
Aluminium pigments for paints —
Part 1:
General aluminium pigments

Pigments d'aluminium pour peintures —
Partie 1: Pigments d'aluminium généraux

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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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 256, *Pigments, dyestuff and extenders*.

This first edition of ISO 1247-1, together with ISO 1247-2, cancels and replaces ISO 1247:1974, which has been technically revised. It also incorporates the Amendment ISO 1247:1974/Amd 1:1982.

The main changes compared to the previous edition are as follows:

- an introduction has been added, giving the reasons for the division;
- [Clause 3](#), Terms and definitions, has been added and the terms “non-volatile-matter” and “hiding power” have been included;
- a distinction between surface-treated and surface-untreated aluminium pigments has been introduced in [Clause 4](#) and [5](#);
- former subclause 4.2, Classes, has been deleted;
- “matter volatile at 105 °C” has been substituted by “non-volatile-matter”;
- former Clause 6, Packing, has been deleted;
- [Clause 7](#), Sampling, has been reduced to a reference to ISO 15528;
- the requirements and test methods of “pigment specific surface area”, “particle size distribution” and “hiding power” and the corresponding test methods have been added to [Table 1](#) and [Table 1](#) has been renamed “Requirements and test methods”;
- the test method for “manganese” has been deleted from the metallic impurities in [Table 1](#);
- the flame atomic absorption spectrometric method has been introduced to determine metal impurities;
- former Clause 13, Test for absence of leaching power, has been deleted;

- Table 3, test portions, has been deleted;
- [Clause 16](#), Determination of hiding power, has been added;
- the normative references have been updated and the text has been editorially revised.

A list of all parts in the ISO 1247 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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Introduction

Since the beginning of the new millennium, with the progress in technology and product development, there is a boom of new types of aluminium pigments which, serving as heat-resistant or rust-proof coatings or as colorants, find their ways into applications in a wide range of industrial sectors. Aluminium pigments can be conveniently divided into two groups according to their forms: general aluminium pigments produced in a milling process and vacuum metallized aluminium pigments (VMP) (see ISO 1247-2). As the technical requirements are different for aluminium pigments used in different fields, it is necessary to develop an international standard for each of the two groups.

To meet the needs in the marketing of aluminium pigments, characteristics such as “particle size distribution” and “hiding power”, as well as their corresponding testing methods, are introduced in this document.

To improve the safety of the aluminium pigments, the organic solvents and auxiliary agents of low flashpoints have been substituted by reagents with high flashpoints, the “matter volatile at 105 °C” has been replaced by “non-volatile-matter”, for which testing methods are developed according to ISO 3251.

The previous edition (i.e. ISO 1247:1974) uses different spectrophotometric methods to determine the content of lead, iron and copper, and the ethylenediamine tetraacetic acid (EDTA) titration method to determine zinc. These methods are cumbersome to operate. The method used to determine lead involves the use of potassium cyanate, a highly toxic agent seldom used since the 1990s, and the method used to determine zinc is applicable only to samples with a total zinc content of 0,10 % or above, and does not suit the status quo of the aluminium pigment industry. While these old methods are retained in this document, the flame atomic absorption spectrometric method is introduced as a new option for the determination of the above-mentioned metallic impurities. This method is faster, operation-friendly, has a wider range of detection, and is gaining more and more popularity.

To use as little toxic reagent as possible, this document resorts to using 2-Butoxyethanol or n-butyl acetate to replace acetone, which, as material likely to be used for making illegal drugs, is now forbidden or restricted by many countries.

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Aluminium pigments for paints —

Part 1: General aluminium pigments

1 Scope

This document specifies the requirements and corresponding test methods for aluminium pigments suitable for use in paints including:

- a) general, decorative and protective paints, and
- b) special finishing paints.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 385, *Laboratory glassware — Burettes*

ISO 648, *Laboratory glassware — Single-volume pipettes*

ISO 793, *Aluminium and aluminium alloys — Determination of iron — Orthophenanthroline photometric method*

ISO 795, *Aluminium and aluminium alloys — Determination of copper content — Oxalyldihydrazide photometric method*

ISO 808, *Aluminium and aluminium alloys — Determination of silicon — Spectrophotometric method with the reduced silicomolybdic complex*

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

ISO 1784, *Aluminium alloys — Determination of zinc — EDTA titrimetric method*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 9277, *Determination of the specific surface area of solids by gas adsorption — BET method*

ISO 13320, *Particle size analysis — Laser diffraction methods*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

ISO 18451-1, *Pigments, dyestuffs and extenders — Terminology — Part 1: General terms*

ISO 18451-2, *Pigments, dyestuffs and extenders — Terminology — Part 2: Classification of colouring materials according to colouristic and chemical aspects*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18451-1, ISO 18451-2 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.1

non-volatile matter

NV

residue by mass obtained by evaporation under specified conditions

Note 1 to entry: Instead of the term “non-volatile matter” different terms, such as solid, dry residue, dry matter, solid matter, stoving residue are being used commonly with the respective abbreviations. The term “non-volatile matter” which is also applied in ISO 3251 should be used together with the abbreviation “NV” instead of these terms.

[SOURCE: ISO 4618:2014, 2.176]

3.2

hiding power

ability of a pigmented medium to hide the colour or the colour differences of a substrate

[SOURCE: ISO 18314-2:2015, 2.1.6]

4 Description

Aluminium pigments are composed of finely divided aluminium metal. The particles of aluminium metal are lamellar in shape when examined microscopically. It is typically divided by the predominant flake shape into “cornflake” (flakes with predominantly ragged and uneven flake shape) and “silverdollar” (flakes with predominantly round and smooth surface).

