BRITISH STANDARD

BS EN
1122:2001

Plastics — Determination of cadmium — Wet decomposition method

The European Standard EN 1122:2001 has the status of a British Standard

ICS 83.080.01

National foreword

This British Standard is the official English language version of EN 1122:2001. It supersedes DD ENV 1122:1995 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/21, Plastics test methods, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

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

Cross-references

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This British Standard, having been prepared under the direction of the Sector Committee for Materials and Chemicals, was published under the authority of the Standards Committee and comes into effect on 15 May 2001

Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 7 and a back cover.

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EN 1122

March 2001

ICS 83.080.01

Supersedes ENV 1122:1995

English version

Plastics — Determination of cadmium — Wet decomposition method

Plastiques — Détermination du cadmium — Méthode par décomposition par voie humide

Kunststoffe — Bestimmung von Cadmium — Nassaufschlussverfahren

This European Standard was approved by CEN on 18 January 2001.

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COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Ref. No. EN 1122:2001 E

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 249, Plastics, the Secretariat of which is held by IBN.

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of an identical text or by endorsement, at the latest by September 2001, and conflicting national standards shall be withdrawn at the latest by September 2001.

This European Standard replaces ENV 1122:1995.

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

1 Scope

This European Standard describes a method for the determination of the total Cadmium (Cd) content in plastics in the range of 10 mg Cd/kg to 3 000 mg Cd/kg. It is not suitable for polyfluorinated plastic materials.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

ISO 3696:1987 — *Water for analytical laboratory use — Specification and test methods.*

ISO 3856-4:1984 — *Paints and varnishes — Determination of "soluble" metal content — Part 4: Determination of cadmium content — Flame atomic absorption spectrometric method and polarographic method.*

3 Principle

Wet decomposition of organic compounds and dissolution of cadmium compounds in a sample. Atomization of a solution in the flame of an atomic absorption spectrophotometer and the measurement of the absorbance at a wavelength of 228,8 nm.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity (ISO 3696:1987).

4.1 Sulfuric acid d = 1,83 g/l 95 % (m/m). H_2SO_4 <分析纯>

4.2 Nitric acid d = 1,40 g/l 65 % (m/m). HNO_3 <分析纯>

4.3 Hydrogen peroxide d = 1,10 g/l 30 % (m/m). H_2O_2 <分析纯>

4.4 Cadmium metal with a purity of 99,9 % or a commercial cadmium standard stock solution (1 g Cd/l) (ISO 3856-4:1984). Cd元素标准品 1000 ppm

4.5 Cadmium standard solutions (0,5 mg Cd/l and 1,0 mg Cd/l) (ISO 3856-4:1984). 0.5 ppm 和 1.0 ppm Cd 标液

5 Apparatus

5.1 Apparatus for wet decomposition (e.g. Figure 1), Kjeldahl flask or any other suitable apparatus for wet decomposition. 湿式分解装置

5.2 Hot plate 加热板

5.3 Fume cupboard, preferably with air washing. 通风柜

5.4 Flame atomic absorption spectrophotometer with background correction, e.g. D2 or Zeemann (ISO 3856-4:1984). 火焰原子吸收分光仪 <AA机>

5.5 Hollow cathode lamp or EDL for cadmium (ISO 3856-4:1984). 空心阴极元素灯

5.6 Analytical balance with range of 1 mg. 分析天平 <量程 0.001 g>

5.7 Membrane filter with a pore size of 0,45 µm.

滤纸 <0.45 µm>

6 Test procedure

6.1 Test sample

Use homogeneous samples of at least 2 g for the analysis. Cut the sample into smaller pieces with a knife or scissors preferably in pieces less than 0,1 g.

6.2 Test portion

Weigh approximately 0,5 g of the test samples (6.1) to the nearest mg into a decomposition apparatus, e.g. a Kjeldahl flask (5.1). Carry out the analysis in duplicate.

6.3 Wet decomposition

The necessary time and reagent consumption for the decomposition depends on the particle size of the sample and mainly on the plastic materials.

6.3.1 Method A

Wet decomposition by a mixture of sulfuric acid, nitric acid and hydrogen peroxide. The following method describes the decomposition in the decomposition apparatus (Figure 1), but any other suitable apparatus can be used (5.1). Carry out the decomposition in a fume cupboard (5.3).

