



BSI Standards Publication

Packaging — Test method to determine the porosity of the internal coating of flexible aluminium tubes

Part 2: Copper sulphate test

National foreword

This British Standard is the UK implementation of EN 15384-2:2017. Together with BS EN 15384-1:2017, it supersedes BS EN 15384:2007 which is withdrawn.

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A list of organizations represented on this subcommittee can be obtained on request to its secretary.

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English Version

**Packaging - Test method to determine the porosity of the
internal coating of flexible aluminium tubes - Part 2:
Copper sulphate test**

Emballage - Méthode d'essai pour déterminer la
porosité du revêtement intérieur des tubes souples en
aluminium - Partie 2 : Essai au sulfate de cuivre

Packmittel - Prüfverfahren zur Bestimmung der
Porosität der Innenbeschichtung von Aluminiumtuben
- Teil 2: Kupfersulfatverfahren

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European foreword

This document (EN 15384-2:2017) has been prepared by Technical Committee CEN/TC 261 "Packaging", the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by July 2017, and conflicting national standards shall be withdrawn at the latest by July 2017.

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1 Scope

This European Standard is applicable for internally coated cylindrical aluminium tubes, mainly used for the packing of pharmaceutical, cosmetic, hygiene, food or other household products.

The internal coating is used as a barrier and should avoid any contact between aluminium and the product. This standard defines the copper sulphate method to detect the electrolyte conductivity as one criterion for the quality of the internal coating.

The electrolyte conductivity of the internal coating is only one criterion for evaluation of the quality of an internal coating. It does not give any information on the quantity or size of any pores or uncoated areas, nor any hint on possible reactions between the aluminium tube and the product. The electrolyte conductivity should never be used as the sole criterion for quality evaluation of the internal coating, but always with other parameters e.g. film thickness, acetone and/or ammonia resistance and of course results of enhanced stability studies.

2 Principle

The electrolyte conductivity of internally coated aluminium tubes is tested by an enamel conductometer. The aluminium tubes are filled with an electrolyte solution up to a fixed level at its open end. One electrode is connected to the tube nozzle, the second electrode is dipped into the solution. A defined voltage is applied for a fixed time. The induced current is a measure for the quality (pores and/or film thickness) of the internal coating.

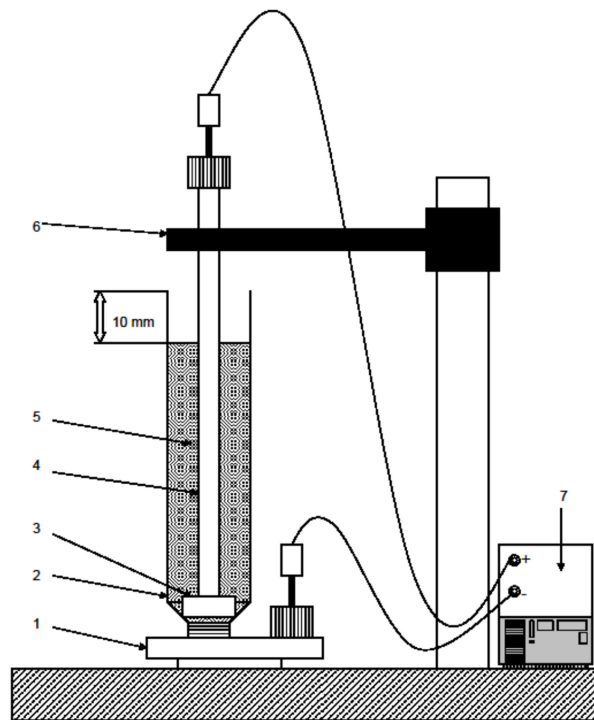
3 Apparatus

3.1 Enamel conductometer

3.2 Moveable electrode

3.3 Electrolyte

NOTE A schematic diagram of the test equipment is given in Figure 1.



Key

- 1 electrode (negative pole)
- 2 aluminium tube
- 3 insulating spacer
- 4 movable electrode (positive pole)
- 5 electrolyte
- 6 electrode holder
- 7 processor unit

Figure 1 — Test device

4 Procedure

All parameters having a significant influence on the test results for the copper sulphate method are listed in Table 1.

