International Standard ISO 4620

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Cadmium pigments — Specifications and methods of test

Pigments de cadmium — Spécifications et méthodes d'essai

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Foreword

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Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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INTERNATIONAL STANDARD

Cadmium pigments — Specifications and methods of test

1 Scope and field of application

This International Standard specifies the requirements and the corresponding methods of test for cadmium pigments suitable for general use.

2 References

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

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

ISO 787, *General methods of test for pigments and extenders —*

Part 1: Comparison of colour of pigments.

Part 2: Determination of matter volatile at 105 °C.

Part 5: Determination of oil absorption value.

Part 7: Determination of residue on sieve — Water method — Manual procedure.

Part 8: Determination of matter soluble in water — Cold extraction method.

Part 9: Determination of pH value of an aqueous suspension.

Part 16: Determination of relative tinting strength (or equivalent colouring value) and colour on reduction of coloured pigments — Visual comparison method.

Part 20: Comparison of ease of dispersion (oscillatory shaking method).

Part 21: Comparison of heat stability of pigments using a stoving medium.

ISO 842, *Raw materials for paints and varnishes — Sampling.*

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

ISO 3696, *Water for laboratory use — Specifications.*[1]

ISO 3856, *Paints and varnishes — Determination of "soluble" metal content —*

Part 1: Determination of lead content — Flame atomic absorption spectrometric method and dithizone spectrophotometric method.

Part 2: Determination of antimony content — Flame atomic absorption spectrometric method and Rhodamine B spetrophotometric method.

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

Part 4: Determination of cadmium content — Flame atomic absorption spectrometric method and polarographic method.

Part 6: Determination of total chromium content of the liquid portion of the paint — Flame atomic absorption spetrometric method.

Part 7: Determination of mercury content of the pigment portion of the paint and of the liquid portion of water-dilutable paints — Flameless atomic absorption spectrometric method.

Part 8: Determination of selenium content — Flame atomic absorption spectrometric method and X-ray fluorescence method.[2]

Part 9: Determination of arsenic content — Atomic absorption and silver diethyldithiocarbamate spectrometric methods.[1]

ISO 4793, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation.*

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

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[1] At present at the stage of draft.

[2] In preparation.

3 Definition

cadmium pigment: An inorganic coloured pigment consisting predominantly of cadmium sulfide or of mixed crystals of cadmium sulfide and cadmium selenide, in which the cadmium may be partially replaced by zinc.

NOTES

1 To adjust the relative tinting strength and/or to obtain certain application properties, cadmium pigments may contain colourless additives.

2 Cadmium pigments are characterized by a very low solubility in water and diluted acids and by a very high heat stability.

3 Yellow cadmium pigments consist mainly of mixed crystals of cadmium sulfide and zinc sulfide (Pigment yellow No. 37, part 2, ref. 77 199)[1] or of cadmium sulfide (Pigment orange No. 20, part 2, ref. 77 199).[1]

Orange to maroon cadmium pigments consist mainly of mixed crystals of cadmium sulfide and cadmium selenide (Pigment red No. 108, part 2, ref 77 202[1]).

4 Classification

This International Standard specifies the following three types of pigment, which shall be free from organic materials, other coloured inorganic pigments and brightening agents:

Type 1: Cadmium pigments, concentrated. These pigments contain not more than 30 % (*m*/*m*) of extenders to adjust their relative tinting strength.

Type 2: Cadmium pigments, extended. These pigments contain not more than 70 % (*m*/*m*) of extenders to facilitate processing.

Type 3: Cadmium pigments, unextended. These are essentially pure and do not contain any extenders.

NOTE — Type 1 and type 2 pigments are generally suitable for use in paints, plastics, rubber, etc. Type 3 pigments are mainly suitable for use in ceramics.

5 Required characteristics and their tolerances

5.1 For cadmium pigments complying with this International Standard, the essential requirements are specified in table 1 and the conditional requirements are referred to in table 2. For the conditional requirements to be specified, prior agreement between the interested parties is necessary.

5.2 The agreed reference pigment, which is referred to in table 2, shall conform with the requirements specified in table 1.

6 Sampling

Take a representative sample of the product to be tested as described in ISO 842.

7 Determination of total cadmium and zinc contents

NOTE — The method given in this clause allows the separate determination of the alkali earth metals present. However, it should be used only for cadmium pigments with a zinc content greater than 5 %. For pigments with lower zinc contents, a suitable atomic absorption spectrometric method should be agreed between the interesed parties.

