BRITISH STANDARD

BS EN ISO
9455-6:1997

Soft soldering fluxes — Test methods —

Part 6: Determination and detection of halide (excluding fluoride) content

The European Standard EN ISO 9455-6:1997 has the status of a British Standard

ICS 25.160.50

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National foreword

This British Standard is the English language version of EN ISO 9455-6:1997. It is identical with ISO 9455-6:1995. It partially supersedes BS 5625:1985 which will be withdrawn following publication of other European standards in this series.

The UK participation in its preparation was entrusted by Technical Committee NFE/27, Tin and tin alloys, to Subcommittee NFE/27/4, Solders and soldering fluxes, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request.

Cross-references

Attention is drawn to the fact that annex ZA lists normative references to international publications with their corresponding European publications. The British Standards which implement these international or European publications may be found in the BSI Standards Catalogue under the section entitled "International Standards Correspondence Index", or using the "Find" facility of the BSI Standards Electronic Catalogue.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, the EN title page, page 2, the ISO title page, pages ii to iv, pages 1 to 10 and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

This British Standard, having been prepared under the direction of the Engineering Sector Board, was published under the authority of the Standards Board and comes into effect on 15 April 1997

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EN ISO 9455-6

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Descriptors: See ISO document

English version

Soft soldering fluxes – Test methods – Part 6: Determination and detection of halide (excluding fluoride) content (ISO 9455-6:1995)

Flux de brasage tendre – Méthodes d'essai – Partie 6: Dosage et détection des halogénures (à l'exception des fluorures) (ISO 9455-6:1995)

Flußmittel zum Weichlöten – Prüfverfahren – Teil 6: Bestimmung und Nachweis des Halogenidgehaltes (außer Fluorid) (ISO 9455-6:1995)

This European Standard was approved by CEN on 1996-12-12. CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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European Committee for Standardization
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Europäisches Komitee für Normung

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Foreword

The text of the International Standard from Technical Committee ISO/TC 44 “Welding and allied processes” of the International Organization for Standardization (ISO) has been taken over as an European Standard by Technical Committee CEN/TC 121 “Welding”, the secretariat of which is held by DS.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by July 1997, and conflicting national standards shall be withdrawn at the latest by July 1997.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 9455-6:1995 has been approved by CEN as a European Standard without any modification.

NOTE Normative references to International Standards are listed in annex ZA (normative).

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INTERNATIONAL STANDARD

ISO 9455-6

First edition
1995-09-15

Soft soldering fluxes — Test methods —

Part 6:
Determination and detection of halide (excluding fluoride) content

Flux de brasage tendre — Méthodes d'essai —
Partie 6: Dosage et détection des halogénures (à l'exception des fluorures)

Reference number
ISO 9455-6:1995(E)

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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 9455-6 was prepared by Technical Committee ISO/TC 44, *Welding and allied processes,* Subcommittee 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 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;*

— *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.

Descriptors: Soldering, soldering fluxes, tests, chemical analysis, detection, determination of content, halides, volumetric analysis.

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1 Scope

This part of ISO 9455 specifies three quantitative methods for the determination of the ionic halide (excluding fluoride) content of soldering fluxes. Halides are calculated as chlorides. A useful qualitative test method for the detection of ionic halides is also described.

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 water-soluble 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 µS/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 ($AgNO_3$) 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.

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 method given in annex A, to derive the correlation factor f_1. This is stored in an amber glass bottle.

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

NOTE 2 1 ml of silver nitrate solution (**3.2.3**) is equivalent to 0,000 709 × 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.

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.

Add 100 ml of propan-2-ol (**3.2.2**), or water, according to the solubility characteristics of the flux (see note 4). Cover with a watch glass and allow to dissolve with gentle agitation.

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 potions 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

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 water-soluble 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.

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 µS/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.

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

The silver nitrate solution is standardized using the method given in annex A, to derive the correlation factor f_2. This is stored in an amber glass bottle.

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

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

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

8 g of ammonium thiocyanate (NH_4CNS) 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 50 ml of water, followed by 5 ml of nitric acid (**4.2.7**).

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:

$$f_3 = \frac{25 \times f_2}{\text{volume of } NH_4CNS}$$

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:

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

which simplifies to:

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

where:

V is the volume, in millilitres, of ammonium thiocyanate solution (**4.2.3**) used in the titration of the blank, minus the volume used in the titration of the flux sample;

d is the 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 ($AgNO_3$) 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.

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 method given in annex A, to derive the correlation factor f_1. This is stored in an amber glass bottle.

