INTERNATIONAL STANDARD 510

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Red lead for paints

Minium pour peintures

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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 510 was drawn up by Technical Committee ISO/TC 35, *Paints and varnishes*, and was circulated to the member bodies in December 1974.

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

Australia	Iran	South Africa, Rep. of
Austria	Ireland	Spain
Brazil	Italy	Sweden
Bulgaria	Netherlands	Switzerland
Egypt, Arab Rep. of	New Zealand	Turkey
France	Poland	Yugoslavia
Germany	Portugal	
India	Romania	

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

United Kingdom

This International Standard cancels and replaces ISO Recommendation R 510-1966, of which it constitutes a technical revision.

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Red lead for paints

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the requirements and corresponding test methods for two types of red lead pigment, suitable for use in paints.

2 REFERENCES

ISO/R 150, *Raw, refined and boiled linseed oil.*

ISO 787, *General methods of test for pigments.*

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

3 DEFINITIONS

3.1 red lead : A red to orange-red pigment which consists of lead orthoplumbate (Pb_3O_4) and lead monoxide (PbO) and in which no impurities other than those arising from normal manufacturing processes are present.

3.2 non-setting red lead; high-percentage red lead : Red lead which, when mixed with linseed oil, does not cause undue thickening.

4 REQUIRED CHARACTERISTICS AND THEIR TOLERANCES

Red lead shall have the characteristics shown in the table.

5 SAMPLING

A representative sample of the pigment shall be taken in accordance with ISO 842.

TABLE — **Required characteristics and their tolerances***

Characteristic		Requirement according to type		Test method
		Non-setting red lead (High-percentage red lead)	High-dispersive red lead	
Lead dioxide	% (*m/m*)	min. 32,5	min. 33,5	Clause 6
Lead orthoplumbate	% (*m/m*)	min. 93,2	min. 96,0	Clause 8
Sum of lead orthoplumbate and free lead monoxide	% (*m/m*)	min. 99	min. 99	Clause 8
Matter volatile at 105 °C	% (*m/m*)	max. 0,2	max. 0,2	ISO 787, Part II
Matter soluble in water	% (*m/m*)	max. 0,3	max. 0,3	ISO 787, Part III
Residue on sieve (63 μm)**	% (*m/m*)	max. 0,75	max. 0,3	ISO 787, Part VI or VII
Oil absorption value		To be agreed between the interested parties		ISO 787, Part V
Sedimentation volume***	ml	***	min. 30***	Clause 9
Non-setting properties		After exposure in air for 14 days, the mixture described in clause 10 shall, after stirring, still be in a condition suitable for application by brush		Clause 10

* All percentages are calculated on the original sample.

** Two alternative methods for determination of residue on sieve are specified in ISO 787, identified respectively as the "oil method" and the "water method". Both methods are acceptable for the purpose of this International Standard, but it is recommended that the method to be used in a particular case be specified in the contract or otherwise agreed between the interested parties; it shall in any case be mentioned in the test report.

*** High-percentage red lead differs from high-dispersive red lead by, among other factors, having a lower sedimentation volume.

METHODS OF TEST

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

6 DETERMINATION OF LEAD DIOXIDE (PbO_2) CONTENT

6.1 Reagents

6.1.1 Sodium thiosulphate, 0,1 N solution.

6.1.2 Acetic acid, 300 g/l solution.

6.1.3 Sodium acetate, 600 g/l solution.

Dissolve 600 g of sodium acetate trihydrate ($C_2H_3O_2Na.3H_2O$) in water and make up to 1 l.

6.1.4 Iodine, 0,1 N standard volumetric solution.

6.1.5 Starch indicator solution.

Shake 10 g of soluble starch and 10 mg of mercury(II) iodide with about 30 ml of water to obtain a homogeneous suspension. Pour it into 1 l of boiling water. Boil the solution for 3 min and allow to cool.

6.2 Procedure

6.2.1 *Determination*

Weigh, to the nearest 1 mg, 0,5 to 0,8 g of the laboratory sample and transfer to a 250 ml conical flask. Add successively the following reagents :

25 ml of the sodium thiosulphate solution (6.1.1), accurately measured with a pipette,

25 ml of the sodium acetate solution (6.1.3),

20 ml of the acetic acid solution (6.1.2).

