SLOVENSKI STANDARD
SIST ENV 1122:1999

01-maj-1999

Določevanje kadmija v polimernih materialih z metodo mokrega razklopa

Determination of cadmium in plastics with the method of the wet decomposition

Bestimmung von Cadmium in Kunststoffen nach dem Naßaufschluß

Détermination du cadmium dans les plastiques d'apres la méthode de décomposition par voie humide

Ta slovenski standard je istoveten z: **ENV 1122:1995**

ICS:

83.080.01	Polimerni materiali na splošno	Plastics in general

SIST ENV 1122:1999 **en**

EUROPEAN PRESTANDARD **ENV 1122**

PRÉNORME EUROPÉENNE

EUROPÄISCHE VORNORM January 1995

ICS 83.080

Descriptors: Plastics, chemical analysis, determination of content, cadmium, atomic absorption, spectrophotometry, flame photometric analysis

English version

Determination of cadmium in plastics with the method of the wet decomposition

Détermination du cadmium dans les plastiques d'après la méthode de décomposition par voie humide

Bestimmung von Cadmium in Kunststoffen nach dem Naßaufschluß

This European Prestandard (ENV) was approved by CEN on 1994-05-03 as a prospective standard for provisional application. The period of validity of this ENV is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the ENV can be converted into an European Standard (EN).

CEN members are required to announce the existance of this ENV in the same way as for an EN and to make the ENV available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the ENV) until the final decision about the possible conversion of the ENV into an EN is reached.

CEN members are the national standards bodies of Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

CEN

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

Central Secretariat: rue de Stassart,36 B-1050 Brussels

Foreword

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

According to the CEN/CENELEC Internal Regulations, the following countries are bound to announce this European Prestandard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, 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 polyfluorated 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.

ISO 648:1977	Laboratory glassware - One-mark pipettes.
ISO 1042:1983	Laboratory glassware - One-mark volumetric flask.
ISO 3856-4:1984	Determination of cadmium content - Flame atomic absorption spectroscopic method.
ISO 3696:1987	Water for analytical laboratory use-specification and test methods.

3 Principle

Wet decomposition of organic compounds and disolution 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 destilled water or water of equivalent purity. (ISO 3696:1987)

4.1 Sulphuric acid d = 1.83 g/l 95 % (m/m)

4.2 Nitric acid. d = 1.40 g/l 65 % (m/m)

4.3 Hydrogen peroxide d = 1.10 g/l 30 % (m/m)

4.4 Cadmium metal with a purity of 99.9 % or a commercial cadmium standard stock solution (1 g Cd/l) (ISO 3856/4)

4.5 Cadmium standard solutions (0.5 and 1.0 mg Cd/l) (ISO 3856/4)

5 Apparatus

5.1 Apparatus for wet decomposition e.g.(figure 1), Kjeldahl flask, beaker or any other suitable apparatus for wet decomposition

5.2 Hot plate

5.3 Fume cupboard, preferable with air washing

5.4 Flame atomic absorption spectrophotometer with background correction e.g. D2 or Zeemann (ISO 3856-4)

5.5 Hollow cathode lamp or EDL for cadmium (ISO 3856-4)

5.6 Analytical balance in range of 1 mg

5.7 Membrane filter with a pore size of 0.45 µm

6 Test procedure

6.1 Test Sample

Use a homogeneous sample of at least 2 g for the analysis. Cut the sample in smaller pieces with a knife or scissor preferable 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 1 mg into a decomposition apparatus, a Kjeldahl flask or a 400 ml beaker (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 plastics materials.

6.3.1 Method A:

Wet decomposition by a mixture of sulphuric 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 sulphuric acid (4.1). Connect the flask to the condensate reservoir, run water into the condenser, close the tap of R 1 and R 2 and add 10 ml of nitric acid ((4.2) into the funnel. Using tap of R 1, allow 1 - 2 ml of the nitric acid to run, then moderately heat until the material changes into a black mass and white fumes of SO3 are liberated. Now stop heating and allow 1 - 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 ml of nitric acid, open the 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 quantitative 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 partial filtration by using a dry membrane filter (5.7).

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

6.3.2 Method B:

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

Place the beaker and its contents on the hot plate (5.2), add 10 ml of the sulphuric acid (4.1), cover the beaker with a watchglas 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 beaker from the hot plate and allow to cool for about 10 minutes. Add slowly, from a 5 ml pipette, four 5 ml portions of hydrogen peroxide solution (4.1.3), allowing the reaction to subside after each addition.

Note: Because of the danger from splattering, the beaker should be kept covered between additions of the 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 if no organic matter remains. Allow to cool to room temperature and dilute with water taking precaution. Rinse the beaker and the watchglass with water and decant quantitative 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 partial filtration by 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 clause 6.3, by the method described in clause 3 of ISO 3856/4 (3).

Note: The sulphuric acid in the test and reagent blank solution might influence the results by 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 isotope specific methode. The used technique must be noted in the test report.

7 Expression of the Results

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

$$100 \times \frac{f \times (C - B)}{M}$$

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

B is the cadmium concentration in milligrams per litre of the reagent blank solution obtained by clause 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 do not differ more than 20 % based on the average (results between 10 and 50 mg Cd/kg) or 10 % (results between 50 and 3000 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 single 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*	$r^* = \frac{r}{m} \cdot 100\ \%$
10 - 50 mg Cd/kg	20 %	25 %	$R^* = \frac{R}{m} \cdot 100\ \%$
50 - 3000 mg Cd/kg	10 %	25 %	
m is 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 all contain at least the following information:

a) type and identification of the products tested

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

c) the results of the tests 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

e) date of the test and name of the operator