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ISO 9455-16

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Soft soldering fluxes — Test methods —

Part 16:
Flux efficacy tests, wetting balance method

Flux de brassage tendre — Méthodes d'essai —

Partie 16: Essais d'efficacité des flux, méthode à la balance de mouillage

Reference number
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Foreword

ISO (the International Organisation 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 9455-16 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*
- *Part 17: Surface insulation resistance, comb test and electrochemical migration test of flux residues*

Annexes A and B form an integral part of this part of ISO 9455.

Soft soldering fluxes — Test methods —

Part 16:
Flux efficacy tests, 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 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 edition of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 1634-1:1987, *Wrought copper and copper alloy plate, sheet and strip — Part 1: Technical conditions of delivery for plate, sheet, and strip for general purposes.*

ISO 9453:1990, *Soft solder alloys — Chemical compositions and forms.*

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

IEC 60068-2-3:1969, *Environmental testing. Part 2: Tests. Test Ca: Damp heat, steady state.*

IEC 60068-2-20:1979, *Environmental testing. Part 2: Tests. Test T: Soldering*

IEC 60068-2-54:1985, *Environmental testing. Part 2: Tests. Test Ta: Soldering - Solderability testing by the wetting balance method.*

3 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 prescribed test piece, appropriate to the class of flux under test.

4 Reagents

In the test use only reagents of recognized analytical quality and only distilled or de-ionized water.

4.1 Acid cleaning solution.

Add cautiously, while stirring, 75 ml of sulphuric 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.

4.2 Acetone.

5 Apparatus

Usual laboratory apparatus and, in particular, the following.

5.1 Wetting balance and ancillary instrumentation, as described in IEC 600682 54.

NOTE The apparatus is to be calibrated in accordance with the manufacturer's instructions.

5.2 Solder bath, containing molten tin-lead solder complying with ISO 9453, grade S-Sn60Pb40E or S-Sn63Pb37E, maintained at a temperature of (235 ± 3) °C. The dimensions of the solder bath shall be such that no portion of the test piece (clause 6) 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.

5.3 Acid-free filter paper.

6 Test pieces

Test pieces cut from a copper and of rectangular shape sheet are used. The dimensions of each test piece shall be as follows:

- width: (10,0 ± 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), the test pieces shall be cut from copper sheet complying with ISO 1634-1: Grade Cu-ETP, temper HA.

The sheet 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.

7 Procedure

7.1 Preparation of the test pieces

7.1.1 Cleaning

The test pieces shall be handled with clean tongs throughout. Select sufficient test pieces (see clause 6) to allow ten per test flux and ten per standard flux. Degrease them in acetone (4.2) and allow to dry. Immerse them for 20 s in the acid cleaning solution (4.1) at room temperature. Remove the test pieces from the acid cleaning solution (4.1), wash for about five seconds under running tap water. Rinse with distilled or deionized water then acetone (4.2) and dry with acid-free filter paper (5.3).

NOTE If required, the test pieces may be stored in acetone after rinsing them in deionized water. When needed they should be removed from the acetone and dried with acid-free filter paper (5.3).

Subject all the test pieces to one of the ageing procedures given in 7.1.2 to 7.1.4. as agreed between the flux supplier and the user.

7.1.2 Ageing the surface by sulfidation process

Carry out the procedure given in annex B on all the cleaned test pieces (see 7.1.1).

7.1.3 Steam ageing the surface

Carry out the steam ageing procedure given in IEC 60068-2-20 (1979), clause 4.5.1, ageing 1b, for a period of 4 h on all the cleaned test pieces (see 7.1.1).

7.1.4 Damp heat, steady state ageing

Subject all the cleaned pieces (see 7.1.1) to the test chamber conditions specified in IEC 60068-2-3 (1969), clause 2, for a period selected from 1 h, 4 h or 24 h.

7.2 Test method

7.2.1 Carry out the following test procedure on each of the ten test pieces. Complete all ten tests within 45 min of the preparation stage (see 7.1).

7.2.2 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 ± 3) °C.

NOTE 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 (see clause 8) should be agreed between the purchaser and the supplier.

7.2.3 Remove one of the test pieces from the acetone, dry it between two sheets of acid-free filter paper (5.3) 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 selected depth for immersion of the test piece in the solder (see 7.2.5). 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.