Stir gently to dissolve the test portion. Rub down coarse particles of the pigment by means of a glass rod flattened at the end (after this, carefully rinse the rod). If the last traces of the pigment are difficult to dissolve, the addition of not more than 0,5 g of potassium iodide is recommended. When the lead oxides are completely dissolved (except of course insoluble constituents, such as metallic lead particles, etc.), titrate the excess sodium thiosulphate with the standard volumetric iodine solution (6.1.4), using the starch solution (6.1.5) as indicator.

6.2.2 *Blank test*

In parallel with the determination, carry out a blank test following the same procedure and using the same quantities of all the reagents, but omitting the test portion.

6.3 Expression of results

The lead dioxide content, a, expressed as a percentage by mass of PbO_2, is given by the formula

$$a = 0{,}119\,6 \times 100 \times \frac{(V_2 - V_1)\,T}{m_1}$$

$$= \frac{11{,}96\,(V_2 - V_1)\,T}{m_1}$$

where

V_1 is the volume, in millilitres, of the standard volumetric iodine solution (6.1.4) used for the determination;

V_2 is the volume, in millilitres, of the standard volumetric iodine solution (6.1.4) used for the blank test;

T is the exact normality, in moles of H^+ per litre, of the standard volumetric iodine solution (6.1.4);

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

0,119 6 is the number of milligrams of lead dioxide equivalent to 1 ml of 1 N iodine solution.

7 DETERMINATION OF TOTAL LEAD CONTENT BY THE SULPHATE METHOD[1)]

7.1 Reagents

7.1.1 Hydrogen sulphide.

7.1.2 Hydrochloric acid, 3 M.

7.1.3 Nitric acid, 4 M.

7.1.4 Nitric acid, 4 M, saturated with bromine.

7.1.5 Sulphuric acid, 500 g/l solution.

7.1.6 Potassium hydroxide, 100 g/l solution.

7.1.7 Ammonium acetate, 335 g/l solution.

7.1.8 Sodium sulphide, 100 g/l solution.

7.1.9 Ethanol or **denatured spirit,** approximately 95 % (*V*/*V*).

7.1.10 Hydrogen peroxide, 30 g/l solution, free from sulphuric acid.

7.2 Procedure

7.2.1 Weigh, to the nearest 1 mg, 0,5 g of the laboratory sample and transfer it to a beaker of 400 ml nominal capacity. Add 10 ml of the nitric acid (7.1.3) and add the hydrogen peroxide solution (7.1.10), drop by drop while heating gently, until all the red lead has dissolved. Cover the beaker, boil the solution gently for 5 min to decompose any excess hydrogen peroxide and rinse the cover.

NOTE – Should the red lead be found to contain impurities, treat the contents of the beaker as follows :

Filter off any remaining material insoluble in nitric acid and wash the filter with hot water until free from soluble lead. Evaporate the filtrate to dryness. Add 2 ml of the hydrochloric acid (7.1.2), stir to mix and again evaporate to dryness on a water bath. Repeat this operation once more. Add a further 2 ml of the hydrochloric acid, followed by 200 ml of water. Boil the contents of the beaker to dissolve the lead chloride and pass in hydrogen sulphide (7.1.1), until cold. Filter the precipitate of lead sulphide on paper and wash with a saturated solution of hydrogen sulphide.

If antimony is present, wash the bulk of the precipitate back into the beaker and digest with 10 ml of the potassium hydroxide solution (7.1.6) and 10 ml of the sodium sulphide solution (7.1.8) for 10 min without boiling.

Again filter the lead sulphide onto the same paper and wash with the sodium sulphide solution diluted with 10 times its volume of water. Pierce the paper with a pointed glass rod and wash as much as possible of the lead sulphide down into the original beaker. Then dissolve the remaining lead sulphide from the paper with the nitric acid saturated with bromine (7.1.4) and warm the beaker to dissolve all the lead sulphide.

