INTERNATIONAL STANDARD

ISO 9455-6

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Soft soldering fluxes — Test methods —

Part 6:

iTeh S Determination and detection of halide (excluding fluoride) content (standards.iteh.ai)

Flux de brasage tendre — Méthodes d'essai https://standards.iteh.ai/catalog/standards/sist/ce105b5e-bcd1-4b8f-ad68-Partie 6: Dosage et détection des halogénures (à l'exception des fluorures)



Reference number ISO 9455-6:1995(E)

Foreword

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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 FVIEW a vote.

International Standard ISO 9455-6 was prepared by Technical Committee ISO/TC 44, Welding and allied processes, Subcommittee SC 12, Soldering and brazing materials.

https://standards.iteh.ai/catalog/standards/sist/ce105b5e-bcd1-4b8f-ad68-ISO 9455 consists of the following parts, under Sthe ageneral/4title-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 and detection 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

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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 forms an integral part of this part of ISO 9455.

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Soft soldering fluxes — Test methods —

Part 6:

Determination and detection of halide (excluding fluoride) content

1 Scope

This part of ISO 9455 specifies three quantitative to revision methods for the determination of the ionic halide (excluding fluoride) content of soldering fluxes. Halides possibility are calculated as chlorides. A useful qualitative test **S**. It standards is method for the detection of ionic halides is also demaintain rescribed. ISO 9455-6:1995

Method A is a potentiometric titration method for the determination of halide (excluding fluoride) content and is applicable to flux classes 1 and 2, defined in ISO 9454-1. This method, which is to be considered the reference method for these fluxes, is suitable for halide contents generally within the range 0,05 % (m/m) to 2 % (m/m) in the non-volatile matter of the flux.

Method B is a titration method for the determination of the total halide (excluding fluoride) content of water-soluble fluxes. It is applicable to flux classes 2.1.2.A and 3.1.1.A, as defined in ISO 9454-1.

Method C is a titration method for the determination of the halide (excluding fluoride) content of watersoluble fluxes containing phosphates and is applicable to flux class 3.2.1.A, as defined in ISO 9454-1.

Method D is a qualitative test, using silver chromate test paper, for the presence of ionic halides. The technique may be used for all classes of flux.

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 5725-2:1994, Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.

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

ISO 9455-1:1990, Soft soldering fluxes — Test methods — Part 1: Determination of non-volatile matter, gravimetric method.

ISO 9455-2:1993, Soft soldering fluxes — Test methods — Part 2: Determination of non-volatile matter, ebulliometric method.

3 Method A: Potentiometric method (Reference method)

3.1 Principle

A prepared, weighed sample of the flux is dissolved in a suitable solvent. The resulting solution is titrated with standard silver nitrate solution, using a silver electrode, the mV readings being recorded simultaneously. From the graph of volume of titrant readings against mV readings, the point of inflexion is determined and the percentage halide content, expressed as chloride, is calculated. The method is not suitable for the determination of fluoride.

This method is applicable to flux classes 1 and 2, as defined in ISO 9454-1.

3.2 Reagents

3.2.1 General

Only reagents of recognized analytical quality and distilled or deionized water with a conductivity less than 10 μ S/cm are to be used.

3.2.2 Propan-2-ol.

3.2.3 Silver nitrate solution (0,02 mol/l).

3,4 g of silver nitrate (AgNO₃) are dissolved in water (see note 1) then transferred to a 1 litre volumetric flask and diluted to the mark with water. It is mixed thoroughly.

3.3.4 Magnetic or mechanical stirrer, with variable speed drive, the stirrer bar to be PTFE-coated.

3.4 Procedure

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

Weigh, to the nearest 0,001 g, approximately 0,5 g of the solid flux sample or 2 g of the liquid flux sample, avoiding loss of volatile matter during the weighing, and transfer it to a 250 ml low-form beaker.

NOTE 3 This procedure is suitable for halide ion concentrations generally within the range 0,1 % (m/m) to 1 % (m/m) in the non-volatile content of the solution to be titrated. For flux samples having halide ion concentrations outside this range, the sample mass taken should be adjusted, as follows:

a) for flux samples having a halide ion concentration in the range 0.05 % (m/m) to 0.1 % (m/m), the mass of sample taken should be double; and

b) for flux samples having a halide ion concentration in the range 1 % (m/m) to 2 % (m/m), the mass of sample taken should be halved.

