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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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Contents

Page

Foreword

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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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

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

This third edition cancels and replaces the second edition (ISO 9455-16:2013), of which it constitutes a minor revision.

The main changes compared to the previous edition are as follows:

— Clause 2 has been updated;

— the coding of the fluxes has been updated in accordance with ISO 9454-1:2016;

— the format of this document has been updated.

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.

Soft soldering fluxes — Test methods —

Part 16:
Flux efficacy test, wetting balance method

1 Scope

This document 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 documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 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 60068-2-69, *Environmental testing — Part 2-69: Tests — Test Te/Tc: Solderability testing of electronic components and printed boards by the wetting balance (force measurement) method*

IEC 60068-2-78:2012, *Environmental testing — Part 2-78: Tests; Test Cab: Damp heat, steady state*

3 Terms and definitions

No terms and definitions are listed in this document.

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 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

5 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.

The fluxes under test shall correspond to one of the classes defined in ISO 9454-1 and fulfil the requirements of ISO 9454-1.

6 Reagents

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

6.1 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.

6.2 Acetone.

6.3 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.

7 Apparatus

Usual laboratory apparatus and, in particular, the following.

7.1 Wetting balance and ancillary instrumentation, in accordance with IEC 60068-2-69.

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

7.2 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 8) 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 may 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.

7.3 Acid-free filter paper.

8 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.

9 Procedure

9.1 Preparation of the test pieces

9.1.1 Cleaning

The test pieces shall be handled with clean tongs throughout. Select sufficient test pieces (see Clause 8) to allow 10 per test flux and 10 per standard flux. Degrease them in acetone (6.2) and allow to dry. Immerse them for 20 s in the acid cleaning solution (6.1) 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 (6.2) and dry with acid-free filter paper (7.3).

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 (7.3).

Subject all the test pieces to one of the ageing procedures given in 9.1.2 to 9.1.4 as agreed between the flux supplier and the customer.

9.1.2 Ageing the surface by sulfidation process

Carry out the procedure given in Annex B on all the cleaned test pieces (see 9.1.1).

9.1.3 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 9.1.1).

9.1.4 Damp-heat, steady-state ageing

Subject all the cleaned pieces (see 9.1.1) to the test chamber conditions specified in IEC 60068-2-78:2012, Clause 4, for a period selected from 1 h, 4 h or 24 h.

9.2 Test method

9.2.1 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 9.1).

9.2.2 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 7.2).

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.

9.2.3 Remove one of the test pieces from the acetone, dry it between two sheets of acid-free filter paper (7.3) 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 9.2.5). 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.

9.2.4 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 (7.2). 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 can 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.

9.2.5 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.

9.2.6 Repeat steps 9.2.2 to 9.2.5 for each of the remaining nine test pieces.

10 Reference value using standard flux

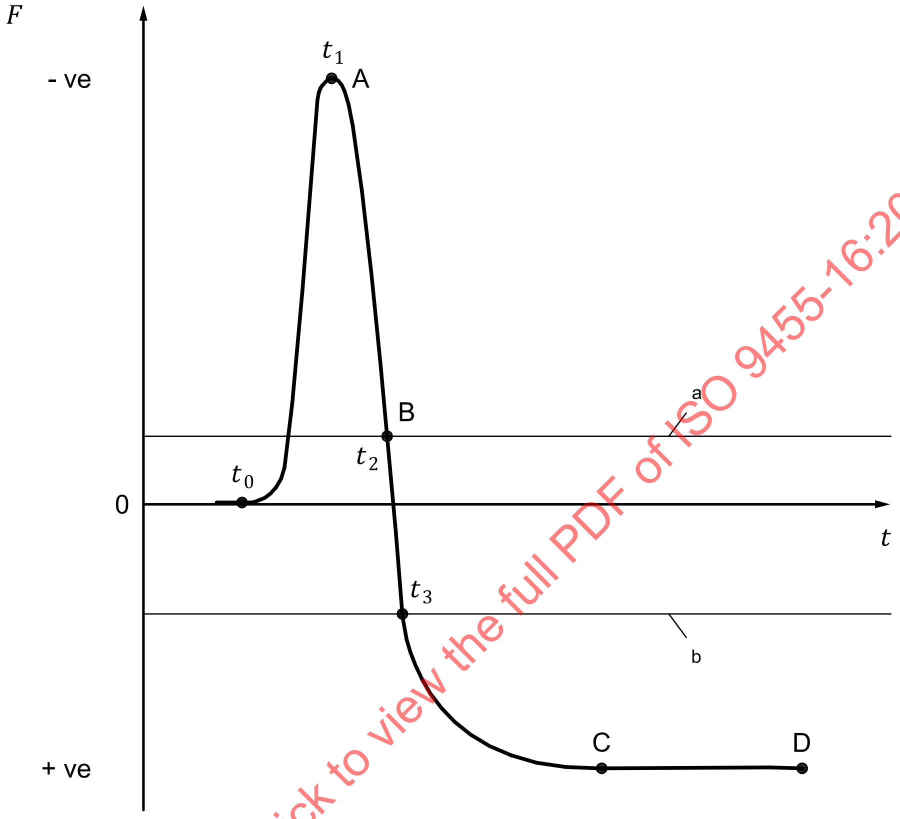
Carry out the procedure described in Clause 9 using a further 10 test pieces (see Clause 8) but using a standard flux instead of the flux under test. If the flux under test is of type 1 or type 2 (as defined in ISO 9454-1), the standard flux prepared as described in Annex A shall be used. If the flux under test is of type 111 or 121 (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 112, 113, 122 or 123 (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 by the supplier and customer (see 9.2.2, second paragraph).

