INTERNATIONAL STANDARD 1247

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Aluminium pigments for paints

Pigments d'aluminium pour peintures

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, International Standard ISO 1247 replaces ISO Recommendation R 1247-1971 drawn up by Technical Committee ISO/TC 35, *Paints and varnishes.*

The Member Bodies of the following countries approved the Recommendation :

Austria	Iran	South Africa, Rep. of
Brazil	Israel	Spain
Chile	Italy	Sweden
Denmark	Netherlands	Switzerland
Egypt, Arab Rep. of	New Zealand	Turkey
Germany	Peru	United Kingdom
Greece	Poland	U.S.S.R.
India	Portugal	

The Member Body of the following country expressed disapproval of the Recommendation on technical grounds :

France*

* Subsequently, this Member Body approved the Recommendation.

Printed in Switzerland

Aluminium pigments for paints

1 SCOPE AND FIELD OF APPLICATION

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

a) general purpose, decorative and protective paints, and

b) speciality finishing paints.

2 REFERENCES

ISO/R 760, *Determination of water by the Karl Fischer method.*

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

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

ISO/R 795, *Chemical analysis of aluminium and its alloys — Photometric determination of copper (Oxaldihydrazide method applicable to copper content between 0,002 and 0,8 %).*

ISO/R 798, *Chemical analysis of aluminium and its alloys — Gravimetric determination of zinc in aluminium alloys (Zinc content between 0,50 and 6,5 %).*

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

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

ISO 886, *Aluminium and aluminium alloys — Determination of manganese — Photometric method (Manganese content between 0,005 and 1,5 %).*

ISO 1250, *Mineral solvents for paints — White spirits and related hydrocarbon solvents.*

3 DESCRIPTION

Aluminium pigments are composed of finely divided aluminium metal. The particles of aluminium metal are lamellar in shape when examined microscopically. The material may be in the form of a powder or a paste and have leafing or non-leafing characteristics.

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

4 CLASSIFICATION

4.1 Types

This International Standard covers four types of aluminium pigments, as follows :

— type 1 : aluminium powder, leafing

— type 2 : aluminium paste, leafing

— type 3 : aluminium powder, non-leafing

— type 4 : aluminium paste, non-leafing

4.2 Classes

Pigments of types 1 and 2 are further classified by their water-covering capacity as shown in table 1.

TABLE 1 — **Classes of types 1 and 2**

Type	Class	Water-covering capacity
		m^2/g
1	a	up to 0,8
	b	over 0,8 up to 1,5
	c	over 1,5 up to 2,2
	d	over 2,2
2	p	up to 1,7
	q	over 1,7 up to 2,4
	r	over 2,4

NOTE — Attention is drawn to the reproducibility limits given in 11.8.

5 REQUIRED CHARACTERISTICS AND THEIR TOLERANCES

The material shall have the characteristics given in the appropriate column of table 2.

The liquid contained in paste pigment shall be category A mineral solvent complying with ISO 1250, or other appropriate liquid as may be agreed between the interested parties.

TABLE 2 – **Required characteristics and their tolerances**

Characteristic	Requirement according to type				Test method
	Type 1 Leafing powder	**Type 2** Leafing paste	**Type 3** Non-leafing powder	**Type 4** Non-leafing paste	
Matter volatile at 105 °C, % (*m/m*)	max. 1,0	max. 35,0[1)]	max. 1,0	max. 35,0[1)]	ISO 787 Part II
Matter soluble in organic solvents, % (*m/m*)	max. 6,0	max. 4,0	max. 1,5	max. 6,0	Sub-clause 8.1 (types 1 and 2) and sub-clause 8.2 (types 3 and 4)
Appearance of paint prepared in an agreed vehicle	To match closely the appearance of paint prepared similarly from an agreed sample				Clause 9
Residue on sieve[2)]	Nil on 250 µm	Nil on 180 µm	Nil on 250 µm	Nil on 180 µm	Clause 10
Water-covering capacity, m^2/g	Within the limits for the agreed class (see 4.2)		–	–	Clause 11
Leafing power, %	min. 65	min. 65	Nil	Nil	Clause 12 (types 1 and 2) and clause 13 (types 3 and 4)
Water content, % (*m/m*)	max. 0,2	max. 0,15	max. 0,2	max. 0,15	Clause 14
Metallic impurities, % (*m/m*) 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		Limits to be agreed between the interested parties		For lead : clause 15[3)] For iron : ISO 793[3)] For copper : ISO/R 795[3)] For silicon : ISO 808[3)] For manganese : ISO 886[3)] For zinc : ISO/R 798[3)]

1) Alternative limits for volatile content of pastes may be agreed between the interested parties.