7.2.4 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 (5.2). Allow it to remain there for (20 ± 5) s, so that the solvent in the flux may evaporate before the test commences (see note). During this drying period, adjust the suspension force signal and recorder trace to the desired zero position.

NOTE Some types of flux may require a shorter, or a longer, drying time than (20 ± 5) s. In these cases, the drying time should be agreed between the flux supplier and the user.

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

7.2.5 By either raising the solder bath, or lowering the test piece, dip the test piece into the molten solder at a speed of (20 ± 5) mm/s to a selected 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 ± 5) mm/s. Record the wetting force against time for the period during which the test piece is in contact with the solder.

7.2.6 Repeat operations 7.2.2 to 7.2.5 for each of the remaining nine test pieces.

8 Reference value using standard flux

Carry out the procedure described in clause 7, using a further ten test pieces (see clause 6), and using a standard flux instead of the sample flux under test. If the flux under test is of type 1 or type 2, 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, 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, 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 agreed upon by the purchaser and supplier. (See the note to 7.2.2.)

9 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 are positive.

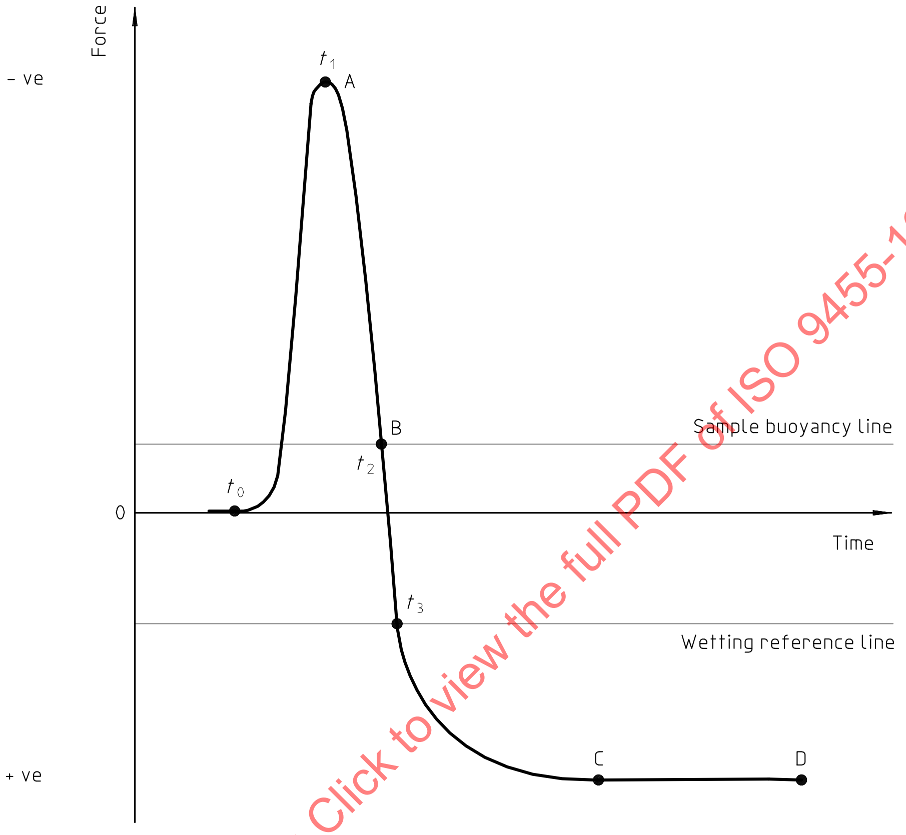

Figure 1 — Recorded trace for wetting balance method showing significant points

The following are the points of significance in figure 1.

- Time t_0 is the time 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 sample 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 mN = 0,08[1] $\times d \times a$

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[1] This value applies only to molten 60/40 tin/lead solder at 235 °C.

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.

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

Table 1 — Force dependence on 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 testpiece 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.

10 Expression of results

Carry out the following operations.

10.1 For each of the ten recorded traces obtained for the sample flux, draw the sample buoyancy line through point B (see clause 9) and draw the wetting reference line 5,6 mN below the buoyancy line (see figure 1). Read off from each of the traces the time, in seconds:

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

b) between t_0 and t_3 (i.e. the time to reach 5,6 mN wetting force). Calculate the mean values for a) and b) obtained from the ten traces.

c) Measure the force corresponding to point C (i.e. the maximum wetting force) for each trace. Calculate the mean value for the maximum wetting force.

