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STANDARD**

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495-2**

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**Organic pigments for paints —**

**Specification —**

Part 2:

**Phthalocyanines**

ICS 87.060.10

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Reference number

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In order to match with technological development and to keep continuous progress in industries, standards are subject to periodic review. Users shall ascertain that they are in possession of the latest edition

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## Foreword

Rwanda Standards are prepared by Technical Committees and approved by Rwanda Standards Board (RSB) Board of Directors in accordance with the procedures of RSB, in compliance with Annex 3 of the WTO/TBT agreement on the preparation, adoption and application of standards.

The main task of technical committees is to prepare national standards. Final Draft Rwanda Standards adopted by Technical committees are ratified by members of RSB Board of Directors for publication and gazettment as Rwanda Standards.

DRS 495-2 was prepared by Technical Committee RSB/TC 056, *Paints, varnishes, Adhesives and Related Products*.

In the preparation of this standard, reference was made to the following standard:

IS 3574 (Part 2): Organic pigments for paints — Specification — Part 2: Phthalocyanines

The assistance derived from the above source is hereby acknowledged with thanks.

DRS 495 consists of the following parts, under the general title *Organic pigments for paints — Specification*:

— *Part 1: Azo pigments*

— *Part 2: Phthalocyanines*

## Committee membership

The following organizations were represented on the Technical Committee on *Paints, varnishes, Adhesives and Related Products* (RSB/TC 056) in the preparation of this standard.

University of Rwanda/College of Sciences and Technology (UR/CST)

University of Rwanda/College of Education (UR/CE)

EARTHENABLE

Standards for Sustainability (SfS)

AMACO Paints

Rwanda Inspectorate, Competition and Consumer Protection Authority (RICA)

REX Paints

Star Construction and Consultancy Ltd

IPRC Kigali

MEI Paints

Rwanda Standards Board (RSB) – Secretariat

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## Introduction

Phthalocyanine pigments have attained a distinct position in organic dyes for paints where clear tone, high light fastness and chemical inertness are required.

Phthalocyanine ( $H_2Pc$ ) is a large, aromatic, macrocyclic, organic compound with the formula  $(C_8H_4N_2)_4H_2$  and is of theoretical or specialized interest in chemical dyes and photoelectricity. It is composed of four isoindole units linked by a ring of nitrogen atoms.  $(C_8H_4N_2)_4H_2 = H_2Pc$  has a two-dimensional geometry and a ring system consisting of 18  $\pi$ -electrons. The extensive delocalization of the  $\pi$ -electrons affords the molecule useful properties, lending itself to applications in dyes and pigments. Metal complexes derived from  $Pc^{2-}$ , the conjugate base of  $H_2Pc$ , are valuable in catalysis, organic solar cells, and photodynamic therapy

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# Organic pigments for paints — Specification — Part 2: Phthalocyanines

## 1 Scope

This Draft Rwanda Standard prescribes requirements, sampling and test methods for phthalocyanine pigments used in paints and related products. It applies to 9 types of pigments as it is described in clause 4.

## 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 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*

ISO 787-7, *General methods of test for pigments and extenders — Part 7: Determination of residue on sieve — Water method — Manual procedure*

ISO 787-2, *General methods of test for pigments and extenders — Part 2: Determination of matter volatile at 105 degrees C*

ISO 787-3, *General methods of test for pigments and extenders — Part 3: Determination of matter soluble in water — Hot extraction method*

ISO 787-9, *General methods of test for pigments and extenders — Part 9: Determination of pH value of an aqueous suspension*

ISO 787-5, *General methods of test for pigments and extenders — Part 5: Determination of oil absorption value*

ISO 787-25, *General methods of test for pigments and extenders — Part 25: Comparison of the colour, in full-shade systems, of white, black and coloured pigments — Colorimetric method*

ISO 787-15, *General methods of test for pigments and extenders — Part 15: Comparison of resistance to light of coloured pigments of similar types*

