SLOVENSKI STANDARD
SIST ISO 6503:1996

01-maj-1996

Barve in laki - Določanje skupne vsebnosti svinca - Plamenska atomska absorpcijska spektrometrijska metoda

Paints and varnishes -- Determination of total lead -- Flame atomic absorption spectrometric method

Peintures et vernis -- Détermination du plomb total -- Méthode par spectrométrie d'absorption atomique dans la flamme

Ta slovenski standard je istoveten z: **ISO 6503:1984**

ICS:

87.040	Barve in laki	Paints and varnishes

SIST ISO 6503:1996 **en**

International Standard 6503

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

Paints and varnishes — Determination of total lead — Flame atomic absorption spectrometric method

Peintures et vernis — Détermination du plomb total — Méthode par spectrométrie d'absorption atomique dans la flamme

First edition — 1984-10-15

UDC 667.6 : 543.422 : 546.815 **Ref. No. ISO 6503-1984 (E)**

Descriptors : paints, varnishes, chemical analysis, determination of content, lead, atomic absorption method.

Price based on 6 pages

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 6503 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*.

Printed in Switzerland

Contents

This page intentionally left blank

Paints and varnishes — Determination of total lead — Flame atomic absorption spectrometric method

1 Scope and field of application

This International Standard describes a flame atomic absorption spectrometric method for the determination of total lead in paints and related products.

The method is applicable to products having total lead contents in the range of about 0,01 to 2 % (m/m).

NOTE — This method may also be applicable to products with a total lead content of more than 2 % (m/m), but it should only be used when the precision does not exceed the appropriate values given in 7.2.

Two methods are given for the treatment of the test portion; the dry ashing method (clause 4) should be used as the referee method in cases of dispute.

For the determination of lead in the test solution, the dithizone spectrophotometric method specified in ISO 3856/1 may be used as an alternative method.

2 References

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

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

ISO 1512, *Paints and varnishes — Sampling.*

ISO 1513, *Paints and varnishes — Examination and preparation of samples for testing.*

ISO 3696, *Water for laboratory use — Specifications.*[2]

ISO 3856/1, *Paints and varnishes — Determination of "soluble" metal content — Part 1 : Determination of lead content — Flame atomic absorption spectrometric method and dithizone spectrophotometric method.*

ISO 5725, *Precision of test methods — Determination of repeatability and reproducibility by inter-laboratory tests.*

3 Principle

Decomposition of a test portion by either the dry ashing method (clause 4) or the wet oxidation method (clause 5), and determination of the lead by flame atomic absorption spectrometry.

4 Dry ashing method

4.1 Principle

Evaporation of a test portion to dryness and ashing at 475 °C to remove all organic matter. Extraction of any lead in the residue with hydrochloric acid.

4.2 Reagents

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

4.2.1 Sodium carbonate, anhydrous.

4.2.2 Magnesium carbonate.

4.2.3 Sulfur.

4.2.4 Liquid paraffin.

4.2.5 Sodium sulfide, 10 g/l solution.

4.2.6 Hydrochloric acid, approximately 180 g/l.

Add 450 ml of concentrated hydrochloric acid [36 % (m/m), ϱ approximately 1,18 g/ml] to an approximately equal amount of water and dilute to 1 000 ml.

4.2.7 Hydrochloric acid, approximately 18 g/l.

Add 100 ml of the hydrochloric acid (4.2.6) to water and dilute to 1 000 ml.

---

[1] At present at the stage of draft. (Partial revision of ISO/R 385-1964.)

[2] At present at the stage of draft.

4.2.8 Nitric acid, approximately 315 g/l.

Add 1 volume of concentrated nitric acid [about 65 % (*m*/*m*), ϱ approximately 1,40 g/ml] to 2 volumes of water.

4.2.9 Lead, standard stock solution containing 1 g of Pb per litre.

Either

a) transfer the contents of an ampoule of standard lead solution containing exactly 1 g of Pb into a 1 000 ml one-mark volumetric flask containing some water and 30 ml of the nitric acid (4.2.8), dilute to the mark with water and mix well;

or

b) weigh, to the nearest 1 mg, 1,598 g of lead nitrate [$Pb(NO_3)_2$] (previously dried for 2 h at 105 °C), dissolve in water in a 1 000 ml one-mark volumetric flask, add 30 ml of the nitric acid (4.2.8), dilute to the mark with water and mix well.

1 ml of this standard stock solution contains 1 mg of Pb.

4.2.10 Lead, standard solution containing 100 mg of Pb per litre.

Prepare this solution on the day of use.

Pipette 10 ml of the standard stock solution (4.2.9) into a 100 ml one-mark volumetric flask, dilute to the mark with the hydrochloric acid (4.2.7) and mix well.

1 ml of this standard solution contains 100 µg of Pb.

4.3 Apparatus

Ordinary laboratory apparatus and

4.3.1 Crucibles, silica, preferably new.

4.3.2 Muffle furnace, capable of being maintained at 475 ± 25 °C.

4.3.3 Hotplate, with energy regulation control.

4.4 Sampling

Take a representative sample of the product to be tested by the method described in ISO 1512.

Examine and prepare the sample for testing as described in ISO 1513.

4.5 Procedure

4.5.1 Preliminary tests

If the composition of the product to be tested is not known, carry out qualitative tests for the presence of cellulose nitrate and antimony. If the results of these tests do not confirm the absence of cellulose nitrate and antimony, carry out the full procedure.

4.5.2 Test portion

Carry out the procedure in duplicate.