The material may be in the form of a powder or a paste and have leafing or non-leafing characteristics and the surface of the aluminium flakes may be untreated or treated by technical processing.

Mica and other adulterants shall be absent. If, in solution of the sample in hydrochloric acid as described in 15.3.3, a non-fatty residue is obtained, the residue shall be examined.

5 Classification

This document covers four types of general aluminium pigments, as follows:

- Type 1: aluminium powder, leafing, surface untreated or treated;
- Type 2: aluminium paste, leafing, surface untreated or treated;
- Type 3: aluminium powder, non-leafing, surface untreated or treated;
- Type 4: aluminium paste, non-leafing, surface untreated or treated.

NOTE Untreated refers to milled aluminium pigments with necessary milling agents. Treated refers to the involvement of an additional process to modify the aluminium surface in order to protect the pigments, for example encapsulation

6 Requirements and test methods

The material shall have the characteristics given in the appropriate column of [Table 1](#).

The liquid contained in paste pigment shall be agreed between the interested parties.

Table 1 — Requirements and test methods

Characteristic	Requirement according to type				Test method
	Type 1	Type 2	Type 3	Type 4	
Non-volatile matter, % mass fraction	min. 99,0	min. 25,0	min. 99,0	min. 25,0	Clause 8
Matter soluble in organic solvents, % mass fraction ^b	max. 6,0	max. 4,0	max. 1,5	max. 6,0	9.1 (Type 1 and Type 2) and 9.2 (Type 3 and Type 4)
Comparison of appearance	To match closely the appearance of paint prepared similarly from an agreed sample				Clause 10
Residue on sieve ^a	To be agreed between the interested parties				Clause 11
Pigment specific surface area, m ² /g	To be agreed between the interested parties				ISO 9277
Water-covering capacity, m ² /g	To be agreed between the interested parties		–	–	Clause 12
Leafing power, %	min. 65	min. 65	–	–	Clause 13 ^a
Water content, % mass fraction ^c	max. 0,2	max. 0,15	max. 0,2	max. 0,15	Clause 14
Metallic impurities, % mass fraction on dry pigment	max. 1,0 for Cu + Fe + Pb + Si + Zn max. 0,03 for Pb Separate limits for metals other than lead may be agreed between the interested parties		To be agreed between the interested parties		For lead ^b : Clause 15 , Clause 17 , For iron ^b : Clause 17 , ISO 793 For copper ^b : Clause 17 , ISO 795 For silicon ^b : ISO 808 For zinc ^b : Clause 17 , ISO 1784
Hiding power, g/m ²	To be agreed between the interested parties				Clause 16
Particle size distribution (instrumental method)	To be agreed between the interested parties				ISO 13320

^a Aluminium pigments surface-treated are not included.
^b These methods are recommended for reference purposes, but other methods may be used by agreement between the interested parties.

7 Sampling

Take a representative sample of the product to be tested according to ISO 15528.

8 Determination of non-volatile-matter content

8.1 Apparatus

8.1.1 Weighing bottle, squat form, wide-mouthed, with ground glass stopper.

8.1.2 Air oven, designed to carry out the test in safe conditions, and capable of being controlled at the specified or agreed temperature ± 2 °C (for temperatures up to 150 °C) or $\pm 3,5$ °C (for temperatures above 150 °C and up to 200 °C). The air oven shall be fitted with forced-ventilation equipment, except for phenolic resins when an oven with natural convection with a perforated metal shelf placed at one-third of the height of the oven may be used.

8.1.3 Balance, accurate to 1 mg or better.

8.1.4 Desiccator, containing an efficient desiccant.

8.2 Procedure

8.2.1 Number of determinations

Carry out the determination in duplicate.

8.2.2 Test portion

Heat the weighing bottle (8.1.1), with the stopper removed, in the oven (8.1.2) at 105 °C for 2 h. Allow to cool in the desiccator (8.1.4), insert the stopper and weigh to the nearest 1 mg.

Spread 5 g to 10 g of the sample in a uniform layer on the bottom of the weighing bottle, insert the stopper and weigh to the nearest 1 mg.

It may be necessary to reduce the mass of the test portion for pigments and extenders with a high bulk volume. The use of a test portion smaller than that specified shall be stated in the test report.

8.2.3 Determination

Heat the weighing bottle and contents, with the stopper removed, in the oven at (140 ± 2) °C or agreed temperature for a minimum of 2 h. Allow to cool in the desiccator, insert the stopper and weigh to the nearest 1 mg. Repeat the heating for at least 30 min, allow to cool in the desiccator, insert the stopper and again weigh to the nearest 1 mg. Repeat this procedure until two successive weighings differ by no more than 5 mg. Record the lower mass.

If the results of the two determinations differ by more than 10 % of the higher value, repeat the whole procedure (see 8.2).

If the material under test is unstable at 140 °C, the test conditions shall be agreed between the interested parties and shall be stated in the test report.

8.3 Expression of results

Calculate the non-volatile-matter content, w_0 , expressed as a mass fraction, in per cent, using [Formula \(1\)](#):

$$w_0 = \frac{m_2 - m_0}{m_1 - m_0} \times 100 \quad (1)$$

where

m_0 is the mass, in grams, of the empty weighing bottle;

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

m_2 is the mass, in grams, of the weighing bottle with the residue.

