INTERNATIONAL STANDARD

ISO 4630

Third edition
2015-12-01

Clear liquids — Estimation of colour by the Gardner colour scale

Liquides clairs — Évaluation de la couleur au moyen de l'échelle de couleur Gardner

Reference number
ISO 4630:2015(E)

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

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 on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

ISO 4630 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 10, *Test methods for binders for paints and varnishes*, in collaboration with ASTM D 01.34, *Naval stores*. It has been harmonized with ASTM D 1544-04, *Standard test Method for Color of Transparent Liquids (Gardner Color Scale)* and ASTM D 6166-12, *Standard Test Method for Color of Naval Stores and Related Products (Instrumental Determination of Gardner Color)*.

This third edition of ISO 4630 cancels and replaces ISO 4630-1:2004 and ISO 4630-2:2004, which have been technically revised. The main changes are:

a) both standards have been combined into one standard;

b) the spectrophotometric method (formerly described in ISO 4630-2:2004) is the only one standardized now;

c) the original visual comparison of colours (formerly described in ISO 4630-1:2004) has been deleted, and the description of manufacture of the original Gardner colour standards has been moved to Annex A.

Clear liquids — Estimation of colour by the Gardner colour scale

1 Scope

This International Standard specifies a method for estimating the colour of optically clear, yellow/brownish coloured liquid products by means of the Gardner colour scale using colour-measuring instruments. The method uses the Gardner colour scale described in Annex A.

It is applicable to drying oils, varnishes and solutions of fatty acids, polymerized fatty acids, resins, tall oil, tall oil fatty acids, rosin and related products. The results might be invalid if other products are tested.

The method described provides a more precise way of measuring Gardner colour than a visual sample comparison using human eyes. It is applicable to products having colours from Gardner 1 to Gardner 18. The Gardner scale is not applicable to products with colours darker than 18. For products with colours lighter than Gardner 1 the method specified in ISO 6271 applies.

2 Normative references

The following referenced documents, in whole or in part, are normally referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 13632, *Binders for paints and varnishes — Rosin — Sampling and sample preparation for colour measurement*

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

CIE Publication No. 15:2004, *Colorimetry*

3 Principle

The colour of a liquid sample is measured using an instrument capable of measuring transmitted colour and reporting in Gardner colours or in a colour system that can be converted into Gardner colours.

4 Apparatus and materials

4.1 Colour-measuring instrument, spectrophotometer capable of measuring transmitted colour (0°/180° geometry) and reporting the results in the Gardner colour scale. If such an instrument is not available, one may be used which is capable of measuring transmitted colour and reporting in tristimulus values using standard illuminant C and the 2° observer, described in CIE Publication No. 15:2004.

4.2 Absorption cells, 10 mm light path length recommended, unless a different path length is specified by the instrument manufacturer or

4.3 Glass tubes, 11 mm path length. Glass test tubes designed for a specific instrument may be used.

5 Sampling

Take a representative sample of the product to be tested, as described in ISO 15528.

In the case of rosin, take a representative sample, as specified in ISO 13632.

6 Procedure

Baseline calibration of the instrument shall be performed following the instrument manufacturer's recommendations.

If the material to be tested is cloudy, first filter it (see Note). Then, using the same type of glass tube or absorption cell as used for the baseline calibration, fill the glass tube or absorption cell with the product. Take care not to touch the measurement area of the glass tube or absorption cell.

If the material shows any visual haziness, remove the haze e.g. by filtration, centrifugation, heating, ultrasonic treatment or any other suitable means (see Note).

If the haziness cannot be removed, the measured value will be unreliable.

Then, using the same type of glass tube or absorption cell as used for the baseline calibration, fill the glass tube or absorption cell with the product. Take care not to touch the measurement area of the glass tube or absorption cell.

Avoid creating air bubbles when filling the glass tube or absorption cell. If air bubbles are formed and remain trapped, remove them by heating, vacuum, ultrasonic treatment or any other suitable means (see Note).

NOTE Some sample pre-treatments can change the colour.

Insert the glass tube or absorption cell in the instrument and measure the Gardner colour, following the instrument manufacturer's recommended procedure.

Regular checks as per the instrument manufacturer's recommendations should be carried out. These will normally be in the form of checks with certified reference materials.

7 Expression of results

Report the colour in Gardner colour units to the nearest tenth of a Gardner unit as given by the instrument.

8 Precision

8.1 General

The precision of the test method was determined by interlaboratory testing in accordance with ISO 5725-2.

Three different materials were tested by 13 laboratories.

8.2 Repeatability limit, *r*

The repeatability limit *r* is the value below which the absolute difference between two single test results, each the mean of duplicates, obtained on identical material by one operator in one laboratory within a short interval of time using the standardized test method can be expected to lie with a probability of 95 %.

The repeatability for three repeated measurements, made in accordance with this International Standard and expressed as the repeatability limit *r*, is 0,1 Gardner units.

The repeatability standard deviation among test results, related to the above number by the factor 2,8, is 0,02 Gardner units.