2) Additional limits for residue on sieves of smaller aperture may be agreed between the interested parties.

3) These methods are recommended for reference purposes, but other methods may be used by agreement between the interested parties.

6 PACKING

The material shall be packed in air-tight containers.

7 SAMPLING

7.1 For the purpose of testing a pigment in accordance with this International Standard, a sample representative of the bulk material shall be taken by the appropriate procedure specified in ISO 842. The sample shall have a mass of not less than 250 g and shall be packed in the manner specified in ISO 842.

For both pastes and powders, a sampling tube shall be used which enables the whole depth (and, preferably, the longest diagonal) of the container to be sampled. For powders, which may be compacted, a sharply pointed sampling tube shall be used, and the container shall be rolled vigorously before sampling.

Suitable designs of sampling tubes are illustrated in figure 1.

7.2 Except where otherwise agreed between the interested parties, the agreed sample referred to in table 2 and clause 9 shall comply in all respects with the requirements of this International Standard. The sample shall have a mass of not less than 250 g and shall be packed in the manner specified in ISO 842.

METHODS OF TEST

8 DETERMINATION OF MATTER SOLUBLE IN ORGANIC SOLVENTS

8.1 Method 1 (for use with leafing pigments, types 1 and 2)

8.1.1 *Principle*

The sample is treated with hydrochloric acid to dissolve the metal and the residual oily and fatty matter is extracted with acetone, dried and weighed.

8.1.2 *Reagents*

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

8.1.2.1 **Hydrochloric acid,** approximately 6 N solution.

8.1.2.2 **Acetone**

8.1.3 *Procedure*

8.1.3.1 TEST PORTION

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

8.1.3.2 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 (8.1.2.1) in small portions, heating gently to complete the reaction after each addition until all the metal is dissolved. Not more than 60 ml of acid should be necessary.

Cool the beaker and contents to room temperature and filter the contents through an acid-washed, grease-free filter paper. Thoroughly wash the beaker, the cover and the filter paper 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 acetone (8.1.2.2) and transfer the washings to the filter paper. Wash the paper at least five times with warm acetone, 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 acetone 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.

8.1.4 *Expression of results*

Calculate the matter soluble in organic solvents, as a percentage by mass, by the following formula :

$$\frac{100\, m_1}{m_0}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the residue.

8.2 **Method 2** (for use with non-leafing pigments, types 3 and 4)

8.2.1 *Principle*

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

8.2.2 *Reagents*

8.2.2.1 **Solvent mixture,** obtained by mixing 3 parts by volume of toluene with 1 part of diethyl ether, ρ 0,720 g/ml.

8.2.2.2 **Light petroleum spirit,** boiling range 40 to 60 °C.

8.2.3 *Apparatus*

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

8.2.4 *Procedure*

8.2.4.1 TEST PORTION

Weigh, to the nearest 1 mg, about 2 g of the material into a 250 ml beaker and disperse it in 20 ml of the solvent mixture (8.2.2.1), with frequent intermittent swirling of the contents of the beaker.

8.2.4.2 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 (8.2.3) 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 (8.2.2.2).

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.

8.2.5 *Expression of results*

Calculate the matter soluble in organic solvents, as a percentage by mass, by the following formula :

$$\frac{100\, m_1}{m_0}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the residue.

9 COMPARISON OF APPEARANCE

9.1 Test portion

Weigh an amount of sample agreed between the interested parties.

9.2 Assessment

Disperse the test portion in a paint vehicle to be agreed between the interested parties (see note below) in the agreed proportions, by simple 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.

NOTE — 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.

10 DETERMINATION OF RESIDUE ON SIEVE

10.1 Reagents

10.1.1 Mineral solvent, category A, complying with ISO 1250.

10.1.2 Acetone

10.2 Apparatus

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

10.2.2 Beaker, 400 ml.

10.2.3 Test sieve of nominal aperture 180 μm (for pastes) or 250 μm (for powders). See note to 10.3.1.)

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

10.3 Procedure

10.3.1 *Test portion*

Weigh 10 g of the sample, to the nearest 0,1 g, in the 400 ml beaker (10.2.2).

NOTE — 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.