ISO 787-21, *General methods of test for pigments and extenders — Part 21: Comparison of heat stability of pigments using a stoving medium*

ISO 7579, *Dyestuffs — Determination of solubility in organic solvents — Gravimetric and photometric methods*

RS OIML R 87, *Quantity of product in pre-packages*

### 3 Terms and definitions

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

#### 3.1

##### term

text of the definition

### 4 Types

The 9 types of phthalocyanine pigments covered by this standard are described in table 1:

| Name of pigment   | Cristal modification | Remarks                             |
|-------------------|----------------------|-------------------------------------|
| Pigment Blue 15   | Alpha                | Solvent unstable                    |
| Pigment Blue 15.1 | Alpha                | Non-crystallizing                   |
| Pigment Blue 15.2 | Alpha                | Non-crystallizing, non-flocculating |
| Pigment Blue 15.3 | Beta                 | Non-crystallizing                   |
| Pigment Blue 15.4 | Beta                 | Non-crystallizing, non-flocculating |
| Pigment Blue 15.6 | Epsilon              | -                                   |
| Pigment Blue 16   | -                    | Metal free phthalocyanine           |
| Pigment Green 7   | -                    | Chlorinated                         |
| Pigment Green 36  | -                    | Partially brominated                |

### 5 Requirements

#### 5.1 General requirements

**5.1.1** The pigment shall be in the form of fine dry powder and free from grit or shall be in such condition that can be readily reduced to powder form by crushing under a pallet knife, without any grinding action. Low dusting type organic pigments in the form of soft granules, may be also acceptable.

**5.1.2** The pigment dried shall essentially consist of copper phthalocyanine or copper free phthalocyanine or polychloro/halogenated phthalocyanine. Presence of other ingredients, free from organic or inorganic colouring matter, incorporated during manufacturing process to improve pigmenting properties, constituting more than 5% of total pigment, by mass, shall be declared.

#### 5.2 Specific requirements

The pigments shall comply with the requirements given in table 2 when tested in accordance with the methods prescribed therein.



Table 2 — Specific requirements for phthalocyanine pigments for paints

| S/N | Parameters   |                              | Requirements   | Test methods |
|-----|--|------------------------------|--|--------------|
| 1   | Residue, (on sieve 325 mesh), % by mass, max.                            |                              | 0.1  | ISO 787-7    |
| 2   | Volatile matter, % by mass, max.   |                              | 1.0  | ISO 787-2    |
| 3   | Matter soluble in water, % by mass, max.                                 |                              | 1.0  | ISO 787-3    |
| 4   | pH of aqueous extract (but within $\pm 1\%$ of value of approved sample) |                              | 5 – 9  | ISO 787-9    |
| 5   | Oil absorption (but within $\pm 1\%$ of value of approved sample)        |                              | 30 – 50  | ISO 787-5    |
| 6   | Colour by visual assessment  | Transparency/opacity         | Close match to the approved sample   | ISO 787-25   |
|     |  | Shade (hue)                  | Do   |              |
|     |  | Dullness/brightness (chroma) | Do   |              |
|     |  | Colour strength              | Close match to the approved sample but within $\pm 5\%$ of value of approved sample  |              |
| 7   | Colour by spectro photometry <sup>1</sup>                                | Lightness/darkness, dL       | As agreed between purchaser and supplier but within $\pm 0.5\%$ CIELAB Unit          | ISO 787-25   |
|     |  | Dullness/brightness, dC.     | As agreed between purchaser and supplier but within $\pm 0.5\%$ CIELAB Unit          |              |
|     |  | Shade, dH                    | As agreed between purchaser and supplier but within $\pm 0.5\%$ CIELAB Unit          |              |
|     |  | Colour strength              | As agreed between purchaser and supplier but within $\pm 0.5\%$ of approved standard |              |
|     |  | Total colour difference, dE  | As agreed between purchaser and supplier but within 1 – 2 of CIE Unit                |              |
| 8   | Light and weather fastness   |                              | Not inferior to approved standard sample   | ISO 787-15   |
| 9   | Acid/alkali resistance   |                              | Do   | Annex A      |
| 10  | Heat resistance  |                              | Do   | ISO 787-21   |
| 11  | Solvent fastness   |                              | Do   | ISO 7579     |
| 12  | Resistance to crystallization and flocculation                           |                              | Do   | Annex B      |