8.3 Reproducibility limit, *R*

The reproducibility limit *R* is the value below which the absolute difference between two test results, each the mean of duplicates, obtained on identical material by operators in different laboratories using the standardized test method can be expected to lie with a probability of 95 %.

The reproducibility for three repeated measurements, made in accordance with this International Standard and expressed as the reproducibility limit *R*, is 0,5 Gardner units.

The reproducibility standard deviation among test results, related to the above number by the factor 2,8, is 0,18 Gardner units.

8.4 Bias

Since there is no accepted reference material suitable for determining the bias of the procedure in this test method, bias has not been determined.

9 Test report

The test report shall contain at least the following information:

a) all details necessary to identify the product examined;

b) a reference to this International Standard (ISO 4630);

c) any type of pretreatment of the test sample;

d) the result of the test as indicated in Clause 7;

e) any deviation from the test method specified;

f) any unusual features (anomalies) observed during the test;

g) the date of the test.

Annex A
(normative)

Gardner colour standards

A.1 Reagents

In preparing these standards, use only reagents of recognized analytical grade and only water of at least grade 3 purity as defined in ISO 3696.

A.1.1 Hydrochloric acid, diluted 1 + 17

Mix 1 volume of concentrated hydrochloric acid, 38 % (by mass), ρ = 1,19 g/ml, with 17 volumes of water.

A.1.2 Potassium hexachloroplatinate solution

Dissolve 790 mg of potassium hexachloroplatinate (K_2PtCl_6) in diluted hydrochloric acid (A.1.1) in a 100 ml one-mark volumetric flask. Warm the solution until all the potassium hexachloroplatinate has dissolved. Cool to 20 °C, dilute to the mark with the same hydrochloric acid and mix well.

The stock solution A.1.2 prepared in this way shall have tristimulus values which lie within the limits specified in Table A.1 when measured in accordance with 4.1 and 4.2 (10 mm optical path length) in the spectrophotometer.

Table A.1 — Tristimulus tolerance limits for potassium hexachloroplatinate stock solution (A.1.2)

X	*Y*	*Z*
80,9 ± 0,5	87,1 ± 0,5	24,5 ± 1,5

A.1.3 Cobalt(II) chloride solution

Dissolve 40 g of cobalt(II) chloride hexahydrate ($CoCl_2 \cdot 6H_2O$) in 120 g of diluted hydrochloric acid (A.1.1).

A.1.4 Iron(III) chloride solution

Dissolve 1 000 g of iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$) in 240 g of diluted hydrochloric acid (A.1.1), heating gently if necessary. Adjust the concentration so that the solution has exactly the same colour (visually assessed) as a freshly prepared 30 g/l solution of potassium dichromate ($K_2Cr_2O_7$) in concentrated sulfuric acid (ρ = 1,84 g/ml).

The stock solution A.1.4 prepared in this way shall have tristimulus values *X*, *Y*, *Z* which lie within the limits specified in Table A.2 when measured in accordance with to 4.1 and 4.2 (10 mm optical path length) in the spectrophotometer.

Table A.2 — Tristimulus tolerance limits for iron(III) chloride stock solution (A.1.4)

X	*Y*	*Z*
10,0 ± 2,5	5,3 ± 1,5	0,0 + 0,2

A.2 Preparation of liquid colour standards

A.2.1 Gardner colour standards 1 to 8

Into each of a series of one-mark volumetric flasks of the capacities indicated in Table A.3, transfer from a microburette the volume of potassium hexachloroplatinate solution (A.1.2) shown in Table A.3, make each up to the mark with diluted hydrochloric acid (A.1.1) and mix well.

Table A.3 — Composition of Gardner colour standards 1 to 8

Gardner colour standard number	Volume of potassium hexachloroplatinate solution ml	Volume of volumetric flask ml
1	3,48	50
2	5,47	50
3	8,42	50
4	6,58	25
5	9,60	25
6	5,35	10
7	8,10	10
8	10,00	10

A.2.2 Gardner colour standards 9 to 18

Into a series of 100 ml one-mark volumetric flasks, introduce from burettes the volumes of iron(III) chloride solution (A.1.4) and cobalt(II) chloride solution (A.1.3) shown in Table A.4. Make each up to the mark with diluted hydrochloric acid (A.1.1) and mix well.

Table A.4 — Composition of Gardner colour standards 9 to 18

Gardner colour standard number	Volume of iron(III) chloride solution ml	Volume of cobalt(II) chloride solution ml	Volume of hydrochloric acid ml
9	3,8	3,0	93,2
10	5,1	3,6	91,3
11	7,5	5,3	87,2
12	10,8	7,6	81,6
13	16,6	10,0	73,4
14	22,2	13,3	64,5
15	29,4	17,6	53,0
16	37,8	22,8	39,4
17	51,3	25,6	23,1
18	100,0	0,0	0,0

A.2.3 Storage

Gardner colour standards are stable for 6 months when stored in the dark but should preferably be prepared immediately before use.