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Soft soldering fluxes — Test methods —

Part 16:
Flux efficacy test, wetting balance method

Flux de brasage tendre — Méthodes d'essai —

Partie 16: Essai d'efficacité du flux, méthode à la balance de mouillage

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 9455-16 was prepared by Technical Committee ISO/TC 44, *Welding and allied processes*, Subcommittee SC 12, *Soldering materials*.

This second edition cancels and replaces the first edition (ISO 9455-16:1998), which has been technically revised.

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 test, solder spread method*

— *Part 11: Solubility of flux residues*

— *Part 13: Determination of flux spattering*

— *Part 14: Assessment of tackiness of flux residues*

— *Part 15: Copper corrosion test*

— *Part 16: Flux efficacy test, wetting balance method*

— *Part 17: Surface insulation resistance comb test and electrochemical migration test of flux residues*

Requests for official interpretations of any aspect of this part of ISO 9455 should be directed to the Secretariat of ISO/TC 44/SC 12 via your national standards body. A complete listing of these bodies can be found at www.iso.org.

Soft soldering fluxes — Test methods —

Part 16:
Flux efficacy test, wetting balance method

1 Scope

This part of ISO 9455 specifies a method for the assessment of the efficacy of a soft soldering flux, known as the wetting balance method. It gives a qualitative assessment of the comparative efficacy of two fluxes (for example, a standard and a test flux), based on their capacity to promote wetting of a metal surface by liquid solder. The method is applicable to all flux types in liquid form classified in ISO 9454-1.

NOTE It is hoped that future developments using improved techniques for obtaining a reproducible range of test surfaces will enable this method for assessing flux efficacy to be quantitative. For this reason, several alternative procedures for preparing the surface of the test piece are included in the present method.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 9453, *Soft solder alloys — Chemical compositions and forms*

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

IEC 60068-2-20:2008, *Environmental testing — Part 2-20: Tests — Test T: Test methods for solderability and resistance to soldering heat of devices with leads*

IEC 60068-2-54, *Environmental testing — Part 2-54: Tests — Test Ta: Solderability testing of electronic components by the wetting balance method*

IEC 60068-2-78:2001, *Environmental testing — Part 2-78: Tests; Test Cab: Damp heat, steady state*

3 Symbols

d	depth of immersion, in millimetres, of the test piece below the undisturbed solder level
A	cross sectional area, in square millimetres, of the test piece at the solder line
ρ	density, in grams per millilitre, of the solder under test at the test temperature
F	wetting force, in millinewtons
t_0	time at which the test piece first makes contact with the surface of the liquid solder in the bath
t_1	time at which the solder starts to wet the test piece (point A, see Figure 1), at which point the trace begins to fall
t_2	time at which the recorded force is equal to the upward force due to buoyancy
t_3	time at which the trace crosses the reference line

4 Principle

The efficacy of the liquid flux under test is compared with that of a standard liquid flux, using a wetting balance in conjunction with a specified test piece, appropriate to the class of flux under test.

5 Reagents

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

5.1 **Acid cleaning solution**, prepared as follows:

Add cautiously, while stirring, 75 ml of sulfuric acid (ρ = 1,84 g/ml) to 210 ml of water and mix. Cool the solution to room temperature. Add 15 ml of nitric acid (ρ = 1,42 g/ml) and mix thoroughly.

5.2 **Acetone**.

5.3 **Propan-2-ol**, complying with the following requirements:

- propan-2-ol: 99,5 % (by mass) minimum;
- acid content: 0,002 % (by mass) maximum;
- non-volatile content: 0,2 % (by mass) maximum.

Isopropyl alcohol (also propan-2-ol, 2-propanol or the abbreviation IPA) is a common name for this chemical compound, which has the molecular formula C_3H_8O.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 **Wetting balance and ancillary instrumentation**, as described in IEC 60068-2-54.

Calibrate the apparatus in accordance with the manufacturer's instructions.

6.2 **Solder bath**.

As a minimum, the bath temperature shall be capable of being maintained at a temperature corresponding to the liquidus temperature of the alloy under test plus 35 °C. The test temperature shall be reported in the test report.

The dimensions of the solder bath shall be such that no portion of the test piece (see Clause 7) is less than 15 mm from the wall of the bath and the depth of the liquid solder in the bath shall be not less than 30 mm.

The solder used for the test and the test temperature shall be one of the following:

- Sn60Pb40 (see ISO 9453) at 235 °C ± 3 °C;
- Sn96,5Ag3,0Cu0,5 (see ISO 9453) at 255 °C ± 3 °C;
- any other solder and temperature combinations as agreed between the customer and the flux supplier.

6.3 **Acid-free filter paper**.

7 Test pieces

The test pieces shall be made of copper.

