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Dyestuffs — Determination of solubility in organic solvents — Gravimetric method

Colorants — Détermination de la solubilité dans les solvants organiques — Méthode gravimétrique

Reference number
ISO 7579:1990(E)

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 7579 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*.

Annexes A and B form an integral part of this International Standard.

International Organization for Standardization
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Introduction

Many dyestuffs, when present in excess, dissolve in solvents to give concentrations that are independent of the ratio of dyestuff to solvent (saturation concentrations). In these instances, the saturation concentration is defined as the solubility. However, some dyestuff/solvent systems do not show a definite saturation concentration and the dyestuff continues to dissolve in the solvent as the proportion of the dyestuff is increased. A graphical method for determining the solubility of these dyestuffs is presented in this International Standard.

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Dyestuffs — Determination of solubility in organic solvents — Gravimetric method

1 Scope

This International Standard specifies a method for determining the solubility of dyestuffs in organic solvents. It is applicable to dyestuffs that do not change chemically under the influence of the solvent, are stable and are non-volatile under the specified drying conditions.

The method is suitable for concentrations between 1 g and 1 000 g of dyestuff per litre of solvent. It is not suitable for the determination of insoluble matter in a dyestuff.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 758:1976, *Liquid chemical products for industrial use — Determination of density at 20 °C.*

ISO 787-2:1981, *General methods of test for pigments and extenders — Part 2: Determination of matter volatile at 105 °C.*

ISO 842:1984, *Raw materials for paints and varnishes — Sampling.*

ISO 1625:1977, *Plastics — Aqueous dispersions of polymers and copolymers — Determination of residue at 105 °C.*

3 Definition

For the purposes of this International Standard, the following definition applies.

solubility: The maximum mass of a dyestuff that is soluble in a given volume of a solvent under specified conditions.

Solubility is expressed in grams per litre of solvent (see annex A).

No distinction is made between "true" solubility and "colloidal" solubility.

4 Principle

Different amounts of the dyestuff are each dispersed in a defined volume of a solvent at 23 °C. After mixing for 24 h, each dispersion is centrifuged and the solids content of the supernatant liquid is determined gravimetrically.

5 Solvent

The organic solvent for the dyestuff shall be completely volatile at 105 °C and the density of the solvent at 23 °C shall be known. The density of the solvent may be determined by the method described in ISO 758, but at a temperature of 23 °C instead of 20 °C. There shall be no chemical interaction between the dyestuff and the solvent.

As organic solvents are normally not chemically pure, the grade of purity (including the type and quantity of any major secondary constituents) shall be given in the test report.

6 Apparatus

Ordinary laboratory apparatus and glassware together with the following.

6.1 Containers, cylindrical, of capacity about 250 ml, of inert material, with tightly fitting lids.

6.2 Stirrer, with adjustable speed control.

A high-speed stirrer shall not be used as it will raise the temperature of the solution.

6.3 Device for rotating the containers (6.1) or **mechanical shaking device.**

6.4 Laboratory centrifuge, capable of imparting a relative centrifugal acceleration of about 10 km/s². A centrifuge with a rotational frequency of 2 000 r/min to 3 000 r/min is suitable.

6.5 Centrifuge tubes, of transparent and inert material, with tightly fitting lids.

6.6 Weighing bottles, squat form, with stoppers.

6.7 Steam bath.

6.8 Air oven, capable of maintaining a temperature of 105 °C ± 2 °C.

7 Sampling

Take a representative sample of the product to be tested, as described in ISO 842.

8 Procedure

8.1 Preliminary determination

If the approximate solubility of the dyestuff in the solvent (clause 5) is not known, carry out a preliminary determination using the procedure described in 8.2.1.2 and 8.2.1.3 with one of the following series of test portions of the dyestuff.

— Dyestuff of low solubility: 1 g, 10 g and 50 g of dyestuff per litre of solvent;

— Dyestuff of high solubility: 100 g, 500 g and 1 000 g of dyestuff per litre of solvent.

Take as the approximate solubility of the dyestuff the concentration of the dispersion in which the undissolved matter after centrifuging is greater than about one-quarter of the total quantity of dyestuff dispersed.

8.2 Determination

8.2.1 Preparation of test suspensions

8.2.1.1 When the approximate solubility of the dyestuff is known or has been determined in the preliminary determination (8.1), carry out the following procedure.

