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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

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Foreword

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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).

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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** $[(CH_3CH_2CH_2CH_2)_4N(OH)]$ 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 [$(CH_3)_2CHOH$], neutralized with tetrabutyl ammonium hydroxide solution (4.2.1) to a faint pink colour, using phenolphthalein as indicator.

4.2.3 Ethanol (C_2H_5OH), anhydrous, neutralized with tetrabutyl ammonium hydroxide solution (4.2.1) to a faint pink colour using phenolphthalein as indicator.

4.2.4 Toluene ($C_6H_5CH_3$), 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.