EXAMPLE Test pieces are cut from a rectangular copper sheet. The dimensions of each test piece are as follows:

- width: 10,0 mm ± 0,1 mm;
- length: constant between 15 mm and 30 mm, to suit the equipment used;
- thickness: either 0,10 mm ± 0,02 mm or 0,30 mm ± 0,05 mm.

When testing fluxes of type 1 or 2 (as defined in ISO 9454-1), full details of the test pieces shall be given in the test report.

The sheet or other article used for preparing the test pieces shall be clean and free from contamination. In order to obtain accurate results, the test pieces shall be cut cleanly without leaving significant burrs.

8 Procedure

8.1 Preparation of the test pieces

8.1.1 Cleaning

The test pieces shall be handled with clean tongs throughout. Select sufficient test pieces (see Clause 7) to allow 10 per test flux and 10 per standard flux. Degrease them in acetone (5.2) and allow to dry. Immerse them for 20 s in the acid cleaning solution (5.1) at room temperature. Remove the test pieces from the acid cleaning solution and wash for about five seconds under running tap water. Rinse with distilled or deionized water then acetone (5.2) and dry with acid-free filter paper (6.3).

If required, the test pieces may be stored in acetone after rinsing them in deionized water. When needed, they shall be removed from the acetone and dried with acid-free filter paper (6.3).

Subject all the test pieces to one of the ageing procedures given in 8.1.2 to 8.1.4 as agreed between the flux supplier and the customer.

8.1.2 Ageing the surface by sulfidation process

Carry out the procedure given in Annex B on all the cleaned test pieces (see 8.1.1).

8.1.3 Steam ageing the surface

Carry out the steam ageing procedure given in IEC 60068-2-20:2008, 4.1.1, ageing procedure 1b, for a period of 4 h on all the cleaned test pieces (see 8.1.1).

8.1.4 Damp-heat, steady-state ageing

Subject all the cleaned pieces (see 8.1.1) to the test chamber conditions specified in IEC 60068-2-78:2001, Clause 4, for a period selected from 1 h, 4 h or 24 h.

8.2 Test method

8.2.1 Carry out the following test procedure on each of the 10 test pieces. Complete all 10 tests within 45 min of the preparation stage (see 8.1).

8.2.2 If the flux under test is of type 1 or type 2 (as defined in ISO 9454-1), maintain the temperature of the solder bath at 235 °C ± 3 °C or 255 °C ± 3 °C (see 6.2).

When testing fluxes which are not type 1 or type 2 (as defined in ISO 9454-1), the bath temperature requirements and the standard flux to be used for comparison shall be agreed between the flux supplier and the customer.

8.2.3 Remove one of the test pieces from the acetone, dry it between two sheets of acid-free filter paper (6.3) and place it in the wetting balance specimen clip so that the long edges are vertical. Dip the test piece in the flux solution under test at room temperature, to a depth of no less than 3 mm greater than the depth selected for immersion of the test piece in the solder (see 8.2.5). Avoid excess flux by withdrawing the test piece cornerwise from the flux. If excess flux is still visible, touch the corner of the test piece on clean filter paper.

8.2.4 Attach the specimen clip to the wetting balance, ensuring that the bottom edge of the test piece is horizontal and approximately 20 mm above the solder bath (6.2). Allow it to remain there for 20 s± 5 s so that the solvent in the flux can evaporate before the test commences. Some types of flux might require a drying time which is shorter or longer than 20 s± 5 s. In these cases, the drying time shall be agreed between the flux supplier and the customer. During this drying period, adjust the suspension force signal and recorder trace to the desired zero position.

Immediately before starting the test, scrape the surface of the solder bath with a blade of suitable material to remove oxides.

8.2.5 Either by raising the solder bath or by lowering the test piece, dip the test piece into the molten solder at a speed of 20 mm/s ± 5 mm/s to a depth of either 3 mm ± 0,2 mm or 4 mm ± 0,2 mm.

Hold the test piece in this position for 5 s to 10 s and then withdraw it at a speed of 20 mm/s ± 5 mm/s. Record the wetting force against time for the period during which the test piece is in contact with the solder.