10.3.2 *Determination*

Half fill two of the containers (10.2.1) with the mineral solvent and half fill the third container with the acetone (10.1.2). Mix the test portion with 100 ml of the mineral solvent (10.1.1). Add a further 50 ml of the mineral solvent with vigorous stirring. Pour the suspension slowly onto the surface of the test sieve (10.2.3), 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 acetone container for 2 to 3 min. Wash down the sides of the sieve with a small stream of acetone and collect the residue on one side. Transfer the residue, by washing with a minimum quantity of acetone, to the tared sintered glass filter (10.2.4) 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 ± 2 °C and heat at this temperature for 1 h.

Weigh the residue to the nearest 1 mg.

10.4 Expression of results

Calculate the residue on sieve, as a percentage by mass, by the following formula :

$$\frac{100\, m_1}{m_0}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the residue on the sieve.

11 DETERMINATION OF WATER-COVERING CAPACITY

11.1 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.

11.2 Reagents

11.2.1 Petroleum spirit, boiling range 80 to 100 °C.

11.2.2 Butan-2-ol.

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

11.3 Apparatus

Ordinary laboratory apparatus, and in particular :

11.3.1 Evaporating dish, porcelain, approximately 200 mm diameter.

11.3.2 Brushes, small, camel hair.

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

11.3.4 Trough, rectangular in shape, supported on levelling screws and approximately 650 mm long, internal width 120 mm and 13 to 15 mm deep with vertical sides about 13 mm thick, machined and finished smooth on the upper surface (see figure 2). (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.)

11.3.5 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.

11.3.6 Watch-glass, approximately 50 mm diameter,

11.4 Pre-treatment of sample

Place the test sample (about 0,5 g for powder, about 1 g for paste) in the evaporating dish (11.3.1); add, in a few portions, 50 ml of the petroleum spirit (11.2.1) and thoroughly disperse using a brush (11.3.2). Allow to stand for 10 min. Filter through the sintered glass crucible (11.3.3) 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.

11.5 Preparation of trough

Prepare the trough (11.3.4) for use by cleaning, drying, heating to 45 to 50 °C, rubbing with the paraffin wax (11.2.3) 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 (11.3.5). 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.

NOTE – 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 11.6.2.

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.

11.6 Procedure

11.6.1 *Test portion*

Weigh, to the nearest 0,1 mg, on the watch-glass (11.3.6), a quantity of the treated sample which has been slurried and filtered three times (as specified in 11.4) and which will give a final film length on the trough of between 150 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 and 300 mm.

11.6.2 *Determination*

Add from a dropping bottle a quantity of the butan-2-ol (11.2.2) 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.

NOTES

1 The barriers shall not be used for stirring the film.

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

3 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 11.4). Again 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 11.4, 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 m^2/g are obtained.

11.7 Expression of results

Calculate the water-covering capacity, in square metres per gram, by the following formula :

$$\frac{lb}{10^6\, m}$$

where

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

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

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

Report the mean value, rounded to the nearest 0,01 m^2/g, as the water-covering capacity.

11.8 Precision

11.8.1 *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 m^2/g.

11.8.2 *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 m^2/g.

12 DETERMINATION OF LEAFING POWER

12.1 Reagents

12.1.1 White spirit, the aromatics content of which has been adjusted to 20 % (*V/V*) by the addition of xylene. The relative density should then be within the range d_{20} = 0,780 to 0,790.

12.1.2 Coumarone-indene or hydrocarbon resin, with an acid value not greater than 0,5, completely soluble in the white spirit (12.1.1) when made up as the leafing test vehicle (12.1.3) 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 International Standard, a coumarone-indene resin, designated B2-TN/75 (obtainable from Verkaufsvereinigung für Teerverwertung, Essen, Germany), was used and found satisfactory but other resins may also be found satisfactory.

12.1.3 Leafing test vehicle, obtained by dissolving 50 g of the resin (12.1.2) in 100 ml of the white spirit (12.1.1). 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.

12.2 Apparatus

12.2.1 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.

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

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

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

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

12.3 Procedure

The day before the tests are to be carried out, place 5 ml of the leafing test vehicle (12.1.3) in the glass cylinder (12.2.2) and close with the unslotted cork (12.2.3), then leave overnight.

12.3.1 *Test portion*

Remove the top surface layer from the pigment and weigh a quantity of the sample according to table 3 in the evaporating dish or spouted capsule (12.2.5).

TABLE 3 – **Test portions**

Type	Class	Mass of test portion [1]
		g
1	a	3,0
	b	2,5
	c	2,0
	d	1,5
2	p	3,0
	q	2,5
	r	2,0

1) If the water-covering capacity of the material is near the borderline between two classes (see 4.2), two series of leafing power tests shall be carried out using the mass of pigment indicated for each of the two classes, and the results of both series of tests shall be reported.

