SLOVENSKI STANDARD
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Talila za mehko spajkanje - Preskusne metode - 3. del: Določevanje kislinskega števila s potenciometrično in vizualno titracijo (ISO/DIS 9455-3:2019)

Soft soldering fluxes - Test methods - Part 3: Determination of acid value, potentiometric and visual titration methods (ISO/DIS 9455-3:2019)

Flussmittel zum Weichlöten - Prüfverfahren - Teil 3: Bestimmung des Säurewertes, potentiometrische und visuelle Titrationsmethoden (ISO/DIS 9455-3:2019)

Flux de brasage tendre - Méthodes d'essai - Partie 3: Détermination de l'indice d'acide par des méthodes de titrage potentiométrique et visuel (ISO/DIS 9455-3:2019)

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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).

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For an explanation on 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 the following URL: 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*.

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:

— Automated titration procedure has been added to Clause 3.4.

ISO 9455 consists of the following parts.

— *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 (excluding fluoride) 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 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: Surface insulation resistance comb test and electrochemical migration test of flux residues*

Soft soldering fluxes — Test methods —

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

1 Scope

This part of IS0 9455 specifies two methods for the determination of the acid value of a flux of types 1 and 2 only, as defined in IS0 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 documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 9454-1, *Soft soldering fluxes — Classification and requirements — Part 1: Classification, labelling and packaging*

IS0 9455-1, *Soft soldering fluxes — Test methods — Part 1: Determination of non-volatile matter, gravimetric method*

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

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 As fluxes of classes 1.1.3.1 and 1.2.3.1 (see IS0 9454-1) may lose some acidity during the determination of non-volatile matter, the non-volatile matter obtained from carrying out the procedure of IS0 9455-1 or IS0 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** $[(CH_3CH_2CH_2CH_2)_4N(OH)]$ 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 (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 [$(CH_3)_2CHOH$], neutralized with tetrabutyl ammonium hydroxide solution (3.2.1) to a faint pink colour, using phenolphthalein as indicator.

3.2.3 Ethanol (C_2H_5OH), anhydrous, neutralized with tetrabutyl ammonium hydroxide solution (3.2.1) to a faint pink colour using phenolphthalein as indicator.

3.2.4 Toluene ($C_6H_5CH_3$), 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.

3.3.5 Automated titration system with evaluating processor unit.

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 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 (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.

3.4.1 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 (3.2.1) 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 may conveniently be determined by using the derivative curve.