8.2.6 Repeat operations 8.2.2 to 8.2.5 for each of the remaining nine test pieces.

9 Reference value using standard flux

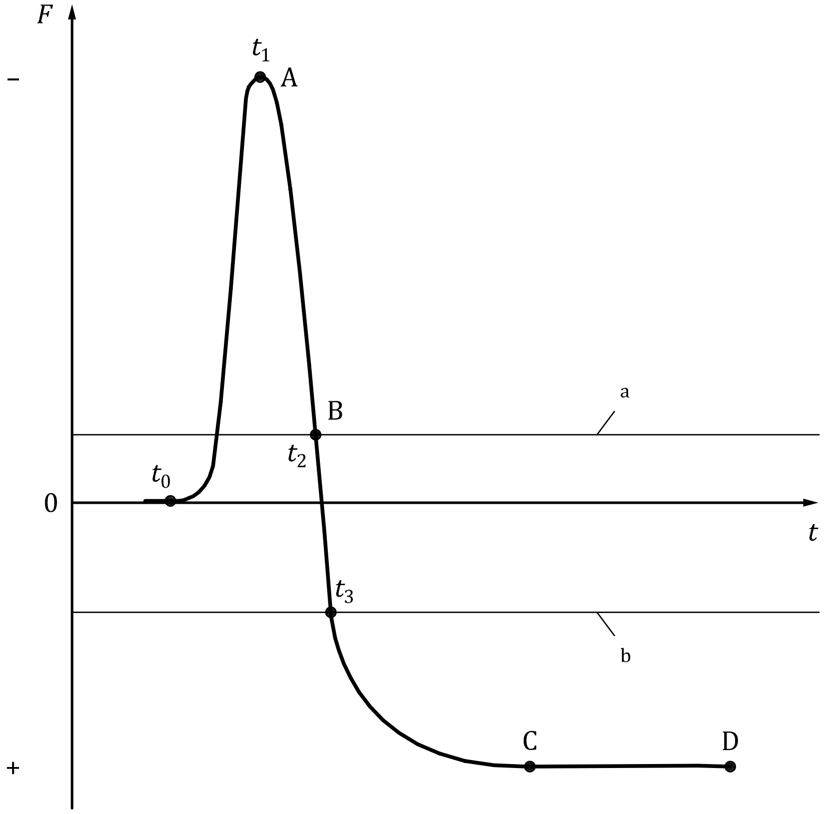
Carry out the procedure described in Clause 8 using a further 10 test pieces (see Clause 7) but using a standard flux instead of the flux under test. If the flux under test is of type 1 or type 2 (as defined in ISO 9454-1), the standard flux prepared as described in Annex A may be used. If the flux under test is of type 1.1.1 or 1.2.1 (as defined in ISO 9454-1), use the standard flux prepared as described in A.5.1. If the flux under test is of type 1.1.2, 1.1.3, 1.2.2 or 1.2.3 (as defined in ISO 9454-1), use the standard flux prepared as described in A.5.2.

If the flux under test is not of type 1 or type 2, use a standard flux as agreed upon by the supplier and customer (see 8.2.2, second paragraph).

10 Presentation of results

A typical trace of wetting force against time is given in Figure 1.

In Figure 1, non-wetting (upward) forces are shown as negative and wetting (downward) forces as positive.



Key

F	force
t	time

[a] Test piece buoyancy line.

[b] Wetting reference line.

Figure 1 — Recorded trace for wetting balance method showing significant points

The following are the points of significance in Figure 1.

- Time t_0 is the moment at which the test piece first makes contact with the surface of the liquid solder in the bath. It is indicated by a sharp deviation of the recorder trace from the zero-force line.
- Time t_1 is the moment at which the solder starts to wet the test piece and corresponds to point A at which the trace begins to fall.
- Time t_2, corresponding to point B, is the moment at which the recorded force is equal to the upward force due to buoyancy. The position of the test piece buoyancy line is calculated from the density of the solder and the depth of immersion of the test piece as follows.

Force at point B, in millinewtons, is equal to

$$\rho dA \times 9{,}81/1\,000$$

where

d is the depth of immersion, in millimetres, of the test piece below the undisturbed solder level;

A is the cross-sectional area, in square millimetres, of the test piece at the solder line;

ρ is the density, in grams per millilitre, of the solder under test at the test temperature.

Time t_3 is the moment at which the trace crosses the reference line. The line is drawn at a distance corresponding to a force, F, which depends on the test piece thickness and the immersion depth, as given in Table 1.

Table 1 — Values of the wetting force as a function of test piece thickness and depth of immersion

Thickness mm	**Immersion depth** mm	**Force *F*** mN
0,1	3	5,23
0,3	3	5,01
0,1	4	5,17
0,3	4	4,85

NOTE The force F is equal to 2/3 of the theoretical maximum wetting force on the test piece, assuming a wetting angle of zero and a liquid/vapour surface tension of 0,4 mN/mm.

Point C corresponds to the maximum value of the wetting force attained during the specified immersion period.

Point D corresponds to the end of the specified immersion period.

11 Calculation and expression of results

11.1 Carry out the operations and calculations described in 11.2 and 11.3.

11.2 For each of the 10 recorded traces obtained for the sample flux, draw the test piece buoyancy line through point B (see Clause 10) and draw the wetting reference line 5,6 mN below the buoyancy line (see Figure 1). Read off the following times, in seconds:

a) the time between t_0 and t_1 (i.e. the time to the start of wetting);

b) the time between t_0 and t_3 (i.e. the time to reach a wetting force of 5,6 mN).

Calculate the mean values for a) and b) obtained from the 10 traces.