If the two results (duplicates) differ by more than 2 % to the mean (absolute), repeat the procedure described in [8.2](#).

Calculate the mean of two valid results (replicates) and report the test result to the nearest 0,1 % mass fraction.

9 Determination of matter soluble in organic solvents

9.1 Method 1 (for use with leafing pigments, Type 1 and Type 2)

9.1.1 Principle

The sample is treated with hydrochloric acid to dissolve the metal, and the residual oily and fatty matter is extracted with 2-Butoxyethanol or n-butyl acetate, dried and weighed.

9.1.2 Reagents

All reagents shall be of recognized analytical quality. Distilled water or water of equivalent purity shall be used in the test.

9.1.2.1 Hydrochloric acid (HCl), CAS-No 7647-01-0¹⁾, approximately 6 mol/l solution.

9.1.2.2 2-Butoxyethanol (C₆H₁₄O₂), CAS-No 111-76-2, or **n-butyl acetate (C₆H₁₂O₂)**, CAS-No 123-86-4.

9.1.3 Procedure

9.1.3.1 Test portion

Weigh, to the nearest 1 mg, about 2 g of the material into a 400 ml beaker.

9.1.3.2 Determination

Add 100 ml of hot water to the test portion and cover the beaker, for example with a watch-glass. Add the hydrochloric acid ([9.1.2.1](#)) in small portions, heating gently to complete the reaction after each addition until all the metal is dissolved. The maximum amount of hydrochloric acid should not exceed 60 ml.

Cool the beaker and contents to room temperature and filter the contents through an acid-washed, grease-free filter paper. Wash the beaker, the cover and the filter paper thoroughly with cold water.

Allow the paper to drain and dry completely in the filter funnel, heating gently, if necessary, to a temperature not exceeding 50 °C. Remove as much water as possible from the beaker by shaking it.

Place a weighed 100 ml beaker under the funnel. Wash the original beaker and the cover with warm 2-Butoxyethanol or n-butyl acetate ([9.1.2.2](#)) and transfer the washings to the filter paper. Wash the paper at least five times with warm 2-Butoxyethanol or n-butyl acetate, filling it about half full each time. Finally rinse the tip of the funnel. Gently warm the beaker and its contents on a water bath without using a free flame, until the 2-Butoxyethanol or n-butyl acetate has been evaporated as completely as possible. Continue the evaporation by heating the beaker at a temperature of (105 ± 2) °C for 1 h, then cool and weigh.

1) CAS-No — Chemical Abstracts Service Registry Number

9.1.4 Expression of results

Calculate the matter soluble in organic solvents, w_1 , expressed as a percentage mass fraction, using [Formula \(2\)](#):

$$w_1 = \frac{m_4}{m_3} \times 100 \quad (2)$$

where

m_3 is the mass, in grams, of the test portion;

m_4 is the mass, in grams, of the residue.

9.2 Method 2 (for use with non-leaving pigments, Type 3 and Type 4)

9.2.1 Principle

The sample is dispersed in solvent and the solvent-extractable matter is filtered off, dried and weighed.

9.2.2 Reagents

9.2.2.1 Solvent mixture, obtained by mixing 3 parts by volume of toluene, CAS-No 108-88-3, with 1 part of diethyl ether, CAS-No 60-29-7, $\rho = 0,720$ g/ml.

9.2.2.2 Light petroleum spirit, boiling range 40 °C to 60 °C, CAS-No 64742-49-0.

9.2.3 Apparatus

9.2.3.1 Sintered glass filter crucible, of porosity grade P 16 (pore size index 10 µm to 16 µm).

9.2.4 Procedure

9.2.4.1 Test portion

Weigh, to the nearest 1 mg, about 2 g of the material into 250 ml beaker and disperse it in 20 ml of the solvent mixture ([9.2.2.1](#)), with frequent intermittent swirling of the contents of the beaker.

9.2.4.2 Determination

When complete dispersion has been obtained, add a further 10 ml of the solvent mixture, thoroughly agitate by swirling the beaker and then allow to stand for 1 h for the metallic flakes to settle.

Decant the supernatant liquid into the sintered glass filter crucible ([9.2.3.1](#)) and filter by suction into a clean flask.

When all the liquid has been filtered, add a further 30 ml of the solvent mixture to the residue in the beaker and repeat the swirling so as to redisperse the aluminium pigment. Filter the dispersion through the sintered glass filter crucible, washing the beaker with the light petroleum spirit ([9.2.2.2](#)).

Transfer the filtrate from the flask to a 250 ml beaker and evaporate to minimum bulk (about 50 ml). Transfer the concentrated filtrate to a weighed 100 ml beaker and wash the 250 ml beaker with the light petroleum, transferring the washings into the 100 ml beaker. Evaporate the contents of the 100 ml beaker just to dryness, heat in an oven at a temperature of (105 ± 2) °C for 1 h, then cool and weigh.

9.2.5 Expression of results

Calculate the matter soluble in organic solvents, w_2 , expressed as a percentage mass fraction, using [Formula \(3\)](#):

$$w_2 = \frac{m_6}{m_5} \times 100 \quad (3)$$

where

m_5 is the mass, in grams, of the test portion;

m_6 is the mass, in grams, of the residue.

10 Comparison of appearance

10.1 Test portion

Weigh an amount of sample agreed between the interested parties.

