UDC 667.622

Ref. No.: ISO / R 510 - 1966 (E)

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 510

RED LEAD

1st EDITION

October 1966

For each individual country the only valid standard is the national standard of that country.

Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The ISO Recommendation R 510, *Red Lead*, was drawn up by Technical Committee ISO/TC 35, *Paints, Varnishes and Related Products and their Raw Materials*, the Secretariat of which is held by the Nederlands Normalisatie-instituut (NNI).

Work on this question by the Technical Committee began in 1950 taking into account the studies which had been made by the former International Federation of the National Standardizing Associations (ISA) and led, in 1954, to the adoption of a Draft ISO Recommendation.

This first Draft ISO Recommendation (No. 32) was circulated to all the ISO Member Bodies for enquiry in March 1954. As the results of this consultation were not considered satisfactory, the Technical Committee successively put forward a second and a third Draft ISO Recommendation, which were circulated in August 1961 and in November 1963 respectively.

This third Draft was approved, subject to a few modifications of an editorial nature, by the following Member Bodies:

Argentina	Denmark	Portugal
Australia	France	Spain
Austria	Germany	Sweden
Belgium	India	Turkey
Bulgaria	Indonesia	U.A.R.
Canada	Italy	United Kingdom
Chile	Japan	U.S.S.R.
Colombia	Morocco	Yugoslavia
Czechoslovakia	Netherlands	

No Member Body opposed the approval of the Draft.

The third Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in October 1966, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 510 October 1966

RED LEAD

1. SCOPE

This ISO Recommendation establishes the more important requirements for three qualities of red lead and the methods of test for these requirements.

2. DEFINITION

Red lead. A red to orange-red pigment which consists of lead orthoplumbate (Pb_3O_4) and lead monoxide (PbO) in which no impurities other than those resulting during the course of normal manufacture are present.

3. REQUIRED CHARACTERISTICS AND THEIR TOLERANCES *

Red lead should have the following characteristics:

Property		Ordinary red lead	High percentage red lead (non-setting)	Dispersed red lead	Clause describing test method
Lead dioxide, min.	%	26	32.5	33.5	5.1
Lead orthoplumbate, min.	%	74.5	93.2	96.0	5.4
Sum of lead orthoplumbate and free lead monoxide, min.	%	99	99	99	5.4
Matter volatile at 105 °C, max.	%	0.3	0.3	0.3	5.5
Matter soluble in water, max.	%	0.3	0.3	0.3	5.6
Residue on sieve, max.	%	1.5	0.75	0.75	5.7
Oil absorption value		To be agreed between purchaser and vendor			5.8
Sedimentation volume, ** min.	ml	—	**	30 **	5.9
Non-setting properties		—	After exposure in air for 14 days, the mixture described in clause 5.10 should still be in a condition suitable for application by brush.		5.10

* All percentages are calculated from the original sample.

** High-percentage red lead and dispersed red lead differ, among other things, by the fact that the sedimentation volume of the latter is higher than that of the former.

4. SAMPLING

See ISO Recommendation R. . . , *Sampling Raw Materials for Paints and Varnishes.**

5. TEST METHODS

5.1 Determination of lead dioxide (PbO_2) content

5.1.1 *Reagents*

5.1.1.1 *Sodium thiosulphate solution* 0.1 N.

5.1.1.2 *Standard iodine solution* 0.1 N.

5.1.1.3 *Starch solution* for indicator. Shake 10 g of soluble starch and 10 mg of mercuric iodide with about 30 ml of water to obtain a homogeneous suspension. Pour it into 1 litre of boiling water. Boil the solution for 3 min and after that allow to cool.

5.1.1.4 *Acetic acid solution*, 30 g/100 ml.

5.1.1.5 *Sodium acetate solution.* Dissolve 600 g of crystallized sodium acetate hydrate ($C_2H_3O_2Na.3H_2O$) in distilled water and make up to 1 litre.

5.1.2 *Procedure*

Weigh to the nearest 1 mg, 0.5 to 1.0 g of red lead and transfer to a 250 ml conical flask. Add successively the following reagents:

30 ml of the sodium thiosulphate solution (accurately measured),

25 ml of the sodium acetate solution,

20 ml of the acetic acid solution.

Stir gently to dissolve the red lead in the liquid. Rub down coarse particles of the pigment by means of a glass rod flattened at the end (after this, the rod is carefully rinsed). If the last traces of the pigment are difficult to dissolve, a little potassium iodide may be added. The addition of not more than 0.5 g of potassium iodide is recommended. When the lead oxides are completely dissolved (of course with the exception of insoluble constituents, such as, e.g., metallic lead particles, etc.), titrate the excess of sodium thiosulphate with the standard iodine solution (0.1 N), using the starch solution as an indicator.

At the same time perform a blank determination.