11 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 Formula (1):

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

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.

12 Calculation and expression of results

12.1 Carry out the procedures and calculations described in 12.2 and 12.3.

12.2 For each of the 10 recorded traces obtained for the sample flux, draw the test piece buoyancy line through point B (see Clause 11) 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.

12.3 Repeat the operations and calculations described in 12.2 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 12.2 a), b) and c) obtained for the flux under test with those obtained from the 10 results using the standard flux.

Then, assess the efficacy of the flux under test as better than, as good as, or worse than, the standard flux, in relation to the speed and strength of wetting.

13 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 document, i.e. ISO 9455-16);

c) the test piece dimensions and, when fluxes of type 1 or type 2 were tested, full details of the test pieces (see Clause 7);

d) the ageing treatment (see 9.1.2 to 9.1.4);

e) the test temperature (see 6.2);

f) the depth of immersion of the test piece in the molten solder (see 9.2.5);

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 can 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 1111 and type 1123, 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 5 and 10).

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 6.3.

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 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).

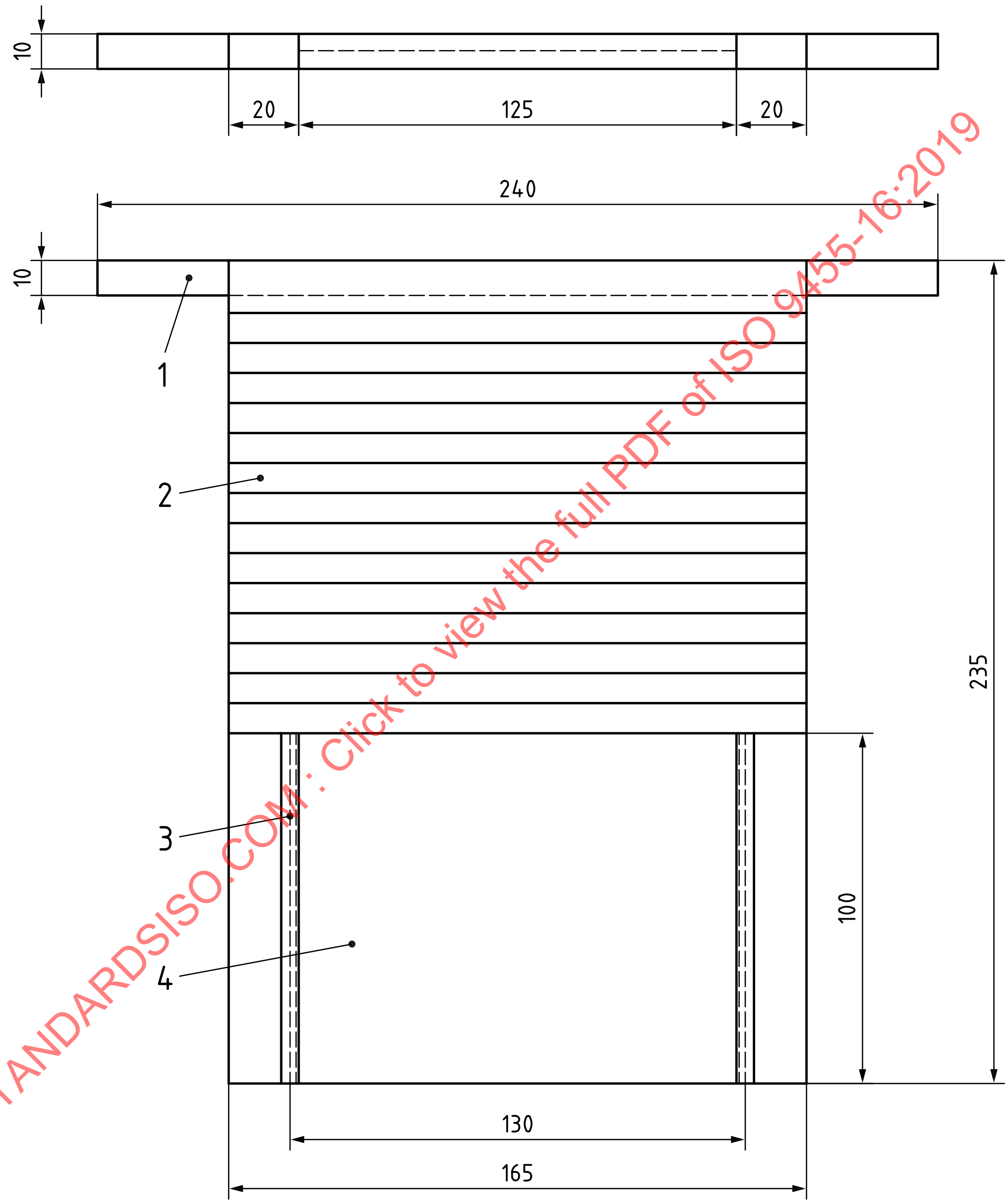
B.1.2.8 Stop-watch.