Measure the force corresponding to point C (i.e. the maximum wetting force). Calculate the mean value of the maximum wetting force.

11.3 Repeat the operations and calculations described in 11.2 for the 10 recorder traces obtained from the standard flux in order to obtain the mean values for a), b) and c) for the standard flux.

Compare the mean results for 11.2 a), b) and c) obtained for the flux under test with those obtained from the 10 results using the standard flux.

Hence assess the efficacy of the flux under test as:

- better than,
- as good as,
- worse than

the standard flux, in relation to the speed and strength of wetting.

12 Test report

The test report shall include the following information:

a) the identification of the sample tested, including copper test specification, identification number and flux identifiers;

b) the test method used (a reference to this part of ISO 9455, i.e. ISO 9455-16:2013);

c) the test piece dimensions and, when fluxes of type 1 or type 2 were tested, full details of the test pieces (see Clause 6);

d) the ageing treatment (see 8.1.2 to 8.1.4);

e) the test temperature (see 5.2);

f) the depth of immersion of the test piece in the molten solder (see 8.2.5);

g) details of the standard flux used for comparison;

h) the results obtained;

i) the nominal temperature range during the test and the actual temperature range recorded during the test;

j) any unusual features noted during the procedure;

k) details of any operation not specified in the method, or any optional operation which might have influenced the results;

l) the date of the test.

Annex A
(normative)

Method for the preparation of standard rosin (colophony) based liquid fluxes having 25 % (by mass) non-volatile content

A.1 General

This annex gives a method for the preparation of two standard rosin (colophony) based liquid fluxes having 25 % (by mass) non-volatile content, one non-activated and the other halogen-activated (i.e. type 1.1.1.A and type 1.1.2.A, as defined in ISO 9454-1, respectively). The specifications for the flux constituents are based on IEC 60068-2-20:2008, Annex B.

The standard flux may be used as a reference against which the efficacy of the flux under test may be compared (see Clauses 4 and 9).

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

Ordinary laboratory apparatus, including an oven for use at 110 °C ± 2 °C, is required.

A.4 Reagents

Use only reagents of recognized analytical grade.

A.4.1 Rosin (colophony), water white grade gum rosin, or equivalent, complying with the following requirements:

- 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 a 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, see 5.3.

A.5 Procedure

A.5.1 Non-activated rosin

Weigh out 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

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 % (by mass) of active chloride.

A.6 Storage

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

Annex B
(normative)

Method for the production of test pieces with a controlled-contaminated surface for the wetting balance test (artificial sulfidation method)

B.1 Copper test pieces for the artificial sulfidation method

B.1.1 Reagents

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

B.1.1.1 Nitric acid, ρ = 1,33 g/ml.

B.1.1.2 Sulfuric acid, ρ = 1,84 g/ml.

B.1.1.3 White dextrin.

B.1.1.4 Denatured ethanol.

B.1.1.5 Acetone.

B.1.1.6 Ammonium persulfate, powdered.

B.1.1.7 Degreasing product, with a pH between 7 and 8.

B.1.1.8 Copper sulfate pentahydrate.

B.1.2 Apparatus

Usual laboratory apparatus and, in particular, the following:

B.1.2.1 Heated magnetic stirrer, with a PTFE-coated magnetic bar.

B.1.2.2 Current-stabilized power supply, delivering 10 A at 3 V.

B.1.2.3 Temperature-controlled bath, capable of being controlled from 20 °C to 30 °C.

B.1.2.4 Precision balance, accurate to within 0,01 g.

B.1.2.5 Anode support bars, of copper or brass.

B.1.2.6 Borosilicate glass beakers, 1 l and 5 l.

B.1.2.7 Electrolysis frame for cathode, of copper or brass (see Figure B.1).

