EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 15384

November 2007

ICS 55.120

English Version

Packaging - Flexible aluminium tubes - Test method to determine the porosity of the internal coating

Emballage - Tubes souples en aluminium - Méthode d'essai pour déterminer la porosité du revêtement intérieur

Packmittel - Aluminiumtuben - Prüfverfahren zur Bestimmung der Porosität der Innenbeschichtung

This European Standard was approved by CEN on 30 September 2007.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

© 2007 CEN All rights of exploitation in any form and by any means reserved worldwide for CEN national Members.

Ref. No. EN 15384:2007: E

Contents

Page

Foreword 3
1 Scope 4
2 Principle 4
3 Apparatus 4
4 Procedure 5
5 Tolerances 7
6 Test report 8
Bibliography 9

Foreword

This document (EN 15384:2007) 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of an identical text or by endorsement, at the latest by May 2008, and conflicting national standards shall be withdrawn at the latest by May 2008.

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

This standard is based on the professional recommendations of the European Tube Manufacturers Association (etma).

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This standard is applicable for internally coated 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 two alternative methods – copper sulphate and sodium chloride method – to detect the electrolyte conductivity as one criterion for the quality of the internal coating.

NOTE 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.

NOTE In the early 80´s the copper sulphate method was developed and standardized (e.g. DIN 55436-3 [1]). Subsequently this method became a nationally and internationally well accepted quality criterion for the internal coating of metal tubes and cans. In the past few years a new method, the sodium chloride method, came into being, holding several advantages concerning accuracy, reproducibility and repeatability as well as on health and environmental aspects. As the changeover from the copper sulphate to the sodium chloride method will require a transitional period, this standard describes both methods. It is planned to delete the copper sulphate method as a national and/or international standard in the next revision of this document.