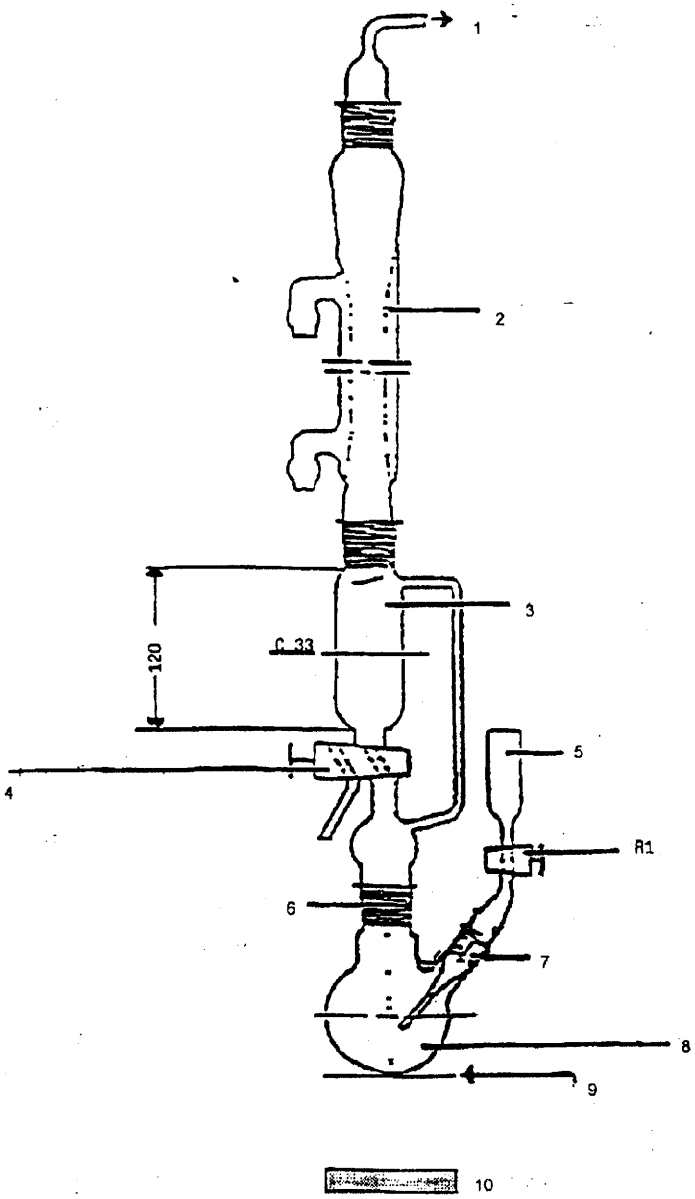
To flask B of the decomposition apparatus (Figure 1), add 10 ml of sulfuric acid (4.1). Connect the flask to the condensate reservoir, run water into the condenser, close taps R 1 and R 2 and add 10 ml of nitric acid (4.2) into the funnel. Using tap R 1, allow 1 ml to 2 ml of the nitric acid to run, then moderately heat until the material changes into a black mass and white fumes of SO_3 are liberated. Now stop heating and allow 1 ml to 2 ml of the nitric acid to fall in drops. Reheat until white fumes appear. Repeat this procedure until a light yellow coloured solution is obtained.

After the addition of the last portion of nitric acid, open tap R 2 and allow the condensate to run into the flask, first dropwise in order to prevent a too violent reaction, then more rapidly. Close tap R 2, reheat until white fumes are produced. Allow to cool for some minutes and add about 5 ml of hydrogen peroxide (4.3) into the flask using the funnel. Complete the decomposition by reheating for about 5 minutes.

Stop heating, open tap R 2 and allow the condensate to drop slowly into the flask. After cooling to room temperature, rinse the apparatus with water and decant quantitatively into a 100 ml one-mark volumetric flask. Dilute to the mark by adding distilled water and mix well.

If insoluble matter exists at this stage which might disturb the atomic absorption method, then remove it by filtration, using a dry membrane filter (5.7).

Prepare a reagent blank solution in the same way without using a test sample.