Alternatively a commercially available concentrate is **CarrAdd 100 mL of b**opan-2-ol (3.2.2), or water, according used to prepare 0,02 mol/l standard silver nitrate solution. ISO 94530te (4)5 Cover with a watch glass and allow to dishttps://standards.iteh.ai/catalog/standSolves.with.gentle.agitation.d68-

The silver nitrate solution is standardized using the $7_{acab/so-9455-6-1995}$ method given in annex A, to derive the correlation NOTE 4 For factor f_1 . This is stored in an amber glass bottle.

NOTES

1 The silver nitrate (AgNO_3) should be dried at 110 °C \pm 5 °C for 16 h and cooled in a desiccator before weighing.

2 1 ml of silver nitrate solution (3.2.3) is equivalent to 0,000 709 $\times f_1$ g of chloride ion.

3.3 Apparatus

Ordinary laboratory apparatus and

3.3.1 Millivolt meter.

3.3.2 Silver electrode.

3.3.3 Mercury/mercurous sulfate 1 mol/l sodium sulfate electrode, or a reversible hydrogen electrode. NOTE 4 For fluxes of class 1.2, as defined in ISO 9454-1 the choice of solvent should be agreed between the flux manufacturer and the user.

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 silver nitrate solution (3.2.3), adding 1 ml portions and recording the mV meter readings after each addition. As the end-point is approached, reduce the additions of titrant to 0,1 ml and continue titrating past the end-point.

Plot the 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 5 The point of inflexion of the curve may conveniently be determined by using the derivative curve.

Carry out a blank determination, using all reagents, for comparison purposes.

3.5 Calculation of results

The halide (excluding fluoride) content, expressed as the percentage, by mass, of chloride in the flux, is given by:

$$\frac{0,070 \ 9 \times V}{m} \ f_1$$

where

- *V* is the volume, in millilitres, of silver nitrate solution used, less the volume needed to titrate the blank;
- f_1 is the correlation factor for the silver nitrate solution (see 3.2.3);
- *m* is the mass, in grams, of the sample taken.

The halide content of the flux sample is given by the mean of the three results obtained on the triplicate test samples.

The halide content may also be expressed as the percentage, by mass, of chloride in the non-volatile matter by:

$$\frac{7,09 \times V}{mS} f_1$$

where

The test is applicable to fluxes of classes 2.1.2.A and 3.1.1.A only, as defined in ISO 9454-1.

Some liquid fluxes contain free hydrochloric acid and the halide content will, consequently, be greater than the amount equivalent to the zinc and other chlorides present.

4.2 Reagents

4.2.1 General

Only reagents of recognized analytical quality and distilled or deionized water with a conductivity less than 10 μ S/cm are to be used.

4.2.2 Silver nitrate solution (0,1 mol/l).

16,99 g of silver nitrate $(AgNO_3)$ are dissolved in water (see note 1) then transferred to a 1 litre volumetric flask and diluted to the mark with water. This is mixed thoroughly.

f1 **iTeh STANDARD** Alternatively, a commercially available concentrate is used to prepare 0,1 mol/l standard silver nitrate sol-(standards.itetion.ai)

ISO 9455-6:1995 https://standards.itch.ai/catalog/standards/sist/enubod_given_in_annex_A, to derive the correlation is the percentage non-volatileomatter.comp-9455-6-1995-2. This is stored in an amber glass bottle.

S is the percentage non-volatile matter content of the sample, determined as described in ISO 9455-1 or ISO 9455-2. For solid flux samples, S = 100.

4 Method B: Titration method for halide (excluding fluoride) content of watersoluble fluxes

4.1 Principle

A prepared sample of the flux is diluted to a known volume with water and a specified volume of sulfuric acid of known concentration. The solution is then treated with a known quantity of silver nitrate solution and the excess silver nitrate is titrated with ammonium thiocyanate. A blank determination using water in place of the flux is carried out simultaneously and the percentage halide content, expressed as chloride, is calculated. The method is not suitable for the determination of fluoride.

