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# International Standard



# 4619

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

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## Driers for paints and varnishes

*Siccatifs pour peintures et vernis*

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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.

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It has been approved by the member bodies of the following countries :

Australia	India	Portugal
Brazil	Iran	Romania
Bulgaria	Ireland	South Africa, Rep. of
Canada	Israel	Sweden
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The member body of the following country expressed disapproval of the document on technical grounds :

United Kingdom

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# Driers for paints and varnishes

## 1 Scope and field of application

This International Standard specifies the requirements and the corresponding test methods for driers for paints, varnishes and related products. The requirements relate to driers in the solid or liquid form.

## 2 References

ISO 150, *Raw, refined and boiled linseed oil for paints and varnishes — Specifications and methods of test.*

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

ISO 1250, *Mineral solvents for paints — White spirits and related hydrocarbon solvents.*

ISO 1523, *Paints and varnishes — Determination of flashpoint — Closed cup method.*

ISO 2431, *Paints and varnishes — Determination of flow time by use of flow cups.*<sup>1)</sup>

ISO 2592, *Petroleum products — Determination of flash and fire points — Cleveland open cup method.*

ISO 2811, *Paints and varnishes — Determination of density.*

ISO 3219, *Polymers in the liquid, emulsified or dispersed state — Determination of viscosity using a rotational viscometer working at defined shear rate.*

ISO 3251, *Paint media — Determination of volatile and non-volatile matter.*

ISO 4793, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation.*<sup>2)</sup>

## 3 Descriptions

### 3.1 Definition

**drier** : A compound, usually organometallic and soluble in organic solvents and binders, which is added to products drying by oxidation in order to accelerate the process.

### 3.2 General

#### 3.2.1 Solid driers

Solid driers are materials which may be manufactured in a hard, soft (highly viscous) or powder form.

#### 3.2.2 Liquid driers

Liquid driers are materials which are supplied as solutions in organic solvents, usually white spirit.

NOTE — Emulsifiable driers are also available, but no requirements for this type are given in this International Standard.

All these types of driers, when dissolved in solvents (normally hydrocarbons), impart specific drying properties depending on the metal used.

#### 3.2.3 Metals used

The following metals are used : cobalt, manganese, lead, zinc, calcium, cerium (or other rare earths), iron, zirconium, vanadium, barium, etc.

NOTE — In this International Standard methods for determination of metal content are given only for those metals which are in common use.

1) At present at the stage of draft. (Revision of ISO 2431-1972.)

2) At present at the stage of draft.

**3.2.4 Acids used**

The following acids are used : fatty acids of linseed oils, tall oil fatty acids, resinic acids, naphthenic acids, 2-ethylhexanoic acid, fatty iso-acids with 9 carbon atoms, fatty acids with 9 to 11 carbon atoms, etc.

**4 Required characteristics and their tolerances**

**4.1** Driers for paints shall have the characteristics shown in the table.

**4.2** Driers named according to the commercial name of the main acid used shall contain at least 90 % of this acid, except for driers based on naphthenic acids, which shall contain at least 70 % of these acids, expressed as a percentage of the total mass of acid present.

NOTE — If desired, the type and content of the acids may be determined by gas chromatographic (GC) analysis, except in the case of naphthenic acids.

**5 Sampling**

Take a representative sample of the drier in accordance with ISO 842.

**6 Methods of test for solid driers**

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

**6.1 Appearance and consistency**

Examine the sample visually for uniformity. If the consistency is specified, a method for its determination shall be agreed between the interested parties.

**6.2 Colour**

Dissolve 1 part by mass of the drier in 1 part by mass of white spirit or other agreed solvent and compare the colour against an agreed sample or colour standard.