7.1 Principle

Dissolution of a test portion in hydrochloric acid and nitric acid. After neutralization, addition of potassium cyanide and titration of the alkali earth metals with EDTA solution. Addition of formaldehyde solution to destroy the cadmium and zinc cyanide complexes. Titration of the sum of cadmium and zinc ions with EDTA solution. Destruction of the cadmium-EDTA complex in the titrated solution with sodium diethyldithiocarbamate and precipitation of the cadmium with ammonia solution. Back-titration of the excess of EDTA corresponding to the cadmium, with magnesium sulfate solution.

7.2 Reagents

During the analysis, use only reagents of recognized analytical grade and only water of at least grade 3 purity according to ISO 3696.

WARNING — Use the reagents in accordance with the appropriate health and safety regulations.

7.2.1 Hydrochloric acid, approximately 37 % (*m*/*m*), ϱ approximately 1,18 g/ml.

7.2.2 Hydrochloric acid, diluted, c(HCl) approximately 0,1 mol/l.

7.2.3 Nitric acid, approximately 65 % (*m*/*m*), ϱ approximately 1,40 g/ml.

7.2.4 Sodium hydroxide, 10 % (*m*/*m*) solution.

7.2.5 Ammonia solution, approximately 25 % (*m*/*m*), ϱ approximately 0,91 g/ml.

7.2.6 EDTA, standard volumetric solution,
c(EDTA) = 0,1 mol/l.

Dissolve 37,225 g of disodium ethylenediamine-tetraacetate dihydrate (EDTA disodium salt) in water in a 1 000 ml one-mark volumetric flask, dilute to the mark and mix well.

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[1] Classification according to the Colour Index. The Colour Index is published by the Society of Dyers and Colourists, Bradford, England and the American Association of Textile Chemists and Colorists, Lowell, Mass. (USA), revised edition 1971.

Table 1 — Essential requirements

Characteristic		Requirement	Test method
Total (Cd + Zn + Se + S)	type 1, % (m/m)	min. 70	Clause 10
	type 2, % (m/m)	min. 30	
	type 3, % (m/m)	min. 98	
Matter volatile at 105 °C	% (m/m)	max. 0,5	ISO 787/2
Matter soluble in water (cold extraction method)	% (m/m)	max. 0,3	ISO 787/8
Matter soluble in 0,07 mol/l hydrochloric acid[1]			
Antimony (Sb)	% (m/m)	max. 0,05	Clause 12
Arsenic (As)[2]	% (m/m)	max. 0,01	Clause 13
Barium (Ba)	% (m/m)	max. 0,01	Clause 14
Cadmium (Cd)	% (m/m)	max. 0,1	Clause 15
Chromium (Cr)	% (m/m)	max. 0,1	Clause 16
Lead (Pb)[2]	% (m/m)	max. 0,01	Clause 17
Mercury (Hg)	% (m/m)	max. 0,005	Clause 18
Selenium (Se)	% (m/m)	max. 0,01	Clause 19
pH of the aqueous suspension		5 to 8	ISO 787/9
Residue on sieve (45 µm)	% (m/m)	max. 0,1	ISO 787/7

Table 2 — Conditional requirements

Characteristic	Requirement	Test method
Colour	Shall closely match that of the agreed reference pigment (5.2)	ISO 787/1
Relative tinting strength	Shall closely match that of the agreed reference pigment (5.2)	ISO 787/16
Ease of dispersion	Shall not be inferior to that of the agreed reference pigment (5.2)	ISO 787/20
Oil absorption value	Shall not differ by more than 15 % from the value agreed between the interested parties[3]	ISO 787/5
Heat stability	Shall show no greater change in colour or relative tinting strength than that of the agreed reference pigment (5.2)	ISO 787/21, carried out at 400 ± 25 °C in an inert atmosphere for 5 min

1) Where national legislation specifies different limits for the content of metals "soluble" in 0,07 mol/l hydrochloric acid and if the limits are less than those specified in this International Standard, then those limits will take precedence over those in this specification. In certain cases, limits for additional metals may be specified, for example zinc, for which a test method is described in clause 20.

2) It may be necessary to determine the arsenic or lead contents in the pigment itself, instead of in the hydrochloric acid extract, because of the national legislation of the country in which the pigment is to be used. The methods of determination should be agreed between the interested parties.

