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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*



Reference number
ISO 9455-3:1992(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 9455-3 was prepared by Technical Committee ISO/TC 44, *Welding and allied processes*, Sub-Committee SC 12, *Soldering and brazing materials*.

ISO 9455 consists of the following parts, under the general title *Soft soldering fluxes — Test methods*:

- *Part 1: Determination of non-volatile matter, gravimetric method*
- *Part 2: Determination of non-volatile matter, ebulliometric method*
- *Part 3: Determination of acid value, potentiometric and visual titration methods*
- *Part 5: Copper mirror test*
- *Part 6: Determination of halide content*
- *Part 8: Determination of zinc content*
- *Part 9: Determination of ammonia content*
- *Part 10: Flux efficacy tests, solder spread method*
- *Part 11: Solubility of flux residues*
- *Part 12: Steel tube corrosion test*

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- *Part 13: Determination of flux spattering*
- *Part 14: Assessment of tackiness of flux residues*
- *Part 15: Copper corrosion test*
- *Part 16: Flux efficacy tests, wetting balance method*
- *Part 17: Determination of surface insulation resistance of flux residues (Comb test)*

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Soft soldering fluxes — Test methods —

Part 3:

Determination of acid value, potentiometric and visual titration methods

1 Scope

This part of ISO 9455 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

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 9455. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 9455 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 9454-1:1990, *Soft soldering fluxes — Classification and requirements — Part 1: Classification, labelling and packaging*.

ISO 9455-1:1990, *Soft soldering fluxes — Test methods — Part 1: Determination of non-volatile matter, gravimetric method*.

ISO 9455-2:—¹⁾, *Soft soldering fluxes — Test methods — Part 2: Determination of non-volatile matter, ebulliometric method*.

1) To be published.

3 Method A: Potentiometric titration method

3.1 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.

NOTE 1 As fluxes of classes 1.1.3 and 1.2.3 (see ISO 9454-1) may 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.

3.2 Reagents

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

3.2.1 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 (3.2.2). 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.

3.2.2 Propan-2-ol [(CH₃)₂CHOH], neutralized with tetrabutyl ammonium hydroxide solution (3.2.1) to a faint pink colour, using phenolphthalein as indicator.

3.2.3 Ethanol (C₂H₅OH), anhydrous, neutralized with tetrabutyl ammonium hydroxide solution (3.2.1) to a faint pink colour using phenolphthalein as indicator.

3.2.4 Toluene (C₆H₅CH₃), neutralized with tetrabutyl ammonium hydroxide solution (3.2.1) to a faint pink colour using phenolphthalein as indicator.

3.2.5 Ethanol/toluene mixture.

Mix equal volume of the anhydrous ethanol (3.2.3) and toluene (3.2.4).

3.3 Apparatus

Usual laboratory apparatus and, in particular, the following.

3.3.1 Millivoltmeter or pH meter.

3.3.2 Glass electrode.

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

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

3.4 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 propan-2-ol.

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

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 (3.2.2) or the selected solvent (3.2.3 to 3.2.5), according to the solubility

characteristics of the flux. Cover with a watch glass and dissolve the flux by gentle agitation.

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 (3.2.1) adding 1 ml portions and recording the pH, or mV meter readings after each addition. As the end-point is approached, reduce the additions of titrant to 0,1 ml and continue titrating past the end-point.

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 2 The point of inflexion of the curve may conveniently be determined by using the derivative curve.

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

3.5 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

$$\frac{5\,611\,VM}{mS}$$

where

<i>V</i>	is the volume, in millilitres, of tetrabutyl ammonium hydroxide solution used, minus the volume needed to titrate the blank;
<i>M</i>	is the molarity of the tetrabutyl ammonium hydroxide solution (3.2.1);
<i>m</i>	is the mass, in grams, of the sample taken;
<i>S</i>	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, <i>S</i> = 100).

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

4 Method B: Titration method with visual end-point

4.1 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.

NOTE 3 As fluxes of classes 1.1.3 and 1.2.3 (see ISO 9454-1) may 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 Ethanol, anhydrous, neutralized with potassium hydroxide, 0,1 mol/l in alcohol (4.2.5), to a faint pink colour using phenolphthalein as indicator.

4.2.2 Toluene, neutralized with potassium hydroxide, 0,1 mol/l in alcohol (4.2.5), to a faint pink colour using phenolphthalein as indicator.

4.2.3 Ethanol/toluene mixture. Mix equal volumes of the anhydrous ethanol (4.2.1), and toluene (4.2.2).

4.2.4 Propan-2-ol, neutralized with potassium hydroxide, 0,1 mol/l in alcohol (4.2.5), to a faint pink colour using phenolphthalein as indicator.

4.2.5 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 (4.2.1). 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 (4.2.1) and standardize this solution against an accurately weighed amount of benzoic acid (about 0,5 g) dissolved in ethanol (4.2.1).

4.2.6 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.

4.3 Apparatus

Ordinary laboratory apparatus is required.

4.4 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 indicator (4.2.6) and titrate with the potassium hydroxide solution (4.2.5) until a faint pink colour persists throughout the titrated solution for 15 s.

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

4.5 Calculation of results

The acid value of the sample, expressed in milligrams of potassium hydroxide per gram of non-volatile matter, is given by:

$$\frac{5\,611\,VM}{mS}$$

where

V	is the volume, in millilitres, of potassium hydroxide solution (4.2.5) used in the titration, minus the volume used to titrate the blank;
M	is the molarity of the potassium hydroxide solution (4.2.5);
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 on each of the three test samples.

5 Precision

5.1 Method A

Method A of this part of ISO 9455 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

5.2 Method B

Method B of this part of ISO 9455 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

6 Test report

The test report shall include the following information:

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

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