Prepare six suspensions in accordance with 8.2.1.2 by taking test portions of the dyestuff to give concentrations of about 40 %, 60 %, 80 %, 100 %, 120 % and 140 % of the approximate solubility, using the procedure described in 8.2.1.2 and 8.2.1.3. If, at the lowest concentration employed, 10 % or more of the dyestuff remains undissolved, reduce the concentration until more than 90 % of the dyestuff is dissolved. If, at the highest concentration, the residue is less than 25 % of the test portion, then increase the mass of the test portion.

If the solubility limit cannot be obtained because of a large increase in viscosity with increasing mass of the test portion, the last value obtained shall be recorded. (See annex A, clause A.4.)

8.2.1.2 Place a known volume (100 ml to 200 ml) of the solvent in a container (6.1). Using the stirrer (6.2), stir the solvent and add the dyestuff into the vortex, ensuring rapid and complete wetting. Place a loose-fitting cover over the container to prevent excessive loss of solvent and continue stirring for 10 min. At the end of this period, check that there are no large agglomerates of dyestuff. Close the container with its tightly fitting lid and place it on the rotating device (6.3). Rotate the container, maintaining the contents at 23 °C ± 2 °C.

If the container (6.1) is mechanically shaken, this shall be stated in the test report.

8.2.1.3 After agitating the dyestuff and solvent continuously for 24 h, place portions of the mixture in the centrifuge tubes (6.5) and cap each tube. Centrifuge the tubes at 23 °C ± 2 °C for 10 min. Observe if the supernatant liquid is clear; if not, or if in doubt, centrifuge for another 10 min. Decant the supernatant liquid from each tube into a clean, dry container (6.1) and close securely.

8.2.2 Determination of the concentration of the dissolved dyestuff

8.2.2.1 General

Take a known mass of each supernatant liquid prepared in 8.2.1.3 and determine the concentration of the dyestuff using either method A or method B below. Carry out the weighings to the nearest 0,2 mg.

For each determination, the volume of supernatant liquid shall contain at least 30 mg of the dyestuff.

Two determinations shall be carried out on each supernatant liquid. The mean values shall be recorded and used for the calculation as shown in 9.1.

8.2.2.2 Method A (using an oven)

Place a portion (about 3 g) of the clear supernatant liquid into a previously tared weighing bottle (6.6), replace the stopper and weigh. Remove the stopper, place the weighing bottle on the steam bath (6.7) and heat until most of the solvent has evaporated.

Transfer the bottle to the oven (6.8), maintained at 105 °C ± 2 °C, and heat for 1 h.

Place the bottle in a desiccator and, after cooling, weigh the stoppered bottle. Repeat the heating, cooling and weighing operations with heating periods of 30 min until the results of two consecutive weighings do not differ by more than 0,2 mg.

NOTE 1 If the solvent has low volatility, it may be advantageous either to use method B or to remove the final traces of solvent by using a vacuum oven, but care should be taken that none of the dyestuff is lost due to sublimation. Method B should be considered if the dyestuff has a high solubility in the solvent.

8.2.2.3 Method B (using the evaporation apparatus specified in ISO 1625 and described in annex B)

NOTE 2 This method is unsuitable for use with dyestuffs having a low solubility in the solvent because of the smaller quantity of dyestuff solution dried on the apparatus described in ISO 1625 which leads to low precision.

Take a known mass of each supernatant liquid prepared in 8.2.1.3 and evaporate the solvent using the method described in annex B. After removal of all the solvent, dry the residue, cool and weigh. Repeat the heating, cooling and weighing operations until the results of two consecutive weighings do not differ by more than 0,2 mg.

8.2.3 Determination of non-volatile matter of the dyestuff

Use the procedure described in ISO 787-2 and express the non-volatile matter as the ratio of the mass, in grams, of the residue to the mass, in grams, of the test portion, i.e. as a decimal value less than 1.

9 Expression of results

9.1 Calculation

The calculation of the solubility of a dyestuff is dependent on the behaviour of the dyestuff in the particular solvent. If a saturation concentration is obtained, the solubility is determined directly from that concentration (see annex A, clause A.1). If the solubility is dependent on the proportion of dyestuff added, a definite saturation concentration is not obtained and, in this case, the solubility is calculated in accordance with clause A.2 or is recorded together with the proportion of the dyestuff added (see clause A.3).