10.2 Assessment

Disperse the test portion in a paint vehicle to be agreed between the interested parties in the agreed proportions, by appropriate mixing without grinding. Store for a period to be agreed between the interested parties, for example 24 h, at a temperature also to be agreed, for choice 20 °C, in a covered container. After this period remove any surface skin, mix well by shaking or stirring or both, and apply a coat of the mixture by a suitable method to a smooth, clean, non-absorbent panel, allowing thorough drying in a clean atmosphere. Treat the agreed sample in a similar way on the same day. When dry, compare visually the two panels thus prepared for colour, opacity, finish and brightness.

For leafing pigments, the vehicle shall have an acid value less than 7,5 mg of KOH per gram and shall not contain lead driers.

The optical characterization of n.i. aluminium pigments is recommended to be carried out in full shade and colour reduction spray applied in an application method or alternatively with a draw down. A subsequent colorimetry according to ISO/CIE 11664-4 in multiple angles is recommended.

11 Determination of residue on sieve

11.1 Reagent

11.1.1 Mineral solvent, to be agreed between the interested parties.

11.1.2 2-Butoxyethanol (C₆H₁₄O₂), CAS-No 111-76-2, or ***n*-butyl acetate (C₆H₁₂O₂)**, CAS-No 123-86-4.

11.2 Apparatus

11.2.1 Containers, three, of suitable size to accommodate the sieve.

11.2.2 Beaker, 400 ml.

11.2.3 Test sieve of nominal aperture 180 µm (for pastes) or 250 µm (for powders) (see [11.3.1](#)).

11.2.4 Sintered glass filter, of porosity grade P 16 (pore size index 10 µm to 16 µm).

11.3 Procedure

11.3.1 Test portion

Weigh 10 g of the sample, to the nearest 0,1 g, in the 400 ml beaker (11.2.2).

If it is required, by agreement between the interested parties, to determine the residue on a sieve of nominal aperture other than 180 µm (for pastes) or 250 µm (for powders), the procedure to be adopted is similar to that specified, except that the mass of the test portion shall be correspondingly reduced for sieves of smaller nominal aperture.

11.3.2 Determination

Half fill two of the containers (11.2.1) with the mineral solvent and half fill the third container with the 2-Butoxyethanol or n-butyl acetate (11.1.2). Mix the test portion with 100 ml of the mineral solvent (11.1.1). Add a further 50 ml of the mineral solvent with vigorous stirring. Pour the suspension slowly onto the surface of the test sieve (11.2.3), adjusting the speed of transference so that the majority of the suspension passes through. Wash the residue on the sieve by holding the sieve in the first container at a slight angle to the surface of the mineral solvent and shaking the sieve backwards and forwards so that the screen surface passes just under and just above the level of the liquid. Continue this operation for 1 min and then repeat the procedure in the second container for about 2 min.

When it is evident that no more material passes through the sieve, repeat the procedure in the 2-Butoxyethanol or n-butyl acetate container for 2 min to 3 min. Wash down the sides of the sieve with a small stream of 2-Butoxyethanol or n-butyl acetate and collect the residue on one side. Transfer the residue, by washing with a minimum quantity of 2-Butoxyethanol or n-butyl acetate, to the tared sintered glass filter (11.2.4) and apply suction.

As soon as filtration is complete and the surface of the residue is apparently dry, place the filter in an oven at $(105 \pm 2) ^\circ\text{C}$ (or at a temperature appropriate for the drying) and heat at this temperature for 1 h.

Weigh the residue to the nearest 1 mg.

11.4 Expression of results

Calculate the residue on sieve, w_3 , expressed as a percentage mass fraction, using [Formula \(4\)](#):

$$w_3 = \frac{m_8}{m_7} \times 100 \quad (4)$$

where

m_7 is the mass, in grams, of the test portion;

m_8 is the mass, in grams, of the residue on the sieve.

12 Determination of water-covering capacity

12.1 Principle

The water-covering capacity is measured in a standard apparatus on a sample which has been carefully washed with petroleum spirit and filtered under vacuum. The test involves a comparative trial to

determine the number of washings (between three and six) which gives the maximum result for water-covering capacity.

NOTE Experience has shown that it is essential to carry out the test precisely as described below if reproducible results are to be achieved.

12.2 Reagents

12.2.1 Petroleum spirit, boiling range 80 °C to 100 °C, CAS-No 64742-49-0.

12.2.2 Butan-2-ol, CAS-No 78-83-1.

12.2.3 Paraffin wax, laboratory quality, with a melting point of about 50 °C.

12.3 Apparatus

Usual laboratory apparatus, and in particular:

12.3.1 Evaporating dish, porcelain, approximately 200 mm diameter.

12.3.2 Brushes, small, camel hair.

12.3.3 Sintered glass filter crucible, of porosity grade P 10 (pore size index 4 µm to 10 µm).

12.3.4 Trough, rectangular in shape, supported on levelling screws and approximately 650 mm long, internal width 120 mm and 13 mm to 15 mm deep with vertical sides about 13 mm thick, machined and finished smooth on the upper surface (see Figure 1). (The trough used may conveniently be fabricated from block aluminium, in which case the bottom may be covered internally with black adhesive tape so as to exclude reflections which may interfere with measurement of the length of the pigment film.)

