
**Soft soldering fluxes — Test
methods —**

**Part 3:
Determination of acid value,
potentiometric and visual titration
methods**

Flux de brasage tendre — Méthodes d'essai —

*Partie 3: Détermination de l'indice d'acide par des méthodes de
titrage potentiométrique et visuel*

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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 of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 44, *Welding and allied processes*, Subcommittee SC 12, *Soldering materials*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Official interpretations of TC 44 documents, where they exist, are available from this page: <https://committee.iso.org/sites/tc44/home/interpretation.html>.

This second edition cancels and replaces the first edition (ISO 9455-3:1992), which has been technically revised.

The main changes compared to the previous edition are as follows:

- the automated titration procedure has been added to [4.4](#);
- the document has been editorially aligned with the current Directives, Part 2.

A list of all parts in the ISO 9455 series can be found on the ISO website.

Soft soldering fluxes — Test methods —

Part 3:

Determination of acid value, potentiometric and visual titration methods

1 Scope

This document specifies two methods for the determination of the acid value of a flux of types 1 and 2 only, as defined in ISO 9454-1.

Method A is a potentiometric titration method and is to be considered as the reference method.

Method B is an alternative, visual end-point, titration method.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

4 Method A: Potentiometric titration method

4.1 Principle

A prepared, weighed sample of the flux is dissolved in a suitable solvent. The resulting solution is titrated with standard tetrabutyl ammonium hydroxide solution, using a glass electrode, the pH or mV readings being recorded simultaneously. From the graph of volume of titrant against pH or mV readings, the point of inflexion is determined, from which the acid value is calculated.

As fluxes of classes 1131 and 1231 (see ISO 9454-1) can lose some acidity during the determination of non-volatile matter, the non-volatile matter obtained from carrying out the procedure of ISO 9455-1 or ISO 9455-2 on these classes of flux should not be used for this determination.

4.2 Reagents

Use only reagents of recognized analytical quality and only distilled, or deionized, water.

4.2.1 Tetrabutyl ammonium hydroxide $[(\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2)_4\text{N}(\text{OH})]$ 0,1 M (0,1 mol/l).

Use a commercially available standard solution or one prepared from a commercially available concentrated standard solution by dilution with propan-2-ol (4.2.2). Alternatively, prepare an 0,1 mol/l tetrabutyl ammonium hydroxide solution by diluting commercial concentrated solution

with propan-2-ol and standardize this solution against an accurately weighed amount of benzoic acid (about 0,5 g) dissolved in dimethylformamide, previously neutralised to thymol blue.

4.2.2 Propan-2-ol [(CH₃)₂CHOH], neutralized with tetrabutyl ammonium hydroxide solution (4.2.1) to a faint pink colour, using phenolphthalein as indicator.

4.2.3 Ethanol (C₂H₅OH), anhydrous, neutralized with tetrabutyl ammonium hydroxide solution (4.2.1) to a faint pink colour using phenolphthalein as indicator.

4.2.4 Toluene (C₆H₅CH₃), neutralized with tetrabutyl ammonium hydroxide solution (4.2.1) to a faint pink colour using phenolphthalein as indicator.

4.2.5 Ethanol/toluene mixture, made by mixing equal volumes of the anhydrous ethanol (4.2.3) and toluene (4.2.4).

4.3 Apparatus

Usual laboratory apparatus and, in particular, the following.

4.3.1 Millivoltmeter or pH meter.

4.3.2 Glass electrode.

4.3.3 Saturated calomel, or silver chloride/silver, electrode.

4.3.4 Magnetic, or mechanical, stirrer, with variable speed drive.

4.3.5 Automated titration system with evaluating processor unit.

4.4 Procedure

4.4.1 General

By preliminary experiments, determine whether the sample is soluble in propan-2-ol, anhydrous ethanol, toluene or the ethanol/toluene mixture. If it is not completely soluble in any of these solvents, select the one in which it appears to be the most soluble. If it is equally soluble in all four solvents, then use propan-2-ol.

Carry out the following procedure, in triplicate, on the flux example.

Weigh, to the nearest 0,001 g, approximately 0,5 g of the solid flux sample, or 2,0 g of the liquid flux sample taking steps to prevent loss of volatile matter during the weighing. Transfer the weighed sample to a 250 ml low form beaker.

Add 100 ml propan-2-ol (4.2.2) or the selected solvent (4.2.3 to 4.2.5), according to the solubility characteristics of the flux. Cover with a watch glass and dissolve the flux by gentle agitation.

4.4.2 Manual procedure

Place the beaker on the stand of the titration assembly with the electrodes, stirrer and burette in position. Adjust the speed of the stirrer to give vigorous stirring without splashing. Titrate with the tetrabutyl ammonium hydroxide solution (4.2.1) adding 1 ml portions and recording the pH, or mV meter readings after each addition. As the endpoint is approached, reduce the additions of titrant to 0,1 ml and continue titrating past the endpoint.

Plot the pH, or potential values against the volume of titrant added to obtain the titration curve. The point of inflexion of the curve corresponds to the end-point of the titration.

NOTE The point of inflexion of the curve can conveniently be determined by using the derivative curve.

Carry out a blank determination, using all reagents, for comparison purposes.

4.4.3 Automated procedure

Details of the automated procedure must be in accordance with the manual of used titration system.

Place the beaker on the stand of the titration assembly and bring other titration equipment in position as defined in the manual. Adjust the speed of the stirrer to give vigorous stirring without splashing. Before titration starts, several parameters shall be entered into the evaluation unit for calculation and identification of the test sample.

Start the automated titration system. The titration curve is plotted automatically.

