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Influence of metallic materials on water intended for human consumption — Determination of residual surface lead (Pb) — Extraction method

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National foreword

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

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English Version

Influence of metallic materials on water intended for human consumption - Determination of residual surface lead (Pb) - Extraction method

Influence des matériaux métalliques sur l'eau destinée à la consommation humaine - Dosage du plomb (Pb) résiduel de surface - Méthode d'extraction

Einfluss metallischer Werkstoffe auf Wasser für den menschlichen Gebrauch - Bestimmung des Rückstands an Oberflächenblei (Pb) - Extraktionsverfahren

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Foreword

This document (EN 16057:2012) has been prepared by Technical Committee CEN/TC 164 "Water supply", 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of an identical text or by endorsement, at the latest by November 2012, and conflicting national standards shall be withdrawn at the latest by November 2012.

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 European Standard is one of a series of test methods which support associated product standards.

In respect of potential adverse effects on the quality of water intended for human consumption caused by metallic materials, attention is drawn to the fact that the relevant national regulations remain in force until the adoption of verifiable European acceptance criteria. Water intended for human consumption is hereafter referred to as "drinking water" and means the same as the definition given at Article 2(1) of the Council Directive 98/83/EC on the quality of water intended for human consumption.

This document describes a test method to determine the presence of lead (Pb) on the surface of copper alloys.

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, Croatia, 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, Turkey and the United Kingdom.

Introduction

During processing of lead containing copper alloys a lead film might be formed on the surface of the material. These lead layers depend on the individual manufacturing process at the different production sites. The lead film on the inner surface of products will cause a lead release into the drinking water in the first weeks after a new product comes in contact with drinking water (short term behaviour).

The lead release from the bulk material is not affected by the lead film on the surface and depends on the composition of the material. The bulk material can release lead for a long period (long term behaviour). It is possible to test materials for their lead release from the bulk material (EN 15664-1 and -2), so that products made of approved materials do not have to be tested for this characteristic. As the lead films on the surface depend on the individual manufacturing process, it is necessary to test the products or the manufacturing process.

This test method is intended to be used as a process control method to assess the presence of lead films on the inner surface of products intended to come in contact with drinking water or to assess the efficiency of a manufacturing process to remove or minimise surface Pb, e.g. washing process to remove lead on surfaces.

The implementation of an audit controlled process monitoring is an effective way to maintain safety of the products in the first weeks of their lifetime.

This test method has been determined as a result of research programme *Action 14/2005 objective A* sponsored by DG Enterprise of the EU Commission.

1 Scope

This European Standard describes a test method to determine the amount of lead on the surface of test specimens made from lead containing copper alloys.

2 Normative references

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

CEN/TS 13388:2008, *Copper and copper alloys — Compendium of compositions and products*

3 Terms and definitions

3.1

method control sample

MCS

tube of defined geometry made under defined conditions from a material of defined composition to check the extraction method

Note 1 to entry: See 7.1.4.

3.2

process sample

PS

tube of defined geometry processed in the same way as a product

3.3

test specimen

process sample or method control sample

3.4

extract

test solution after the extraction procedure

3.5

extraction time

time of contact between test solution and test specimen

4 Principle

Lead films on the inner surface of a test specimen are dissolved by using a defined test solution. This procedure is repeated and each extract is analyzed for lead. The total mass of the surface lead is calculated from the sum of the Pb removed.

The method is sensitive to the concentration and age of the acids used for making up the test solution and the degree of shaking in the procedure, therefore, the effectiveness of the method is checked using Method-Control-Samples.

5 Reagents

The following chemicals shall be used:

- a) tetrafluoroboric acid (HBF₄) 50% (m/m) CAS number [16872-11-0];
- b) sulphamic acid (H₃N-SO₃) CAS number [5329-14-6].

All chemicals shall be analytical grade.

NOTE Only use HBF₄ solution which has been stored at low temperatures (less than 8 °C) in tightly closed bottles and in the dark to avoid degradation of the acid.

6 Apparatus

The following apparatus shall be used:

- a) Volumetric flask (1l);
- b) Polyethylene (PE) bottles (20 ml to 50 ml);
- c) Test specimen caps made of inert material (i.e. will not release lead or any other material that will adversely affect the test results) e.g. polyethene;
- d) Flat seal made of inert material;
- e) Sealing tape made of Polytetrafluoroethylene (PTFE);
- f) Stop watch;
- g) Optional: Mechanical shaker with a minimum amplitude of 20 mm and a deflection rate of 4/s.

7 Test specimen

7.1 Method-control-sample (MCS)

7.1.1 Use of method control sample

The MCS shall be used to verify the effectiveness of the extraction method.