Dimensions in millimetres

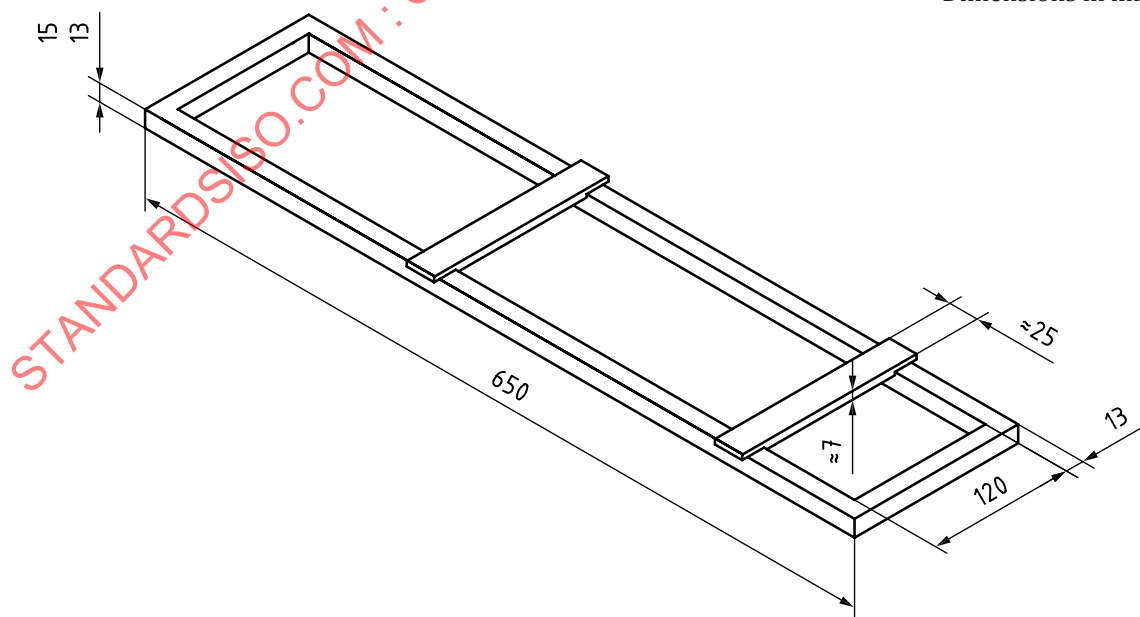


Figure 1 — Diagram of trough for determination of water-covering capacity

12.3.5 Barriers, two, for the trough, made of glass or polished plastics material (for example transparent acrylic resin) of thickness approximately 7 mm, width approximately 25 mm and length slightly more

than the width of the trough, with the extremities of one edge slightly inset so that when a barrier is resting on the sides of the trough the lower edge lies slightly below the sides of the trough.

12.3.6 Watch-glass, approximately 50 mm diameter.

12.4 Pre-treatment of sample

Place the test sample (about 0,5 g for powder, about 1 g for paste) in the evaporating dish ([12.3.1](#)); add, in a few portions, 50 ml of the petroleum spirit ([12.2.1](#)) and thoroughly disperse using a brush ([12.3.2](#)). Allow to stand for 10 min. Filter through the sintered glass crucible ([12.3.3](#)) and suck dry. Disconnect the vacuum line.

Transfer the filter cake to the evaporating dish using a brush and redisperse it with 50 ml of the petroleum spirit, part of which may be used to wash the dish. Filter as before.

Repeat the whole operation specified in the preceding paragraph (i.e. to make a total of three dispersing operations) and leave the filter cake under suction for 30 min after it appears to be dry. Then set aside a small portion of the filter cake. Repeat these same operations once, twice and three times more (i.e. four, five and six dispersing operations respectively), setting aside small portions of the filter cake each time.

Place the small portions of filter cake on separate pieces of gloss paper and mix each with a dry brush.

Allow the portions to dry in a clean, dry atmosphere for 2 h at room temperature, occasionally mixing each with a dry brush.

12.5 Preparation of trough

Prepare the trough ([12.3.4](#)) for use by cleaning, drying, heating to 45 °C to 50 °C, rubbing with the paraffin wax ([12.2.3](#)) and polishing with a soft cloth.

Run water into the trough until the level of the meniscus is appreciably above the sides of the trough, adjusting the levelling screws as necessary.

Sweep the water surface from end to end until it is visibly free from dust, using one barrier ([12.3.5](#)). Place the barrier near one end of the trough and blow away any remaining dust. Place the second barrier alongside the first and slide it along towards the other end of the trough so that there is a clear stretch of water between the two barriers.

It is essential that the two barriers are placed sufficiently widely apart, otherwise the pigment film will not be satisfactorily formed by the procedure specified in [12.6.2](#).

Adjust the water level so that it is just below the upper edges of the trough and in contact with the lower edges of the barriers.

12.6 Procedure

12.6.1 Test portion

Weigh, to the nearest 0,1 mg, on the watch-glass ([12.3.6](#)), a quantity of the treated sample which has been slurred and filtered three times (as specified in [12.4](#)) and which will give a final film length on the trough of between 150 mm and 300 mm. In order to determine the actual quantity of the treated sample to use, carry out a preliminary test using 20 mg of the treated sample.

NOTE It has been found by experience that the manipulation of the film in the trough is facilitated if the length of film when measured is between 150 mm and 300 mm.

12.6.2 Determination

Add from a dropping bottle a quantity of the butan-2-ol (12.2.2) to the test portion such that, after stirring for at least 30 s with a glass rod, a slurry of smooth consistency is obtained (at least 2 ml will usually be required).