If there are no other requirements in the manual, carry out a blank determination, using all reagents, for comparison purposes.

4.5 Calculation of results

The acid value is expressed in milligrams of potassium hydroxide per gram of non-volatile matter, regardless of the alkali used to perform the titration.

The acid value (expressed in milligrams of potassium hydroxide per gram of non-volatile matter) is given by [Formula \(1\)](#)

$$\frac{5\ 611\ VM}{mS} \quad (1)$$

where

- V is the volume, in millilitres, of tetrabutyl ammonium hydroxide solution used, minus the volume needed to titrate the blank;
- M is the molarity of the tetrabutyl ammonium hydroxide solution ([4.2.1](#));
- m is the mass, in grams, of the sample taken;
- S is the percentage non-volatile matter content of the sample, determined as described in ISO 9455-1 or ISO 9455-2 (for solid flux samples, $S = 100$).

The acid value of the flux under test is calculated as the mean of the results obtained of the three test samples.

At the end of an automatic titration the evaluation unit automatically calculates the results. Some units plot the titration curve, too.

5 Method B — Titration method with visual end-point

5.1 Principle

A prepared, weighed sample of the flux is dissolved in propan-2-ol, ethanol, toluene, or an ethanol/toluene mixture. The solution is then titrated with standard potassium hydroxide solution and the acid value is calculated.

As fluxes of classes 113 and 123 (see ISO 9454-1) can lose some acidity during the determination of non-volatile matter, the non-volatile matter obtained from carrying out the procedure of ISO 9455-1 or ISO 9455-2 on these classes of flux should not be used for this determination.

5.2 Reagents

Use only reagents of recognized analytical quality and only distilled, or deionized, water.

5.2.1 Ethanol, anhydrous, neutralized with potassium hydroxide, 0,1 mol/l in alcohol (5.2.5), to a faint pink colour using phenolphthalein as indicator.

5.2.2 Toluene, neutralized with potassium hydroxide, 0,1 mol/l in alcohol (5.2.5), to a faint pink colour using phenolphthalein as indicator.

5.2.3 Ethanol/toluene mixture, made by mixing equal volumes of the anhydrous ethanol (5.2.1) and toluene (5.2.2).

5.2.4 Propan-2-ol, neutralized with potassium hydroxide, 0,1 mol/l in alcohol (5.2.5), to a faint pink colour using phenolphthalein as indicator.

5.2.5 Potassium hydroxide solution, 0,1 mol/l in alcohol.

Use a commercially available standard solution or one prepared from a commercially available concentrated standard solution by dilution with ethanol (5.2.1). Alternatively, prepare a 0,1 mol/l potassium hydroxide solution by dissolving $3 \text{ g} \pm 0,1 \text{ g}$ potassium hydroxide (KOH) in 500 ml of ethanol (5.2.1) and standardize this solution against an accurately weighed amount of benzoic acid (about 0,5 g) dissolved in ethanol (5.2.1).

5.2.6 Phenolphthalein indicator solution. Add 1 g of phenolphthalein to approximately 50 ml methanol and mix. When dissolved, dilute to 100 ml with methanol and mix.

5.3 Apparatus

Ordinary laboratory apparatus is required.

5.4 Procedure

By preliminary experiments, determine whether the sample is soluble in propan-2-ol, anhydrous ethanol, toluene or the ethanol/toluene mixture. If it is not completely soluble in any of these solvents, select the one in which it appears to be the most soluble. If it is equally soluble in all four solvents, then use ethanol as the selected solvent.

Carry out the following procedure in triplicate on the flux sample.

Weigh to the nearest 0,001 g sufficient of the flux sample to correspond to approximately 1 g of non-volatile matter (see ISO 9455-1 or ISO 9455-2) taking steps in the case of liquid flux samples to prevent loss of volatile matter during the weighing. Transfer the weighed sample to a suitable flask or beaker and add 100 ml of the selected solvent. Stir until the sample has dissolved as completely as possible. Do not heat.

Add 3 drops of phenolphthalein (5.2.6) and titrate with the potassium hydroxide solution (5.2.5) until a faint pink colour persists throughout the titrated solution for 15 s.

Carry out a blank determination, using all reagents, for comparison purposes.

5.5 Calculation of results

The acid value of the sample, expressed in milligrams of potassium hydroxide per gram of non-volatile matter, is given by [Formula \(1\)](#).

The acid value of the flux under test is calculated as the mean of the results obtained of the three test samples.

6 Precision

6.1 Method A

Method A of this document was subjected to an interlaboratory test programme involving eight laboratories. For five rosin-based fluxes having acid values covering the range 170 mg to 230 mg KOH per gram of non-volatile matter, the estimates for precision data were as follows (expressed in milligrams of potassium hydroxide per gram):

Standard deviations:

— within laboratory	S_w	1,0
— between laboratories	S_b	3,6
Repeatability	r	3,0
Reproducibility	R	10,0

6.2 Method B

Method B of this document was subjected to a limited interlaboratory test programme, involving only five laboratories. For nine rosin-based fluxes having acid values covering the range 160 mg to 300 mg KOH per gram of non-volatile matter, the estimates for precision data were as follows (expressed in milligrams of potassium hydroxide per gram):

Standard deviations:

— within laboratory	S_w	2,1
— between laboratories	S_b	5,8
Repeatability	r	5,8
Reproducibility	R	16,1

7 Test report

The test report shall include the following information:

- the identification of the test sample;
- the test method of this document used (i.e. reference to ISO 9455-3; method A or method B);
- the results obtained;
- any unusual features noted during the determination;
- details of any operation not included in this document, or regarded as optional.