**Table — Required characteristics and their tolerances**

Characteristic	Requirement	Test method	
		solid driers	liquid driers
Appearance	Clear and uniform, no suspended matter or sediment	clause 6.1	clause 7.1
Consistency, if required		To be agreed between the interested parties	—
Colour	As agreed between the interested parties	clause 6.2	clause 7.2
Solubility (miscibility) in solvent, raw linseed oil and other drying media	No separation or deposit	clause 6.3	clause 7.3
Stability of solution	Clear solution, no clouding, gelation or sedimentation	clause 6.4	clause 7.4
Suspended solid matter	of liquid driers % (m/m)	max. 0,1	
	of solid driers		
Viscosity, only for liquid driers		—	clause 7.5
Volatile matter at 105 °C		clause 6.6	clause 6.6
Flashpoint		ISO 2592	ISO 1523
Density		To be agreed between the interested parties	ISO 2811
Acidity or basicity		clause 6.7	clause 6.7
Drying characteristics		To be agreed between the interested parties	
Metal content (range)	up to 10 % (m/m)	± 0,2 % <sup>1)</sup>	
	above 10 to 20 % (m/m)	± 0,3 % <sup>1)</sup>	
	above 20 to 30 % (m/m)	± 0,4 % <sup>1)</sup>	
	above 30 % (m/m)	± 0,5 % <sup>1)</sup>	
		clause 8 or 9	

1) Tolerance (absolute value) on the metal content declared or agreed.

### 6.3 Solubility (miscibility) in solvents, raw linseed oil or other drying media

Slowly heat, raising the temperature at a rate of 1 °C/min, 5 g of the drier and 20 g of an agreed solvent (or drying medium) under reflux on a sand bath, with stirring, until a homogeneous solution is obtained.

Allow to cool to room temperature and examine the solution for any separation or deposit.

### 6.4 Stability of solution

Allow three portions of the solution obtained by the method specified in 6.3 to stand for 7 days in stoppered bottles, one at each of the following temperatures :

- a) 0 °C;
- b) ambient temperature;
- c) 50 °C.

After 1 day and again after 7 days, examine the solutions for clarity, clouding, sedimentation or gelation.

NOTE — The bottle used for the test at 50 °C should be able to withstand the pressure generated.

### 6.5 Suspended solid matter

Weigh, to the nearest 0,1 g, 5 g of solid drier (or 10 g of liquid drier) into a glass flask and dissolve in (or dilute with) 100 g of white spirit or an agreed solvent. Stopper the flask, allow to stand at ambient temperature for 3 days, then filter off the sediment or suspended matter using a glass filter crucible of porosity P 16 (see ISO 4793). Wash the residue on the filter with the solvent and dry it at 105 °C for 3 h. Cool to ambient temperature and weigh to the nearest 1 mg.

Calculate the suspended solid matter, as a percentage by mass, by the formula

$$\frac{m_1}{m_0} \times 100$$

where

$m_0$  is the mass, in grams, of the test portion;

$m_1$  is the mass, in grams, of the residue.

### 6.6 Volatile matter

Proceed according to ISO 3251, taking a flat-bottomed dish of glass or aluminium and a test portion of  $1 \pm 0,02$  g. Place the dish with the test portion in the air oven, maintained at  $105 \pm 2$  °C. Leave it in the oven at this temperature for 3 h.

### 6.7 Acidity or basicity

#### 6.7.1 Principle

A solution of the drier in toluene/2-propanol is passed through a strong acid cation exchanger and the total acid in the eluate is determined. From the total acid determined, the acid combined with the metal is subtracted.

If a negative value for the acidity is obtained, the drier tested is a basic drier.

The method is suitable for driers containing barium, calcium, cobalt, lead or zinc as metals, but is not applicable to driers containing cerium, iron, manganese or zirconium as metals.

In such cases, a method should be agreed between the interested parties.

#### 6.7.2 Reagents

**6.7.2.1 Cation exchanger** : strong acid, ring-sulphonated polystyrene resin (for example Merck I, Dowex 50, Amberlite IR 120).