3) The oil absorption value of cadmium pigments lies generally in the range 15 to 25 g/100 g.

7.2.7 Magnesium sulfate, standard volumetric solution, $c(\mathrm{MgSO_4}) = 0{,}1$ mol/l.

7.2.8 Formaldehyde, 30 % (m/m) solution.

7.2.9 Methyl orange, 0,01 % (m/m) solution.

7.2.10 Buffered indicator tablets, containing mordant black II (Eriochrome Black T)[1] and a buffer substance.

NOTE — Instead of an indicator tablet, 0,25 g of a mixture of 1 g of mordant black II (Eriochrome Black T) and 100 g of ammonium chloride may be added. In this case the colour changes in 7.3.2 and 7.3.3 are from red to blue and in 7.3.4 from blue to red.

7.2.11 Buffer solution, pH 10.

Dissolve 70 g of $\mathrm{NH_4Cl}$ in 774 ml of the ammonia solution (7.2.5) and dilute to 1 000 ml with water.

7.2.12 Sodium diethyldithiocarbamate.

7.2.13 Potassium cyanide.

WARNING — Potassium cyanide is a deadly poison.

7.3 Procedure

Carry out the determination in duplicate.

7.3.1 Weigh, to the nearest 0,1 mg, 0,3 to 0,4 g of the sample into a 250 ml beaker and add about 10 ml of the hydrochloric acid (7.2.1). Heat carefully until the hydrochloric acid is evaporated. Add 2 ml of the nitric acid solution (7.2.3) to the residue, cover the beaker with a watch glass and heat on a hotplate for 10 min or until a clear solution is obtained. Add 100 ml of water, boil for a few minutes, filter off any remaining residue if present, using a fine filter paper, wash first with the diluted hydrochloric acid (7.2.2) then with water, and collect the solution and washwater.

7.3.2 Add 1 or 2 drops of the methyl orange solution (7.2.9) to the solution obtained according to 7.3.1 and neutralize with the sodium hydroxide solution (7.2.4). Add a few millilitres of the buffer solution (7.2.11), one indicator tablet (7.2.10) or 0,25 g of the mordant black II mixture and about 2 g of the potassium cyanide (7.2.13). Titrate the solution with the EDTA solution (7.2.6) until the colour changes from red to greyish green (see the note to 7.2.10).

7.3.3 Add about 20 ml of the formaldehyde solution (7.2.8) to the solution obtained according to 7.3.2. Titrate the solution with the EDTA solution (7.2.6) until the colour changes from red to greyish green (see the note to 7.2.10). Record the volume of EDTA solution used (V_0).

7.3.4 Add approximately 1 g of the sodium diethyldithiocarbamate (7.2.12) and a few drops of the ammonia solution (7.2.5) to the titrated solution (7.3.3) and titrate with the magnesium sulfate solution (7.2.7) until the colour changes from greyish green to red (see the note to 7.2.10). Record the volume of magnesium sulfate solution used (V_1).

7.4 Expression of results

Calculate the total cadmium content of the pigment, using the equation

$$w_{\mathrm{Cd,t}} = \frac{V_1 \times 1{,}124}{m}$$

Calculate the total zinc content of the pigment, using the equation

$$w_{\mathrm{Zn,t}} = \frac{(V_0 - V_1) \times 0{,}654}{m}$$

where

m is the mass, in grams, of the test portion;

V_0 is the volume in millilitres, of the EDTA solution (7.2.6) required for the titration of the sum of (Cd + Zn) in 7.3.3;

V_1 is the volume in millilitres, of the magnesium sulfate solution (7.2.7) required for the titration of Cd in 7.3.4;

$w_{\mathrm{Cd,t}}$ is the total cadmium content of the pigment, expressed as a percentage by mass;

$w_{\mathrm{Zn,t}}$ is the total zinc content of the pigment, expressed as a percentage by mass;

1,124 is the factor (× 100) for the conversion of millilitres of EDTA solution (7.2.5) to grams of Cd;

0,654 is the factor (× 100) for the conversion of millilitres of EDTA solution (7.2.5) to grams of Zn.

Take the mean of the two determinations, but if the duplicate determinations differ by more than 0,4 %, repeat the procedure.

8 Determination of selenium content

8.1 Principle

Dissolution of a test portion in bromine water. After driving off the bromine, reduction of the selenium present and precipitation with hydrazinium dichloride followed by weighing.

