UDC 667.622.271

Ref. No. : ISO/R 1247-1971 (E)

Transformed

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 1247

ALUMINIUM PIGMENTS

1st EDITION

May 1971

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

Printed in Switzerland

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

BRIEF HISTORY

The ISO Recommendation R 1247, *Aluminium pigments,* was drawn up by Technical Committee ISO/TC 35, *Paints and varnishes,* the Secretariat of which is held by the Nederlands Normalisatie-instituut (NNI).

Work on this question led to the adoption of Draft ISO Recommendation No. 1247, which was circulated to all the ISO Member Bodies for enquiry in October 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

The following Member Body opposed the approval of the Draft :

France

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as an ISO RECOMMENDATION.

CONTENTS

METHODS OF TEST

ISO Recommendation R 1247 May 1971

ALUMINIUM PIGMENTS

1. SCOPE

This ISO Recommendation specifies the requirements and the corresponding methods of test for aluminium pigments used in paints including

(*a*) general purpose, decorative and protective paints, and

(*b*) speciality finishing paints.

2. DESCRIPTION

The material should be composed essentially of finely divided aluminium metal. The particles of aluminium metal should be lamellar in shape as examined microscopically. The material should be in the form of a powder or a homogeneous paste.

NOTE. – Mica and other adulterants should be absent. If when carrying out the solution of the sample in hydrochloric acid as described in clause 14.1.3.3 a non-fatty residue is obtained, the residue should be examined.

3. CLASSIFICATION

3.1 Types

This ISO Recommendation 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

3.2 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 clause 10.9.

4. REQUIRED CHARACTERISTICS AND THEIR TOLERANCES

The material should have the characteristics given in the appropriate column of Table 2.

The liquid contained in paste pigment should be category A mineral solvent complying with ISO Recommendation R 1250, *Mineral solvents for paints (white spirits and related hydrocarbon solvents)*, or other appropriate liquid as may be agreed between purchaser and vendor.

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

Characteristic	Requirements 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 % max.	1.0	35.0*	1.0	35.0*	ISO/R 787** Part II
Matter soluble in organic solvents % max.	6.0	4.0	1.5	6.0	clause 7.1 (Types 1 and 2) clause 7.2 (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				section 8
Residue on sieve***	Nil on 250 μm	Nil on 180 μm	Nil on 250 μm	Nil on 180 μm	section 9
Water-covering capacity, m^2/g	Within the limits for the agreed class (see clause 3.2)		–	–	section 10
Leafing power	65 % min.	65 % min.	Nil	Nil	section 11 (Types 1 and 2) section 12 (Types 3 and 4)
Water content % max.	0.2	0.15	0.2	0.15	section 13
Metallic impurities, % max. on dry pigment	1.0 for Cu + Fe + Pb + Si + Zn 0.03 for Pb Separate limits for metals other than lead may be agreed between purchaser and vendor		Limits to be agreed between purchaser and vendor		section 14

* Alternative limits for volatile content of pastes may be agreed between purchaser and vendor.

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

*** Additional limits for residue on sieves of smaller aperture may be agreed between purchaser and vendor.

5. PACKING

The material should be packed in air-tight containers.

6. SAMPLING

6.1 For the purpose of testing a pigment in accordance with this ISO Recommendation, a sample representative of the bulk material should be taken by the appropriate procedure described in ISO Recommendation R 842, *Sampling raw materials for paints and varnishes.* The sample should have a mass of not less than 250 g and should be packed in the manner described in ISO Recommendation R 842.

For both pastes and powders a sampling tube should 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 should be used, and the container should be rolled vigorously before sampling.

Suitable designs of sampling tubes are illustrated in Figure 1.

6.2 Except where otherwise agreed between purchaser and vendor, the agreed sample referred to in Table 2 and section 8 should comply in all respects with the requirements of this ISO Recommendation. The sample should have a mass of not less than 250 g and should be packed in the manner described in ISO Recommendation R 842.

METHODS OF TEST

7. DETERMINATION OF MATTER SOLUBLE IN ORGANIC SOLVENTS

7.1 **Method 1** (for use with leafing pigments, Types 1 and 2)

7.1.1 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.