**6.7.2.2 2-Propanol.**

**6.7.2.3 Toluene.**

**6.7.2.4 Hydrochloric acid**, approximately 5 % (m/m) solution.

**6.7.2.5 Potassium hydroxide**, approximately 0,2 mol/l standard volumetric solution in 96 % (V/V) ethanol.

**6.7.2.6 Phenolphthalein**, 1 % (m/m) solution in 96 % (V/V) ethanol.

#### 6.7.3 Apparatus

Ordinary laboratory apparatus and

**Ion-exchange column.** A suitable column is shown in the figure.

#### 6.7.4 Preparation of the ion-exchange column

Fill the ion-exchange column (6.7.3) with a quantity of the swollen ion-exchange resin (6.7.2.1) so that the height of resin in the column is about 170 mm. Pour 250 ml of the hydrochloric acid solution (6.7.2.4) gradually into the exchange column in order to change the resin into the hydrogen form. Drain off slowly, at about 1 or 2 drops/s ( $\approx 5$  ml/min). When the acid has drained off completely, wash the resin successively with several 350 ml portions of water. The final washings shall not be acid to litmus paper. Then displace the water in the exchange column by 50 ml of the 2-propanol (6.7.2.2) and finally displace the 2-propanol with 50 ml of a mixture (1 + 1) of the 2-propanol and the toluene (6.7.2.3).

NOTE — Do not use this column for more than about 50 milliequivalents of total metal.

Dimensions in millimetres

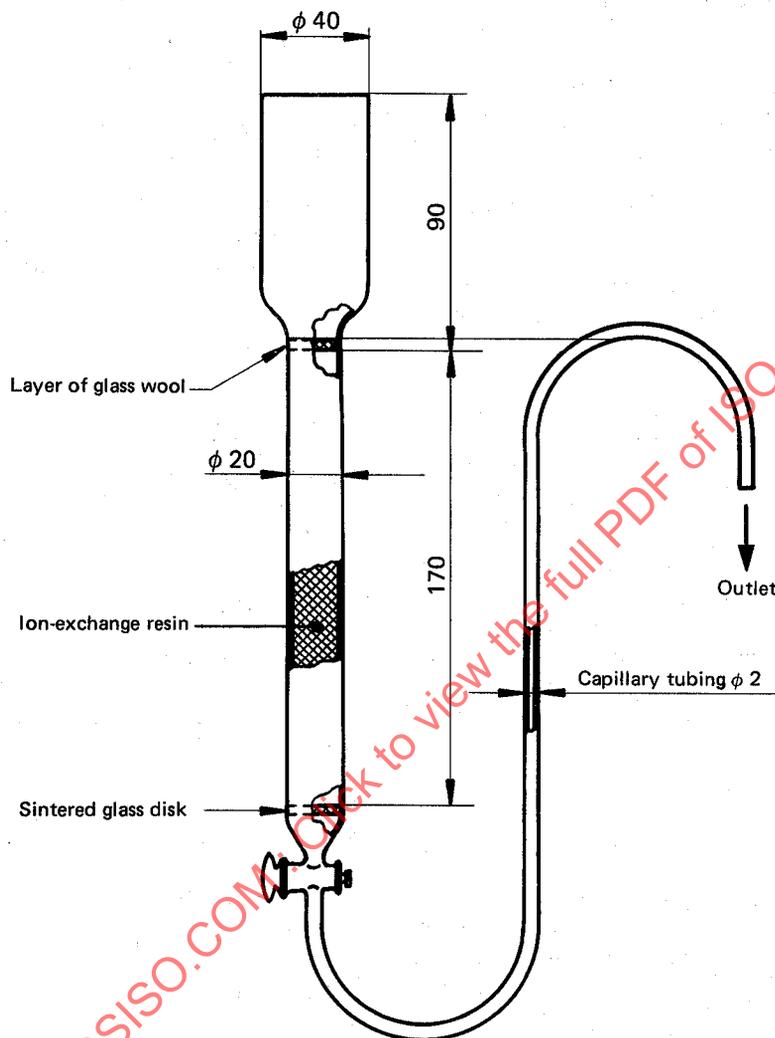


Figure — Suitable ion-exchange column (see 6.7.3)

### 6.7.5 Procedure

Weigh, to the nearest 1 mg, about 8 g of the drier to be tested into a 100 ml one-mark volumetric flask and dissolve it in 50 ml of the toluene (6.7.2.3). Dilute to the mark with the 2-propanol (6.7.2.2) and mix well. Pipette 25 ml of the solution into the ion-exchange column (6.7.4) and adjust the rate of flow to 5 ml/min. Collect the eluate in a 500 ml conical flask. When all the liquid has soaked through the resin, wash the exchange column with 150 ml of a mixture (1 + 1) of the 2-propanol and the toluene. Add a few drops of the phenolphthalein solution (6.7.2.6) to the eluate and titrate with the potassium hydroxide solution (6.7.2.5) to the end-point.