NOTES

6 The silver nitrate (AgNO_3) should be dried at 110 °C \pm 5 °C for 16 h and cooled in a desiccator before weighing.

7 1 ml of silver nitrate solution (4.2.2) is equivalent to 0,003 545 \times f_2 grams of chloride ion.

4.2.3 Ammonium thiocyanate solution (approximately 0,1 mol/l).

8 g of ammonium thiocyanate (NH₄CNS) are dissolved in water then transferred to a 1 litre volumetric flask and diluted to the mark with water. This is mixed thoroughly.

Alternatively, a commercially available concentrate is used to prepare 0,1 mol/l standard ammonium thiocyanate solution.

4.2.4 Sulfuric acid solution [20 % (V/V)]. 200 ml of sulfuric acid (density 1,84 g/ml) is cautiously added, with stirring, to 400 ml of water. It is then mixed, cooled, diluted to 1 litre and mixed thoroughly.

4.2.5 Ammonium ferric sulfate indicator solution.

10 g of ammonium ferric sulfate $[NH_4Fe(SO_4)_2 \cdot 12H_2O]$ are dissolved in 100 ml water.

4.2.6 Nitrobenzene

WARNING — Care should be exercised in the handling and disposal of this hazardous reagent.

4.2.7 Nitric acid (density 1,42 g/ml).

4.3 Apparatus

Ordinary laboratory apparatus.

4.4 Procedure

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

By means of a pipette, transfer 25 ml of the sample to a 500 ml volumetric flask. Add 10 ml of sulfuric acid solution (4.2.4), dilute to the mark with water and mix.

By means of a pipette, transfer 10 ml of this solution to a glass-stoppered 500 ml conical flask and add^{ISO 9455-6:1995} 50 ml of water, followed by 5 ml of nitricació (4:2:7)log/standards/sist/ce1 b0et5957acab/iso-9455-6-1

To a similar flask transfer 60 ml of water and 5 ml of nitric acid (4.2.7) and carry out, simultaneously, a blank determination following the same procedure and using the same quantities of reagents as used for the flux sample.

By means of a burette or a pipette, add 50 ml of silver nitrate solution (4.2.2) to the contents of the flask.

NOTE 8 2 ml of nitrobenzene (4.2.6) may be added to assist coagulation of the precipitate.

Re-stopper the flask and shake it vigorously. Add 5 ml of ammonium ferric sulfate indicator solution (4.2.5) and titrate with ammonium thiocyanate solution (4.2.3), shaking the flask vigorously between additions of the titrant. The end-point is indicated when a faint orange-red colour first persists throughout the solution in the flask.

4.5 Standardization of the ammonium thiocyanate solution (4.2.3)

Transfer 70 ml of water and 5 ml of nitric acid (4.2.7) to a 500 ml conical flask. By means of a pipette, add 25 ml of silver nitrate solution (4.2.2). Add 5 ml of

ammonium ferric sulfate indicator solution (4.2.5) and titrate with the ammonium thiocyanate solution (4.2.3), as in the procedure given in 4.4.

Calculate the correlation factor f_3 for the ammonium thiocyanate, as follows:

$$f_3 = \frac{25 \times f_2}{\text{volume of NH}_4\text{CNS}}$$

4.6 Calculation of results

The halide (excluding fluoride) content, expressed as the percentage by mass of chloride in the flux, is given by the following formula:

$$\left[\frac{(\text{titre}_{\text{blank}} - \text{titre}_{\text{sample}}) \times 0,003 \ 545 \times f_3}{0,5 \ d}\right] \times 100$$

which simplifies to:

where:

$$\frac{0,709 \times V}{d} f_3$$

solution solution

- t/ce105bfe_bcd1_4b8f-ad68-5-6-195the density, in grams per millilitre, of the original flux sample, at 20 °C, determined by the use of a hydrometer;
- f_3 is the correlation factor for the ammonium thiocyanate obtained in 4.5.

The halide content of the flux sample is given by the mean of the three results obtained on the triplicate test samples.