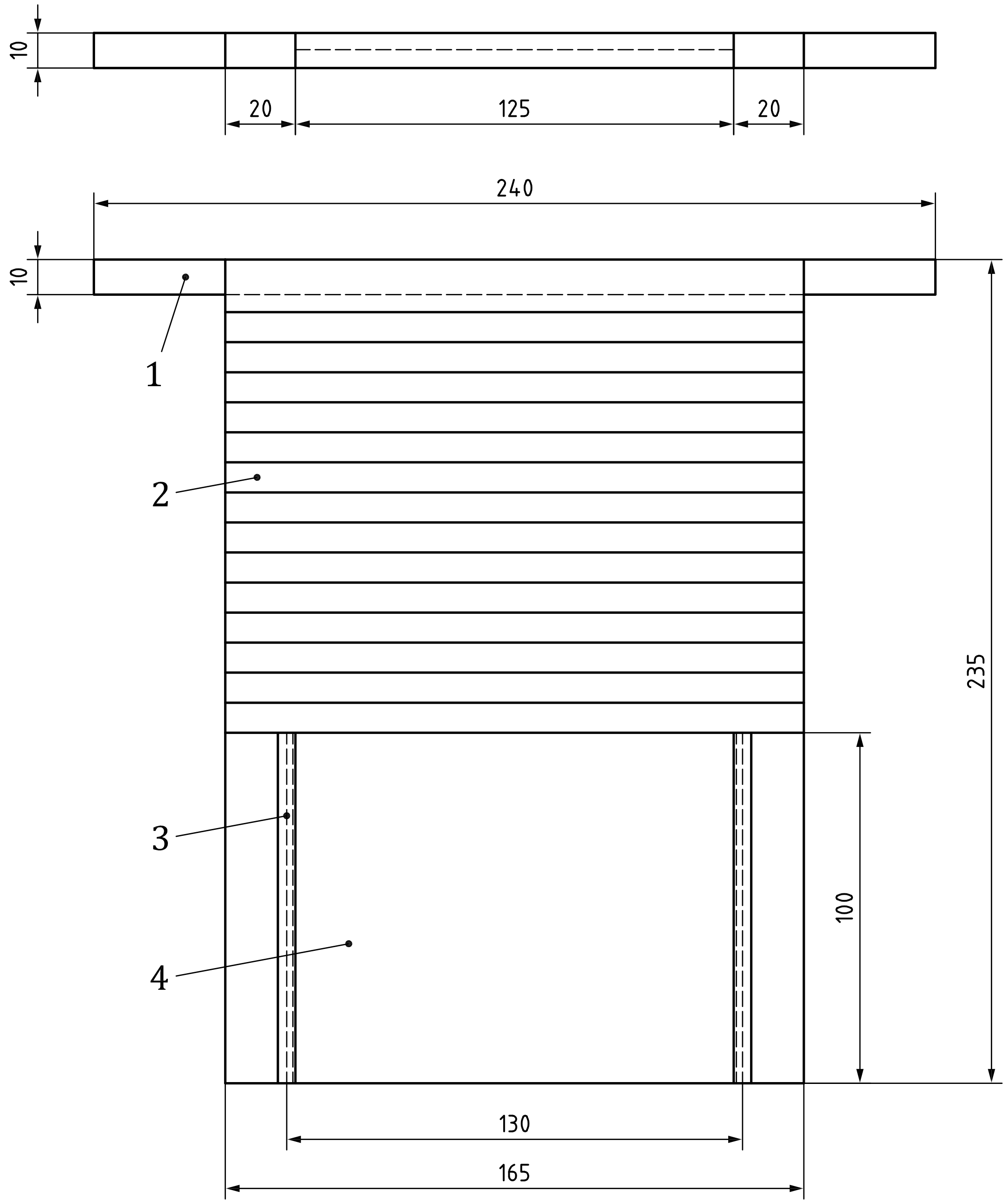
B.1.2.8 Stop-watch.

B.1.2.9 Anode hooks, of copper or stainless steel.

B.1.2.10 Crocodile clip.

B.1.2.11 Plastic containers, 3 l to 5 l.

Dimensions in millimetres



Key

1 location for contacts

2 electrolytic adhesive tape

3 conductive adhesive tape for frame/blank contact

4 copper blank, size 130 mm × 100 mm

Figure B.1 — Electrolysis frame and blank assembly

B.1.2.12 Thermometers, graduated every 0,5 °C from 0 °C to +100 °C.

B.1.2.13 Absorbent paper, filter paper quality.

B.1.2.14 Inhibitor paper, “for silver”.

B.1.2.15 Conductive adhesive tape.

B.1.2.16 Electrolytic adhesive tape.

B.1.2.17 Copper anodes, cut from copper sheet.

B.1.2.18 Copper foil blanks.

B.1.3 Preparation of baths

B.1.3.1 Electrolytic copper-plating bath

B.1.3.1.1 Solution A

Introduce 1,5 l of deionized water into a 5 l beaker (see B.1.2.6) with a premarked 3,2 l volume.

Using the heated stirrer (B.1.2.1), heat the water to approximately 50 °C.

Gradually add 640 g ± 0,5 g of copper sulfate (B.1.1.8), stirring until completely dissolved. Remove the beaker from the stirrer.

B.1.3.1.2 Solution B

Introduce 400 ml of deionized water into a 1 l beaker (see B.1.2.6).

While stirring, gradually add 160 g ± 0,5 g of sulfuric acid (B.1.1.2). Allow to cool.

When the temperature has dropped to approximately 50 °C, add 6,40 g ± 0,01 g of white dextrin (B.1.1.3).

Place the beaker on the heated stirrer (B.1.2.1) and maintain the temperature at about 50 °C until all the dextrin has dissolved. Remove the beaker from the stirrer.

B.1.3.1.3 Electrolysis bath

Pour solution B into solution A, stir thoroughly and dilute to 3,2 l with deionized water.