### 6.7.6 Expression of results

The acidity or basicity, expressed in milligrams of potassium hydroxide (KOH) per gram, is given by the formula

$$56,1 \left( \frac{4 \times V \times T}{m} - \frac{10 \times c \times n}{A} \right)$$

where

$V$  is the volume, in millilitres, of the potassium hydroxide solution (6.7.2.5) required for the titration;

$T$  is the exact concentration of the potassium hydroxide solution (6.7.2.5), in moles of KOH per litre;

$c$  is the metal content, as a percentage by mass, of the drier, as determined in clause 8;

$n$  is the valency of the metal in the drier;

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

$A$  is the relative atomic mass of the metal in the drier.

NOTE — For mixed driers the factor  $\frac{c \times n}{A}$  should be calculated, taking into account the composition of the mixed drier.

## 7 Methods of test for liquid driers

### 7.1 Appearance

Examine the sample visually for uniformity, clarity, suspended matter or sediment.

### 7.2 Colour

Compare the colour of the liquid drier against that of an agreed sample or colour standard.

### 7.3 Solubility (miscibility) in solvents, raw linseed oil or other drying media

Prepare a mixture as follows :

Raw linseed oil, complying with ISO 150 (see note 1) : 16 parts by volume

Mineral solvents, complying with ISO 1250 [aromatic content 25 % (V/V) maximum] (see note 2) : 4 parts by volume

The drier under test (in standard strength) (see note 3) : 1 part by volume

Allow the mixture to stand at room temperature for 6 h and then examine it for any separation or deposit.

#### NOTES

1 In the case of driers containing calcium, barium or rare earths, it is recommended that an air-drying alkyd resin [non-volatile content at least 60 % (m/m)] be used instead of linseed oil.

2 The exact aromatic content is to be agreed between the interested parties.

3 For the purpose of this test, the standard strength is defined, in % (m/m), as

6	Co	6	Ce (or other rare earths)
6	Mn	6	Fe
24	Pb	6	Zr
8	Zn	12,5	Ba
4	Ca		

Dilute driers of higher concentration than those above with the mineral solvents until the standard strength is obtained.

### 7.4 Stability of solution

Prepare three mixtures of 10 g of liquid driers and 10 g of mineral solvents, complying with ISO 1250 (see note 2 in 7.3) and allow to stand for 7 days in stoppered bottles at the following temperatures :

- 0 °C;
- ambient temperature;
- 50 °C.

After this period, examine the solutions for clarity, clouding, sedimentation or gelation.

NOTE — The bottle used for the test at 50 °C should be able to withstand the pressure generated.

### 7.5 Viscosity

A suitable method shall be agreed between the interested parties, for example by the procedure specified in ISO 2431 (flow cup method), ISO 3219 (rotational viscometer working at known shear rate) or falling ball methods.

## 8 Methods for determination of metal content of driers containing only one metal

NOTE — The methods are not selective for only one metal. In case of doubt, a qualitative test for the presence of other metals may be carried out. Other methods, e.g. atomic absorption spectrometry (AAS), may be used by agreement between the interested parties.

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity. All indicator solutions should preferably be freshly prepared but shall in no case be older than 2 weeks.

### 8.1 Cobalt (EDTA titrimetric method)

#### 8.1.1 Reagents

8.1.1.1 Hydroxylammonium chloride (OHNH<sub>3</sub>Cl).

8.1.1.2 2-Propanol.

8.1.1.3 Ethanol, 96 % (V/V) solution.

8.1.1.4 Hydrochloric acid, approximately 1 mol/l solution.

8.1.1.5 Hexamethylenetetramine, 40 % (m/m) aqueous solution.

8.1.1.6 EDTA, 0,100 mol/l standard volumetric solution.

Dissolve 37,225 0 g of disodium ethylene diamine-tetraacetate dihydrate (EDTA disodium salt), in water in a

1 000 ml one-mark volumetric flask, dilute to the mark and mix well.

Store the solution in a polyethylene bottle.