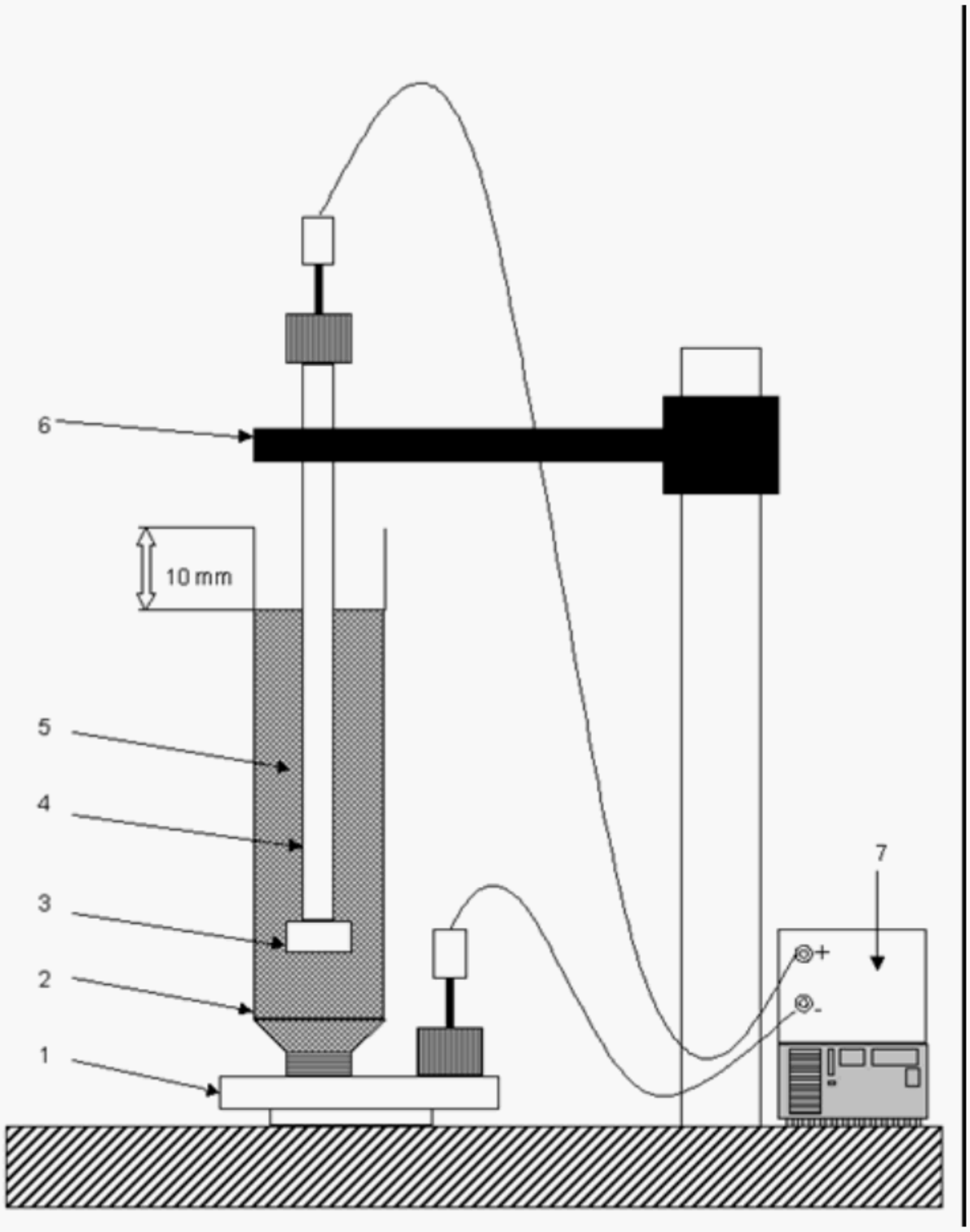
3 Apparatus

— Enamel conductometer

— Moveable electrode

— Electrolyte

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



Key

1 Electrode
2 Aluminium tube
3 Insulating spacer
4 Moveable electrode
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 and sodium chloride methods are listed in Table 1.

Table 1 — Test conditions for the copper sulphate and the sodium chloride method

Parameter	Copper sulphate method	Sodium chloride method
Temperature	23 ± 2 °C	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	6 300 mV DC 4,0 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 plastic cap 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	 dipping electrode is the negative pole dipping electrode stainless steel adapter electrode stainless steel 6 mm ± 0,3 mm for all tube sizes
Electrolyte	Copper sulphate solution (10,0 ± 0,01) g Copper sulphate Pentahydrate p.a. $CuSO_4\ 5H_2O$ 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 $CuSO_4\ 5H_2O$ 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.	Sodium chloride solution (10,2 ± 0,01) g Sodium chloride p.a. NaCl, CAS[a] 7647-14-5 Dissolve the NaCl in deionized water and make up to (1 000 ± 2) ml with deionized water.

[a] CAS is Chemical Abstracts Service – CAS registry numbers are unique numerical identifiers for chemical compounds, polymers, biological sequences, mixtures and alloys.
[b] HAc is the most common abbreviation for acetic acid in the context of acid-base reactions.

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, 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, tests for one tube can be repeated with the sodium chloride 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 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 Ø [mm]	Mean Value [mA]	Single Value [mA]
Ø ≤ 28	≤ 25	≤ 80
28< Ø ≤ 45	≤ 35	≤ 80
45< Ø ≤ 50	≤ 45	≤ 80

Tolerances for the sodium chloride method are given in Table 3:

Table 3 — Tolerances for the sodium chloride method

Tube diameter Ø [mm]	Maximum electrolyte conductivity [mA]			
	Cylindrical tubes		Conical tubes	
	Mean value	Single value	Mean value	Single value
Ø ≤ 20	10	25	15	30
20< Ø ≤ 30	15	30	20	40
30< Ø ≤ 40	20	40	25	50
40< Ø ≤ 45	25	50	30	60
45< Ø ≤ 50	35	80	-	-

NOTE 1 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 2 Test apparatus from different suppliers may give different results. In this case comparative measurements and correlations factors may be required.

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

[1] DIN 55463-3, *Verpackungsprüfung; Aluminiumtuben; Bestimmung der Porigkeit der Innenschutzlackierung; Elektrolytisches Verfahren*

[2] (Packaging test; tubes made of aluminium; disposition of porosity of the inside protective lacquer; electrolytical test)

[3] EN 12374, *Packaging – Flexible tubes – Terminology*

[4] EN 13046, *Packaging – Flexible cylindrical metallic tubes – Dimensions and tolerances*

[5] EN 13047, *Packaging – Flexible conical metallic tubes – Dimensions and tolerances*

[6] ISO 2859-1, *Sampling procedures for inspection by attributes – Part 1: Sampling schemes indexed by acceptance quality limit (AQL) for lot-by-lot inspection*