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[1] Sodium 1-(1-hydroxy-2-naphthylazo)-6-nitronaphthalene-4-sulfonate.

8.2 Reagents

During the analysis, use only reagents of recognized analytical grade and only water of at least grade 3 purity according to ISO 3696.

WARNING — A number of reagents used in this determination, as well as selenium itself, are hazardous. Use the reagents in accordance with the appropriate health and safety regulations. Care must be taken when handling these materials and, wherever possible, operations shall be carried out in a fume cupboard.

8.2.1 Hydrochloric acid, approximately 37 % (*m*/*m*), ϱ approximately 1,18 g/ml.

8.2.2 Hydrochloric acid, diluted, $c(\text{HCl})$ approximately 0,1 mol/l.

8.2.3 Bromine.

8.2.4 Hydrazinium dichloride, ($H_2N{\cdot}NH_2{\cdot}2HCl$).

WARNING — Hydrazinium dichloride is a severe poison.

8.2.5 Carbon dioxide or **nitrogen,** as the compressed gas.

8.3 Procedure

WARNING — All procedures involving bromine shall be carried out in a fume cupboard. Use safety gloves.

Carry out the determination in duplicate.

Weigh to the nearest 0,1 mg, about 0,5 g of the sample, suspend it in 10 ml of water in a 100 ml conical flask fitted with a ground glass stopper and add 1 ml of the bromine (8.2.3) by means of a safety pipette. Close the flask and allow it to stand for at least 3 h or overnight.

Dilute the solution with about 20 ml of water. Fit a Liebig condenser on to the conical flask and insert a glass tube with a drawn-out tip through the condenser almost to the bottom of the flask. Connect the tube to the source of the carbon dioxide or nitrogen (8.2.5). While gently heating, pass carbon dioxide or nitrogen through the solution until the bromine has been driven off completely. Test with potassium iodine-starch paper for the presence of bromine at the top of the condenser.

Add 5 ml of the hydrochloric acid (8.2.1) to the bromine-free solution and filter off any remaining residue, if present, using a fine filter paper. Wash the residue and filter paper first with the diluted hydrochloric acid (8.2.2) and then with hot water until free from chloride.

To the filtrate and washings add 20 to 30 ml of the hydrochloric acid (8.2.1) for each 100 ml of solution. Add about 5 g of the hydrazinium dichloride (8.2.4) to the cooled solution and heat the solution on a water bath or on a gently heated sand bath. When the precipitated red selenium has been totally converted to the black form, filter off the selenium using a previously weighed sintered crucible of porosity 7 to 8 µm complying with the requirements of ISO 4793 and wash the selenium with hot water. Collect the filtrate and washings, and retain for the determination of sulfur content (see clause 9).

Dry the crucible and selenium at 105 °C for 1 h, and after cooling to room temperature in a desiccator, weigh to the nearest 0,1 mg. Repeat the drying, cooling and weighing procedure until successive weighings differ by not more than 0,1 mg.

8.4 Expression of results

Calculate the total selenium content of the pigment, using the equation

$$w_{\text{Se,t}} = \frac{m_2}{m_1} \times 100$$

where

m_1 is the mass, in grams, of the test portion;

m_2 is the mass, in grams, of the precipitate;

$w_{\text{Se,t}}$ is the total selenium content of the pigment, expressed as a percentage by mass.

Take the mean of the two determinations but, if the duplicate determinations differ by more than 0,4 %, repeat the procedure.

9 Determination of sulfur content

NOTE — Using this method, both the sulfide content and the sulfate content are determined, with the exception of insoluble sulfates, such as barium sulfate.

9.1 Reagents

During the analysis, use only reagents of recognized analytical grade and only water of at least grade 3 purity according to ISO 3696.

WARNING — Use the reagents in accordance with the appropriate health and safety regulations.

9.1.1 Methyl orange, 0,01 % (*m*/*m*) solution.

9.1.2 Ammonia solution, approximately 25 % (*m*/*m*), ϱ approximately 0,91 g/ml.

9.1.3 Hydrochloric acid, $c(\text{HCl})$ approximately 1 mol/l.

9.1.4 Barium chloride, 10 % (*m*/*m*) solution.

9.2 Procedure

Carry out the determination in duplicate.

Add 5 to 6 drops of the methyl orange (9.1.1) to the filtrate and washings obtained during the determination of selenium content, described in 8.3, and neutralize with the ammonia solution (9.1.2). To this solution add 3 to 5 ml of the hydrochloric acid

(9.1.3) for each 100 ml of solution. Heat to boiling. Add, drop by drop, while stirring, about 15 ml of the barium chloride solution (9.1.4) and boil again.