**8.1.1.7 Zinc sulphate**, approximately 0,05 mol/l standard volumetric solution.

a) Preparation

Dissolve 14,38 g of zinc sulphate heptahydrate ( $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ ) in water in a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

b) Standardization

Pipette 20 ml of the zinc sulphate solution into a 250 ml flask. Dilute to about 100 ml with water and add successively 10 ml of the buffer solution (8.1.1.8) and 5 or 6 drops of the Mordant black solution (8.1.1.9). Titrate with the EDTA solution (8.1.1.6) until the first change of the red colour to blue.

c) Calculation of concentration

The concentration,  $T_1$ , of the zinc sulphate solution, in moles of  $\text{ZnSO}_4$  per litre, is given by the formula

$$T_1 = \frac{V_1 \times T_0}{20}$$

where

$V_1$  is the volume, in millilitres, of the EDTA solution (8.1.1.6) required for the titration;

$T_0$  is the concentration of the EDTA solution (8.1.1.6), in moles of EDTA per litre.

**8.1.1.8 Buffer solution**, pH 10.

Add 54 g of ammonium chloride and 350 g of ammonia solution ( $\rho$  approximately 0,91 g/ml) to water in a 1 000 ml one-mark volumetric flask. Dilute to the mark and mix.

Store the solution in a polyethylene bottle.

**8.1.1.9 Mordant black 11\***, indicator solution.

Dissolve 0,5 g of Mordant black and 2,25 g of the hydroxyl-ammonium chloride (8.1.1.1) in 100 ml of the ethanol (8.1.1.3).

**8.1.1.10 Xylenol orange/potassium nitrate mixture** (1 + 100), for use as indicator.

## 8.1.2 Procedure

Weigh, to the nearest 1 mg, a test portion of the drier containing approximately 120 mg of cobalt into a 250 ml conical flask. Add 25 ml of the hydrochloric acid solution (8.1.1.4) and heat to boiling.

Dilute with 100 ml of the 2-propanol (8.1.1.2), add 15 ml of the hexamethylenetetramine solution (8.1.1.5) and 0,050 g of the xylenol orange/potassium nitrate mixture (8.1.1.10). Titrate with the EDTA solution (8.1.1.6) until the violet colour just changes to yellow.

If desired, back titrate with the zinc sulphate solution (8.1.1.7) until the first change of the yellow colour to red.

## 8.1.3 Expression of results

The cobalt content, expressed as a percentage by mass of Co, is given by the formula

$$\frac{[(V_2 \times T_0) - (V_3 \times T_1)] \times 0,058\ 94}{m_2} \times 100$$

$$= \frac{(V_2 \times T_0) - (V_3 \times T_1)}{m_2} \times 5,894$$

where

$V_2$  is the volume, in millilitres, of the EDTA solution (8.1.1.6) required;

$V_3$  is the volume, in millilitres, of the zinc sulphate solution (8.1.1.7) required (if used);

$T_0$  is the concentration of the EDTA solution (8.1.1.6), in moles of EDTA per litre;

$T_1$  is the concentration of the zinc sulphate solution (8.1.1.7), in moles of  $\text{ZnSO}_4$  per litre;

$m_2$  is the mass, in grams, of the test portion.

## 8.2 Manganese (EDTA titrimetric method)

### 8.2.1 Reagents

**8.2.1.1 Ascorbic acid or hydroxylammonium chloride** ( $\text{OHNH}_2\text{Cl}$ ).

**8.2.1.2 Toluene.**

**8.2.1.3 2-Propanol.**

\* Listed in Colour Index as C.I. 14645. Trade names include Chrome Fast Black CAT, KIT & TS, Eriochrome Black DW, T and TDW, Potting Black C, etc.

