

Australian/New Zealand Standard®

Paints and related materials—Methods of test

Method 501.1: Soluble lead content

PREFACE

This Standard was prepared by the Joint Australia/New Zealand Standards Committee CH/3 on Paints and Related Materials, under the direction of the Multitechnics Standards Policy Board, to supersede the 1980 edition of AS 1580.501.1, *Methods of test for paints, varnishes, lacquers and related materials*, Method 501.1: *Soluble lead content (gravimetric method)*.

This Standard is identical with, and has been reproduced from, ISO 3856/1—1984, *Paints and varnishes—Determination of 'soluble' metal content*, Part 1: *Determination of lead content—Flame atomic absorption spectrometric method and dithizone spectrophotometric method*.

Under arrangements made between Standards Australia/Standards New Zealand and the international Standards bodies, ISO and IEC, as well as certain other Standards organizations, users of this Joint Standard are advised of the following:

- (a) In Australia, copyright is vested in Standards Australia. In New Zealand, copyright is vested in Standards New Zealand.
- (b) For the purpose of this Joint Standard, the text of ISO 3856/1 should be modified as follows:
 - (i) *Substitute* a fullpoint (.) for a coma (,) wherever it appears as a decimal marker.
 - (ii) *Replace* reference to other publications by references to Australian/New Zealand Standards as follows:

Reference to International Standard		Australian/New Zealand Standard	
ISO		AS	
385/1	Laboratory glassware—Burettes— Part 1: General requirements	2165	Burettes and bulb burettes
648	Laboratory glassware—One-mark pipettes	2166	One-mark pipettes
1042	Laboratory glassware—One-mark volumetric flasks	2164	One-mark volumetric flasks
4800	Laboratory glassware—separating funnels and dropping funnels	2407	Separating funnels and dropping funnels
		AS/NZS	
6713	Paints and varnishes—Preparation of acid extracts from paints in liquid or powder form	1580	Paints and related materials Methods of test
		1580.501.2	Method 501.2: Preparation of acid extracts

- (iii) *Delete* Clause 0.
- (iv) In Clause 1 and Clause 5, *substitute* AS/NZS 1580.501.1 for ISO 3856.
- (v) *Delete* the existing text of Clause 3.2 and 4.2 and *substitute* the following:
'During analysis, use only reagents of recognized analytical grade. Only distilled or deionized water shall be used.'

0 Introduction

This International Standard is a part of ISO 3856, *Paints and varnishes — Determination of "soluble" metal content*.

1 Scope and field of application

This part of ISO 3856 describes two methods for the determination of the lead content of the test solutions, prepared according to ISO 6713 or other suitable International Standards.

The methods are applicable to paints having "soluble" lead contents in the range of about 0,05 to 5 % (m/m).

The flame atomic absorption spectrometric (AAS) method (clause 3) should be used as the referee method in cases of dispute. Other methods can be used by agreement between the interested parties. If a spectrophotometric method is agreed, the dithizone spectrophotometric method (clause 4) should be used.

2 References

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

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

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

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

ISO 4800, *Laboratory glassware — Separating funnels and dropping funnels*.

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

3 Flame atomic absorption spectrometric method

3.1 Principle

Aspiration of the test solution into an acetylene/air flame. Measurement of the absorption of the selected spectral line, emitted by a lead hollow-cathode or lead discharge lamp, in the region of 283,3 nm.

3.2 Reagents and materials

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

3.2.1 Hydrochloric acid, $c(\text{HCl}) = 0,07 \text{ mol/l}$.

Use the hydrochloric acid, identical to that used for the preparation of the test solutions in accordance with ISO 6713. (See 3.4.2.)

3.2.2 Acetylene, commercial grade, in a steel cylinder.

3.2.3 Compressed air.

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

2) At present at the stage of draft.

3.2.4 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, dilute to the mark with the hydrochloric acid (3.2.1) and mix well;

or

b) weigh, to the nearest 1 mg, 1,598 g of lead nitrate [Pb(NO₃)₂] (previously dried for 2 h at 105 °C), dissolve in the hydrochloric acid (3.2.1) in a 1 000 ml one-mark volumetric flask, dilute to the mark with the same hydrochloric acid and mix well.

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

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

Prepare this solution on the day of use.

Pipette 100 ml of the standard stock solution (3.2.4) into a 1 000 ml one-mark volumetric flask, dilute to the mark with the hydrochloric acid (3.2.1) and mix well.

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

3.3 Apparatus

Ordinary laboratory apparatus and

3.3.1 Flame atomic absorption spectrometer, suitable for measurements at a wavelength of 283,3 nm and fitted with a burner fed with acetylene and air.

3.3.2 Lead hollow-cathode lamp or lead discharge lamp.

3.3.3 Burette, of capacity 50 ml, complying with the requirements of ISO 385/1.

3.3.4 One-mark volumetric flasks, of capacity 100 ml, complying with the requirements of ISO 1042.

3.4 Procedure

3.4.1 Preparation of the calibration graph

3.4.1.1 Preparation of the standard matching solutions

Prepare these solutions on the day of use.

Into a series of six 100 ml one-mark volumetric flasks (3.3.4), introduce from the burette (3.3.3), respectively, the volumes of the standard lead solution (3.2.5) shown in the following table, dilute each to the mark with the hydrochloric acid (3.2.1) and mix well.

Standard matching solution No.	Volume of the standard lead solution (3.2.5)	Corresponding concentration of Pb in the standard matching solution
	ml	µg/ml
0 *	0	0
1	2,5	2,5
2	5	5
3	10	10
4	20	20
5	30	30

* Blank matching solution.

3.4.1.2 Spectrometric measurements

Install the lead spectral source (3.3.2) in the spectrometer (3.3.1) and optimize the conditions for the determination of lead. Adjust the instrument in accordance with the manufacturer's instructions and adjust the monochromator to the region of 283,3 nm in order to obtain the maximum absorbance.

Adjust the flow of the acetylene (3.2.2) and of the air (3.2.3) according to the characteristics of the aspirator-burner, and ignite the flame. Set the scale expansion, if fitted, so that the standard matching solution No. 5 (see table) gives almost a full-scale deflection.

Aspirate into the flame each of the standard matching solutions (see 3.4.1.1) in ascending order of concentration, and repeat with the standard matching solution No. 4 to verify that the instrument has achieved stability. Aspirate water through the burner between each measurement, taking care to keep the rate of aspiration uniform.

3.4.1.3 Calibration graph

Plot a graph having the masses, in micrograms, of Pb contained in 1 ml of the standard matching solutions as abscissae and the corresponding values of the absorbances, reduced by the reading for the blank matching solution, as ordinates.

3.4.2 Test solutions

3.4.2.1 Pigment portion of the liquid paint and paint in powder form

Use the solutions obtained by the procedure described in sub-clause 8.2.3 or 8.3.2 of ISO 6713, as appropriate.

3.4.2.2 Liquid portion of the paint

Use the solutions obtained by the procedure described in sub-clause 9.3 of ISO 6713.

3.4.2.3 Other test solutions

Use the solutions obtained by other specified or agreed procedures (for example, by the procedure described in sub-clause 8.3.1.4 of ISO 6713).