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Soft soldering fluxes — Test methods —

Part 9:
Determination of ammonia content

Flux de brasage tendre — Méthodes d'essai —

Partie 9: Dosage de l'ammoniac

Reference number
ISO 9455-9:1993(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 9455-9 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*

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Soft soldering fluxes — Test methods —

Part 9:
Determination of ammonia content

1 Scope

This part of ISO 9455 specifies a distillation method for the determination of the ammonia content of solid, paste or liquid fluxes. The method is applicable to fluxes of class 3.1.1 only, 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 9454-1:1990, *Soft soldering fluxes — Classification and requirements — Part 1: Classification, labelling and packaging.*

3 Principle

The prepared flux solution is distilled with sodium hydroxide to expel the ammonia present in the flux. The resulting distillate is passed into a standard sulfuric acid solution. The excess acid is then titrated with sodium hydroxide solution and the ammonia content of the flux is calculated.

4 Reagents

4.1 General

Use only reagents of recognized analytical quality and only distilled, or deionized, water.

4.2 Sodium hydroxide solution, 1,0 mol/l standard solution, commercially available.

Alternatively, use an approximately 1,0 mol/l solution of sodium hydroxide, prepared by the following method. Dissolve 40 g of sodium hydroxide in water and cool. Transfer the solution to a 1 litre volumetric flask, dilute to the mark and mix well. Standardize this solution with 0,5 mol/l sulfuric acid solution (4.3).

4.3 Sulfuric acid, 0,5 mol/l, standard solution, commercially available.

Alternatively, use an approximately 0,5 mol/l solution of sulfuric acid prepared by the following method. Cautiously add 30 ml of sulfuric acid ($\varrho = 1,84$ g/ml) to 400 ml of water and mix. Cool and transfer to a 1 litre volumetric flask, dilute to the mark and mix well. Standardize this solution with a standard solution prepared from anhydrous sodium carbonate.

NOTE 1 1 ml of 0,5 mol/l sulfuric acid is equivalent to 0,053 5 g of ammonium chloride.

4.4 Sulfuric acid, 50 % (*V/V*) solution.

Adopting appropriate safety precautions, carefully add 500 ml of sulfuric acid ($\varrho = 1,84$ g/ml) to 500 ml of water. Mix well.

WARNING — This is a potentially dangerous procedure and should be carried out by a trained person.

4.5 Sodium hydroxide solution, 10 mol/l.

Dissolve 400 g of sodium hydroxide in water. Dilute to 1 litre and mix well. This solution should be prepared in a water-cooled polyethylene beaker and stored in a polyethylene bottle.

4.6 Methyl orange indicator solution, 0,1 g/100 ml.

Dissolve 0,1 g of methyl orange in 100 ml water. Mix well.

5 Apparatus

In addition to ordinary laboratory apparatus, the apparatus shown in figure 1 is required.

6 Procedure

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

6.1 Preparation of flux test solution

6.1.1 Solid fluxes

Weigh 10 g of the solid flux sample into a 400 ml beaker. Add water and sufficient sulfuric acid solution (4.4) to clear the solution. Transfer to a 500 ml volumetric flask, dilute to the mark and mix.

6.1.2 Paste fluxes

For water-soluble paste fluxes, weigh 10 g of the paste flux sample into a 400 ml beaker. Add water and sufficient sulfuric acid solution (4.4) to clear the solution. Transfer to a 500 ml volumetric flask, dilute to the mark and mix.

NOTE 2 For non-water-soluble paste fluxes, the method of preparation of the flux test solution can require modification. In such cases, advice should be sought from the manufacturer.

6.1.3 Liquid fluxes

By means of a pipette, transfer 25 ml of the liquid flux sample to a 500 ml volumetric flask. Dilute to the mark and mix.