Cover and allow to stand for at least 24 h.

The electrolysis bath shall be used within 15 days of preparation. It is sufficient to treat 20 to 30 blanks. Cover the bath between each electrolysis.

B.1.3.2 Degreasing bath

Prepare and use the degreasing bath in accordance with the manufacturer’s instructions.

B.1.3.3 Stripping bath

In a 5 l beaker (see B.1.2.6), dissolve 170 g of ammonium persulfate (B.1.1.6) in 1 l of deionized water. Add sulfuric acid (B.1.1.2) at the rate of 5 ml of sulfuric acid per litre. Bring the solution to the final desired total volume by adding deionized water.

NOTE The dissolution of the ammonium persulfate is endothermic.

This bath shall not be used below a temperature of 22 °C (see B.1.4).

B.1.3.4 Decontamination bath

In a 5 l beaker (see B.1.2.6), dilute 200 ml of sulfuric acid (B.1.1.2) to 1 l with deionized water by slowly adding the acid to the water with continuous stirring.

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

B.1.4 Copper plating of blanks

Attach a blank(B.1.2.18) to the electrolysis frame (see Figure B.1), using conductive adhesive tape (B.1.2.15).

The anodes (B.1.2.17) and the blank shall be prepared together, as described below:

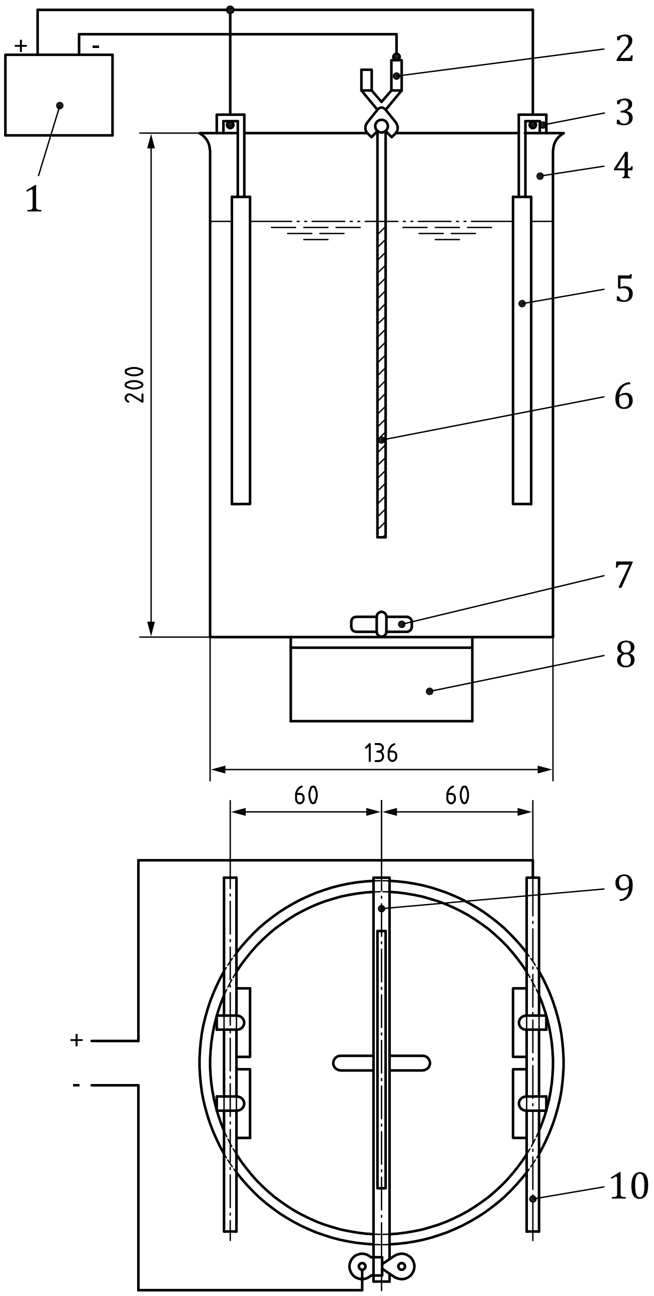
- immerse in the degreasing bath maintained between 22 °C and 27 °C for 5 s to 10 s;
- rinse for 2 min in a flow of deionized water;
- immerse for 1,5 min in the stripping bath (B.1.3.3) maintained between 22 °C and 27 °C;
- rinse for 1 min in a flow of deionized water;
- immerse for 1 min in the decontamination bath maintained between 22 °C and 27 °C;
- use immediately for copper plating by placing the anodes in a 5 l beaker (see B.1.2.6) containing the copper plating bath and making the electrical connections as shown in Figure B.2;
- connect the blank, then immerse at minimum voltage in the electrolysis bath as shown in Figure B.2;
- as soon as the blank is put in the bath, gradually increase the current to 2 A/dm^2 (the surface to be considered is the total immersed surface to be copper-plated, including the metallic zones of the frame in contact with the bath);
- electrolyse for 30 min while stirring, with the bath maintained at 22 °C to 27 °C;
- turn off the power supply, take the frame out of the bath and rinse under a flow of deionized water for approximately 30 s;
- dry the frame quickly between sheets of absorbent paper (B.1.2.13), without rubbing;
- take the copper-plated blank out of the frame and dry by pressing it between sheets of absorbent paper (B.1.2.13).