7.1.2 REAGENTS

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

7.1.2.1 *Hydrochloric acid* solution, approximately 6N.

7.1.2.2 *Acetone.*

7.1.3 PROCEDURE

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

7.1.3.2 *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 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 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 hour, then cool and weigh.

7.1.4 CALCULATION

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

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

where

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

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

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

7.2.1 PRINCIPLE

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

7.2.2 REAGENTS

7.2.2.1 *Solvent mixture.* Mix 3 parts by volume of toluene with 1 part of diethyl ether, $d = 0.720$.

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

7.2.3 APPARATUS

Sintered glass filter crucible, of porosity grade P.16 (maximum pore size 10 to 16 μm), in accordance with ISO Recommendation R . . .*, *Porosity grading of laboratory sintered (fritted) filters.*

7.2.4 PROCEDURE

7.2.4.1 *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, with frequent intermittent swirling of the contents of the beaker.

7.2.4.2 *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 hour for the metallic flakes to settle.

Decant the supernatant liquid into the sintered glass filter crucible 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.

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 hour, then cool and weigh.

7.2.5 CALCULATION

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

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

where

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

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

8. COMPARISON OF APPEARANCE

8.1 *Test portion.* Weigh an amount of sample agreed between purchaser and vendor.

8.2 *Assessment.* Disperse the test portion in a paint vehicle to be agreed between purchaser and vendor (see Note) in the agreed proportions, by simple mixing without grinding. Store for a period to be agreed between purchaser and vendor, for example 24 hours, 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 should have an acid value less than 7.5 mg of KOH per gramme and should not contain lead driers.

---

* At present at the stage of draft proposal.

9. DETERMINATION OF RESIDUE ON SIEVE

9.1 Reagents

9.1.1 *Mineral solvent,* Category A, complying with ISO Recommendation R 1250, *Mineral solvents for paints (white spirits and related hydrocarbon solvents).*

9.1.2 *Acetone.*

9.2 Apparatus

9.2.1 *Containers,* three, of suitable size to accommodate the sieve.

9.2.2 *Beaker,* 400 ml.

9.2.3 *Test sieve* of nominal aperture 180 μm (for pastes) or 250 μm (for powders). (See Note to clause 9.3.1.)

9.2.4 *Sintered glass filter,* of porosity grade P.16 (maximum pore size 10 to 16 μm), in accordance with ISO Recommendation R . . .*, *Porosity grading of laboratory sintered (fritted) filters.*

9.3 Procedure

9.3.1 *Test portion.* Weigh 10 g of the sample, to the nearest 0.1 g, in a 400 ml beaker.

NOTE. – If it is required, by agreement between purchaser and vendor, 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 described, except that the mass of the test portion should be correspondingly reduced for sieves of smaller nominal aperture.

9.3.2 *Determination.* Half fill two of the containers with the mineral solvent and half fill the third container with the acetone. Mix the test portion with 100 ml of the mineral solvent. Add a further 50 ml of the mineral solvent with vigorous stirring. Pour the suspension slowly onto the surface of the test sieve, 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 minute and then repeat the procedure in the second container for about 2 minutes.

When it is evident that no more material passes through the sieve, repeat the procedure in the acetone container for 2 to 3 minutes. 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 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 hour.

Weigh the residue to the nearest 1 mg.

9.4 Calculation

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

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

where

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

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

---

* At present at the stage of draft proposal.

10. DETERMINATION OF WATER-COVERING CAPACITY

10.1 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.

10.2 Reagents

10.2.1 *Petroleum spirit*, boiling range 80 to 100 °C.

10.2.2 *2-Butanol.*

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

10.3 Apparatus

Usual laboratory equipment, and in particular :

10.3.1 *Evaporating dish*, porcelain, approximately 200 mm diameter.

10.3.2 *Brushes*, small, camel hair.

10.3.3 *Sintered glass filter crucible*, of porosity grade P.10 (maximum pore size 4 to 10 μm), in accordance with ISO Recommendation R . . .*, *Porosity grading of laboratory sintered (fritted) filters.*

10.3.4 *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.)