After the precipitate has settled (allow to stand overnight, if necessary), filter through a fine filter paper and wash the precipitate first with the hydrochloric acid (9.1.3) and then with hot water. Ash the filter paper slowly with access to air and heat the residue to about 800 °C. After cooling to room temperature weigh the residue.

9.3 Expression of results

Calculate the total sulfur content of the pigment, using the equation

$$w_{S,t} = \frac{m_3}{m_1} \times 13{,}74$$

where

m_1 is the mass, in grams, of the test portion (see 8.3);

m_3 is the mass, in grams, of the precipitate ($BaSO_4$);

$w_{S,t}$ is the total sulfur content of the pigment, expressed as a percentage by mass;

13,74 is the factor (× 100) for the conversion of grams of $BaSO_4$ to grams of S.

Take the mean of the two determinations but if the duplicate determinations differ by more than 0,4 %, repeat the procedure.

10 Calculation of the total content of cadmium, zinc selenium and sulfur

Calculate the total Cd, Zn, Se and S content expressed as a percentage by mass, using the formula

$$w_{Cd,t} + w_{Zn,t} + w_{Se,t} + w_{S,t}$$

where

$w_{Cd,t}$ is the total cadmium content of the pigment, determined according to clause 7;

$w_{Zn,t}$ is the total zinc content of the pigment, determined according to clause 7;

$w_{Se,t}$ is the total selenium content of the pigment, determined according to clause 8;

$w_{S,t}$ is the total sulfur content of the pigment, determined according to clause 9.

11 Preparation of the hydrochloric acid extract

Prepare the hydrochloric acid extract of the pigment sample, for all metals including lead, by the procedure described in ISO 6713, sub-clause 8.2.

12 Determination of "soluble" antimony content

Determine the "soluble" antimony content of the pigment sample by the method described in ISO 3856/2, clause 3 (flame atomic absorption spectrometric method). Calculate the "soluble" antimony content, using the first equation given in sub-clause 3.6.1.1 of ISO 3856/2, but multiply the result by the volume, in millilitres, of the extract taken (assumed to be 20 ml).

13 Determination of "soluble" arsenic content

Determine the "soluble" arsenic content of the pigment sample by the method described in ISO 3856/9. Calculate the "soluble" arsenic content, using the first equation given in sub-clause 3.5.1.1 of ISO 3856/9, but multiply the result by the volume, in millilitres, of the extract taken (assumed to be 20 ml).

14 Determination of "soluble" barium content

Determine the "soluble" barium content of the pigment sample by the method described in ISO 3856/3. Calculate the "soluble" barium content, using the first equation given in sub-clause 7.1.1 of ISO 3856/3, but multiply the result by the volume, in millilitres, of the extract taken (assumed to be 20 ml).

15 Determination of "soluble" cadmium content

NOTE — For the determination of acid-soluble cadmium content, two methods are provided. The AAS method (15.1) should be used as the referee method in cases of dispute. Other methods can be used by agreement between the interested parties. If a complexometric method is agreed, the method described in 15.2 should be used.

15.1 Flame atomic absorption spectrometric method

Determine the "soluble" cadmium content of the pigment sample by the method described in ISO 3856/4, clause 3 (flame atomic absorption spectrometric method). Calculate the "soluble" cadmium content, using the first equation given in sub-clause 3.5.1.1 of ISO 3856/4, but multiply the result by the volume, in millilitres, of the extract taken (assumed to be 20 ml).

15.2 Complexometric method

15.2.1 Principle

Titration of the sum of cadmium and zinc ions with EDTA solution. Destruction of the cadmium-EDTA complex in the titrated solution with sodium diethyldithiocarbamate and back-titration of the excess of EDTA, corresponding to the cadmium, with magnesium sulfate solution.

Titration of the alkali earth metals in a further volume of the test solution with EDTA solution and calculation by difference of the volume of EDTA solution required for the titration of zinc.

15.2.2 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water of at least grade 3 purity according to ISO 3696.

WARNING — Use the reagents in accordance with the appropriate health and safety regulations.