10.2 Repeat the operations and calculations described in10.1 for the ten recorder traces obtained from a standard flux, in order to obtain the mean values for a), b) and c) for a standard flux.

Compare the mean results for 10.1 a), b) and c) obtained for the sample flux under test with those obtained from the ten results using the standard flux.

Hence assess the efficacy of the sample flux as:

better than,

as good as,

worse than

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

11 Test report

The test report shall include the following information:

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

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

c) the test piece dimensions;

d) the ageing treatment (see 7.1.2 to 7.1.4);

e) the depth of immersion of the test piece in the molten solder (see 7.2.5);

f) details of the standard flux used for comparison;

g) the results obtained;

h) any unusual features noted during the procedure;

i) details of any operation not specified in the method, or any optional operation which may have influenced the results.

Annex A
(normative)

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

A.1 General

This annex gives a method for the preparation of two standard rosin (colophony) based liquid fluxes having 25 % (m/m) non-volatile content, one nonactivated and the other halogen activated (i.e. being respectively class 1.1.1.A and class 1.1.2.A, as defined in ISO 9454-1). The specifications for the flux constituents are based on IEC 60068-2-20:1979, 4th edition, annex C.

The standard flux may be used as a reference against which the efficacy of the flux under test may be compared. See clauses 3 and 8.

A.2 Principle

The nonactivated 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, which shall comply 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, which shall comply with the following requirements.

- propan-2-ol: 99,5 % (m/m) minimum
- acid content: 0,002 % (m/m) maximum
- non-volatile content: 0,2 % (m/m) maximum

A.5 Procedure

A.5.1 Nonactivated rosin (colophony)

Weigh 25 g ± 0,1 g rosin (A.4.1) and dissolve it with gentle mixing in 75 g ± 0,1 g propan-2-ol (A.4.3).

A.5.2 Halogen activated rosin (colophony)

Weigh 0,39 g ± 0,01 g diethylamine hydrochloride (A.4.2) and dissolve it in 75 g ± 0,1 g propan-2-ol (A.4.3). Then add 25 g ± 0,1 g rosin (A.4.1) and dissolve it with gentle mixing. This flux solution contains 0,5 % (*m*/*m*) active chloride.

A.6 Storage

The standard flux solutions, prepared as described in A.5 must 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 a controlled contaminated surface sample for the wetting balance test (artificial sulfidation method)

B.1 Copper samples for the artificial sulfidation method

B.1.1 Reagents

Use only reagents of recognized analytical quality and only distilled or de-ionized 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 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 PTFE coated magnetised 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 to 5 l.

