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Soft soldering fluxes — Test methods —

Part 1:

Determination of non-volatile matter, gravimetric
method

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Flux de brasage tendre — Méthodes d'essai —

ISO 9455-1:1990

Partie 1: Dosage des matières non volatiles par gravimétrie
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INTERNATIONAL

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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.

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 9455-1 was prepared by Technical Committee ISO/TC 44, *Welding and allied processes*.

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 titration method*
- 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: *Ease and efficiency of flux residue removal*
- Part 12: *Steel tube corrosion test*

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- *Part 13: Determination of flux spattering*
- *Part 14: Determination of tackiness — Chalk powder method*
- *Part 16: Flux efficacy tests — Wetting balance method*

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Soft soldering fluxes — Test methods —

Part 1:

Determination of non-volatile matter, gravimetric method

1 Scope

This part of ISO 9455 specifies a gravimetric method for the determination of the content of non-volatile matter in soft soldering fluxes. It is applicable to liquid and paste fluxes of type 1, as defined in ISO 9454-1.

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:—¹⁾, *Soft soldering fluxes — Classification and requirements — Part 1: Classification, labelling and packaging.*

3 Principle

A prepared, weighed sample of the flux is heated in a boiling water bath and then in an oven to drive off volatile matter. The cooled sample is re-weighed and the percentage of non-volatile matter is calculated.

4 Apparatus

Ordinary laboratory apparatus and

1) To be published.

4.1 Petri dish, thin section glass nominally 100 mm diameter.

4.2 Weighing pipette, 20 ml to 25 ml capacity, for sampling liquid fluxes.

4.3 Boiling water bath.

4.4 Drying oven, maintained at a temperature of $110\text{ °C} \pm 2\text{ °C}$.

4.5 Desiccator, containing silica gel as the desiccant.

5 Procedure

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

Dry the glass Petri dish (4.1) in the oven (4.4). Cool the dish in the desiccator to room temperature and weigh to the nearest 0,001 g. Transfer to the dish sufficient of the sample, weighed to the nearest 0,001 g, to yield between 0,5 g and 1,0 g of non-volatile matter, taking steps to prevent loss of volatile matter during the weighing. For liquid samples the use of the weighing pipette (4.2) is recommended.

Heat the dish and contents on a boiling water bath (4.3) to evaporate the bulk of the volatile matter.

NOTE 1 For samples already of a low volatile content, this preliminary drying may be omitted.

Remove the Petri dish (4.1) from the boiling water bath (4.3) and wipe the outside of the dish to remove condensed water.

Place the dish and contents in the oven and dry for 3 h at $110\text{ °C} \pm 2\text{ °C}$. Cool in the desiccator to room temperature and weigh the dish and residue to the nearest 0,001 g.

NOTE 2 The drying temperature used in the procedure, 110 °C , is applicable to fluxes containing propan-2-ol or other solvents of a similar boiling point. For fluxes with solvents of a higher boiling point, an alternative temperature may be agreed upon between the supplier and the purchaser. Fluxes of classes 1.1.3 and 1.2.3 (see ISO 9454-1), if activated with organic acids, and of classes 1.1.2 and 1.2.2 if they contain mixed organic and halide activators (see ISO 9454-1), may lose a proportion of the activator content during the procedure described in clause 5.

6 Expression of results

The non-volatile matter in the flux sample, expressed as a percentage by mass, is given by the formula

$$\frac{m_2}{m_1} \times 100\%$$

where

m_2 is the mass, in grams, of the residue after drying;

m_1 is the mass, in grams, of the sample taken.

7 Precision

This method was subjected to a limited interlaboratory test programme, involving five laboratories.

Repeatability and reproducibility were calculated in accordance with ISO 5725²⁾; the results are given in table 1.

8 Test report

The test report shall include the following information:

- the identification of the test sample;
- the test method used (i.e. reference to this part of ISO 9455);
- details of any variation of the drying temperature/drying time used (see note 2 to clause 5);
- the results obtained;
- any unusual features noted during the determination;
- details of any operation not included in this part of ISO 9455, or regarded as optional.

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 Table 1 — Precision data

Parameter	Non-volatile matter content of test flux		
	5 % (m/m)	15 % (m/m)	25 % (m/m)
Within laboratory:			
— standard deviation, S_w	0,06	0,09	0,12
— repeatability, r	0,19	0,25	0,35
Between laboratories:			
— standard deviation, S_b	0,08	0,20	0,47
— reproducibility, R	0,25	0,55	1,30

2) ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

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