INTERNATIONAL STANDARD

ISO 9455-2

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Soft soldering fluxes — Test methods —

Part 2:
Determination of non-volatile matter, ebulliometric method

Flux de brasage tendre — Méthodes d'essai —

Partie 2: Dosage des matières non volatiles par ébulliométrie

Reference number
ISO 9455-2:1993(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 9455-2 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*

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- *Part 12: Steel tube corrosion test*
- *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* [Technical Report]
- *Part 17: Determination of surface insulation resistance of flux residues (Comb test)*
- *Part 18: Electrochemical migration test for flux residues*

Annex A of this part of ISO 9455 is for information only.

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Soft soldering fluxes — Test methods —

Part 2:
Determination of non-volatile matter, ebulliometric method

1 Scope

This part of ISO 9455 specifies an ebulliometric method for the determination of the content of non-volatile matter in soft soldering fluxes. It is applicable to liquid fluxes of classes 1.1.1, 1.1.2 and 1.1.3, as defined in ISO 9454-1, and only those fluxes containing solvents of relatively high volatility, such as propan-2-ol. The method is applicable only to fluxes with a non-volatile content of 10 % or more.

NOTE 1 For the determination of the content of non-volatile matter of liquid fluxes of these classes, where the solvent has a lower volatility than propan-2-ol, the method given in ISO 9455-1 should be used.

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 4791-1:1985, *Laboratory apparatus — Vocabulary relating to apparatus made essentially from glass, porcelain or vitreous silica — Part 1: Names for items of apparatus.*

ISO 9454-1:1990, *Soft soldering fluxes — Classification and requirements — Part 1: Classification, labelling and packaging.*

IEC 68-2-20:1979, *Environmental testing — Part 2: Tests — Test T: Soldering.*

3 Principle

A weighed amount of the flux sample is evaporated in an ebulliometer until the entire volatile part is driven off, which is determined by following the graph of distillation temperature against time. The flux residue is considered as the non-volatile content of flux.

4 Apparatus

Usual laboratory apparatus and, in particular, the following.

4.1 Martin ebulliometer (ISO 4791-1:1985, 5.04.10), but with a spherical distillation flask, as shown in figure 1.

NOTE 2 It has been found that better heat exchange characteristics are obtained between the heater and the distillation flask if a spherical flask is used rather than the pear-shaped flask normally supplied with the Martin ebulliometer.

4.2 Thermostatically controlled heater, suitable for heating the distillation flask of the ebulliometer.

4.3 Laboratory thermometer, covering the range 50 °C to 150 °C with 0,5 °C graduations, for use with the ebulliometer.

NOTE 3 A corrosion-resistant thermocouple, connected to a digital indicator, or recorder, may be used in place of the laboratory thermometer.

4.4 Laboratory balance, accurate to 0,1 mg.

5 Procedure

Carry out the following procedure in duplicate.

All the glass apparatus used shall be clean and dry. Remove the distillation flask from the Martin ebulliometer (4.1) and weigh it to the nearest 0,001 g. Introduce between 40 g and 50 g of the flux sample into the flask and re-weigh the flask to the nearest 0,001 g. This weighing shall be carried out quickly, so as to avoid loss of solvent by evaporation.

Attach the flask to the ebulliometer and fit the thermometer, or thermocouple, (4.3) as shown in figure 1, through a new rubber plug in the top of the distillation column.

Adjust the thermometer or thermocouple height so that its tip is on a level with the entry of the cross tube leading to the condenser. Turn on the water in the condenser. Turn the heater on. When the flux begins to boil, regulate the heat so as to collect the condensate at the rate of 1 drop per second for the first millilitre and 2 drops per second thereafter.

Record the temperature and time at approximately 2 min intervals and plot the distillation curve (temperature versus time) during the course of the distillation. A typical distillation curve is given in figure 2.

NOTE 4 This curve may be obtained directly if a thermocouple (see 4.3) is used and connected to a suitable recorder.

Stop the distillation when the temperature drops back to 75 °C. Remove the distillation flask immediately and put it into a desiccator while it cools down to room temperature. Weigh the distillation flask containing the non-volatile residue.

6 Calculation of results

The non-volatile matter content for each determination, in % (*m/m*), is equal to

$$\frac{(P_3 - P_1)}{(P_2 - P_1)} \times 100$$

where

P_1 is the mass, in grams, of the empty distillation flask;

P_2 is the mass, in grams, of the flask and the flux sample;

P_3 is the mass, in grams, of the flask and the non-volatile residue.

The values calculated for the duplicate determinations on the flux sample should not differ by more than 0,2 % (*m/m*).