**8.2.1.4 EDTA**, 0,100 mol/l standard volumetric solution. See 8.1.1.6.

**8.2.1.5 Zinc sulphate**, approximately 0,05 mol/l standard volumetric solution. See 8.1.1.7.

**8.2.1.6 Buffer solution**, pH 10. See 8.1.1.8.

**8.2.1.7 Mordant black 11**, indicator solution. See 8.1.1.9.

## 8.2.2 Procedure

Weigh, to the nearest 1 mg, a test portion of the drier containing approximately 80 mg of manganese into a 250 ml conical flask. Dissolve the test portion in 10 ml of the toluene (8.2.1.2) and dilute with 50 ml of the 2-propanol (8.2.1.3). Add approximately 0,100 g of the ascorbic acid or the hydroxylammonium chloride (8.2.1.1). Add successively 10 ml of the buffer solution (8.2.1.6) and 5 or 6 drops of the Mordant black 11 solution (8.2.1.7). Titrate with the EDTA solution (8.2.1.4) until the colour just changes from red to blue.

If desired, back titrate with the zinc sulphate solution (8.2.1.5) until the first change of the blue colour to red.

## 8.2.3 Expression of results

The manganese content, expressed as a percentage by mass of Mn, is given by the formula

$$\frac{[(V_4 \times T_0) - (V_5 \times T_1)] \times 0,054\ 93}{m_3} \times 100$$

$$= \frac{(V_4 \times T_0) - (V_5 - T_1)}{m_3} \times 5,493$$

where

$V_4$  is the volume, in millilitres, of the EDTA solution (8.2.1.4) required;

$V_5$  is the volume, in millilitres, of the zinc sulphate solution (8.2.1.5) required (if used);

$T_0$  is the concentration of the EDTA solution (8.2.1.4), in moles of EDTA per litre;

$T_1$  is the concentration of the zinc sulphate solution (8.2.1.5), in moles of  $ZnSO_4$  per litre;

$m_3$  is the mass, in grams, of the test portion.

## 8.3 Lead (EDTA titrimetric method)

### 8.3.1 Reagents

**8.3.1.1 Toluene.**

**8.3.1.2 2-Propanol.**

**8.3.1.3 Acetic acid**, about 4 mol/l solution.

**8.3.1.4 EDTA**, 0,100 mol/l standard volumetric solution. See 8.1.1.6.

**8.3.1.5 Zinc sulphate**, approximately 0,05 mol/l standard volumetric solution. See 8.1.1.7.

**8.3.1.6 Buffer solution**, pH 10. See 8.1.1.8.

**8.3.1.7 Mordant black 11**, indicator solution. See 8.1.1.9.

## 8.3.2 Procedure

Weigh, to the nearest 1 mg, a test portion of the drier containing 300 to 350 mg of lead into a 250 ml conical flask. Dissolve the test portion in 10 ml of the toluene (8.3.1.1) and dilute with 50 ml of the 2-propanol (8.3.1.2). Add 5 ml of the acetic acid solution (8.3.1.3) and 25 ml of the EDTA solution (8.3.1.4) by means of a pipette. Add successively 10 ml of the buffer solution (8.3.1.6) and 5 or 6 drops of the Mordant black 11 solution (8.3.1.7). Titrate with the zinc sulphate solution (8.3.1.5) until the first change of the blue colour to red.

## 8.3.3 Expression of results

The lead content, expressed as a percentage by mass of Pb, is given by the formula

$$\frac{[(25 \times T_0) - (V_6 \times T_1)] \times 0,207\ 2}{m_4} \times 100$$

$$= \frac{(25 \times T_0) - (V_6 \times T_1)}{m_4} \times 20,72$$

where

$T_0$  is the concentration of the EDTA solution (8.3.1.4), in moles of EDTA per litre;

$T_1$  is the concentration of the zinc sulphate solution (8.3.1.5), in moles of  $ZnSO_4$  per litre;

$V_6$  is the volume, in millilitres, of the zinc sulphate solution (8.3.1.5) required;

$m_4$  is the mass, in grams, of the test portion.

## 8.4 Zinc (EDTA titrimetric method)

### 8.4.1 Reagents

**8.4.1.1 Toluene.**

**8.4.1.2 2-Propanol.**

**8.4.1.3 EDTA**, 0,100 mol/l standard volumetric solution. See 8.1.1.6.

**8.4.1.4 Zinc sulphate**, approximately 0,05 mol/l standard volumetric solution. See 8.1.1.7.

**8.4.1.5 Buffer solution**, pH 10. See 8.1.1.8.

**8.4.1.6 Mordant black 11**, indicator solution. See 8.1.1.9.

**8.4.2 Procedure**

Weigh, to the nearest 1 mg, a test portion of the drier containing approximately 100 mg of zinc into a 250 ml conical flask. Dissolve the test portion in 10 ml of the toluene (8.4.1.1) and dilute with 50 ml of the 2-propanol (8.4.1.2). Add successively 10 ml of the buffer solution (8.4.1.5) and 5 or 6 drops of the Mordant black 11 solution (8.4.1.6). Titrate with the EDTA solution (8.4.1.3) until the colour just changes from red to blue.