10.3.5 *Barriers*, two, for the trough, made of glass or polished plastics material (for example transparent acrylic resin) a 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.

10.3.6 *Watch-glass*, approximately 50 mm diameter.

10.4 Pre-treatment of sample

Place the test sample (about 0.5 g for powder, about 1 g for paste) in the evaporating dish; add, in a few portions, 50 ml of the petroleum spirit and thoroughly disperse using a brush. Allow to stand for 10 minutes. Filter through the sintered glass crucible 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 described in the preceding paragraphe (i.e. to make a total of three dispersing operations) and leave the filter cake under suction for 30 minutes 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 hours at room temperature, occasionally mixing each with a dry brush.

---

* At present at the stage of draft proposal.

10.5 Preparation of trough

Prepare the trough for use by cleaning, drying, heating to 45 to 50 °C, rubbing with the paraffin wax 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. 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 described in clause 10.6.2.

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.

10.6 Procedure

10.6.1 *Test portion.* Weigh, to the nearest 0.1 mg on the watch-glass, a quantity of the treated sample which has been slurried and filtered three times (as described in clause 10.4) 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.

10.6.2 *Determination.* Add from a dropping bottle a quantity of the 2-butanol to the test portion such that, after stirring for at least 30 seconds 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 should not be used for stirring the film.
2. Draughts should be avoided at all times and no attempt should be made to distribute the film by blowing on it.
3. Movement of the barriers should be minimal consistent with obtaining an unbroken pigment film. Overworking of the film is likely to produce low results.

Repeat the operations described 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 clause 10.4). 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 clause 10.4, 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.

10.7 Calculation

Calculate the water-covering capacity, in square metres per gramme, by means of 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 grammes, of the test portion.

10.8 Expression of results

Report the mean value, rounded to the nearest 0.01 m^2/g, as the water-covering capacity.

10.9 Precision

10.9.1 *Repeatability.* The results of tests on the same sample by the same operator using the same apparatus should not be considered suspect unless the extreme values differ by more than 0.1 m^2/g.

10.9.2 *Reproducibility.* The results of tests on the same sample in different laboratories should not be considered suspect unless the extreme values differ by more than 0.3 m^2/g.

11. DETERMINATION OF LEAFING POWER

11.1 Reagents

11.1.1 *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.

11.1.2 *Coumarone-indene or hydrocarbon resin,* with an acid value not greater than 0.5, completely soluble in the white spirit (11.1.1) when made up as the leafing test vehicle (11.1.3) and without re-precipitation after standing for 24 hours at 20 ± 2 °C.

NOTE. – In the laboratory tests which were carried out to establish this ISO Recommendation, 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.

11.1.3 *Leafing test vehicle,* obtained by dissolving 50 g of the resin (11.1.2) in 100 ml of the white spirit (11.1.1). The relative density of the vehicle should be within the range d_{20} = 0.877 to 0.883.

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

11.2 Apparatus

11.2.1 *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 should be abraded to a satin finish by using aluminium oxide, silicon carbide or emery powder, of grade 000 or equivalent, wetted with mineral solvent.

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

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

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

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

11.3 Procedure

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

11.3.1 *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.

TABLE 3 – **Test portions**

Type	**Class**	**Mass of test portion***
		g
1	a	3.0
	b	2.5
	c	2.0
	d	1.5
2	p	3.0
	q	2.5
	r	2.0

* If the water-covering capacity of the material is near the borderline between two classes (see clause 3.2), two series of leafing power tests should be carried out using the mass of pigment indicated for each of the two classes, and the results of both series of tests should be reported.

11.3.2 *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, 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 immediately to the bottom of the mixture in the tube.

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

NOTE. – Not more than 2 or 3 drops of the mixture should 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 should not be in contact with the vehicle at the bottom of the cylinder at any time. Allow to stand for 6 minutes 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.

11.4 Calculation

Calculate the leafing power, as a percentage, by means of 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.

11.5 Expression of results

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.