<sup>1</sup> These parameters may be tested, if specified by purchaser, to support visual assessment, using a suitable spectrophotometer.

## 6 Packaging and labelling

### 6.1 Packaging

**6.1.1** The pigment shall be packaged in a suitable container that prevents it from deterioration during storage, transportation and normal handling.

**6.1.2** The quantity of product packaged in a container shall be in accordance with the requirements of RS OIML R 87.

### 6.2 Labelling

**6.2.1** Each container shall be labelled legibly and indelibly with the following information in any of the three languages officially accepted in the Republic of Rwanda namely: Kinyarwanda, French and English:

- a) name of the product;
- b) name and address of the manufacturer and/or registered trader mark;
- c) net content;
- d) organic/inorganic colouring matter, % by mass, if added;
- e) batch number
- f) manufacture and expiry dates;
- g) instructions for use and disposal;
- h) special precautions, if any; and
- i) storage condition

## 7 Sampling

Sampling shall be done in accordance with ISO 15528.

## Annex A (normative)

### Acid and alkali resistance test

#### A.1 General

Resistance to acids and alkalis is determined by treating the material under test and the approved sample with dilute hydrochloric acid and dilute sodium hydroxide solution, respectively. The change in the colour and staining power of the material thus treated is compared with the change observed in the case of the approved sample, similarly treated, and tested against the original.

#### A.2 Reagents

**A.2.1 Diluted hydrochloric acid** – Dilute 6mL of hydrochloric acid AR 35% to 100 mL with water.

**A.2.2 Sodium Hydroxide solution** – 10% (m/v)

#### A.3 Procedure

##### A.3.1 Resistance to acid

**A.3.1.1** Carry out the test with approved sample and test sample simultaneously. Weigh 1.0 g pigment under test on a Whatman No. 1 filter paper or equivalent (10 cm x 10 cm). Fold it (as shown in Figure 1) then fold in another direction with the sides overlapping and tie it with a string (keeping one end free and long) so as to enclose the pigment fully protecting it from coming out. Immerse the pigment packet so prepared, suspending at about the middle in 50 mL dilute hydrochloric acid taken in a Nessler's test tube of about 2.5 cm dia and 16 cm height. The extended string can be used for shifting and lifting the packet in acid. Close the test tube with a cork and maintain at room temperature for 24 h or in a water bath at 50 °C for 2 h. Temperature and duration can be changed, as desired, depending upon mutually agreed requirements. Take out the pigment packet and shake the tube to make the acid solution uniform. Compare the colouration of bleed, if any, between the samples.

**A.3.1.2** Wash the treated pigment in the packet with water till free of acid, dry at  $100 \pm 2$  °C on an oven. Test this dried pigment against untreated pigment and compare the change with that obtained in the case of the approved sample when similarly treated and tested.