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

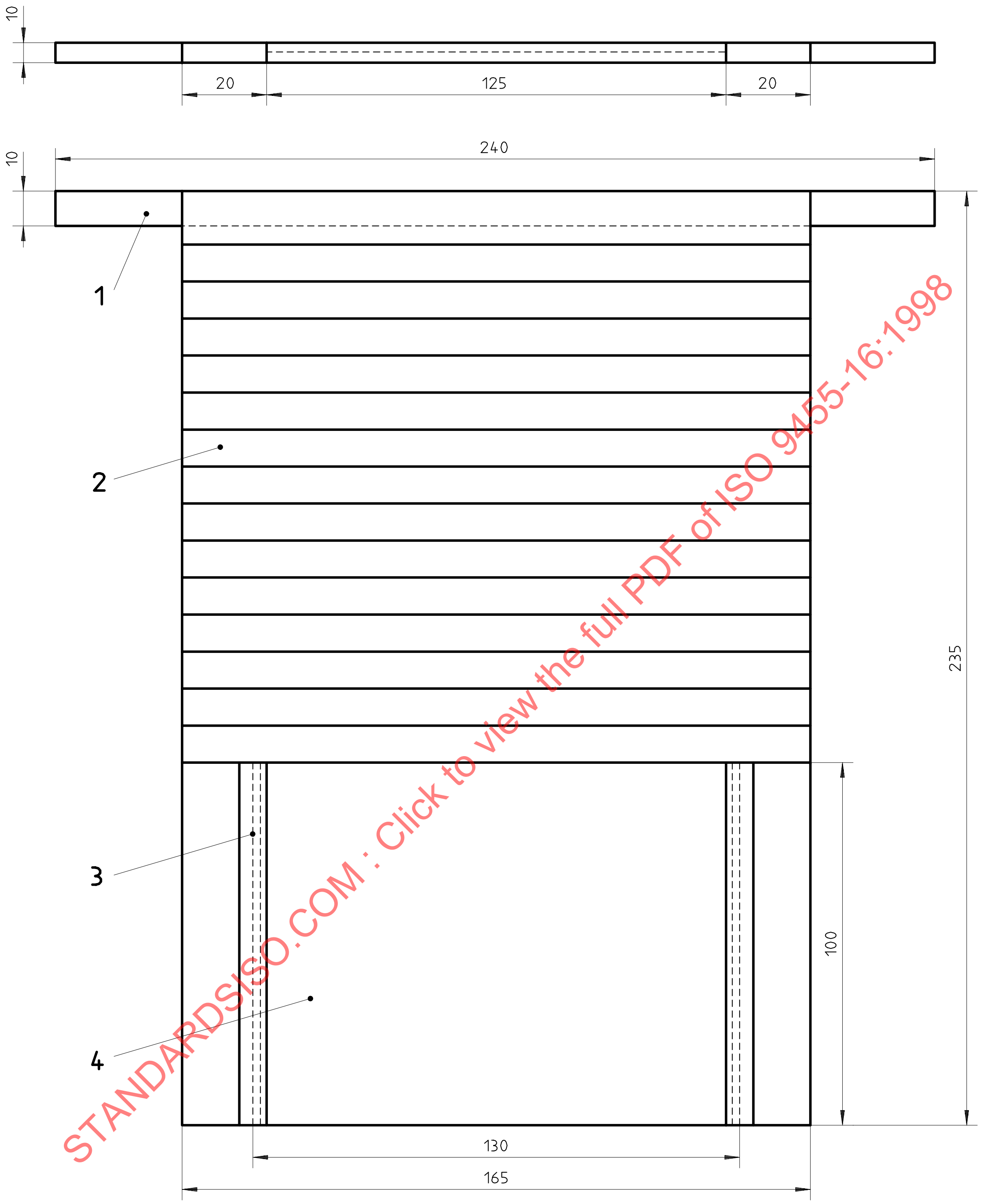
B.1.2.8 Stop-watch.

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 × 100

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 Anodes of copper, cut from copper sheet conforming to ISO 1634-1, grade Cu-ETP temper HA, dimensions: 90 mm × 40 mm × 20 mm.

B.1.2.18 Foil blanks of copper, conforming to ISO 1634-1, grade Cu-ETP temper HA, dimensions: 130 mm × 100 mm × 20 mm.

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 de-ionized water into a 5 l beaker (B.1.2.6) with a premarked 3,2 l volume.

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

Gradually add 640 g ± 0,5 g of copper sulfate (B.1.1.10), while stirring, until total dissolution occurs. Remove the beaker from the stirrer.

B.1.3.1.2 Solution B

Introduce 400 ml of de-ionized water to a 1 l beaker (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 drops 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 dissolves. 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 de-ionized water.

Cover and allow to stand for at least 24 h.

NOTE The electrolysis bath should be used within 15 days of preparation. It is sufficient to treat 20 to 30 blanks. The bath should be covered between each electrolysis.

B.1.3.2 Degreasing bath

Prepare and use the degreasing bath according to the manufacturer's instructions.

B.1.3.3 Stripping bath

In a 5 l beaker (B.1.2.6), dissolve 170 g of ammonium persulfate (B.1.1.6) in 1 l de-ionized 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 with the addition of de-ionized water.

NOTES

1 The dissolution of the ammonium persulfate is endothermic.

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

B.1.3.4 Decontamination bath

In a 5 l beaker (B.1.2.6), dilute 200 ml of sulfuric acid (B.1.1.2) to 1 l of de-ionized water by slowly adding 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 the blanks (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 blanks shall be prepared together, as described in the method 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 de-ionized 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 de-ionized 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 beaker (B.1.2.6) containing the copper plating bath and making the electrical connections according to figure B.2;
- connect the blank, then immerse at minimum voltage in the electrolysis bath according to figure B.2;
- as soon as the blank is put in the bath, gradually increase the current to reach a value of 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 out the blank and rinse under a flow of de-ionized water for approximately 30 s;
- dry the frame quickly between sheets of absorbent paper (B.1.2.13), without rubbing. Take out the copper plated blank, dry by pressing it between sheets of absorbent paper.