Store copper blanks carefully between sheets of inhibitor paper (B.1.2.14).

The deposit thickness shall be approximately 12 µm.

As long as any manual contact is avoided, blanks may be stored indefinitely.

Dimensions in millimetres



Key

1	stabilized power supply	6	blank (cathode)
2	crocodile clip	7	magnetic bar
3	stainless anode support	8	magnetic stirrer
4	5 l glass beaker	9	cathode support bar
5	anode	10	anode support bar

Figure B.2 — Electrolysis system

B.2 Controlled contamination

B.2.1 Reagents

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

B.2.1.1 Nitric acid, ρ = 1,33 g/ml.

B.2.1.2 Methyl ethyl ketone.

B.2.1.3 Starch solution, 1 g/100 ml.

B.2.1.4 Sodium hydroxide solution, 40 g/l.

B.2.1.5 Acetate solution, prepared from the following:

- zinc acetate dihydrate: 24 g
- acetic acid: 10 ml
- deionized water: 1 l

B.2.1.6 Decinormal iodine solution, $c(1/2\ I_2) = 0{,}1$ mol/l, previously calibrated.

B.2.1.7 Decinormal solution of sodium thiosulfate, $c(1/2\ Na_2SO_3) = 0{,}1$ mol/l.

B.2.1.8 Sodium sulfide nonahydrate.

B.2.2 Apparatus

Usual laboratory apparatus and, in particular, the following.

B.2.2.1 Mechanical magnetic stirrer, with a PTFE-coated magnetic bar.

B.2.2.2 Temperature-controlled bath, capable of being controlled from 20 °C ± 0,5 °C to 30 °C ± 0,5 °C.

B.2.2.3 Precision balance, accurate to within 0,1 mg.

B.2.2.4 Borosilicate glass beakers.

B.2.2.5 Stop-watch.

B.2.2.6 Polyethylene cup, for weighing.

B.2.2.7 Graduated tubes.

B.2.2.8 Erlenmeyer flask, 500 ml.

B.2.2.9 One-mark volumetric flasks, 500 ml and 1 l, with stoppers.

B.2.2.10 Tweezers, made of stainless steel.

B.2.2.11 Tweezers, made of plastic.

B.2.2.12 Absorbent paper, acid-free filter paper quality.

It is recommended that the absorbent paper be acid-free, in order to avoid further contamination.

B.2.2.13 Inhibitor paper, "for silver".

B.2.2.14 Polyethylene bag.

B.2.3 Preparation of metallic strips

Carefully cut 100 mm × 10 mm strips from a copper-plated blank, avoiding any manual contact. The strip width shall be constant throughout, with straight edges. The edges shall not contain burrs or notches.

NOTE 1 The edge quality is of prime importance to the results of the test.

Check the width of the strips and, in cases of variation greater than 1 % of the nominal value, use the real width in the calculations.

NOTE 2 It is advisable to carry out the chemical preparation of the copper strips (degreasing and deoxidation) after the preparation of the sulfidation bath. This allows consecutive treatment of the strips from degreasing to packaging before measurement.

Using the stainless-steel tweezers (B.2.2.10), immerse one strip in methyl ethyl ketone (B.2.1.2). Remove the strip and dry it. Immediate drying by pressing between two sheets of absorbent paper is recommended. This avoids mottling, which sometimes appears during drying in air. This mottling can be difficult to remove.

Using the plastic tweezers (B.2.2.11), immerse the strip in a 20 % (by volume) nitric acid solution, while stirring, until all surface marks have been removed. A uniform salmon pink surface shall be obtained. If a uniform salmon pink surface is not achieved, discard the strip and repeat the process.

Rinse the strip several times in deionized water. Replace the rinse solution every 10 strips. Press the strip between two sheets of absorbent paper (B.2.2.12) until completely dry.

If the strips have to wait while the sulfidation bath is prepared, protect them between two sheets of dry absorbent paper.

B.2.4 Preparation of solutions

B.2.4.1 Preparation of solution A

Weigh, to the nearest 0,1 mg, approximately 1,4 g of dry sodium sulfide crystals (B.2.1.8). Let this mass be m grams.

Place the crystals in a 500 ml one-mark volumetric flask (see B.2.2.9). Add 50 ml of deionized water and stir to dissolve. Dilute with deionized water to the mark on the flask and stir to homogenize.