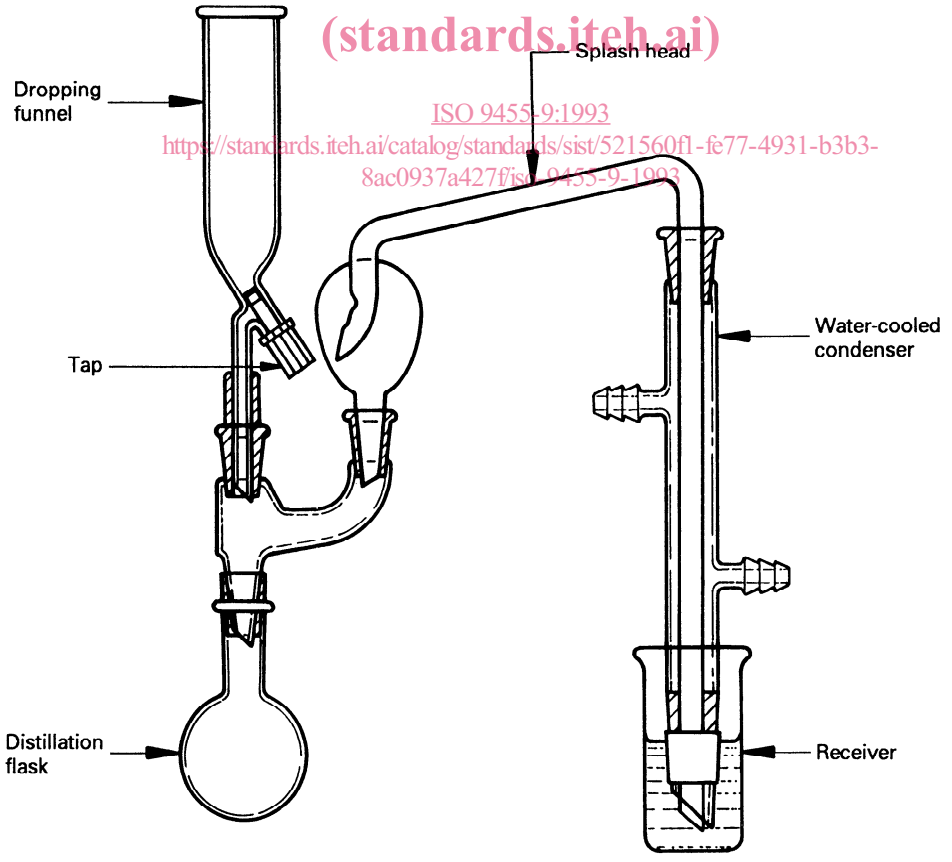

Figure 1 — Apparatus for determination of ammonia content

6.2 Determinations

Set up the apparatus as shown in figure 1. Ensure that the open end of the water-cooled condenser is well below the level of the solution contained in the receiver.

By means of a pipette or burette, transfer 15,0 ml of the sulfuric acid solution (4.3) and 100 ml of water to the receiver.

Transfer 100 ml of the flux test solution (6.1) to the distillation flask. Introduce 30 ml of the 10 mol/l sodium hydroxide solution (4.5) to the dropping funnel, with the tap closed.

Open the tap of the dropping funnel and run all but approximately 2 ml of the sodium hydroxide solution contained in the funnel into the flask. Heat the contents of the flask to boiling and boil briskly for 10 min, so that the ammonia present in the flux test solution is driven off and collected in the receiver.

NOTE 3 This ammonia reacts quantitatively with a proportion of the sulfuric acid contained in the receiver.

Wash the inside of the water-cooled condenser twice with water and add the washings to the receiver.

Titrate the solution in the receiver with 1 mol/l sodium hydroxide solution (4.2), using methyl orange (4.6) as indicator.

7 Calculation of results

7.1 Solid or paste flux samples

The content of ammonia, w, expressed as a percentage by mass of ammonium chloride, is calculated for solid or paste flux samples, from the following formula:

$$w = \left[\frac{(15 - V) \times 0{,}053\ 5 \times 5}{m} \right] \times 100$$

which can be simplified to

$$w = \frac{26{,}75\ (15 - V)}{m}$$

where

V is the volume, in millilitres, of sodium hydroxide solution (4.2) used in the titration;

m is the mass, in grams, of the solid or paste flux sample taken.

7.2 Liquid flux samples

The content of ammonia, w, expressed as a percentage by mass of ammonium chloride, is calculated for liquid flux samples from the following formula:

$$w = \frac{26{,}75\ (15 - V)}{25\varrho}$$

where ϱ is the density, in g/ml, of the liquid flux sample, at 20 °C, determined with a hydrometer.

8 Precision

8.1 General

This method was subjected to a limited interlaboratory test programme. The repeatability and reproducibility of the method were calculated in accordance with ISO 5725[1] ; the results are given in 8.2.

8.2 Precision data

Tests were carried out on two zinc chloride fluxes, containing nominal ammonia contents of 2,3 % and 7,0 % respectively. Five laboratories took part in the tests with the following results:

Table 1

Parameter		Nominal ammonia content of test flux	
		2,3 %	7,0 %
Within laboratory:			
standard deviation	s_w	0,04	0,13
repeatability	r	0,13	0,36
Between laboratories			
standard deviation	s_b	0,11	0,93
reproducibility	R	0,30	2,62

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

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 of 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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