The difference between the consumed quantities of iodine solution is proportional to the amount of lead dioxide (PbO_2), i.e. lead orthoplumbate (Pb_3O_4).

* At present Second Draft ISO Recommendation No. 731.

5.1.3 *Expression of results*

The content of lead dioxide (PbO_2) of red lead (A), expressed as a percentage by mass, is given by the following formula:

$$A = \frac{1.196\ (V_2 - V_1)}{m_1}$$

where

V_2 = volume in millilitres of 0.1 N iodine solution required by the blank test;
V_1 = volume in millilitres of 0.1 N iodine solution required by the test sample;
m_1 = mass in grammes of the test sample.

5.2 Determination of total lead content by the chromate method *

5.2.1 *Reagents*

5.2.1.1 *Nitric acid*, 4 N.

5.2.1.2 *Acetic acid solution*, 2 g/100 ml.

5.2.1.3 *Congo paper.*

5.2.1.4 *Ammonium acetate solution* 2 M, freshly prepared.

5.2.1.5 *Hydrogen peroxide solution*, 3 g/100 ml, free from sulphuric acid.

5.2.1.6 *Potassium dichromate solution*, 5 g/100 ml.

5.2.2 *Procedure*

Weigh to the nearest 1 mg, 0.5 to 1.0 g red lead and transfer to a 500 ml conical flask. First add 10 ml of the nitric acid and then, drop by drop, hydrogen peroxide solution, until the red lead is dissolved completely. Evaporate this solution to dryness to expel the excess of hydrogen peroxide.

Dissolve the residue in as little nitric acid as possible and add to this solution the ammonium acetate solution until the liquid gives no acid reaction with the Congo paper.

Filter insoluble matter, if present, and wash thoroughly with the ammonium acetate solution. Dilute the filtrate, combined with the washings, with water to about 200 ml and then heat to boiling. Precipitate, from the boiling liquid, lead as lead chromate ($PbCrO_4$) by adding an excess of the potassium dichromate solution. Keep the liquid boiling until the precipitate has turned dark orange-red and then keep heated (on the water bath) for 1½ to 2 hours. After cooling, filter the precipitate on a weighed sintered glass filter crucible of porosity No. 4 (i.e. of maximum pore diameter 5-15 μm), wash with the acetic acid solution, then with hot water, and dry in a drying oven (100 ± 2 °C) or a vacuum desiccator to constant mass.

5.2.3 *Expression of results*

The content of total lead (Pb) of red lead (B) expressed as a percentage by mass, is given by the following formula:

$$B = \frac{63.75\ m_3}{m_2}$$

where

m_3 = mass in grammes of the dried residue of lead chromate;
m_2 = mass in grammes of the test sample.

* This method should be used when the pigment is known to be free from adulteration.

5.3 Determination of total lead content by the sulphate method *

5.3.1 *Reagents*

5.3.1.1 *Hydrochloric acid* 3 N.

5.3.1.2 *Nitric acid* 4 N.

5.3.1.3 *Nitric acid* 4 N, saturated with bromine.

5.3.1.4 *Sulphuric acid*, 50 g/100 ml.

5.3.1.5 *Potassium hydroxide solution*, 10 g/100 ml.

5.3.1.6 *Ammonium acetate solution*, 33.5 g/100 ml.

5.3.1.7 *Hydrogen peroxide solution*, 3 g/100 ml.

5.3.1.8 *Sodium sulphide solution*, 10 g/100 ml.

5.3.1.9 *Ethanol or denatured spirit*, approximately 95 % v/v.

5.3.1.10 *Hydrogen sulphide.*

5.3.2 *Procedure*

5.3.2.1 Weigh to the nearest 1 mg 0.5 g of red lead (m_4 g) and transfer to a 400 ml beaker capacity. Add 10 ml of the nitric acid 4 N, cover the beaker and add the hydrogen peroxide solution drop by drop with gentle heating until all the red lead has dissolved. Boil the solution gently for 5 min to decompose any excess hydrogen peroxide and rinse the cover.

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

Filter off any material remaining 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, 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 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 potassium hydroxide solution and 10 ml of sodium sulphide solution 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 nitric acid saturated with bromine and warm the beaker to dissolve all the lead sulphide into the solution.

5.3.2.2 Add 20 ml of the sulphuric acid to the solution and then evaporate gently without boiling, until copious fumes are evolved. To the contents of the beaker add 100 ml of water followed by 100 ml of ethanol and allow to stand for 2 hours.

Transfer the precipitate to a weighed Gooch crucible packed with asbestos (alternatively, a sintered silica crucible of No. 4 porosity, i.e. of maximum pore diameter 5-15 μm, may be used) and wash with ethanol. Heat the crucible, gently at first and then to 500 °C for 10 min, cool and weigh (m_5 g).

* This method should be used when the purity of the pigment is unknown.