15.2.2.1 Ammonia solution, approximately 25 % (m/m), ϱ approximately 0,91 g(ml.

15.2.2.2 EDTA, standard volumetric solution, $c(\text{EDTA}) = 0{,}01$ mol/l.

Dissolve 3,722 5 g of disodium ethylenediamine tetraacetate dihydrate (EDTA disodium salt) in water in a 1 000 ml, one-mark volumetric flask, dilute to the mark and mix well.

15.2.2.3 Magnesium sulfate, standard volumetric solution, $c(\text{MgSO}_4) = 0{,}01$ mol/l.

15.2.2.4 Sodium diethyldithiocarbamate.

15.2.2.5 Potassium cyanide, 65 g/l solution.

WARNING — Potassium cyanide is a deadly poison.

15.2.2.6 Mordant black II (Eriochrome Black T), mixture.

Mix 1 g of mordant black II with 100 g of ammonium chloride.

15.2.2.7 Methyl orange, 0,01 % (m/m) solution.

15.2.3 Apparatus

NOTE — For general use, Class B laboratory glassware is considered adequate, but, in cases of dispute, Class A apparatus should be used.

Ordinary laboratory apparatus and

15.2.3.1 Pipette, of capacity 20 ml, complying with the requirements of ISO 648.

15.2.4 Procedure

Carry out the determination in duplicate.

Measure with the pipette (15.2.3.1) into a conical flask 20 ml of the hydrochloric acid extract obtained as described in clause 11 and dilute to 50 ml with water. Neutralize with the ammonia solution (15.2.2.1) after adding one or two drops of the methyl orange solution (15.2.2.7). Add a further 1 ml of the ammonia solution and then add 0,25 g of the mordant black II mixture (15.2.2.6). Titrate with the EDTA solution (15.2.2.2) until the colour changes from red to blue. Then add exactly further 3 ml of the EDTA solution, shake carefully, note the total volume of EDTA solution added (V_3) and back-titrate with the magnesium sulfate solution (15.2.2.3) until the red colour is stable. Record the volume of magnesium sulfate solution used (V_4).

Add approximately 1 g of the sodium diethyldithiocarbamate (15.2.2.4). Shake carefully and titrate the liberated EDTA with the magnesium sulfate solution (15.2.2.3) until the colour changes. Record the volume of magnesium sulfate solution used (V_5).

Measure a fresh 20 ml portion of the hydrochloric acid extract (clause 11) with the pipette (15.2.3.1) and dilute to 50 ml with water. Neutralize with the ammonia solution (15.2.2.1) after adding one or two drops of the methyl orange solution (15.2.2.7). Add a further 1 ml of the ammonia solution and add 10 ml of the potassium cyanide solution (15.2.2.5). Add 0,25 g of the mordant black II mixture (15.2.2.6) and titrate with the EDTA solution (15.2.2.2) until the colour changes. Record the volume of EDTA solution used (V_6).

15.2.5 Expression of results

Calculate the "soluble" cadmium content of the pigment, using the equation

$$w_{\text{Cd,s}} = \frac{V_5 \times V_1 \times 0{,}112\,4}{m_4 \times V_2}$$

Calculate the "soluble" zinc content of the pigment, using the equation

$$w_{\text{Zn,s}} = [V_3 - (V_4 + V_5 + V_6)] \times 0{,}654 \times \frac{V_1}{V_2} \times \frac{100}{m_4 \times 1\,000}$$

where

m_4 is the mass, in grams, of the test portion (assumed to be 5,00 ± 0,01 g);

V_1 is the volume, in millilitres, of the hydrochloric acid plus ethanol used for the extraction described in sub-clause 8.2 of ISO 6713 (assumed to be 77 ml);

V_2 is the volume, in millilitres, of the hydrochloric acid plus ethanol extract used for the determination (assumed to be 20 ml);

V_3 is the volume, in millilitres, of the EDTA solution (15.2.2.2) added to the test solution;

V_4 is the volume, in millilitres, of the magnesium sulfate solution (15.2.2.3) used for the titration before addition of sodium diethyldithiocarbamate;

V_5 is the volume, in millilitres, of the magnesium sulfate solution (15.2.2.3) used for the titration, after addition of sodium diethyldithiocarbamate;

V_6 is the volume, in millilitres, of the EDTA solution (15.2.2.2) used for the titration of the potassium cyanide treated solution;

$w_{\text{Cd,s}}$ is the "soluble" cadmium content of the pigment, expressed as a percentage by mass;

$w_{\text{Zn,s}}$ is the "soluble" zinc content of the pigment, expressed as a percentage by mass;