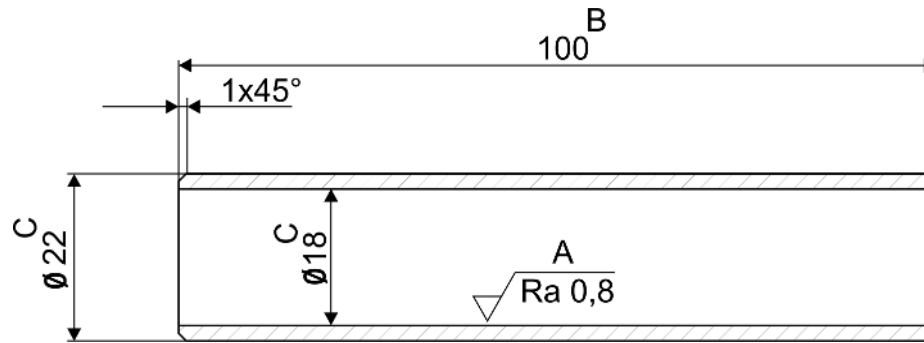
7.1.2 Material of method-control-sample

The MCS shall be made from CW614N - CuZn39Pb3.

The value for lead shall be in the range of 3,0 % – 3,4 % Pb. The remainder of the composition including unavoidable impurities shall have the limits of the specification CW614N according to CEN/TS 13388:2008.

7.1.3 Geometry

The geometry shall be as in Figure 1.



Key

- A – Dry machining
- B – Length (mm)
- C – Diameter (mm)

Figure 1 — Test specimen dimensions

7.1.4 Manufacturing process for the MCS

Starting with bar stock of MCS material with diameter 22 mm, bore out to diameter 16 mm then with a turning-tool to diameter 18 mm. The mean roughness shall be $Ra = 0,8 \mu\text{m} \pm 0,05 \mu\text{m}$ by dry drilling. Make sure that the machining diameter accuracy is at least of the tolerance $\pm 0,5 \text{ mm}$.

Machining parameters:

- a) depth of cut: $a = 1 \text{ mm}$;
- b) rotation speed: $N = 630 \text{ 1/min}$;
- c) feed rate: $f = 0,066 \text{ mm/revolution}$;
- d) tool kit: using an cutting-tool holder for machining with hard-metal-pole and coolant hole; indexable insert (hard metal).

Blow oil-free compressed air through the coolant hole for cooling and swarf removal.

7.2 Process sample (PS)

The process sample shall be manufactured according to the production process to be tested in terms of composition, surface and process parameters.

The geometry shall be as in Figure 1, with the exception of the mean roughness. This parameter shall be to the same as the product specification whose production process is tested.

8 Test procedure

8.1 Method-control-sample (MCS)

The MCS shall be used on a regular basis, the frequency being determined according to the quality assurance procedure of the laboratory, to show the effectiveness of the extraction method.

8.2 Preparation of the test solution

The test solution shall be freshly prepared for each test. Starting with a one-litre volumetric flask carry out the following:

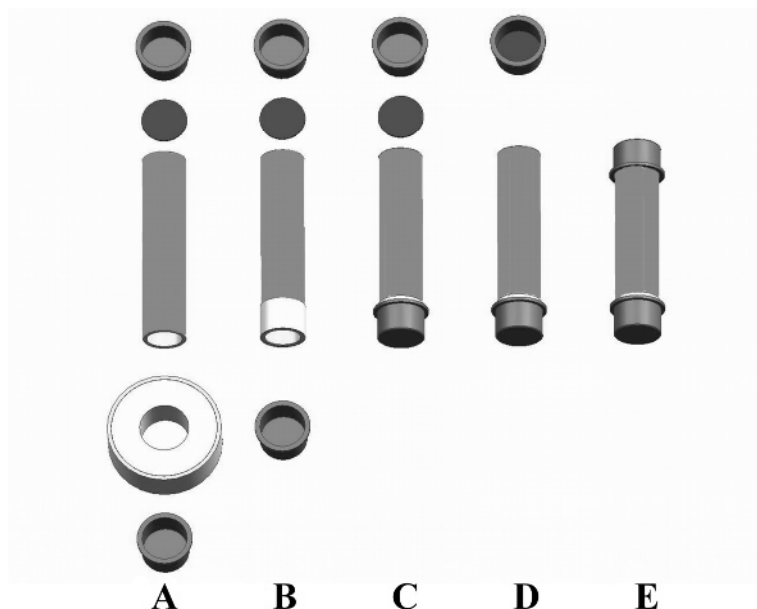
- a) add 10,0 g \pm 0,05 g of sulphamidic acid (5(b));
- b) add approximately 200 ml of demineralized water and shake thoroughly to dissolve the sulphamidic acid;
- c) add 15,0 ml \pm 0,05 ml of tetrafluoroboric acid (5(a));
- d) dilute to 1 litre with demineralized water.