B.2.4.2 Standardization of solution A

Transfer exactly 50 ml of solution A (see B.2.4.1) to an Erlenmeyer flask (B.2.2.8). Add 200 ml of acetate solution (B.2.1.5) and stir to homogenize.

With a microburette, add exactly 15 ml of iodine solution (B.2.1.6) and then 5 ml of starch solution (B.2.1.3).

Titrate the iodine in excess with the sodium thiosulfate solution (B.2.1.7). Let the required thiosulfate volume be V_1 millilitres

Carry out a blank test with 50 ml of deionized water, 200 ml of acetate solution, exactly 15 ml of iodine solution and 5 ml of starch solution. The volume of thiosulfate needed is V_0 millilitres.

The titre *T*, expressed as a percentage, of $Na_2S{\cdot}9H_2O$ in solution A is given by the formula:

$$T = \frac{23}{m} \times \frac{15(15 - V_1)}{V_0}$$

If the difference between the volume of thiosulfate solution required to titrate solution A and the volume required to titrate the blank is greater than 10 %, prepare a fresh solution.

B.2.4.3 Preparation of solution B

Introduce 200 ml of deionized water into a 500 ml one-mark volumetric flask (see B.2.2.9). Add a volume of solution A equal to 20 800/mT ml and 2 ml of sodium hydroxide solution (B.2.1.4). Dilute to volume with deionized water and mix well.

B.2.4.4 Preparation of sulfidization bath

It is recommended that the sulfidization bath be prepared immediately after the preparation of solution B.

Using solution B, prepare the bath corresponding to the chosen sulfidization grade as follows.

Introduce 300 ml of deionized water into a 1 l one-mark volumetric flask (see B.2.2.9). Add 4 ml of sodium hydroxide solution and stir. Add the volume of solution B corresponding to the chosen grade (see Table B.1), dilute to volume with deionized water and mix well.

NOTE 1 Grades III and IV are more commonly used as they are similar to the usual natural contamination of components experienced in electronics.

The sulfidization bath shall be used within 24 h of preparation.

NOTE 2 The grade 0 indicated in Table B.1 is given for information only. This grade is obtained by chemical preparation of the copper strips as described in B.2.3, followed directly by the wettability measurement. This grade is essentially intended for measurement of liquid/vapour surface tension.

Table B.1 — Correlation between the sulfidization grade and the volume of solution B

Sulfidization grade	Volume of solution B ml
0	0
I	3 ± 0,05
II	5 ± 0,05
III	7 ± 0,05
IV	10 ± 0,05
V	13 ± 0,05
VI	19 ± 0,05
VII	25 ± 0,1
VIII	37 ± 0,1
IX	50 ± 0,1
X	75 ± 0,1
XI	100 ± 0,1

B.2.5 Sulfidization

During the entire sulfidization period, the sulfidization bath temperature shall be maintained at 22,5 °C ± 0,5 °C. The tolerance limits for the temperature required for the sulfidization bath are valid for solutions A and B, in which the copper strips will be immersed. It is recommended that the temperature of strips to be immersed in the bath be compatible with that of the bath.

Immerse the metal strip prepared as specified in B.2.3 vertically into the sulfidization bath. Start the stop-watch (B.2.2.5) at the same time as immersion occurs.

Leave the strip in the bath for 30 s ± 0,5 s while stirring slowly; then remove the strip and dry, without rubbing, using absorbent paper (B.2.2.12).

From this point in time, avoid any contamination and, in particular, avoid any manual contact.

Cut the strip with scissors to obtain 20 mm × 10 mm test pieces. Store the test pieces between folds of inhibitor paper (B.2.2.13) to isolate them from one another. Wrap the resulting package in another sheet of inhibitor paper, then place in a polyethylene bag (B.2.2.14). Only open the package at the time of measurement.

Do not use the test pieces within 24 h of storage.

It is recommended that the immersion and the withdrawal of a strip in the sulfidization bath be carried out as quickly as possible in order to reduce the difference in immersion time between the two ends of the strip.

Use of a magnetic stirrer in the sulfidization bath is not recommended as it would cause a stronger action at the bottom of the beaker than at the top. It is recommended that stirring be carried out by gently moving the strip by hand.

Table B.2 gives the typical lifetimes of the test pieces versus the sulfidization grade for a carefully made package.

Table B.2 — Typical lifetimes of test pieces vs sulfidization grade

Sulfidization grade	Lifetime
0	15 minutes
I	36 hours
II	5 days
III	10 days
IV	1 month
V to XI	3 months

Grade 0 is particularly intended for measuring liquid/vapour surface tension. It should preferably be used immediately after strip preparation (see B.2.3) in order to obtain a perfect result, reaching as closely as possible a wetting angle of 0°.

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