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**Preskusne metode za analizo svınca v PVC (polivinilkloridu), ki se uporablja za izolacijo in oplaščenje električnih kablov in kablov z optičnimi vlakni - Metoda A: Določevanje celotnega svınca z atomsko absorpcijsko spektrometrijo z vzbujanjem plamena - Metoda B: Kvalitativna metoda z barvanjem s svinčevim sulfidom**

Test methods for analysis of lead in PVC taken from insulation and sheath of electric and optical fibre cables - Method A: Total lead content determination with flame excitation atomic absorption spectrometry - Method B: Qualitative analysis of lead by lead sulphide staining

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**Test methods for analysis of lead in PVC taken from insulation  
and sheath of electric and optical fibre cables -  
Method A: Total lead content determination  
with flame excitation atomic absorption spectrometry -  
Method B: Qualitative analysis of lead by lead sulphide staining**

Méthodes d'analyse du plomb  
dans le PVC prélevé de l'enveloppe  
isolante et des gaines des câbles  
électriques et à fibres optiques -  
Méthode A: Détermination de la teneur  
totale en plomb par spectrométrie  
atomique d'absorption dans la flamme -  
Méthode B: Analyse qualitative du plomb  
par production de sulfure de plomb

Prüfverfahren für die Analyse von Blei  
in PVC entnommen aus Isolierung  
und Mantel von Kabeln, isolierten  
Leitungen und Lichtwellenleiterkabeln -  
Verfahren A: Bestimmung  
des Gesamtleigehalts mit der Flammen-  
Atomabsorptionsspektrometrie -  
Verfahren B: Qualitative Analyse  
des Bleigehalts durch Bleisulfidverfärbung

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## CENELEC

European Committee for Electrotechnical Standardization  
Comité Européen de Normalisation Electrotechnique  
Europäisches Komitee für Elektrotechnische Normung

Central Secretariat: rue de Stassart 35, B - 1050 Brussels

## Foreword

This European Standard was prepared by the Technical Committee CENELEC TC 20, Electric cables.

The text of the draft was submitted to the formal vote and was approved by CENELEC as EN 50414 on 2006-03-01.

The following dates were fixed:

- latest date by which the EN has to be implemented  
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## Introduction

The determination of small quantities of lead in PVC is possible using a number of analytical techniques. For the purposes of this European Standard two alternative methods are given.

Method A uses flame excitation atomic absorption spectrometry, for the total lead determination in the PVC.

Method B uses a qualitative method for the analysis of lead in the PVC. The method utilises the lead sulfide staining test and is therefore best suited to light coloured or natural materials. It can be considered to be a spot check for the presence of lead or lead compounds.

Other decomposition and determination methods are capable of detecting and analysing small amounts of lead in cable grade PVC. Examples of such methods are X-ray fluorescence spectroscopy, graphite furnace atomic absorption spectrometry, inductively coupled plasma spectrometry and polarography. Contracting parties may agree to use such other methods, but such methods cannot claim conformity to EN 50414. If used, it is recommended that such methods have at least equivalent sensitivity and detection levels as those in this EN.

## 1 Scope

This European Standard describes two methods for the analysis of lead in PVC-insulating and sheathing materials for electric and for optical fibre cables. The samples of PVC are taken from the finished cable.

Method A provides a quantitative method and Method B provides a qualitative method.

NOTE 1 Users may refer to either Method A or Method B or to both methods.

NOTE 2 Other methods of determination not specified here are known to be capable of achieving equivalent results, but do not conform to this EN (see "Introduction").

Both Method A and Method B are applicable to products having a total lead content greater than or equal to 0,01 %.

## 2 Normative references

The following referenced documents are indispensable for the application 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.