12.3.2 *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 (12.2.4), 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 ± 2 °C and dip the steel strip (12.2.1) 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.

NOTE – 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 3), 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.

12.4 Expression of results

Calculate the leafing power, as a percentage, by the following formula :

$$\frac{100\, l_1}{l_0}$$

where

l_0 is the total immersed length, in millimetres;

l_1 is the length of the leafed area, in millimetres.

Report as the percentage leafing power the mean (rounded to the nearest 1 %) of three or four results not differing from this mean by more than ± 3 % absolute.

12.5 Precision

12.5.1 *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.

12.5.2 *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.

13 TEST FOR ABSENCE OF LEAFING POWER

13.1 Reagents

Xylene, suitable commercial grade.

13.2 Procedure

Add 5 g of the sample to 50 ml of the xylene in a suitable container and thoroughly disperse it by vigorous stirring. Allow to settle for 5 min, and then observe the surface of the xylene for flakes of pigment. Absence of flotation of flakes of pigment at the surface is taken as denoting absence of leafing power.

14 DETERMINATION OF WATER CONTENT

CAUTIONARY NOTE – 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/R 760 may be used with the modifications to the procedures given below.

14.1 Electrometric titration method

Add to the reaction vessel sufficient dry chloroform[1] 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.

14.2 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.

14.3 Expression of results

Calculate the water content, as a percentage by mass, by the following formula :

$$\frac{FV}{10\,m}$$

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 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, 6 M solution.

15.1.2 Nitric acid, ρ 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, 100 g/l.

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

15.1.5 Potassium cyanide solution, 100 g/l.

Dissolve the required amount of potassium cyanide 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.

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

15.1.7 Dithizone solution, 0,2 g/l.

Dissolve 0,200 g of dithizone in 200 ml of chloroform 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. 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, 0,5 M, 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 to 20 mm layer of sulphur dioxide solution (dilute 20 ml of saturated sulphur dioxide solution 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, ρ 0,88 g/ml.

15.1.10 Chloroform.

15.2 Apparatus

Ordinary laboratory apparatus, and in particular :

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

NOTE – 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 ± 0,001 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*

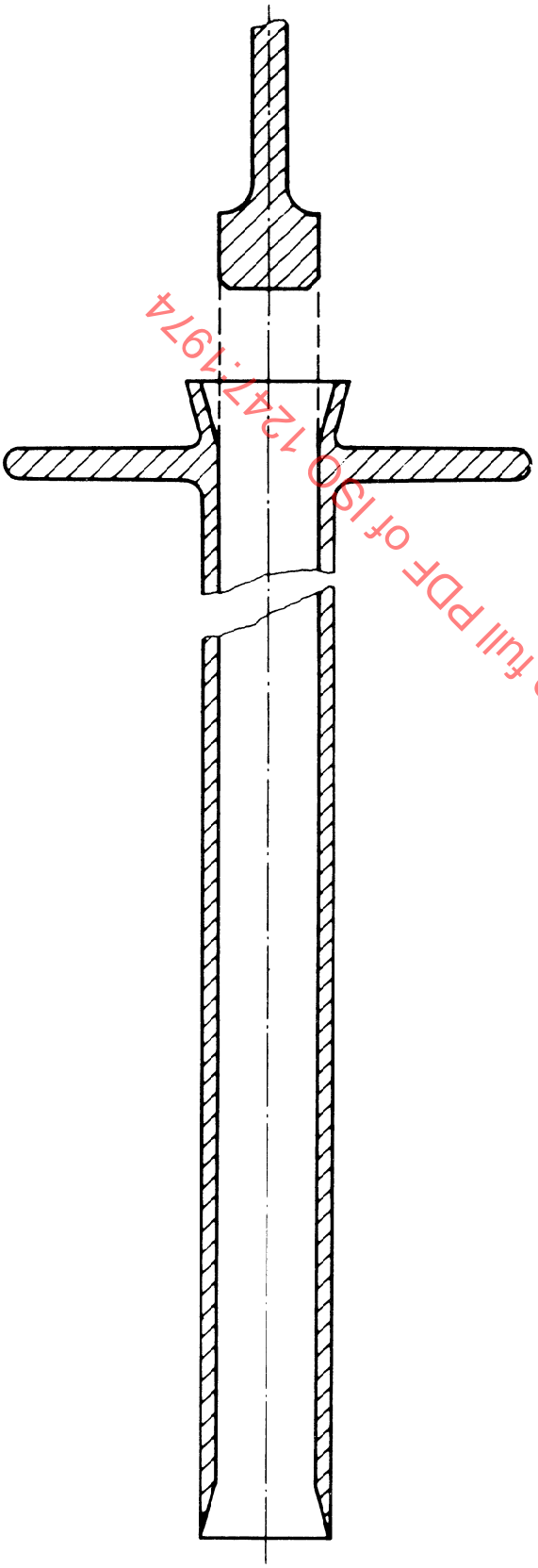
Dissolve the test portion in 15 ml of hydrochloric acid (15.1.1). When dissolution is complete, add 5 drops of nitric acid (15.1.2) and boil for 2 to 3 min. Filter if necessary, washing five times with hot water, combining the filtrate and washings. Adjust the volume to 50 ml, add 15 ml citric acid solution (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 (15.1.5). Boil for 2 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).