8.3 Blank sample

Take a sample of the test solution as blank sample for the determination of the blank value. Determine the lead content of the blank sample. If the lead content exceeds 1 mg/l the test solution cannot be used: discard and start again with fresh reagents.

8.4 Assembly of the test specimen

Assemble the test specimen as shown in Figure 2.



Key

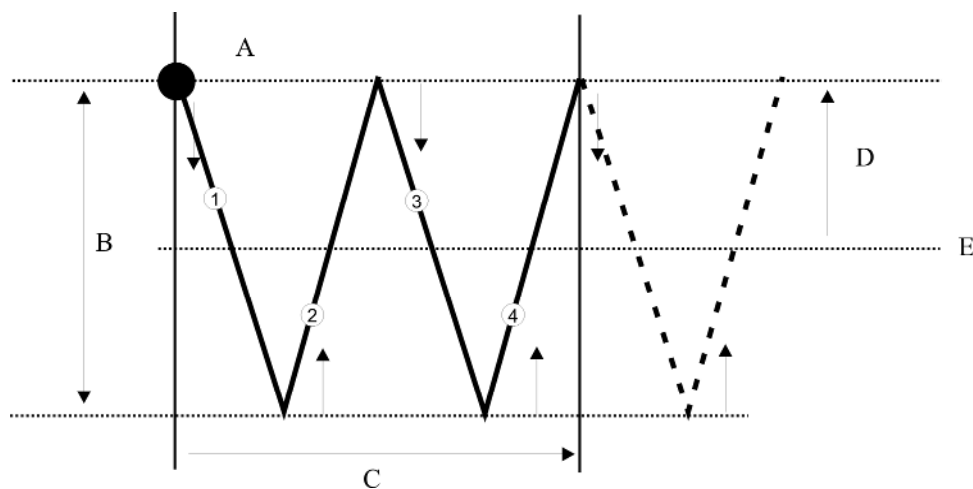
A – step 1 B – step 2 C – step 3 D – step 4 E – step 5

Figure 2 — Illustration of the assembly procedure of test specimen

8.5 Extraction procedure

- a) Prepare 10 labelled PE bottles.
- b) Blow out the test specimen with dry oil-free air and make sure that there is no swarf in the bore.
- c) Wrap sealing tape around one end of the test specimen and close this prepared end with a test specimen cap (see Figure 2, steps 1 to 4).
- d) Add to the test specimen exactly 15,0 ml \pm 0,05 ml of the test solution.
- e) Close the open end with a flat seal and a PE-cap (Figure 2, step 5).
- f) Start shaking within 30 s after adding the test solution.

- g) Shake the test specimen in the longitudinal direction, either horizontally or vertically, with a minimum amplitude of 20 mm. Ensure the test specimen goes through shaking deflections at a rate of 4 per s (see Figure 3).
- h) Shake the test specimen for 120 s.
- i) Open the end with the flat seal and a PE-cap and pour the extract from the test specimen into the first or sequential labelled PE bottle within 30 s after finishing the shaking.
- j) Immediately repeat the extraction procedure with this test specimen from point e) above another nine times (in total 10 times).
- k) Analyze the extract of each of the 10 PE bottles.



Key

- A = 4 deflections in one second
- B = deflection 40 mm minimum
- C = one second
- D = amplitude 20 mm minimum
- E = center
- 1,2,3,4 = number of deflections

The deflection is defined as the sum of the amplitude to one side and to the other side during the shaking procedure.

Figure 3 — Schematic description of the shaking procedure

NOTE It is recommended to test only one test specimen at a time in order to meet the prescribed extraction time.

9 Control criteria for the method

For verification of the method by using MCS the following criteria shall be met:

- First extract: $C_{Pb\ 1} \geq 200\ \text{mg/l}$
- 5th to 10th extract: $C_{Pb\ 5-10} \leq 10\ \text{mg/l}$

