazide spectrophotometric 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 diphenylcarbazide spectrophotometric method for the determination of the "soluble" hexavalent chromium content of the hydrochloric acid extract of the pigment and extender portion of the product, prepared according to clause 7 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, provided that the methods are specific for hexavalent chromium but, in case of dispute, this spectrophotometric 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

Formation of a coloured complex from hexavalent chromium and diphenylcarbazide solution. After addition of orthophosphoric acid and sulphuric acid, spectrophotometric measurement of the colour at a wavelength of about 540 nm.

4 Reagents

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

4.1 Diphenylcarbazide, solution.

Dissolve 0,25 g diphenylcarbazide in a mixture of 50 ml of acetone and 50 ml of water.

4.2 Sodium hydroxide, 2 mol/l solution.

4.3 Sulphuric acid, 1 mol/l solution.

4.4 Orthophosphoric acid, approximately 85 % (*m/m*) solution, ϱ approximately 1,69 g/ml.

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

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

4.6 Hexavalent chromium, standard solution corresponding to 100 mg of Cr(VI) per litre.

Weigh, to the nearest 0,1 mg, 282,9 mg of potassium dichromate, dissolve in water in a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix well.

1 ml of this standard solution contains 100 µg of Cr(VI).

4.7 Hexavalent chromium, standard solution corresponding to 1 mg of Cr(VI) per litre.

Pipette 10 ml of the standard hexavalent chromium solution (4.6) into a 1 000 ml one-mark volumetric flask, dilute to the mark with the hydrochloric acid solution (4.5), and mix well.

Prepare this solution on the day of use.

1 ml of this standard solution contains 1 µg of Cr(VI).

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

5 Apparatus

Ordinary laboratory apparatus and

5.1 Spectrophotometer, suitable for measurements at a wavelength of about 540 nm, fitted with cells of optical path length 10 or 20 mm.

5.2 pH-meter, with glass electrode and reference electrode.

5.3 Burette, of capacity 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.

6 Procedure

6.1 Preparation of the calibration graph

6.1.1 Preparation of standard colorimetric solutions for spectrophotometric measurements in cells of optical path length 10 or 20 mm

Introduce from the burette (5.3), into each of a series of five 50 ml beakers, the volumes of the standard hexavalent chromium solution (4.7) shown in the following table.

Standard colorimetric solution No.	Standard hexavalent chromium solution (4.7)	Corresponding concentration of Cr(VI) in the colorimetric solution
	ml	µg/ml
0*	0	0
1	5	0,1
2	10	0,2
3	15	0,3
4	20	0,4

* Compensation solution.

Treat the contents of each beaker as follows :

Add 5 ml of the sodium hydroxide solution (4.2). Using the pH meter (5.2), adjust the pH value of the solution to 7,0 by the addition of the sulphuric acid solution (4.3). Add 2 ml of the diphenylcarbazide solution (4.1) and 1 to 2 ml of the orthophosphoric acid solution (4.4), together with 5 ml of the sulphuric acid solution (4.3). Transfer to a 50 ml one-mark volumetric flask (5.4), dilute to the mark with water and mix well.

Prepare these solutions on the day of use.

6.1.2 Spectrophotometric measurements

Immediately measure the absorbances of the standard colorimetric solutions (6.1.1) with the spectrophotometer (5.1) at the wavelength of maximum absorption (about 540 nm) against water in the reference cell. Before each measurement, rinse the cells with the standard colorimetric solution. Deduct the absorbance of the compensation solution from those of the other standard colorimetric solutions.

6.1.3 Plotting of the graph

Plot a graph having the masses, in micrograms, of Cr(VI) contained in 1 ml of the standard colorimetric solutions as abscissae, and the corresponding values of absorbance as ordinates. If the procedure has been carried out correctly, the calibration graph should be a straight line.

6.2 Test solution

Use the solution obtained by the procedure specified in subclause 7.2.1 of ISO 6713 or other specified or agreed procedures.

6.3 Determination

Introduce from the burette (5.3), into a 50 ml beaker, a volume of the test solution (6.2) such that its absorbance lies on the calibration graph. Treat the solution as specified in 6.1.1. Measure the absorbance as specified in 6.1.2.

7 Expression of results

7.1 Calculations

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

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

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

where

a_0 is the hexavalent chromium 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 hexavalent chromium concentration, in micrograms per millilitre, of the test solution obtained from the calibration graph;

m_0 is the mass, in grams, of "soluble" hexavalent chromium in the hydrochloric extract;

V_1 is the volume, in millilitres, of the hydrochloric acid solution plus ethanol taken 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 solution plus ethanol taken for the test.

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

$$c_{Cr_1} = m_0 \times \frac{10^2}{m_1} \times \frac{P}{10^2}$$

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

where

c_{Cr_1} is the "soluble" hexavalent chromium 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.

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 3856/6 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 hexavalent 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 3856/5) 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 "soluble" hexavalent chromium content in the pigment and extender portion of the paint;

g) the date of the test.