NOTE 10 The silver nitrate ($AgNO_3$) should be dried at 110 °C ± 5 °C for 16 h and cooled in a desiccator before weighing.

NOTE 11 1 ml of silver nitrate solution (**5.2.2**) is equivalent to 0,000 709 × f_1 grams of chloride ion.

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

1,6 g of ammonium thiocyanate (NH_4CNS) 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 into a glass-stoppered 250 ml conical flask. Add 15 g of ferric nitrate (**5.2.6**) and, by pipette, 5 ml of the silver nitrate solution (**5.2.2**). Stopper the flask and shake it well.

NOTE 12 2 ml of nitrobenzene (**5.2.7**) may be added to assist coagulation of the precipitate.

Titrate the excess silver nitrate with ammonium thiocyanate solution (**5.2.3**) to a faint permanent orange-red 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:

$$f_4 = \frac{5 \times f_1}{\text{volume of } NH_4CNS}$$

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5.6 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:

$$\left[\frac{(\text{titre}_{\text{blank}} - \text{titre}_{\text{sample}}) \times 0{,}000\ 709 \times f_4}{0{,}5m}\right] \times 100$$

which simplifies to:

$$\frac{0{,}141\ 8 \times V}{m} f_4$$

where:

V is the volume, in millilitres, of ammonium thiocyanate solution (**5.2.3**) used in the titration of the blank minus the volume used in the titration of the flux sample;

m is the mass, in grams, of the flux sample originally taken;

f_4 is the correlation factor for the ammonium thiocyanate obtained in **5.5**.

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

6 Method D: Qualitative test for the presence of ionic halides in flux

6.1 Principle

Ionic halides, primarily chloride and bromide in liquid soldering fluxes, are detected at a level of about 0,000 7 g/ml and above, by performing a simple spot test using silver chromate-impregnated paper. This qualitative test can be used for all types of flux, flux residues, flux-cored solder wire and solder pastes.

NOTE 13 The presence of phenols and/or amines in the flux will adversely affect the test results.

6.2 Reagents

6.2.1 *General*

Only reagents of recognized analytical quality and distilled or deionized water are to be used.

6.2.2 *Potassium chromate solution*

2 g potassium chromate (K_2CrO_4) are dissolved in 1 litre of water.

6.2.3 *Silver nitrate solution* (0,01 mol/l)

1,7 g of silver nitrate ($AgNO_3$) 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 0,01 mol/l silver nitrate solution. The silver nitrate solution is kept in an amber glass bottle.

6.2.4 *Standard chloride solution*

0,31 g ± 0,005 g of diethylamine hydrochloride are dissolved in propan-2-ol (**6.2.5**) in a 100 ml volumetric flask and made up to the mark with propan-2-ol.

NOTE 14 1 ml of this solution contains 1 mg chloride ion.

6.2.5 *Propan-2-ol*

6.2.6 *Acetone*

6.3 Apparatus

Ordinary laboratory apparatus, and

6.3.1 *Silver chromate paper*

Silver chromate paper is available commercially but it can also be prepared by the following method. Strips (20 mm to 50 mm wide) or sheets of chromatographic paper are immersed in potassium chromate solution (**6.2.2**) then drained and dried. The dried papers are immersed in the silver nitrate solution (**6.2.3**) then removed and washed with water. The uniform orange-brown coloured paper is then dried in a dark place and cut into 50 mm × 20 mm rectangular test papers. The test papers are stored in an amber glass bottle and kept away from light. The silver chromate papers should be used within four weeks of preparation.

6.3.2 *Soxhlet extraction apparatus.*

6.4 Procedure

6.4.1 ***Preparation of flux test solution***

6.4.1.1 *For liquid flux sample*

Use the flux, as supplied, as the flux test solution.

6.4.1.2 *For flux-cored solder*

Cut a length of the flux-cored solder weighing approximately 150 g and seal the ends by crimping. Wipe the surface clean with a cloth moistened with acetone (**6.2.6**). Place the sample in a beaker, add sufficient water to cover the sample and boil for 5 min to 6 min. Remove the sample, rinse it with acetone (**6.2.6**) and allow to dry.

Protecting the solder surface from contamination, cut the sample into 3 mm to 5 mm lengths, using a scalpel so as not to crimp the cut ends. Place the cut segments in the extraction tube of a clean Soxhlet extraction apparatus (**6.3.2**) and extract the flux with propan-2-ol (**6.2.5**) or other suitable solvent (see note 15) until the return condensate is clear.

Adjust the non-volatile matter content of the extract to 25 % by mass, by evaporation or by dilution with the solvent used during the extraction stage, to produce the flux test solution.

