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Soft soldering fluxes — Test methods —

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

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Partie 6: Dosage et détection des halogénures (à l'exception des fluorures)

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 44, *Welding and allied processes*, Subcommittee SC 12, *Soldering materials*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 121, *Welding and allied processes*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 9455-6:1995), of which it constitutes a minor revision. The changes are as follows:

- [Clause 2](#) has been updated;
- new [Clause 3](#), Terms and definitions, has been inserted;
- the coding of the fluxes has been updated in accordance with ISO 9454-1:2016;
- [4.2](#), [5.2](#), [6.2](#) and [A.3](#) restructured;
- notes changed to body text in [4.4](#), [5.4](#), [5.6](#), [6.4](#) and [7.4.1.4](#);
- title added to [Table 1](#);
- formulae numbered;
- minor editorial changes.

A list of all parts in the ISO 9455 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html. Official interpretations of ISO/TC 44 documents, where they exist, are available from this page: <https://committee.iso.org/sites/tc44/home/interpretation.html>.

Soft soldering fluxes — Test methods —

Part 6:

Determination and detection of halide (excluding fluoride) content

1 Scope

This document 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 considered the reference method for these fluxes, is suitable for halide contents generally within the range of 0,05 % mass fraction to 2 % mass fraction 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 2122 to 2124, 3112 to 3114 and 3212 to 3214, 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 331, 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 can be used for all classes of flux.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

4 Method A: Potentiometric method (Reference method)

4.1 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.

4.2 Reagents

Only reagents of recognized analytical quality and distilled or deionized water with a conductivity less than 10 $\mu\text{S}/\text{cm}$ are to be used.

4.2.1 Propan-2-ol.

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

3,4 g of silver nitrate (AgNO_3) is dissolved in water then transferred to a 1-l volumetric flask and diluted to the mark with water. It is mixed thoroughly. The silver nitrate (AgNO_3) should be dried at $(110 \pm 5)^\circ\text{C}$ for 16 h and cooled in a desiccator before weighing.

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

Standardize the silver nitrate solution using the method given in [Annex A](#) to derive the correlation factor f_1 . This is stored in an amber glass bottle.

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

4.3 Apparatus

The usual laboratory apparatus and, in particular, the following shall be used:

4.3.1 Millivolt meter.

4.3.2 Silver electrode.

4.3.3 **Mercury/mercurous sulfate 1 mol/l sodium sulfate electrode**, or a reversible hydrogen electrode.

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

4.4 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.

This procedure is suitable for halide ion concentrations generally within the range of 0,1 % mass fraction to 1 % mass fraction 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:

- for flux samples having a halide ion concentration in the range of 0,05 % mass fraction to 0,1 % mass fraction, the mass of sample taken should be double; and
- for flux samples having a halide ion concentration in the range of 1 % mass fraction to 2 % mass fraction, the mass of sample taken should be halved.

Add 100 ml of propan-2-ol ([4.2.1](#)), or water, according to the solubility characteristics of the flux. Cover with a watch glass and allow to dissolve with gentle agitation.

For fluxes of class 12, 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 (4.2.2), 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.

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.

4.5 Calculation of results

The halide (excluding fluoride) content, expressed as the percentage by mass of chloride in the flux, is given by [Formula \(1\)](#):

$$\frac{0,0709 \times V}{m} f_1 \quad (1)$$

where

- V is the volume, in ml, 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 [4.2.2](#));
- m is the mass, in g, 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 using [Formula \(2\)](#):

$$\frac{7,09 \times V}{mS} f_1 \quad (2)$$

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, $S = 100$.

5 Method B: Titration method for halide (excluding fluoride) content of water-soluble fluxes

5.1 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 2122 to 2124, 3112 to 3114 and 3212 to 3214 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.

5.2 Reagents

Only reagents of recognized analytical quality and distilled or deionized water with a conductivity less than 10 $\mu\text{S}/\text{cm}$ are to be used.

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

16,99 g of silver nitrate (AgNO_3) is dissolved in water (see 4.2.2) then transferred to a 1-l volumetric flask and diluted to the mark with water. This is mixed thoroughly. The silver nitrate (AgNO_3) should be dried at $(110 \pm 5)^\circ\text{C}$ for 16 h and cooled in a desiccator before weighing.

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

Standardize the silver nitrate solution using the method given in Annex A to derive the correlation factor f_2 . This is stored in an amber glass bottle.

NOTE 1 ml of silver nitrate solution is equivalent to $0,003\ 545 \times f_2$ g of chloride ion.

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

8 g of ammonium thiocyanate (NH_4CNS) is dissolved in water then transferred to a 1-l 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.