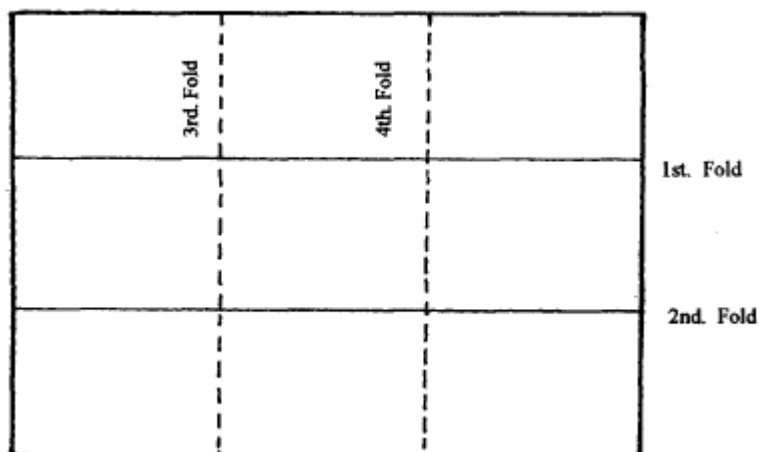


Figure 1 – Folding of filter paper

### A.3.2 Resistance to alkali

Repeat the test by taking dilute sodium hydroxide solution in place of dilute acid in the test method for resistance to acid.

## Annex B (normative)

### Resistance to crystallization and flocculation test

#### B.1 General

Phthalocyanine pigments are directly responsible for loss of staining power or fading due to crystallization which may be accentuated by effects of other pigments or the medium used. To ascertain this property, the material is examined for change in crystal shape and size after refluxing with a strong solvent like xylol. Further this property is tested by comparing the actual performance of a tinted enamel prepared from the material and an agreed medium with that prepared using the approved sample of the pigment and the same medium.

#### B.2 Apparatus

**B.2.1** *Flat-Bottomed Flask*, 250 mL, capacity.

**B.2.2** *Microscope*

#### B.3 Reagents

**B.3.1** *Xylol*

**B.3.2** *Titanium dioxide*, Rutile for paints.

**B.3.3** *Medium short oil alkyd*, dissolved in xylol or any other medium as may be agreed to between the purchaser and the supplier.

#### B.4 procedure

##### B.4.1 Testing for resistance to crystallization

**B.4.1.1** Take about 0.5 g of the pigment in a 250 mL flask and wet it with about 50 mL of xylol. Reflux the contents of the flask for 3 h at 140 °C in a glycerine or oil bath. At the end of this period, remove the flask from the bath and cool the contents. Place few drops of this slurry on a microscope slide. On the same slide, place few drops of the prepared sample with xylol as a control. Examine the two patches under a microscope, at 400 times magnification. Observe the change in the crystal shape and size of the sample under test with the prepared sample. Similarly note the change in the crystal structure and size of the approved sample when similarly treated.

**B.4.1.2** Filter the remaining slurry of the treated sample through a filter paper and keep separately. Prepare a suspension of the prepared sample in xylol and filter the suspension. Check the colour of this filtrate with that obtained after filtration of the treated sample and note bleeding, if any, of both the samples against that of approved samples when similarly treated.

**B.4.1.3** Dry the residue obtained after refluxing with xylol as prescribed in B.4.1.1 on the filter paper in an oven maintained at  $100 \pm 2$  °C for 1h. Compare the staining power of the treated sample against that of the prepared sample. Determine the loss in the case of the approved sample treated similarly.

#### **B.4.2 Testing for resistance to flocculation**

Prepare a tinted enamel using the material and surface treated rutile titanium dioxide in the proportion of 1:100 in the short oil alkyd medium by grinding in a ball-mill or any other suitable equipment. After ensuring thorough grinding of the enamel, reduce it by addition of thinners to spraying consistency. Apply this enamel by spraying on mild steel panel. Rub one corner of the sprayed film with the finger just before it is surface dry. Allow the remaining portion of the material to stand for half an hour. At the end of this period stir the enamel and pour a small quantity over a portion of the sprayed film. Allow it to dry. Observe the difference in colour between the sprayed and rubbed portion of the enamel film with the area on which additional enamel was poured and compare the difference observed against that obtained in the case of the approved sample, similarly treated at the same time.

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## Bibliography

[1] IS 3493, *Method of sampling and test for organic pigments for paints*, 1978

[2]

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