6.4.1.3 *For solder paste samples*

Place 200 ml propan-2-ol (**6.2.5**) or other suitable solvent (see note 15) in a clean Erlenmeyer flask. Add 40 g ± 2 g of the solder paste to the flask, cover with a watch glass and boil for 10 min to 15 min. Allow the powder to settle for 2 min to 3 min and decant the hot solution into a funnel containing filter paper, collecting the flux extract in a clean vessel. Add 50 ml propan-2-ol (**6.2.5**) to the Erlenmeyer flask containing the residue and heat to boiling. Allow the powder to settle for a short time and decant the hot solution through the same filter paper, combining the extracts. The solution may not necessarily be clear.

Adjust the non-volatile matter content of the filtered solution to 25 % (*m*/*m*) as described in **6.4.1.2**, to produce the flux test solution.

6.4.1.4 *For solid flux samples*

Produce a flux test solution having a non-volatile matter content of 25 % (*m*/*m*) by solution in propan-2-ol (**6.2.5**) or other suitable solvent (see note 15).

NOTE 15 If, when preparing the flux test solution, it is found that the flux is not soluble in propan-2-ol, then use another suitable water-miscible solvent and give details of this solvent in the test report 8 f).

6.4.2 *Test procedure*

Place one drop of the flux test solution as described in **6.4.1** and one drop of standard chloride solution (**6.2.4**) a minimum of 20 mm apart on to a silver chromate test paper (**6.3.1**) and wait for 15 s. Rinse the paper with propan-2-ol (**6.2.5**) or with the solvent used in **6.4.1** for preparing the flux test solution. The appearance of an off-white, or white, spot on the orange-brown or chocolate-brown background of the paper indicates the presence of ionic halides. The sample passes the test for halide if the test solution causes no greater change in the paper than does the standard chloride solution.

The test is only valid if the standard chloride solution produces an off-white or white, spot. If it does not, repeat the test using fresh silver chromate paper (**6.3.1**).

NOTE 16 Colophony-based fluxes normally produce a uniform darker spot on the test paper, which does not constitute a positive halide test. A smaller white spot within a darker one is typical of ionic halide activated rosin fluxes.

NOTE 17 The following components in the flux will interfere with the results obtained from this test — free amines (bases), iodides, cyanides and thiocyanates.

7 Precision

7.1 General

Methods A, B and C of this part of ISO 9455 have been subjected to a limited interlaboratory test programme. The repeatability and reproducibility for these methods were calculated according to the principles of ISO 5725-2 and the results are given in **7.2** to **7.4**.

No precision data is given for method D, which is considered to be a qualitative method.

7.2 Method A

Tests were carried out on eight rosin based fluxes, covering a range of halide contents from 0,1 % (*m*/*m*) to 1 % (*m*/*m*) chloride in the non-volatile matter. Five laboratories took part in the tests, with the following results:

Standard deviation		
— within laboratory	s_w	0,003
— between laboratories	s_b	0,012
Repeatability:	*r*	0,01
Reproducibility:	*R*	0,03

7.3 Method B

Tests were carried out on four zinc chloride fluxes, covering a range of halide levels from 5 % (*m*/*m*) to 20 % (*m*/*m*) chloride in the flux. Five laboratories took part in the tests, with the following results:

Level	Mean halide content %	Repeatability (*r*) %	Reproducibility (*R*) %
A	4,48	0,38	0,63
B	8,58	0,25	1,10
C	15,05	0,23	0,84
D	18,45	0,32	2,94

7.4 Method C

Tests were carried out on phosphate-containing fluxes, covering a range of halide contents from 0,05 % (*m*/*m*) to 0,1 % (*m*/*m*), in the flux. Five laboratories took part in the test with the following results.

— Repeatability	*r*	0,008
— Reproducibility	*R*	0,04

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8 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 method A, method B, method C or method D of this part of ISO 9455);

c) the results obtained;

d) any unusual features noted during the determination or test;

e) details of any operation not included in this part of ISO 9455 or regarded as optional;

f) for method D, details of the solvent used in the preparation of the flux test solution (**6.4.1**), if not propan-2-ol.

Annex A (normative) Method for standardizing the silver nitrate solution

A.1 Field of application

This method is to be used for the determination of the concentration of the nominally 0,02 mol/l silver nitrate solution referred to in **3.2.3** and **5.2.2**, and of the nominally 0,1 mol/l silver nitrate solution, referred to in **4.2.2**. The derivation of the factors f_1 and f_2 is described.