Calculate the mean of the two results, to obtain the result for the non-volatile matter content of the flux sample.

NOTE 5 The experimental technique may be verified by carrying out the procedure given in clause 5 on a standard flux, containing 25 % (*m/m*) non-volatile content, prepared as described in annex A.

7 Precision

Interlaboratory tests were carried out on three fluxes containing non-volatile matter within the range 18 % (*m/m*) to 26 % (*m/m*). Six laboratories took part in the tests and the estimates for precision data, expressed as % (*m/m*) non-volatile matter, were as follows:

Standard deviations		
— within laboratory	s_w	0,32
— between laboratories	s_b	1,29
Repeatability	r	0,9
Reproducibility	R	3,6

8 Test report

The test report shall include the following information:

a) 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.

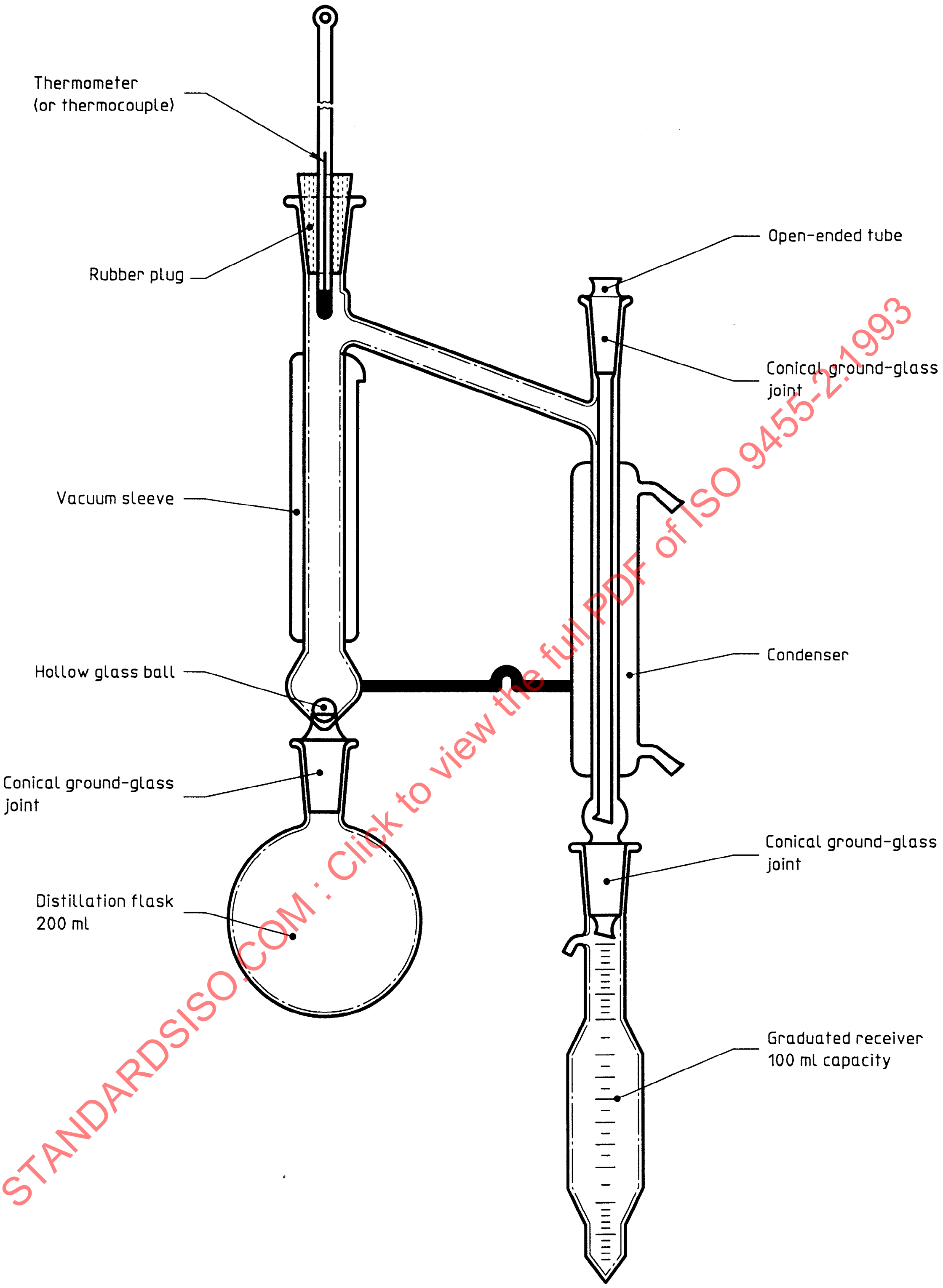

Figure 1 — Martin ebulliometer with spherical distillation flask

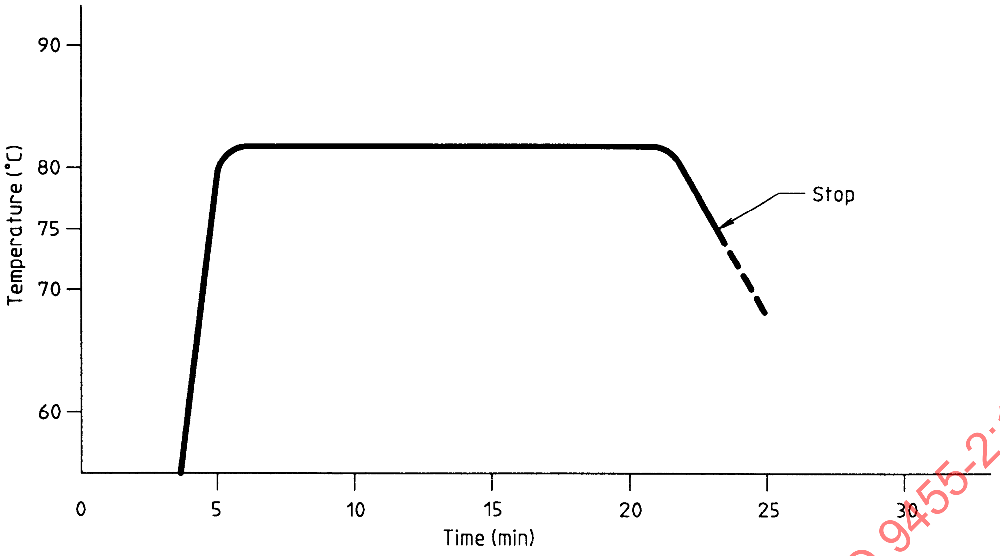

Figure 2 — Typical distillation curve

Annex A
(informative)

Method for the preparation of standard reference rosin (colophony) based liquid fluxes, having 25 % (*m/m*) non-volatile content

A.1 General

This annex gives a method for the preparation of two standard rosin (colophony) based liquid fluxes having 25 % (*m/m*) non-volatile content, one non-activated and the other halogen-activated (i.e. being respectively class 1.1.1.A and class 1.1.2.A, as defined in ISO 9454-1). The specifications for the flux constituents are based on IEC 68-2-20:1979, appendix C.

