INTERNATIONAL STANDARD

ISO 9455-8

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Soft soldering fluxes — Test methods —

Part 8:
Determination of zinc content

Flux de brasage tendre — Méthodes d'essai —

Partie 8: Dosage du zinc

Reference number
ISO 9455-8:1991(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 9455-8 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 8:
Determination of zinc content

1 Scope

This part of ISO 9455 specifies a method for the determination of the zinc content of water soluble fluxes of type 3, as defined in ISO 9454-1. The method is intended to apply to an aqueous solution of flux containing approximately 25 % of zinc, expressed as zinc chloride ($ZnCl_2$). For solid fluxes and for solutions whose concentrations differ appreciably from those stated above, an appropriate adjustment should be made to the amount of sample taken for the test.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this part of ISO 9455. At the time of publication, the edition indicated was 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 edition of the standard 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.*

3 Principle

To an aqueous solution of the flux, containing sulfuric acid, ammonium chloride is added and the solution made alkaline by the addition of ammonia solution. Any precipitated iron is removed as hydroxide. The zinc content of the flux is determined by titrating the solution using EDTA.

4 Reagents

During the test use only reagents of recognized analytical quality and only distilled, or deionized, water.

4.1 Disodium dihydrogen ethylenediaminetetra-acetate solution (EDTA) standard volumetric solution, c(EDTA) = 0,1 mol/l.

Dry approximately 10 g of disodium dihydrogen ethylenediaminetetra-acetate ($C_{10}H_{14}N_2Na_2O_8.2H_2O$) for 2 h at 80 °C. Dissolve 9,308 g of the dried salt in water, transfer to a 250 ml volumetric flask, dilute to the mark and mix well.

NOTE 1 1 ml of 0,1 mol/l EDTA solution is equivalent to 0,006 54 g of zinc, or 0,013 63 g of zinc chloride.

4.2 Buffer solution (pH 10).

Dissolve 7 g of ammonium chloride (NH_4Cl) in 20 ml of water. Add 57 ml of ammonia solution (ϱ 0,91 g/ml) and dilute to 100 ml. Mix well.

4.3 Sulfuric acid, 20 % (V/V) solution

Add cautiously, with stirring, 200 ml of sulfuric acid (ϱ 1,84 g/ml) to 400 ml of water and mix. Cool and dilute to 1 000 ml and mix well.

4.4 Eriochrome black T mixture.

Dissolve 0,25 g of eriochrome black T (also known as mordant black 11 and solochrome black) and 2,25 g hydroxylammonium chloride in a mixture of 45 ml of water and 5 ml of triethanolamine.

NOTE 2 This solution should be freshly prepared from a recently purchased batch of eriochrome black T.

4.5 Hydrochloric acid, ϱ 1,16 g/ml or 1,18 g/ml.

4.6 Ascorbic acid.

4.7 Ammonium chloride, (NH_4Cl).

4.8 Ammonia solution, ϱ 0,91 g/ml.

5 Apparatus

Ordinary laboratory apparatus is required.

6 Procedure

Carry out the following procedure in triplicate.

By means of a pipette, transfer 25,0 ml of the flux sample to a 500 ml volumetric flask.

NOTE 3 This method is intended for flux samples containing approximately 25 % zinc in aqueous solution. For flux samples having a concentration appreciably different from this, make an appropriate adjustment to the volume of sample used for the test.

Solid fluxes should be dissolved in water to give a flux sample having a zinc content around 25 %.

Add 10 ml of sulfuric acid solution (4.3), dilute to the mark with water and mix well.

By means of a pipette, transfer 20,0 ml of this solution to a 250 ml beaker, dilute to approximately 100 ml and add a few drops of hydrochloric acid (4.5). Add approximately 10 g of ammonium chloride (4.7) and make alkaline with ammonia solution (4.8).

If iron is precipitated, boil the solution and filter it through a rapid filter paper, washing the precipitate well with water containing a few drops of ammonia solution (4.8). Combine the filtrate and washings and discard the precipitate.

Add approximately 0,2 g of ascorbic acid (4.6) and 15 ml of buffer solution (4.2). Stir well and add dropwise sufficient eriochrome black T mixture (4.4) to produce a clearly visible red/purple colour. Titrate with the EDTA solution (4.1) to a blue end point.

NOTE 4 The end point of the titration is more easily detected if the progress of the titration is viewed using an incandescent light source.

7 Calculation of results

The percentage, by mass, of zinc in the original flux sample, expressed as zinc chloride, is given by the following formula:

$$\frac{1{,}363\ V}{d}$$

where

V is the volume, in millilitres, of EDTA solution (4.1) used;

d is the density, in grams per millilitre, of the original flux sample at 20 °C, determined by the use of a hydrometer.

NOTE 5 If the original flux were in solid form, or if a different sample volume were used for the test (see clause 6, note 3), then a suitable adjustment will need to be made to the calculation formula.

8 Precision

Tests were carried out on three fluxes containing zinc covering the range 17 % (m/m) to 28 % (m/m). Four laboratories took part in the tests and the estimates for precision data, expressed as a percentage of zinc chloride, were as follows:

Standard deviations:		
— within laboratory	s_w	0,24
— between laboratories	s_b	0,30
Repeatability	r	0,66
Reproducibility	R	0,84

9 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 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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