Distribute this slurry on the water surface of the trough between the barriers by holding the watch-glass and slurry in a sloping position in the water of the trough so that the watch-glass just dips into the water. The slurry should be distributed upon the water surface immediately and almost completely. Raise the rim of the watch-glass just clear of the water and wash the remaining slurry into the trough with water from a wash bottle.

When the surface film ceases moving, raise the water level by running additional water into the trough until the level is appreciably above the sides of the trough. Complete the distribution of the test portion on the water by stirring with a glass rod. It is important that as little work as possible should be done at this stage to avoid overlapping of particles.

Move one barrier towards the other, gently sweeping the pigment film before it and move the barrier backwards and forwards, while wrinkles alternately form and disappear. Adjust the barrier in the position in which the wrinkles have just disappeared.

The barriers shall not be used for stirring the film.

Draughts shall be avoided at all times. No attempt shall be made to distribute the film by blowing on it.

Movement of the barriers shall be minimal consistent with obtaining an unbroken pigment film. Overworking of the film is likely to produce low results.

Repeat the operations specified in the preceding paragraph, using the other barrier. The two barriers should now be positioned parallel to one another and at right angles to the edge of the trough.

Measure the length, in millimetres, of the pigment film between the barriers.

After the measurement has been completed, check that only a negligible quantity of pigment is left adhering to the barriers, the glass rod and the sides of the trough. If this is not the case, discard the result.

Repeat the whole of the previous operation using a portion of the treated sample which has been dispersed and filtered four times (see 12.4). Repeat the whole operation using portions of the treated sample which have been dispersed and filtered five and six times. Note the number of dispersions and filtrations required to give a maximum value for the length of the pigment film.

Repeat the complete test from the beginning of 12.4, using portions of sample which have been dispersed and filtered for the number of times previously found to give the maximum value, until three values differing from their mean by not more than 0,05 m²/g are obtained.

12.7 Expression of results

Calculate the water-covering capacity, w_4 , in square metres per gram, using Formula (5):

$$w_4 = \frac{l_0 \times b}{10^6 \times m_9} \quad (5)$$

where

l_0 is the length, in millimetres, of the pigment film;

b is the width, in millimetres, of the pigment film;

m_9 is the mass, in grams, of the test portion.

Report the mean value, rounded to the nearest 0,01 m²/g, as the water-covering capacity.

12.8 Precision

12.8.1 Repeatability

The difference between results obtained by the same operator within a short time interval with the same apparatus under constant operating conditions on identical test material shall, at the 95 % confidence level, not exceed 0,1 m²/g.

12.8.2 Reproducibility

The difference between results obtained by different operators in different laboratories on identical test material shall, at the 95 % confidence level, not exceed 0,3 m²/g.

13 Determination of leafing power

13.1 Reagents

13.1.1 White spirit, CAS-No 64742-82-1, the aromatics content of which has been adjusted to 20 % (volume fraction) by the addition of xylene. The relative density shall then be within the range $d_{20} = 0,780$ to 0,790.

13.1.2 Coumarone-indene or hydrocarbon resin, with an acid value not greater than 0,5, completely soluble in the white spirit (13.1.1) when made up as the leafing test vehicle (13.1.3) and without re-precipitation after standing for 24 h at (23 ± 2) °C.

NOTE In the laboratory tests which were carried out to establish this document, a coumarone-indene resin, designated B2-TN/75²⁾ was used and found satisfactory, however, other resins can also be used.

13.1.3 Leafing test vehicle, obtained by dissolving 50 g of the resin (13.1.2) in 100 ml of the white spirit (13.1.1). The relative density of the vehicle shall be within the range $d_{20} = 0,877$ to 0,883.

Solution shall be carried out slowly at a temperature not exceeding 50 °C, any loss of solvent being made up on a mass basis.

13.2 Apparatus

13.2.1 Steel strip, of length not less than 140 mm, width (13 ± 0,5) mm, thickness not more than 1,0 mm, rectangular and square ended. The strip shall be abraded to a satin finish by using aluminium oxide, silicon carbide or emery powder, of grade 000 or equivalent, wetted with mineral solvent.

13.2.2 Glass cylinder, with foot, preferably without spout, about 200 mm in height, and 40 mm in internal diameter.

13.2.3 Corks, two, to fit the glass cylinder, one being slotted to hold the steel strip when suspended vertically in the cylinder.

13.2.4 Test tube, about 150 mm in length and 19 mm to 20 mm in external diameter.

13.2.5 Evaporating dish or spouted capsule, 35 ml to 50 ml capacity.

2) Obtainable from Verkaufsvereinigung für Teerverwertung, Essen, Germany. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products. Equivalent products may be used if they can be shown to lead to the same results.

13.3 Procedure

The day before the tests are to be carried out, place 5 ml of the leafing test vehicle (13.1.3) in the glass cylinder (13.2.2) and close with the unslotted cork (13.2.3), then leave overnight.

13.3.1 Test portion

Remove the top surface layer from the pigment. Weigh the sample in the evaporating dish or spouted capsule (13.2.5), the mass of the test portion shall be agreed between the interested parties.