7.2.2 Add 20 ml of the sulphuric acid (7.1.5) to the solution and then evaporate gently without boiling, until copious fumes are evolved. Cool to room temperature, carefully add 100 ml of water followed by 100 ml of the ethanol (7.1.9) and allow to stand for 2 h.

1) This method should be used as the referee method.

By agreement between the interested parties, other methods such as the chromate method specified in ISO 511, *White lead for paints*, or an EDTA method may be used.

Transfer the precipitate to a weighed Gooch crucible packed with asbestos, or to a sintered silica crucible of porosity grade P16, i.e. pore size index 10 to 16 μm, and wash with ethanol (7.1.9). Heat the crucible, gently at first and then to 500 °C for 10 min, cool and weigh.

Pour hot ammonium acetate solution (7.1.7) onto the filter to extract the lead sulphate completely. Wash the residue with hot water, dry, heat to 500 °C for 10 min, cool and reweigh. The difference between these two weighings is the mass of lead sulphate.

7.3 Expression of results

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

$$b = \frac{0{,}683\ 2\ (m_5 - m_6)}{m_4} \times 100 = \frac{68{,}32\ (m_5 - m_6)}{m_4}$$

where

m_4 is the mass, in grams, of the test portion;

m_5 is the mass, in grams, of the first precipitate;

m_6 is the mass, in grams, of the residue after extraction with ammonium acetate;

0,683 2 is the factor for the conversion of lead sulphate to lead.

8 CALCULATION OF LEAD ORTHOPLUMBATE CONTENT AND OF SUM OF FREE LEAD MONOXIDE AND LEAD ORTHOPLUMBATE CONTENTS

The lead orthoplumbate content, c, expressed as a percentage by mass of Pb_3O_4, is given by the formula

$$c = 2{,}866\ a$$

The sum of the free lead monoxide (PbO) and the lead orthoplumbate (Pb_3O_4) contents, expressed as a percentage by mass, is given by the formula

$$1{,}077\ (b - 2{,}599\ a) + c$$

where

a is the lead dioxide content, expressed as a percentage by mass, determined by the method specified in clause 6;

b is the total lead content, expressed as a percentage by mass, determined by the method specified in clause 7.

9 DETERMINATION OF THE SEDIMENTATION VOLUME

9.1 Reagent

Ethanol, 95 % (V/V), ρ_{20} 0,811 4 g/ml, not denatured.

9.2 Apparatus

Glass-stoppered graduated cylinder, of capacity 50 ml, internal height to 50 ml graduation line 150 ± 3 mm.

9.3 Procedure

Weigh 50 ± 0,1 g of the laboratory sample into the cylinder (9.2), add 35 ml of the ethanol (9.1), and shake the mixture for exactly 15 min.

After this period, make up to the 50 ml mark with the ethanol. Allow to stand for 24 h at ambient temperature. After 24 h, read the sedimentation volume to the nearest millilitre.

10 DETERMINATION OF NON-SETTING PROPERTIES

10.1 Reagent

Linseed oil, acid-refined, of acid value between 4 and 5 mg of KOH/g, complying with ISO/R 150.

10.2 Apparatus

Cylindrical tin, of capacity approximately 150 ml and diameter approximately 65 mm.

10.3 Procedure

Prepare at least 150 ml of paint by thoroughly grinding sufficient of the laboratory sample in the linseed oil, (10.1) to form a paste containing 8 to 10 % (m/m) of oil, and further thinning the paste with the same oil to paint consistency. Pour the paint into the tin so that it is filled to within approximately 12 mm of the top. Without placing a lid on the tin, leave the paint exposed to the air for 14 days at ambient temperature. At the end of this period, stir the paint and note whether it has thickened unduly. Apply the stirred paint to a non-absorbent surface and note whether it is still suitable for application by brush.

11 TEST REPORT

The test report shall contain at least the following information :

a) a reference to this International Standard or to a corresponding national standard;

b) the type and identification of the product tested;

c) the results of the tests and whether or not the product tested complies with the relevant specification limits;

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

e) the date of the tests.