If desired, back titrate with the zinc sulphate solution (8.4.1.4) until the first change of the blue colour to red.

**8.4.3 Expression of results**

The zinc content, expressed as a percentage by mass of Zn, is given by the formula

$$\frac{[(V_7 \times T_0) - (V_8 \times T_1)] \times 0,065\ 38}{m_5} \times 100$$

$$= \frac{(V_7 \times T_0) - (V_8 - T_1)}{m_5} \times 6,538$$

where

$V_7$  is the volume, in millilitres, of the EDTA solution (8.4.1.3) required;

$V_8$  is the volume, in millilitres, of the zinc sulphate solution (8.4.1.4) required (if used);

$T_0$  is the concentration of the EDTA solution (8.4.1.3), in moles of EDTA per litre;

$T_1$  is the concentration of the zinc sulphate solution (8.4.1.4), in moles of  $ZnSO_4$  per litre;

$m_5$  is the mass, in grams, of the test portion.

**8.5 Calcium (EDTA titrimetric method)**

**8.5.1 Reagents**

**8.5.1.1 Toluene.**

**8.5.1.2 2-Propanol.**

**8.5.1.3 Hydrochloric acid**, approximately 4 mol/l solution.

**8.5.1.4 EDTA**, 0,100 mol/l standard volumetric solution. See 8.1.1.6.

**8.5.1.5 Zinc sulphate**, approximately 0,05 mol/l standard volumetric solution. See 8.1.1.7.

**8.5.1.6 Buffer solution**, pH 10. See 8.1.1.8.

**8.5.1.7 Mordant black 11**, indicator solution. See 8.1.1.9.

**8.5.2 Procedure**

Weigh, to the nearest 1 mg, a test portion of the drier containing approximately 60 mg of calcium into a 250 ml conical flask. Dissolve the test portion in 10 ml of the toluene (8.5.1.1) and dilute with 50 ml of the 2-propanol (8.5.1.2). Add 3 ml of the hydrochloric acid solution (8.5.1.3). Add successively 10 ml of the buffer solution (8.5.1.6) and 5 or 6 drops of the Mordant black 11 solution (8.5.1.7). Titrate with the EDTA solution (8.5.1.4) until the colour just changes from red to blue.

If desired, back titrate with the zinc sulphate solution (8.5.1.5) until the first change of the blue colour to red.

**8.5.3 Expression of results**

The calcium content, expressed as a percentage by mass of Ca, is given by the formula

$$\frac{[(V_9 \times T_0) - (V_{10} \times T_1)] \times 0,040\ 08}{m_6} \times 100$$

$$= \frac{(V_9 \times T_0) - (V_{10} - T_1)}{m_6} \times 4,008$$

where

$V_9$  is the volume, in millilitres, of EDTA solution (8.5.1.4) required;

$V_{10}$  is the volume, in millilitres, of zinc sulphate solution (8.5.1.5) required (if used);

$T_0$  is the concentration of the EDTA solution (8.5.1.4), in moles of EDTA per litre;

$T_1$  is the concentration of the zinc sulphate solution (8.5.1.5), in moles of  $ZnSO_4$  per litre;

$m_6$  is the mass, in grams, of the test portion.

**8.6 Iron (iodometric method)**

NOTE — The method is suitable for the determination of up to 0,200 g of iron. It is not applicable in the presence of oxidizing materials and anions which combine with iron(III) ions to give complex compounds.

**8.6.1 Principle**

The drier is hydrolysed by boiling in sulphuric acid/ethanol/toluene medium. Any iron(II) is oxidized with cerium(IV) sulphate using ferroin as indicator. Iron(III) is reduced by iodide as follows :