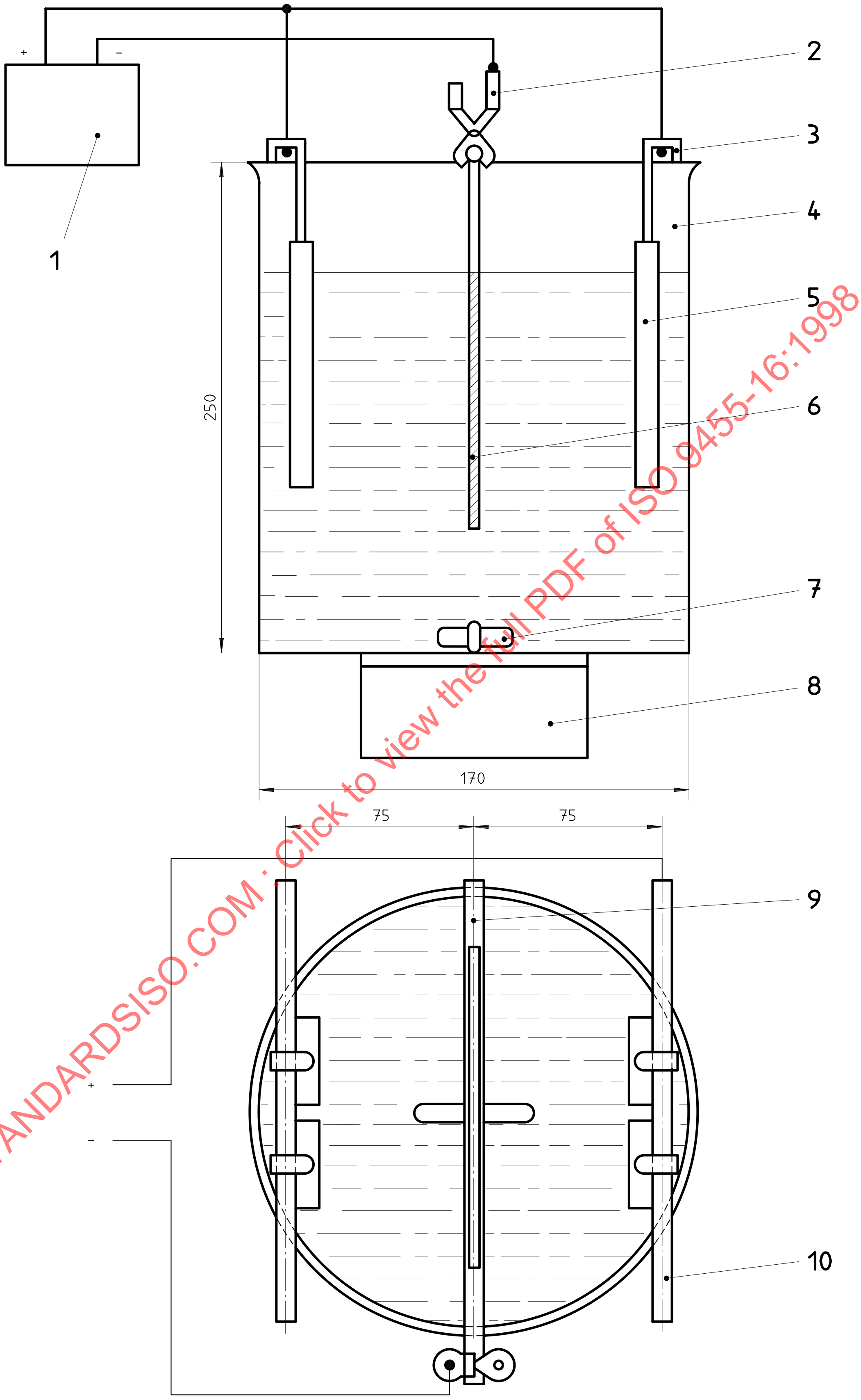
Store the copper blanks carefully between sheets of inhibitor paper (B.1.2.14).

NOTES

1 The deposit thickness should be approximately 12 µm.

2 As long as any manual contact is avoided, the blanks can be stored indefinitely.

Dimensions in milimetres



Key

1 Stabilized power supply
2 Crocodile clip
3 Stainless anode support
4 5 dm^3 glass beaker
5 Anode
6 Blank (cathode)
7 Magnetized bar
8 Magnetic stirrer
9 Cathode support bar
10 Anode support bar

Figure B.2 — Electrolysis system