Mix the sample thoroughly and immediately transfer about 5 g into a weighed silica crucible (4.3.1). Weigh the test portion to the nearest 10 mg.

If the product contains cellulose nitrate (see 4.5.1), mix about 2 g of the liquid paraffin (4.2.4) with the test portion in the crucible.

4.5.3 Ashing

Place the crucible and its contents on the hotplate (4.3.3) in a fume cupboard. Gradually increase the temperature of the hotplate to remove all volatile solvents.

Spread 2 g of the magnesium carbonate (4.2.2) over the contents of the crucible and, over a period of 10 min, progressively introduce the crucible into the muffle furnace (4.3.2) at approximately 350 °C. Raise the temperature of the furnace to 475 ± 25 °C over a further period of 60 min and maintain at this temperature until ashing is complete. Ensure that an adequate supply of air for oxidation is available.

Do not allow the material in the crucible to ignite at any stage.

4.5.4 Extraction

4.5.4.1 If the material does not contain antimony (see 4.5.1), proceed as follows.

Allow the crucible and ash (see 4.5.3) to cool. Transfer the crucible and ash to a 250 ml beaker, add 100 ml of the hydrochloric acid (4.2.6) and, using the hotplate (4.3.3), boil gently for 15 min. Then allow to digest for a further 15 min.

While still hot, filter by decantation through a fine-texture filter paper 1), into a 250 ml beaker. Wash the paper and residue with hot water, collecting the washings in the beaker. Cool the beaker and transfer the filtrate and washings to a 250 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

---

1) The following filter papers have been found to be suitable :

Whatman Nos. 42 and 44
Schleicher and Schüll Nos. 589/3 and 589/6.

4.5.4.2 If the material contains antimony (see 4.5.1), proceed as follows.

Grind the ash (see 4.5.3) to a fine powder, replace it in the same crucible and mix it with approximately 10 g of a mixture of equal parts of the sodium carbonate (4.2.1) and the sulfur (4.2.3). Cover the crucible and heat it over a moderate flame until there is no odour of sulfur dioxide; this should take 1 to 2 h.

Cool the crucible and digest the contents with a small quantity of hot water until the melt is completely broken up. Filter, transferring all the residue to a filter paper with the sodium sulfide solution (4.2.5) and wash the residue with the sodium sulfide solution. Reject the filtrate.

Transfer the filter paper and residue to a 250 ml beaker. Add 15 ml of the nitric acid (4.2.8) and, using the hotplate (4.3.3), boil gently for 15 min. Add 100 ml of the hydrochloric acid (4.2.6) and allow to digest for 30 min. While still hot, filter through a fine-texture filter paper into a 250 ml beaker. Wash the paper and residue with hot water, collecting the washings in the beaker. Cool the beaker and transfer the filtrate and washings to a 250 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

4.5.5 Preparation of test solutions

From each extract solution (4.5.4) take an aliquot portion, of size determined by the expected lead content in the sample, in accordance with table 1.

Table 1

Expected lead content	Aliquot portion
% (m/m)	ml
less than 0,4	25
0,4 to 1	10
1 to 2	5

NOTE — If the lead content is higher than 2 % (m/m), a suitable aliquot portion should be taken.

Place the aliquot portion in a 100 ml one-mark volumetric flask and, if a 5 ml or 10 ml aliquot portion is taken, add 10 ml of the hydrochloric acid (4.2.6). Dilute to the mark with water and mix well.

4.5.6 Preparation of reagent blank

Repeat the procedures described in 4.5.3, 4.5.4.1 or 4.5.4.2 and 4.5.5, but omitting the test portion.

5 Wet oxidation method

5.1 Principle

Wet oxidation of a test portion with a mixture of sulfuric acid and hydrogen peroxide in a beaker (method A) or with a mixture of sulfuric acid and nitric acid in a Kjeldahl flask (method B) to remove all organic matter. Heating to remove the excess sulfuric acid, and extraction of any lead (in the form of lead sulfate) in the residue with EDTA and ammonia solution.

NOTE — The presence of antimony or cellulose nitrate in a sample does not cause interference with this method.

5.2 Reagents

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

5.2.1 Sulfuric acid, about 96 % (m/m), ϱ approximately 1,84 g/ml.

5.2.2 Hydrogen peroxide, approximately 30 % (m/m), or, with the appropriate additional safety precautions, approximately 50 % (m/m) solution.

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

5.2.4 Nitric acid, approximately 315 g/l.

Add 1 volume of the nitric acid (5.2.3) to 2 volumes of water.

5.2.5 Ammonia solution, approximately 85 g NH_3/l solution.

Dilute 380 ml of concentrated ammonia solution [25 % (m/m)] to 1 000 ml with water.

5.2.6 EDTA (Ethylenediaminetetraacetic acid, disodium salt), 37 g/l solution.

5.2.7 Lead, standard stock solution containing 1 g of Pb per litre.

Either

a) tranfer the contents of an ampoule of standard lead solution containing exactly 1 g of Pb into a 1 000 ml one-mark volumetric flask containing some water and 30 ml of the nitric acid (5.2.4), dilute to the mark with water and mix well;

or

b) weigh to the nearest 1 mg, 1,598 g of lead nitrate $[Pb(NO_3)_2]$ (previously dried for 2 h at 105 °C), dissolve in water in a 1 000 ml one-mark volumetric flask, add 30 ml of the nitric acid (5.2.4), dilute to the mark with water and mix well.

1 ml of this standard stock solution contains 1 mg of Pb.

5.2.8 Lead, standard solution containing 100 mg of Pb per litre.

Prepare this solution on the day of use.