A.2 Principle

A known volume of the sodium chloride standard solution is titrated with the silver nitrate solution under test, using potassium dichromate as indicator.

A.3 Reagents

A.3.1 *General*

Only reagents of recognized analytical quality and distilled or deionized water are to be used.

A.3.2 *Sodium chloride (NaCl)*

A.3.3 *Sodium hydroxide solution* [approximately 4 % (m/m)]

4 g sodium hydroxide (NaOH) are added, with cooling, to 100 ml of water and are mixed thoroughly. This is stored in a plastic container.

A.3.4 *Nitric acid solution* [1 % (*V*/*V*)]

1 ml concentrated nitric acid (density 1,42 g/ml) is added to water, diluted to 100 ml with water and mixed.

A.3.5 *Potassium dichromate solution* [approximately 30 % (*m*/*m*)]

Approximately 15 g potassium dichromate ($K_2Cr_2O_7$) are dissolved in 50 ml water and mixed thoroughly.

A.3.6 *Phenolphthalein indicator solution*

1 g of phenolphthalein is added to approximately 50 ml methanol and mixed. When dissolved, this is diluted to 100 ml with methanol and mixed.

A.4 Apparatus

In addition to normal laboratory apparatus the following are required:

A.4.1 *Laboratory oven*, capable of maintaining a temperature of 100 °C ± 5 °C.

A.4.2 *Desiccator*

A.5 Preparation of standard sodium chloride solutions

A.5.1 *For use in standardizing 0,1 mol/l silver nitrate solution*

Approximately 10 g of sodium chloride (**A.3.2**) are dried in the oven (**A.4.1**) for a minimum of 6 h at 100 °C ± 5 °C, and allowed to cool to room temperature in the desiccator (**A.4.2**).

To the nearest 0,001 g, approximately 5,844 g of the dried sodium chloride are weighed and transfered to a clean 1 litre volumetric flask then diluted to the mark with water and mixed thoroughly.

A.5.2 *For use in standardizing 0,02 mol/l silver nitrate solution*

50 ml of the sodium chloride solution (**A.5.1**) are pipetted into a clean 250 ml volumetric flask, diluted to the mark with water and mixed thoroughly.

A.6 Procedure

Fill a 50 ml burette with the silver nitrate solution to be standardized.

According to the nominal concentration of the silver nitrate solution to be standardized, either:

a) for the 0,1 mol/l solution, pipette 25 ml of standard sodium chloride solution (**A.5.1**) into a clean Erlenmeyer flask or

b) for the 0,02 mol/l solution, pipette 25 ml of standard sodium chloride solution (**A.5.2**) into a clean Erlenmeyer flask.

Dilute the contents of the flask to approximately 100 ml with water. Add 2 drops of phenolphthalein indicator solution (**A.3.6**) and add, dropwise, sodium hydroxide solution (**A.3.3**) until the solution turns red.

Add, dropwise, nitric acid solution (**A.3.4**) until the red colour is just completely discharged, then add 6 drops of potassium dichromate solution (**A.3.5**).

Titrate with the silver nitrate solution to be standardized until a red-brown endpoint is reached.

Note the volume of silver nitrate solution used in the titration.

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A.7 Expression of results

The standardization factor f_1 for the nominally 0,02 mol/l silver nitrate solution is given by:

$$f_1 = \frac{25}{X} \times \frac{m}{5,844}$$

where

X is the volume, in millilitres, of silver nitrate solution used;

m is the mass, in grams, of dried sodium chloride used in **A.5.1**.

The standardization factor f_2 for the nominally (0,1 mol/l) silver nitrate solution is given by:

$$f_2 = \frac{25}{Y} \times \frac{m}{5,844}$$

where

Y is the volume, in millilitres, of silver nitrate solution used;

m is the mass, in grams, of dried sodium chloride used in **A.5.1**.

These values for f_1 and f_2 are applicable to the formulae in **3.5**, **4.5** and **5.5** of this part of ISO 9455.

Annex ZA (normative)
Normative references to international publications with their relevant European publications

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

Publication	Year	Title	EN	Year
ISO 9454-1	1990	*Soft soldering fluxes — Classification and requirements — Part 1: Classification, labelling and packaging*	EN 29454-1	1993
ISO 9455-1	1990	*Soft soldering fluxes — Test methods — Part 1: Determination of non-volatile matter, gravimetric method*	EN 29455-1	1993
ISO 9455-2	1993	*Soft soldering fluxes — Test methods — Part 2: Determination of non-volatile matter, ebulliometric method*	EN ISO 9455-2	1995

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