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:

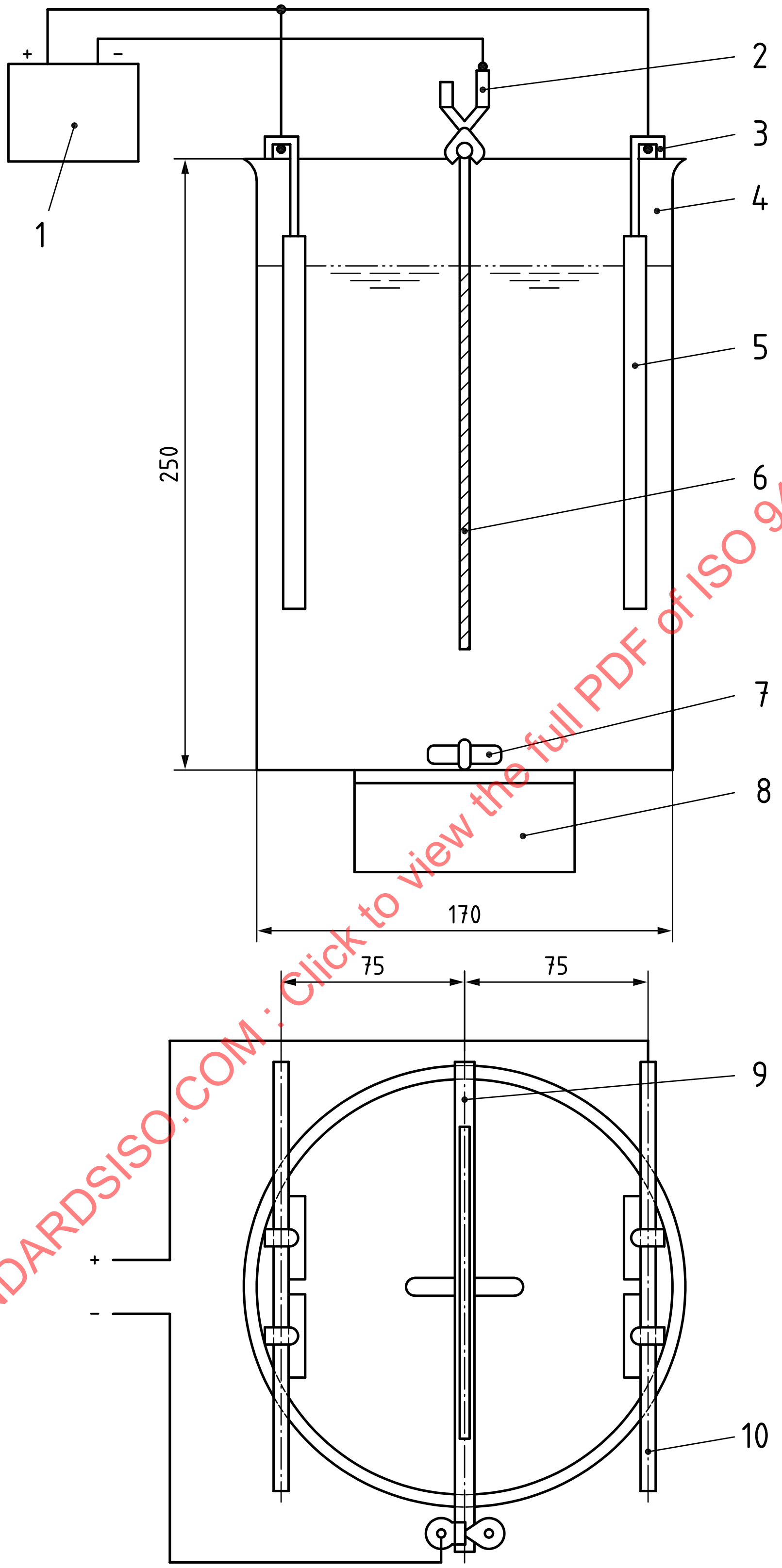
- immerse in the degreasing bath maintained between 22 °C and 27 °C for 5 s to 10 s;
- rinse for 2 min in a flow of deionized water;
- immerse for 1,5 min in the stripping bath (B.1.3.3) maintained between 22 °C and 27 °C;
- rinse for 1 min in a flow of deionized water;
- immerse for 1 min in the decontamination 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

2 crocodile clip

3 stainless anode support

4 5 l glass beaker

6 blank (cathode)

7 magnetic bar

8 magnetic stirrer

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.