
INTERNATIONAL STANDARD



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Paint media — Determination of saponification value

Liants pour peintures — Détermination de l'indice de saponification

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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.

International Standard ISO 3681 was drawn up by Technical Committee ISO/TC 35, *Paints and varnishes*, and was circulated to the Member Bodies in December 1974.

It has been approved by the Member Bodies of the following countries :

Austria	India	South Africa, Rep. of
Brazil	Iran	Spain
Bulgaria	Israel	Sweden
Canada	Netherlands	Switzerland
Czechoslovakia	New Zealand	Turkey
France	Portugal	United Kingdom
Germany	Romania	Yugoslavia

No Member Body expressed disapproval of the document.

Paint media — Determination of saponification value

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of the esterified acid content in paint media whereby free acids and acid anhydrides are necessarily included.

Because different paint media vary in their resistance to saponification, this International Standard is of limited applicability. If necessary, completeness of saponification should be checked by repeating the test under more severe conditions which can be achieved by the use of longer saponification time, more concentrated potassium hydroxide solution, or a higher boiling alcohol as solvent.

The annex to this International Standard specifies a procedure adapted to paint media which saponify with difficulty.

The method is not applicable to those materials which show further reaction with alkalis beyond normal saponification.

2 REFERENCE

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

3 DEFINITIONS

3.1 saponification : The formation of alkali salts from derivatives of organic acids.

3.2 saponification value : The number of milligrams of potassium hydroxide (KOH) required for the saponification of 1 g of the product.

4 SAMPLING

A representative sample of the product to be tested shall be taken as specified in ISO 842.

5 REAGENTS

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

5.1 Toluene, or other suitable unsaponifiable solvent.

5.2 Potassium hydroxide, 0,5 N ethanolic solution.

NOTE — If more severe conditions are needed, use a 2 N ethanolic potassium hydroxide solution or use 1,2-ethanediol (ethylene glycol) or 2,2-oxydiethanol (diethylene glycol) as the solvent.

5.3 Hydrochloric acid, 0,5 N solution in a mixture of 4 parts by volume of methanol and 1 part by volume of water.

The normality T of this solution shall be checked shortly before use.

5.4 Phenolphthalein, indicator solution, 10 g/l in 95 % (V/V) ethanol, or **thymolphthalein** indicator solution, 10 g/l in 95 % (V/V) ethanol.

6 APPARATUS

Ordinary laboratory apparatus and

6.1 Conical flask, capacity 250 ml, with ground-glass joint.

6.2 Reflux condenser, with ground-glass joint.

6.3 Burette, or pipette, capacity 25 or 50 ml.

If required,

6.4 Device for potentiometric titration, with glass electrode and reference electrode.

6.5 Magnetic stirrer.

7 PRELIMINARY TEST

If no special saponification conditions are specified or agreed, use 25 ml of the ethanolic potassium hydroxide solution (5.2) and a boiling time of 1 h. For testing whether the saponification value can be determined under these conditions, intensify the conditions by prolongation of the saponification time to at least 2 h and/or by using a 2 N potassium hydroxide solution or a solution of potassium hydroxide in an alcohol with a boiling point distinctly higher than that of ethanol, for example 1,2-ethanediol (ethylene glycol) or 2,2-oxydiethanol (diethylene glycol).

If no increase in the final value is obtained using the more severe conditions, the test may be carried out according to this International Standard. If an increased value is obtained which is not further increased by prolonging the test, this International Standard may be applied using and noting the conditions applied. If no final result is obtainable even under severe conditions of saponification, the method to be used shall be agreed between the interested parties.

8 PROCEDURE

8.1 Test portion

The mass of the test portion depends on the saponification value to be expected (approximate values are given in the table). This mass shall be chosen so that less than half of the volume of potassium hydroxide solution added is sufficient to saponify the test portion.

Weigh, to the nearest 1 mg, the test portion, into the conical flask (6.1).

Expected saponification value	Mass of test portion
mg KOH/g	g
to 10	20
above 10 to 20	10
above 20 to 50	5
above 50 to 100	2,5
above 100 to 200	1,5
above 200 to 300	1
above 300 to 500	0,5
above 500	0,2

8.2 Determination

Dissolve the test portion (8.1), if necessary, in a measured volume of the toluene or other suitable, unsaponifiable solvent (5.1), warming, if necessary, under the reflux condenser (6.2). Add, from the burette or pipette (6.3),

- 25 ml of the 0,5 N potassium hydroxide solution (5.2), or
- 25 ml of a different potassium hydroxide solution (see clause 7 and the note to 5.2), or

- the specified or agreed volume of a potassium hydroxide solution.

Heat the contents of the flask to boiling and maintain at boiling point under reflux for 1 h or the specified or agreed time, or the time found necessary for the preliminary test (see clause 7).

Titrate the hot solution with the hydrochloric acid solution (5.3) after addition of 3 drops of the phenolphthalein or thymolphthalein solution (5.4), or using a potentiometric method.

NOTE – If the potentiometric titration is used, the glass electrode shall have a suitable response time.

8.3 Blank test

Carry out a blank test in the same way as the determination, but omitting the test portion.

9 EXPRESSION OF RESULTS

The saponification value is given, in milligrams of potassium hydroxide per gram of sample, by the formula :

$$56,1 \frac{(V_0 - V_1) \times T}{m}$$

where

m is the mass, in grams, of the test portion (8.1);

T is the exact normality of the hydrochloric acid solution (5.3);

V_0 is the volume, in millilitres, of the hydrochloric acid solution (5.3) required for the blank test;

V_1 is the volume, in millilitres, of the hydrochloric acid solution (5.3) required for the determination.

10 TEST REPORT

The test report shall include at least the following particulars :

- a) a reference to this International Standard or to a corresponding national standard;
- b) the type and identification of the product tested;
- c) the solvent and the concentration and volume of the potassium hydroxide solution used;
- d) the period of boiling;
- e) the type of titration : potentiometric or by indicator (phenolphthalein, thymolphthalein);
- f) the saponification value, expressed in milligrams of potassium hydroxide per gram of product (mg KOH/g);
- g) any deviation, by agreement or otherwise, from the procedure specified;
- h) the date of the test.