Key

1 towards hood
2 condenser to coil
3 condensate reservoir
4 R2: tap with PTFE fitting
5 25 ml funnel
6 B29 fitting
7 B14 fitting
8 250 ml flask
9 tripod
10 hot plate

Figure 1 — Example of apparatus for wet decomposition

6.3.2 Method B

Wet decomposition by a mixture of sulfuric acid and hydrogen peroxide. The following method describes the decomposition in a Kjeldahl flask but any other suitable apparatus can be used (5.1). Carry out the decomposition in a fume cupboard (5.3).

Place the flask and its contents on the hot plate (5.2), add 10 ml of the sulfuric acid (4.1) and heat at a higher temperature to decompose and carbonize the organic substances. When white fumes are evolved continue heating for about 15 minutes.

Take the flask from the hot plate and allow to cool for about 10 minutes. Add slowly, from the funnel, four 5 ml portions of hydrogen peroxide solution (4.3), allowing the reaction to subside after each addition.

> Note Because of the danger of splattering, any reaction flask should be kept covered between additions of hydrogen peroxide solution.

Heat again for about 10 minutes and allow to cool for about 5 minutes. Add further 5 ml portions of the hydrogen peroxide solution and heat again. Stop this procedure only when no more organic matter remains. Allow to cool to room temperature and dilute cautiously with water. Rinse the flask with water and decant quantitatively into a 100 ml one-mark volumetric flask. Dilute to the mark by adding distilled water and mix well. If insoluble matter exists at this stage which might disturb the atomic absorption method, then remove it by filtration, using a dry membrane filter (5.7).

Prepare a reagent blank solution in the same way without using a test sample.

6.4 Determination

Determine the cadmium concentration of the test solutions and the reagent blank solution obtained according to 6.3, by the method described in clause 3 of ISO 3856-4:1984.

> Note The sulfuric acid in the test and reagent blank solution might influence the results of the flame atomic absorption method; therefore use a background correction (5.4).

For the determination of the cadmium concentration of the test solutions and the blank solutions other suitable techniques can be used, e.g. inductively-coupled plasma (ICP) or an isotope specific method. The used technique shall be noted in the test report.

7 Expression of results

The total cadmium content of the sample is given by the following formula in mg/kg:

$$100\,\frac{f\,(C-R)}{M}$$

where:

C is the cadmium concentration in milligrams per litre of the test solution obtained by 6.4;

B is the cadmium concentration in milligrams per litre of the reagent blank solution obtained by 6.4;

M is the mass, in grams of the test portion;

f is the dilution factor of the test solution and the reagent blank solution used by clause 4.

If the two results don't differ more than 20 % based on the average (results between 10 mg Cd/kg and 50 mg Cd/kg) or 10 % (results between 50 mg Cd/kg and 3 000 mg Cd/kg), then take the mean. Otherwise repeat the analysis.

8 Precision Data of the Test Method

Repeatability, *r*:

The relative standard deviation between results, found on identical test material, by one operator, using the same apparatus, using the method specified in this standard.

Reproducibility, *R*:

The relative standard deviation between independent results, found by two operators working in different laboratories, on identical test material, using the method specified in this standard.

Range of level	r^*	R^*
10 mg Cd/kg to 50 mg Cd/kg	20%	25%
50 mg Cd/kg to 3 000 mg Cd/kg	10%	25%
$r^* = \frac{r}{m} 100\%$	$R^* = \frac{R}{m} 100\%$	

where:

m is the average of all values for each level.

The precision data were determined from an experiment conducted in 1992 involving 7 laboratories, 6 samples and 5 different plastics (PE, PP, PS, PVC and PET). The precision data only refer to the 5 tested plastics. There exists a lot of experience that these data also can be used for the determination of the cadmium content of other sorts of plastics materials with the exemption of the polyfluorated plastics.

9 Test Report

The test reports shall contain at least the following information:

a) type and identification of the products tested;

b) a reference to this European Standard and a reference to the used method (A or B);

c) the results of the test expressed as mg Cadmium/kg plastic material (mean values and single measurement results);

d) any deviation, by agreement or otherwise, from the test procedure specified here;

e) date of the test and name of the operator.

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