Table 1 — Test conditions for the copper sulphate method

Parameter	Copper sulphate method
Temperature	23 ± 2 °C
Voltage and measuring interval internal resistance	4 500 mV DC 2,5 s voltmeter 20 kΩ/V ammeter 2,5 Ω at 100 mA
Electrodes - polarity - material - diameter	dipping electrode is the positive pole dipping electrode copper (a round insulating spacer of 5 ± 2 mm has to be mounted on the bottom of the electrode to avoid direct contact between electrode and tube shoulder) adapter electrode stainless steel 5 mm ± 0,3 mm for tube diameter < 19 mm 10 mm ± 0,5 mm for tube diameter ≥ 19 mm
Electrolyte	Copper sulphate solution (10,0 ± 0,01) g Copper sulphate Pentahydrate p.a. CuSO ₄ 5H ₂ O CAS ^{a)} (7758-99-8) (5,00 ± 0,01)ml Acetic acid, 100 %, p.a., HAC ^{b)} CAS ^{a)} (64-19-7) (0,05 ± 0,01) g/l wetting agent CAS ^{a)} 68425-44-5 Dissolve the CuSO ₄ 5H ₂ O in deionized water, add the HAC and wetting agent and make up to (1 000 ± 2) ml with deionized water. The electrolyte conductivity of the finished solution shall be (4,75 ± 0,25) mS.
<p>^a CAS is Chemical Abstracts Service – CAS registry numbers are unique numerical identifiers for chemical compounds, polymers, biological sequences, mixtures and alloys.</p> <p>^b HAC is the most common abbreviation for acetic acid in the context of acid-base reactions.</p>	

Depending on the method and the kind of apparatus (different apparatus are available) specific adjustments and calibrations may be necessary and should be done as described in the user manuals prior to use. To take a measurement put the tube into the adapter electrode, ensuring there is an electric circuit between the tube and electrode. Insert the dipping electrode centrally into the tube until it touches the tube shoulder, avoiding any contact between the tube wall / tube shoulder and the dipping electrode. Fill the tube with electrolyte up to 10 mm from the end after inserting the electrode. Start the measurement – application of voltage – immediately. Read the result in mA on the display after the measuring interval. The electrolyte can be used for testing 10 tubes. The test on one tube is not repeatable for the copper sulphate method.

5 Tolerances

The tolerances given in this standard are based on long-term experiences and are valid only for membrane tubes. For tubes with special conification angles and nozzles, e.g. without membrane or cannula, higher tolerance limits may be required.

In addition, for some specific internal coatings but also depending on the product, different tolerance limits may be required. Any deviations from this standard shall be agreed mutually between both parties.

Tolerances for the copper sulphate method are given in Table 2:

Table 2 — Tolerances for the copper sulphate method

Tube diameter \varnothing [mm]	Mean Value [mA]	Single Value [mA]
$\varnothing \leq 28$	≤ 25	≤ 80
$28 < \varnothing \leq 45$	≤ 35	≤ 80
$45 < \varnothing \leq 50$	≤ 45	≤ 80

Due to several variables which can affect the result (e.g. corrosion, surface of copper electrode) the accuracy of the methods may vary by several mA. For making well-founded conclusions a representative number of tubes should always be tested.

NOTE 1 Test apparatus from different suppliers may give different results. In this case comparative measurements and correlations factors may be required.

NOTE 2 The tolerances given in Table 2 are only valid for standard tubes and not for tubes with additional treatment such as sterilization.

6 Test report

Unless otherwise agreed between both parties the test report shall contain the following information:

- a) reference to this standard and – if necessary – a specification for the method of sampling and acceptance of the batch,
- b) complete identification of the batch and of the tubes checked,
- c) description of the samples,
- d) registering number of the apparatus and the selected test parameters,
- e) type of internal coating,
- f) test results,
- g) number of tested tubes,
- h) number of defects,
- i) if applicable the decision for acceptance or rejection,
- j) all factors, that may have had an influence on the test results and which are not covered by this standard,
- k) date, place and name of the inspector.

Bibliography

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