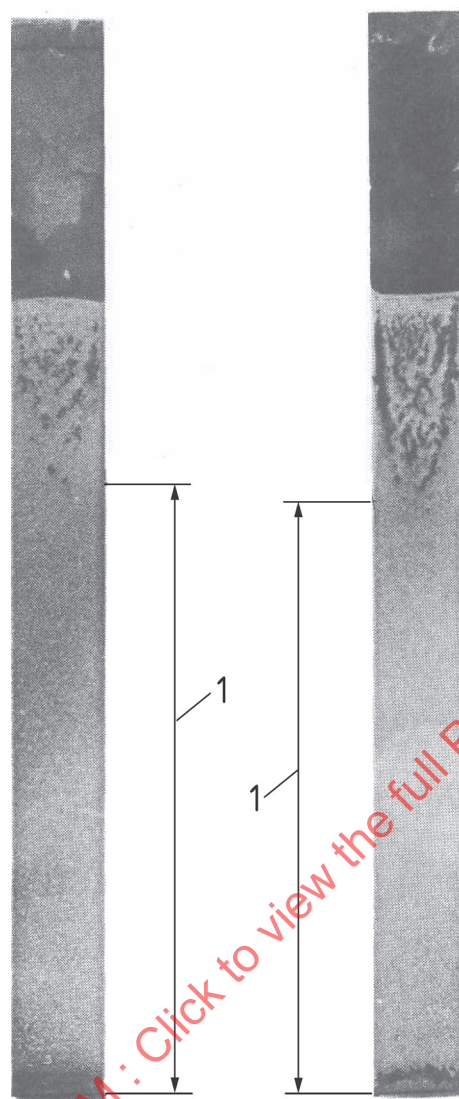
13.3.2 Determination

Measure out 25 ml of the leafing test vehicle and pour about 1 ml (for paste) or 2 ml (for powder) of it onto the pigment. Mix with a small brush until a uniform mixture has been obtained. Repeat with a further similar quantity of leafing test vehicle. Mix well and gradually add the remainder of the 25 ml, mixing carefully without introducing air bubbles into the mixture. Immediately transfer an amount to the test tube (13.2.4), held at an angle of 45°, such that the height in the test tube is 112 mm, avoiding the formation of bubbles. Adjust the temperature as quickly as possible to $(20 \pm 2) ^\circ\text{C}$ and dip the steel strip (13.2.1) immediately to the bottom of the mixture in the tube.

Rotate the steel strip gently for 10 s at about one-quarter turn (90°) per second. The direction of rotation shall be reversed once per second and excessive splashing avoided. Withdraw the strip at a uniform rate (total time (6 ± 1) s without touching the sides of the test tube).

Not more than 2 or 3 drops of the mixture shall drain from the strip.

Using the slotted cork to hold the strip, suspend it vertically in the glass cylinder, sheltered from sunlight, the atmosphere in the cylinder being saturated with the vapour from the leafing test vehicle. The strip shall not be in contact with the vehicle at the bottom of the cylinder at any time. Allow to stand for 6 min and then measure the length of the leafed area, i.e. the completely covered surface free from cracks or breaks (see Figure 2), and the total immersed length. Carry out two determinations with each successive test portion, taking readings to the nearest millimetre on both sides of the steel strip. Calculate the mean of the readings for each test portion.

**Key**

1 leafed area

Figure 2 — Examples of measurement of leafed area**13.4 Expression of results**

Calculate the leafing power, w_5 , as a percentage mass fraction, using [Formula \(6\)](#):

$$w_5 = \frac{l_2}{l_1} \times 100 \quad (6)$$

where

l_1 is the total immersed length, in millimetres;

l_2 is the length of the leafed area, in millimetres.

Report as the result the mean (rounded to the nearest 1 %) of three or four determinations, not differing from this mean by more than ± 3 % absolute.

13.5 Precision

13.5.1 Repeatability

The difference between results obtained by the same operator within a short time interval with the same apparatus under constant operating conditions on identical test material shall, at the 95 % confidence level, not exceed 6 % absolute.

13.5.2 Reproducibility

The difference between results obtained by different operators in different laboratories on identical test material shall, at the 95 % confidence level, not exceed 10 % absolute.

14 Determination of water content

14.1 General

CAUTION — The use of chlorinated hydrocarbon solvents with aluminium powder or paste can be hazardous if heating is involved, since a rapid exothermic reaction may take place. Experience has shown, however, that chloroform presents no danger when used as described below, but precautions should be taken in disposing of waste.

Either of the methods described in ISO 760 may be used with the modifications to the procedures given below.

14.2 Electrometric titration method

Add to the reaction vessel sufficient dry chloroform to cover the electrodes when inserted, switch on the stirrer and titrate with the Karl Fischer reagent until a large deflection of the galvanometer is obtained, which remains above the half-scale reading for at least 30 s. Ignore this titration. Immediately add 10 g of the sample (or such other quantity as will give a titration of not more than 10 ml of the reagent) using chloroform, if necessary, to aid introduction. Allow the material 30 s to disperse and titrate with the Karl Fischer reagent.

Alternatively, if the chloroform used is not dry, allowance should be made for its water content.

14.3 Visual titration method

Add to the reaction vessel 10 g of the sample (or such other quantity as will give a titration of not more than 10 ml of reagent) using chloroform, if necessary, to aid introduction. Add sufficient chloroform to allow complete dispersion of the pigment and titrate with the Karl Fischer reagent until the first permanent appearance of a brown colour.

14.4 Expression of results

Calculate the water content, w_6 , expressed as a percentage mass fraction, using [Formula \(7\)](#):

$$w_6 = \frac{F \times V}{10 \times m_{10}} \quad (7)$$

where

F is the water equivalent, in milligrams per millilitre, of the Karl Fischer reagent;

V is the volume, in millilitres, of the Karl Fischer reagent used;

m_{10} is the mass, in grams, of the test portion.