The standard flux may be used by a laboratory to verify its experimental procedure, in the method for the determination of non-volatile content, described in the main body of this part of ISO 9455.

A.2 Principle

The non-activated flux is prepared by dissolving a special grade of rosin (colophony) in propan-2-ol. The halogen-activated flux is prepared in a similar way, with the addition of diethylamine hydrochloride.

A.3 Apparatus

Usual laboratory apparatus and, in particular, the following.

A.3.1 Oven, capable of being maintained at 110 °C ± 2 °C.

A.4 Reagents

Use only reagents of recognized analytical grade.

A.4.1 Gum rosin (colophony), water-white grade or equivalent, having the following properties:

Acid value:	155 mg KOH /g to 180 mg KOH /g
Softening point:	70 °C minimum
Flow point:	76 °C minimum
Ash:	0,05 % maximum
Solubility:	To give clear 1 + 1 solution in propan-2-ol

A.4.2 Diethylamine hydrochloride, dried for 2 h at 110 °C ± 2 °C.

A.4.3 Propan-2-ol, having the following composition:

Propan-2-ol:	99,5 % (*m/m*) minimum
Acid content:	0,002 % (*m/m*) maximum
Non-volatile content:	0,2 % (*m/m*) maximum

A.5 Procedure

A.5.1 Non-activated rosin (colophony)

Weigh 25 g ± 0,1 g of rosin (A.4.1) and dissolve it with gentle mixing in 75 g ± 0,1 g of propan-2-ol (A.4.3).

A.5.2 Halogen-activated rosin (colophony)

Weigh 0,39 g ± 0,01 g of diethylamine hydrochloride (A.4.2) and dissolve it in 75 g ± 0,1 g of propan-2-ol (A.4.3). Then add 25 g ± 0,1 g of rosin (A.4.1) and dissolve it with gentle mixing. This flux solution contains 0,5 % (*m/m*) of active chloride.

A.6 Storage

The standard flux solutions, prepared as described in A.5 must be stored in a container, properly closed at all times, away from heat or extreme cold.