NOTE 9 As an alternative practice the original 25 ml sample may be weighed and the final calculation adjusted accordingly.

5 Method C: Titration method for the determination of halide (excluding fluoride) content of water-soluble fluxes containing phosphates

5.1 Principle

Oxalic acid is added to complex any copper which may be present in the flux and then ferric nitrate is added to complex the phosphate. The halide content of the flux, calculated as chloride, is then determined volumetrically using silver nitrate and ammonium thiocyanate. The method is not suitable for the determination of fluoride.

This method is applicable to fluxes of class 3.2.1.A as defined in ISO 9454-1.

5.2 Reagents

5.2.1 General

Only reagents of recognized analytical quality and distilled or deionized water with a conductivity less than 10 µS/cm are to be used.

5.2.2 Silver nitrate solution (0,02 mol/l).

3,4 g of silver nitrate (AgNO₃) are dissolved in water (see note 1) then transferred to a 1 litre volumetric flask and diluted to the mark with water. This is mixed thoroughly.

Alternatively, a commercially available concentrate is used to prepare 0,02 mol/l standard silver nitrate solution.

The silver nitrate solution is standardized using the

NOTES

10 The silver nitrate https://daylog.job ai/catalog/standards/sist/ce 110 °C ± 5 °C for 16 h and cooled in a desiccator before 9455 NOTE 12 weighing.

ISO 9455-6:199:

11 1 ml of silver nitrate solution (5.2.2) is equivalent to 0,000 709 $\times f_1$ grams of chloride ion.

5.2.3 Ammonium thiocyanate solution, (approximately 0,02 mol/l).

1,6 g of ammonium thiocyanate (NH₄CNS) are dissolved in water then transferred to a 1 litre volumetric flask and diluted to the mark. This is mixed thoroughly. This solution is standardized with 0,02 mol/l silver nitrate solution (5.2.2), as described in 5.5.

5.2.4 Nitric acid solution (10 % V/V).

100 ml of nitric acid (density 1,42 g/ml) are diluted to 1 litre and mixed thoroughly.

5.2.5 Oxalic acid.

WARNING — Care should be exercised in the handling and disposal of this toxic reagent.

5.2.6 Ferric nitrate.

5.2.7 Nitrobenzene

WARNING - Care should be exercised in the handling and disposal of this hazardous reagent.

5.3 Apparatus

Ordinary laboratory apparatus.

5.4 Procedure

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

Weigh, to the nearest 0,001 g, approximately 10 g of the flux sample.

Transfer it to a 100 ml volumetric flask, add approximately 70 ml of water, stopper the flask and mix. Add oxalic acid (5.2.5) in increments of approximately 0,2 g and mix well, until the blue copper colour just disappears. Dilute to the mark and mix thoroughly. Allow to stand for approximately 10 min and filter through a dry filter paper.

By means of a pipette, transfer 50 ml of the filtrate factor f_1 . This is stored in an amber glass bottle and structure a glass-stoppered 250 ml conical flask. Add 15 g of ferric hitrate (5.2.6) and, by pipette, 5 ml of the silver nitrate solution (5.2.2). Stopper the flask and shake it well.

> 2 ml of nitrobenzene (5.2.7) may be added to assist coagulation of the precipitate.

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Titrate the excess silver nitrate with ammonium thiocyanate solution (5.2.3) to a faint permanent orangered end-point.

Carry out a blank determination following the same procedure, using the same quantities of reagents, but omitting the flux sample.

5.5 Standardization of the ammonium thiocyanate solution (5.2.3)

Dissolve 15 g of ferric nitrate (5.2.6) and approximately half the quantity of oxalic acid (5.2.5) used in 5.4, in 70 ml of 10 % (V/V) nitric acid solution (5.2.4) in a 250 ml conical flask. Add, by pipette, 5 ml of silver nitrate solution (5.2.2) and titrate with ammonium thiocyanate solution (5.2.3) to a faint permanent orange-red end-point.

Calculate the correlation factor f_4 for the ammonium thiocyanate, as follows:

$$f_4 = \frac{5 \times f_1}{\text{volume of NH}_4\text{CNS}}$$