15 Determination of lead (spectrophotometric method)

15.1 Reagents

All reagents shall be of recognized analytical quality. Distilled water or water of equivalent purity shall be used in the test.

15.1.1 Hydrochloric acid (HCl), CAS-No 7647-01-0, 6 mol/l solution.

15.1.2 Nitric acid, CAS-No 7697-37-2, $\rho = 1,4$ g/ml.

15.1.3 Citric acid solution, 200 g/l.

Dissolve 200 g of citric acid in water, and dilute to 1 l. Keep in a borosilicate glass container.

15.1.4 Hydroxylammonium chloride solution, CAS-No 5470-11-1, 100 g/l.

Dissolve 10 g of hydroxylammonium chloride in water and dilute to 100 ml. This solution shall be freshly prepared just before use.

15.1.5 Potassium cyanide solution, 100 g/l.

Dissolve the required amount of potassium cyanide, CAS-No 151-50-8, in water and dilute to 1 l. Take the usual precautions in preparing and keeping these solutions due to the poisonous character of potassium cyanide. Keep in a borosilicate glass container.

15.1.6 Potassium cyanide solution, 5 g/l.

See [15.1.5](#).

15.1.7 Dithizone solution, 0,2 g/l.

Dissolve 0,200 g of dithizone, CAS-No 60-10-6, in 200 ml of chloroform, CAS-No 67-66-3, and agitate in a 1 l separating funnel with 300 ml of dilute ammonia (1+200). Separate the layers and repeat first with 100 ml and then with 50 ml of dilute ammonia, CAS-No 7664-41-7. Discard the chloroform and filter the combined aqueous layers on a wet paper if necessary. Transfer to a clean 1 l separating funnel with 200 ml of chloroform and add hydrochloric acid, $c(\text{HCl}) = 0,5$ mol/l, drop by drop with frequent shaking until the colour turns to green and passes into the chloroform layer. Separate and rinse the aqueous layer a few times with 50 ml portions of chloroform which are then added to the main solution. Filter through a dry paper and dilute to 1 000 ml with pure chloroform.

Keep in a brown bottle and cover with a 10 mm to 20 mm layer of sulfur dioxide solution (dilute 20 ml of saturated sulfur dioxide solution, CAS-No 7446-09-5, to 100 ml). Under these conditions, the dithizone solution is stable for several months.

15.1.8 Dithizone solution, 0,1 g/l.

Dilute a volume of the dithizone solution ([15.1.7](#)) with an equal volume of chloroform.

15.1.9 Ammonia solution, $\rho = 0,88$ g/ml.

15.1.10 Chloroform.

15.2 Apparatus

Ordinary laboratory apparatus, and in particular:

15.2.1 Filter absorptiometer with Cenco³⁾ green filter cat. No. 87 309 B or equivalent, or a **spectrophotometer** with a blue sensitive phototube; cells of 1 cm or 5 cm.

All glassware shall be free from metals reacting with dithizone. It is advisable to rinse each beaker, funnel or flask with a mixture of dilute ammonia (1+200) and dithizone solution (15.1.7), the same mixture being used for all articles unless the dithizone turns red.

15.3 Procedure

15.3.1 Test portion

Weigh $0,5 \text{ g} \pm 0,001 \text{ g}$ of the sample in a 250 ml beaker.

15.3.2 Blank test

Perform a blank test at the same time as the determination.

15.3.3 Determination

Dissolve the test portion in 15 ml of hydrochloric acid (15.1.1). When dissolution is complete, add 5 drops of nitric acid (15.1.2) and boil for 2 min to 3 min. Filter if necessary, washing five times with hot water, combining the filtrate and washings. Adjust the volume to 50 ml, add 15 ml citric acid solution (15.1.3) and neutralize to litmus with ammonia solution (15.1.9), adding 4 drops in excess. Add 2 ml of freshly prepared hydroxylammonium chloride solution (15.1.4). Boil for 2 min to 3 min and cool in a water trough.

Introduce the solution into a 250 ml separating funnel, add 10 ml of dithizone solution (15.1.7) and shake for 30 s. Draw off the chloroform layer into another separating funnel. Continue to shake the aqueous layer with 5 ml portions of dithizone solution (15.1.8), drawing off the chloroform layer into the second funnel before each new addition, until the chloroform layer remains pure green. Make one further extraction with 5 ml of pure chloroform (15.1.10).

Wash the combined chloroform extracts with 25 ml of potassium cyanide solution (15.1.6) and then with 10 ml portions of this solution until successive washings have the same faint amber colour, drawing off the chloroform layer carefully each time into another funnel, and discarding the washings.

Wash once in a clean separating funnel, with distilled water, and filter the chloroform layer through a dry paper. Collect the filtrate in a 100 ml volumetric flask, wash the paper with chloroform, and dilute to the mark with chloroform. Mix well, transfer to 1 cm or 5 cm cells, and read the transmittance with a filter absorptiometer (15.2.1), using the green filter, or read the absorbance with a spectrophotometer (15.2.1) at a wavelength of 515 nm, with chloroform in the reference cell.

Determine the lead concentration from a calibration graph of absorbance against percentage lead.

16 Determination of hiding power

16.1 Apparatus and materials

16.1.1 Commonly used varnish system (preferably air drying or curing system with curing temperature not exceeding 80 °C).

16.1.2 Wire rod.

16.1.3 Balance, sensitivity 0,01 g.

³⁾ Cenco is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.