5.2.3 **Sulfuric acid solution** (20 % volume fraction). 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 l and mixed thoroughly.

5.2.4 Ammonium ferric sulfate indicator solution.

10 g of ammonium ferric sulfate [$\text{NH}_4\text{Fe}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$] is dissolved in 100 ml water.

5.2.5 Nitrobenzene.

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

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

5.3 Apparatus

Ordinary laboratory apparatus.

5.4 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 (5.2.3), 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 (5.2.6).

To a similar flask transfer 60 ml of water and 5 ml of nitric acid (5.2.6) 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 (5.2.1) to the contents of the flask. 2 ml of nitrobenzene (5.2.5) 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 (5.2.4) and titrate with ammonium thiocyanate solution (5.2.1), 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.

5.5 Standardization of the ammonium thiocyanate solution (5.2.2)

Transfer 70 ml of water and 5 ml of nitric acid (5.2.6) to a 500 ml conical flask. By means of a pipette, add 25 ml of silver nitrate solution (5.2.1). Add 5 ml of ammonium ferric sulfate indicator solution (5.2.4) and titrate with the ammonium thiocyanate solution (5.2.2), as in the procedure given in 5.4.

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

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

5.6 Calculation of results

The halide (excluding fluoride) content, expressed as the percentage by mass of chloride in the flux, is given by Formula (3):

$$\left[\frac{(\text{titre}_{\text{blank}} - \text{titre}_{\text{sample}}) \times 0,003\,545 \times f_3}{0,5\,d} \right] \times 100 \quad (3)$$

which simplifies to:

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

where

- V is the volume, in ml, of ammonium thiocyanate solution (5.2.2) used in the titration of the blank, minus the volume used in the titration of the flux sample;
- d is the density, in g/ml, 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 5.5.

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

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

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

6.1 Principle

Oxalic acid is added to complex any copper which is 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 331 as defined in ISO 9454-1.

6.2 Reagents

Only reagents of recognized analytical quality and distilled or deionized water with a conductivity less than 10 $\mu\text{S}/\text{cm}$ are to be used.

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

3,4 g of silver nitrate (AgNO_3) is dissolved in water (see 4.2.2) then transferred to a 1-l volumetric flask and diluted to the mark with water. This is mixed thoroughly. The silver nitrate (AgNO_3) should be dried at $(110 \pm 5)^\circ\text{C}$ for 16 h and cooled in a desiccator before weighing.

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

Standardize the silver nitrate solution using the method given in Annex A to derive the correlation factor f_1 . This is stored in an amber glass bottle.

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

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

1,6 g of ammonium thiocyanate (NH_4CNS) is dissolved in water then transferred to a 1-l volumetric flask and diluted to the mark. This is mixed thoroughly. This solution is standardized with 0,02 mol/l silver nitrate solution (6.2.1), as described in 6.5.

6.2.3 Nitric acid solution (10 % volume fraction).

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

6.2.4 Oxalic acid.

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

6.2.5 Ferric nitrate.

6.2.6 Nitrobenzene.

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

6.3 Apparatus

Ordinary laboratory apparatus.

6.4 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 (6.2.4) 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 (6.2.5) and, by pipette, 5 ml of the silver nitrate solution (6.2.1). Stopper the flask and shake it well. 2 ml of nitrobenzene (6.2.6) may be added to assist coagulation of the precipitate.

Titrate the excess silver nitrate with ammonium thiocyanate solution (6.2.2) 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.

6.5 Standardization of the ammonium thiocyanate solution (6.2.2)

Dissolve 15 g of ferric nitrate (6.2.5) and approximately half the quantity of oxalic acid (6.2.4) used in 6.4, in 70 ml of 10 % volume fraction nitric acid solution (6.2.3) in a 250-ml conical flask. Add, by pipette, 5 ml of silver nitrate solution (6.2.1) and titrate with ammonium thiocyanate solution (6.2.2) to a faint permanent orange-red end-point.

Calculate the correlation factor f_4 for the ammonium thiocyanate, according to Formula (4):

$$f_4 = \frac{5 \times f_1}{\text{volume of NH}_4\text{CNS}} \quad (4)$$

6.6 Calculation of results

The halide (excluding fluoride) content, expressed as the percentage by mass of chloride in the flux, is given by Formula (5):

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

which simplifies to:

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

where

V is the volume, in ml, of ammonium thiocyanate solution (6.2.2) used in the titration of the blank minus the volume used in the titration of the flux sample;

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

f_4 is the correlation factor for the ammonium thiocyanate obtained in 6.5.

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

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

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