where

$C_{Pb,j}$ = concentration of Pb of the jth extract in mg/l

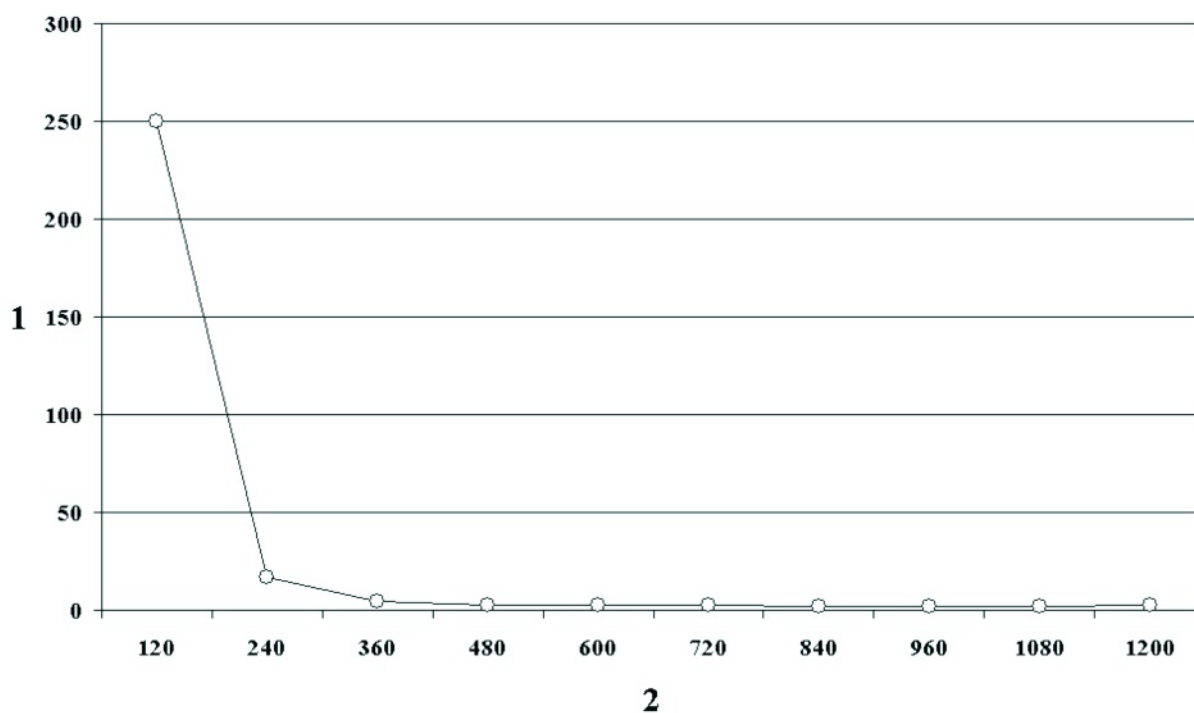
10 Determination of residual surface lead (Pb)

Correct the analytical results for the blank value.

List the Pb contents of the extract as a function of the extraction time as shown in Table 1 and plot the Pb concentration versus cumulative time in a diagram (see Figure 4).

Table 1 — Results table

Extract	1	2	3	4	5	6	7	8	9	10
Extraction time (s)	120	120	120	120	120	120	120	120	120	120
Cumulative time (s)	120	240	360	480	600	720	840	960	1080	1200
c_{Pb} (mg/l)										



Key

1 = Concentration of Pb (mg/l) 2 = Cumulative time (s)

Figure 4 — Diagram (Example)

Calculate the total mass of Pb (m_{Pb}) from extract 1-5 according to:

$$m_{Pb} = \sum_{i=1}^5 c_{Pb,i} * V_S$$

where

$c_{Pb,j}$ = concentration of Pb of the j^{th} extract in mg/l;

m_{Pb} = Total mass of extracted lead in the first five extracts in mg;

V_S = Volume of the test solution in litre (see 8.5, d)).

11 Test report

The test-report shall contain:

- a) name and address of the test institute;
- b) name(s), function(s) and signature(s) or equivalent identification of person(s) authorizing the test report;
- c) unique identification number, signature and title or an equivalent marking of person (s) accepting technical responsibility for the test report and date of issue;
- d) name and address of the customer;
- e) name and address of the manufacturer of the MCS;
- f) reference to this standard;
- g) all details required for the complete identification of the process sample (PS);
 - 1) chemical composition of the PS;
 - 2) surface characteristics of the PS;
 - 3) description of the manufacturing steps used to produce the PS;
 - 4) description of the production process to be tested;
- h) date of starting and finishing the test;
- i) details of any deviation from the procedure specified;
- j) specification of the analytical devices used;
- k) diagram lead concentration versus the cumulative time for testing PS;
- l) diagram lead concentration versus the cumulative time for MCS;
- m) results (results table, diagram, total mass of lead (Pb)).

Bibliography

- [1] EN 15664-1, *Influence of metallic materials on water intended for human consumption — Dynamic rig test for assessment of metal release — Part 1: Design and operation*
- [2] EN 15664-2, *Influence of metallic materials on water intended for human consumption — Dynamic rig test for assessment of metal release — Part 2: Test waters*

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