Calculate the solubility S, in grams per litre, of the dyestuff for each test suspension with which settled matter is observed in the centrifuge tubes in 8.2.1.3, using the equation

$$S = \frac{m_1 \cdot \rho \times 10^3}{(m_0 - m_1)\mathrm{NV}}$$

where

m_0 is the mass, in grams, of the supernatant liquid taken in 8.2.2 ;

m_1 is the mass, in grams, of the dry residue obtained in 8.2.2 ;

ρ is the density, in grams per millilitre, of the solvent at 23 °C;

NV is the non-volatile matter content of the dyestuff (see 8.2.3).

Record the solubility as follows:

Solubility above 1 000 g/l: > 1 000 g/l

Solubility above 10 g/l:
the result to the nearest 1 g/l

Solubility below 10 g/l:
the result to the nearest 0,1 g/l

Solubility below 1 g/l: < 1 g/l

9.2 Precision

No precision data are currently available.

The precision of the method depends very much on the properties of the dyestuff and the solvent. This shall be considered when interpreting the results. Collaborative tests have shown that, in the case of a dyestuff exhibiting a definite saturation concentration (see annex A, clause A.1), a repeatability within ± 5 % is obtained. In the cases described in clause A.2 and clause A.3, the repeatability may well be poorer.

10 Test report

The test report shall contain at least the following information:

a) all details necessary for the identification of the product tested, together with its non-volatile matter content;

b) a reference to this International Standard (ISO 7579);

c) the solvent used and its degree of purity;

d) the method of mixing the dyestuff and solvent;

e) the result of the test, expressed as indicated in 9.1;

f) any deviation from the procedure specified;

g) the date(s) of the test.

Annex A
(normative)

Solubility calculations

A.1 Dyestuff with a definite saturation concentration

For a dyestuff which gives a definite saturation concentration in the chosen solvent, the saturation concentration is reached when an increase in the mass of the test portion results in no further increase in dissolved matter. The solubility may be determined by simply inspecting the results (see table A.1) or by plotting a graph (see figure A.1).

If the saturation concentration corresponds to less than 90 % of the mass of the test portion added to 1 litre of solvent, both these values shall be recorded: for example, a saturation concentration of 170 g/l with a test portion of 200 g/l. Such cases may occur with dyestuffs containing insoluble impurities, for example salts.

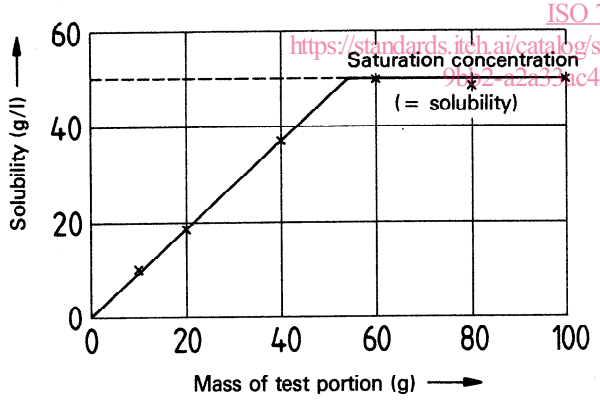

Figure A.1 — Solubility of a dyestuff which gives a definite saturation concentration (graphical presentation of results)

Table A.1 — Solubility of a dyestuff which gives a definite saturation concentration (tabular presentation of results)

Mass of test portion added to 1 litre of solvent g	Mass of dyestuff dissolved in 1 litre of solvent g	Proportion of test portion dissolved in solvent %
10	10	100
20	19	95
40	38	95
60	50	83
80	49	61
100	50	50

A.2 Dyestuff with an "indefinite" saturation concentration

When the results are treated graphically, a dyestuff with an indefinite saturation concentration shows, above a certain point, a distinctly lower rate of increase in the proportion of the test portion dissolved with increasing mass of test portion (see figure A.2). The solubility limit is determined graphically as the concentration at the discontinuity D, provided that the discontinuity represents a concentration at which at least 90 % of the added dyestuff is dissolved. If this is not the case, the solubility limit is determined as outlined in clause A.3.