11.6 Precision

11.6.1 *Repeatability.* The results of tests on the same sample by the same operator using the same apparatus should not be considered suspect unless the extreme values differ by more than 6 % absolute.

11.6.2 *Reproducibility.* The results of tests on the same sample in different laboratories should not be considered suspect unless the extreme values differ by more than 10 % absolute.

12. TEST FOR ABSENCE OF LEAFING POWER

12.1 Reagent

Xylene, suitable commercial grade.

12.2 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 minutes, 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.

13. DETERMINATION OF WATER CONTENT

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 Recommendation R 760, *Determination of water by the Karl Fischer method,* may be used with the modifications to the procedures given below.

13.1 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 seconds. 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 seconds to disperse and titrate with the Karl Fischer reagent.

13.2 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.

---

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

13.3 Calculation

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

$$\frac{F\,V}{10\,m}$$

where

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

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

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

14. DETERMINATION OF METALLIC IMPURITIES

The following methods are recommended for referee purposes, but other methods may be used by agreement between purchaser and vendor.

14.1 Determination of lead (spectrophotometric method)

14.1.1 REAGENTS

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

14.1.1.1 *Hydrochloric acid,* 6 M.

14.1.1.2 *Nitric acid,* $d = 1.4$.

14.1.1.3 *Citric acid* solution, 200 g/l.

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

14.1.1.4 *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.

14.1.1.5 *Potassium cyanide* solution, 100 g/l.

14.1.1.6 *Potassium cyanide* solution, 5 g/l.

Dissolve the required amount of potassium cyanide in water and dilute to 1 litre. Take the usual precautions in preparing and keeping these solutions due to the poisonous character of potassium cyanide. Keep in a borosilicate glass container.

14.1.1.7 *Dithizone* solution, 0.2 g/l.

Dissolve 0.200 g of dithizone in 200 ml of chloroform and agitate in a 1 litre 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 litre 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 1000 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.

14.1.1.8 *Dithizone* solution, 0.1 g/l.

Dilute a volume of the dithizone solution (14.1.1.7) with an equal volume of chloroform.

14.1.1.9 *Ammonia* solution, $d = 0.88$.

14.1.1.10 *Chloroform,* pure.

14.1.2 APPARATUS

Usual laboratory equipment, 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 should 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 (14.1.1.7), the same mixture being used for all articles unless the dithizone turns red.

14.1.3 PROCEDURE

14.1.3.1 *Test portion.* Weigh 0.5 ± 0.001 g of the sample in a 250 ml beaker.

14.1.3.2 *Blank test.* Perform a blank test at the same time as the determination.

14.1.3.3 *Determination.* Dissolve the test portion in 15 ml of hydrochloric acid (14.1.1.1). When dissolution is complete, add 5 drops of nitric acid (14.1.1.2) and boil for 2 to 3 minutes. Filter if necessary, washing five times with hot water, combining the filtrate and washings. Adjust the volume to 50 ml, add 15 ml of citric acid solution (14.1.1.3) and neutralize to litmus with ammonia solution (14.1.1.9), adding 4 drops in excess. Add 2 ml of freshly prepared hydroxylammonium chloride solution (14.1.1.4) and 5 ml of potassium cyanide solution (14.1.1.5). Boil for 2 to 3 minutes and cool in a water trough.

Introduce the solution into a 250 ml separating funnel, add 10 ml of dithizone solution (14.1.1.7) and shake for 30 seconds. Draw off the chloroform layer into another separating funnel. Continue to shake the aqueous layer with 5 ml portions of dithizone solution (14.1.1.8), 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 (14.1.1.10).

Wash the combined chloroform extracts with 25 ml of potassium cyanide solution (14.1.1.6) 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, using the green filter, or read the absorbance with a spectrophotometer at a wavelength of 515 nm (blue-sensitive phototube), with chloroform in the reference cell.

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

14.2 Determination of iron

See ISO Recommendation R 793, *Chemical analysis of aluminium and its alloys – Photometric determination of iron (Orthophenanthroline method applicable to iron content between 0.05 and 2.50 %).*