<u>Publication</u>	<u>Year</u>	<u>Title</u>
EN ISO 1042	1999	Laboratory glassware - One-mark volumetric flasks (ISO 1042:1998)
EN ISO 3696	1995	Water for analytical laboratory use - Specification and test methods (ISO 3696:1987)
ISO 385-1	1984	Laboratory glassware - Burettes - Part 1: General requirements
ISO 5725	Series	Accuracy (trueness and precision) of measurement methods and results
ISO 6503	1984	Paints and varnishes - Determination of total lead - Flame atomic absorption spectrometric method

### 3 Methods of analysis

#### 3.1 General

In case of dispute, the decomposition of the material by the dry calcination method and the analysis of the material by the flame excitation atomic absorption spectrometry (Method A of this standard) shall be the reference method. Method A has been derived from ISO 6503.

#### 3.2 Method A

##### 3.2.1 Principle

The lead content is determined using flame excitation atomic absorption spectrometry following the decomposition of a sample by dry calcination and acid extraction of the residue.

##### 3.2.2 Reagents

During the decomposition and extraction, use only reagents of a recognized analytical quality, and demineralized or distilled water of a purity of at least Grade 3 in accordance with EN ISO 3696.

**3.2.2.1** *sodium carbonate*, anhydrous

**3.2.2.2** *magnesium carbonate*

**3.2.2.3** *sulfur*

**3.2.2.4** *sodium sulfide*, solution at 10 g/l

**3.2.2.5** *hydrochloric acid*, at approximately 180 g/l

450 ml of concentrated hydrochloric acid (36 %, with a specific gravity  $\rho$  of approximately 1,18 g/ml) shall be added to approximately 450 ml of water and shall be made up to 1 000 ml.

**3.2.2.6** *hydrochloric acid*, at approximately 18 g/l

100 ml of hydrochloric acid (made up in 3.2.2.5) shall be added to water, and shall be made up to 1 000 ml.

**3.2.2.7** *nitric acid*, at approximately 315 g/l

One volume of concentrated nitric acid (about 65 %, with a specific gravity  $\rho$  of approximately 1,40 g/ml) shall be added to two volumes of water.

**3.2.2.8** *lead reference solution*, containing 1 g of lead per litre

Exactly 1 g of lead shall be dissolved in 30 ml of the nitric acid prepared in 3.2.2.7 and shall be transferred, using a funnel, to a 1 000 ml volumetric flask. Distilled or deionized water shall be used to dilute the solution to the mark and shall be mixed well.

1 ml of this reference solution shall contain 1 mg of lead.

NOTE ISO 6503 gives further details for the preparation of this solution.

**3.2.2.9** *lead reference solution*, containing 100 mg of lead per litre

This solution shall be prepared fresh each day of its use.

10 ml of the reference solution (see 3.2.2.8) shall be transferred with the aid of a pipette into a 100 ml volumetric flask, and shall be diluted to the mark with hydrochloric acid (see 3.2.2.6) and shall be mixed well.

1 ml of this reference solution shall contain 100 µg of lead.

### 3.2.3 Apparatus

Ordinary laboratory apparatus, and

3.2.3.1 *silica crucibles*, preferably new

3.2.3.2 *muffle furnace*, at  $(475 \pm 25)$  °C

3.2.3.3 *hot plate with thermostatic control*

3.2.3.4 *desiccator*

### 3.2.4 Sampling

Take from the cable a representative sample of the material to be tested. The sample shall be cut into small pieces of approximately 0,05 g.

### 3.2.5 Test procedure

#### 3.2.5.1 Preliminary tests

If the composition of the material to be tested is not known, carry out qualitative tests to determine whether antimony is present. If the results of these tests confirm the presence of antimony, the extraction shall be carried out as specified in 3.2.5.4.2; if not, use the method described in 3.2.5.4.1.

#### 3.2.5.2 Sample

Introduce approximately 1 g of the material to be tested into a pre-weighed silica crucible (see 3.2.3.1). Weigh the sample and crucible to the nearest 10 mg and determine the actual sample weight. Repeat these tasks.