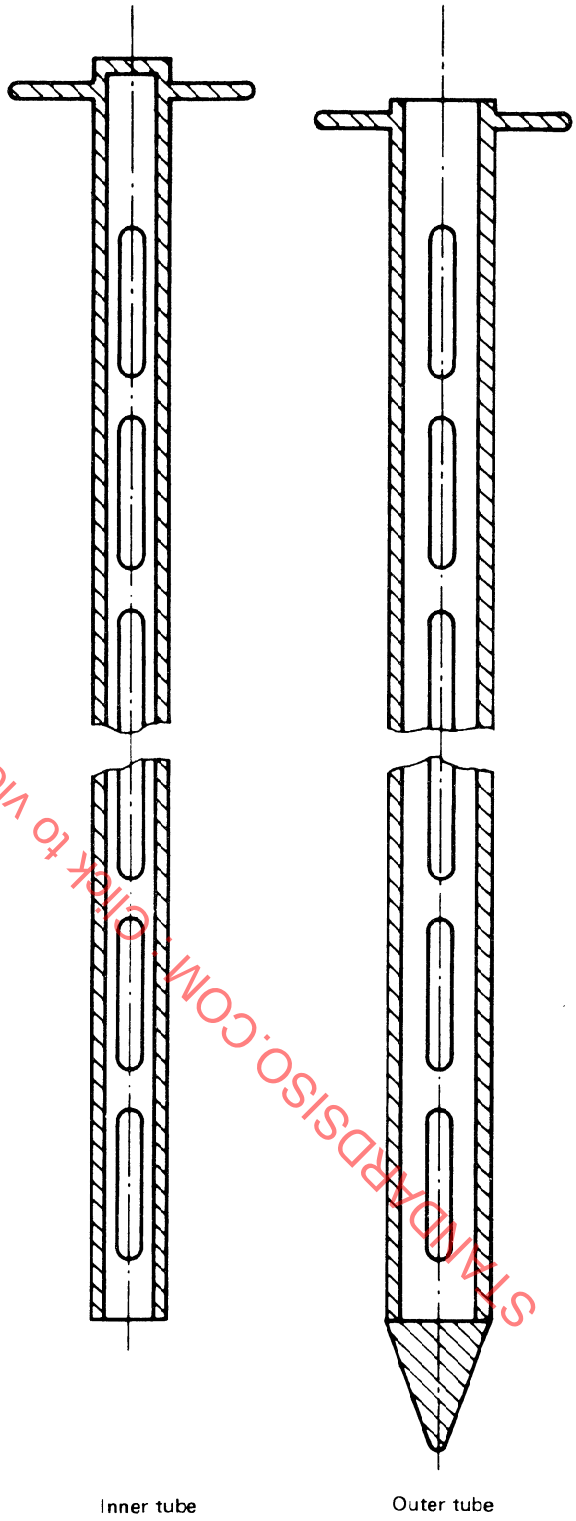
Wash the combined chloroform extracts with 25 ml of potassium cyanide solution (15.1.6) and then with 10 ml portions of this solution until successive washings have the same faint amber colour, drawing off the chloroform layer carefully each time into another funnel, and discarding the washings.

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 or 5 cm cells, and read the transmittance with a filter absorptiometer (15.2), using the green filter, or read the absorbance with a spectrophotometer (15.2) 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.



Sampling tube for paste



(Inner tube, sliding fit in outer tube. All edges square cut.)

Sampling tube for powder

FIGURE 1 — **Suitable sampling tubes**