#### 3.2.5.3 Calcination

Spread 2 g of magnesium carbonate (see 3.2.2.2) on to the sample and introduce the crucible into the muffle furnace (see 3.2.3.2) at ambient temperature. Heat the furnace up to a temperature of 350 °C in approximately 30 min, then increase the furnace temperature up to  $(475 \pm 25)$  °C over 60 min and maintain this temperature until complete calcination has been achieved. Make sure that there is sufficient air supply for oxidation.

NOTE 1 Care should be taken to ensure that the contents of the crucible do not ignite.

NOTE 2 Three hours should be sufficient to ensure complete calcination. If there is any doubt, an extra period of time should be added.

#### 3.2.5.4 Extraction

##### 3.2.5.4.1 Product not containing antimony

If the product does not contain antimony (see 3.2.5.1), proceed as follows.

Allow the crucible and its contents to cool in a desiccator. Transfer the crucible and the ashes into a 250 ml beaker, add 100 ml of hydrochloric acid (see 3.2.2.5), heat and keep boiling gently for 15 min on the hot plate (see 3.2.3.3). Allow to digest for a further 15 min on the hot plate.



While the solution is hot, filter by decantation through a fine texture filter paper into a 250 ml beaker. The filter paper shall consist of undyed cellulose filter paper of a density of  $(85 \pm 15) \text{ g/m}^2$  with an ash content of less than 0,1 % and particle retention of at least 4  $\mu\text{m}$  or better.

Wash the filter paper and the residue with hot water collecting the rinsings in the beaker. Cool the beaker and transfer the filtrate and the rinsings into a 250 ml volumetric flask. Dilute to the 250 ml mark with water and mix well.

#### 3.2.5.4.2 Product containing antimony

If the product does contain antimony (see 3.2.5.1), proceed as follows.

Crush the ash to dust, put it into the crucible and mix it with approximately 10 g of a mixture made of equal portions of sodium carbonate (see 3.2.2.1) and sulfur (see 3.2.2.3). Cover the crucible and heat it over a moderate flame until the smell of sulfur dioxide disappears; this should take from 1 h to 2 h.

Cool the crucible and digest the contents with a small quantity of hot water until complete dissolution of the residual sodium carbonate/sulfur mixture. Filter, by transferring the whole residue on to a filter paper and wash the residue with the sodium sulfide solution (see 3.2.2.4). Discard the filtrate.

Transfer the filter paper and the residue into a 250 ml beaker. Add 15 ml of nitric acid (see 3.2.2.7), heat to boiling and simmer for 15 min on the hot plate (see 3.2.2.3). Add 100 ml of hydrochloric acid (see 3.2.2.5) and allow to digest for 30 min, whilst still on the hot plate. While the solution is hot, filter through a fine texture filter paper into a 250 ml beaker. Wash the filter paper and the residue with hot water, collecting the rinsings in the beaker. Cool the beaker and transfer the filtrate and the rinsings into a 250 ml volumetric flask. Dilute to the 250 ml mark with water and mix well.

#### 3.2.5.5 Preparation of the blank test solution

Repeat the procedures described in 3.2.5.4.2, but omitting the test sample.

### 3.2.6 Determination

#### 3.2.6.1 Principle

An aspiration of the test solution into an acetylene/air flame is followed by measurement of the absorption of the selected spectral line, emitted by a lead hollow cathode lamp or a lead electrodeless discharge lamp within that has a range including 283,3 nm.

#### 3.2.6.2 Reagents and products

During the determination, use only reagents of a recognized analytical quality, and demineralized or distilled water of a purity of at least Grade 3 in accordance with EN ISO 3696 as well as the following gases:

**3.2.6.2.1** *acetylene*, of industrial quality, in a steel bottle

**3.2.6.2.2** *compressed air*

#### 3.2.6.3 Apparatus

Ordinary laboratory apparatus and

**3.2.6.3.1** *flame excitation atomic absorption spectrometer*, suitable for measurements at a wavelength of 283,3 nm and fitted with an acetylene and air-supplied burner

**3.2.6.3.2** *lead hollow cathode lamp or lead electrodeless discharge lamp*