EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 120:1992

July 1992

UDC 674.815:620.1:543.242.3:547.281.1

Supersedes EN 120:1984

Descriptors: Wooden boards, particle boards, fibre boards, plywood, chemical analysis, determination of content, extraction methods, perforators

English version

Wood based panels - Determination of formaldehyde content - Extraction method called the perforator method

Panneaux à base de bois - Détermination de la teneur en formaldéhyde - Méthode d'extraction dite méthode au perforateur

Holzwerkstoffe - Bestimmung des Formaldehydgehalts - Extraktionsverfahren genannt Perforatormethode

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Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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CEN

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

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Contents list

Foreword

This European Standard was prepared by Working Group 5 "Formaldehyde" (Secretariat : Germany) of Technical Committee CEN/TC 112 "Wood-based panels" (Secretariat : Germany).

This European Standard is based on the October 1984 edition. To that times the work was the responsibility of CEN/TC 91 "Particleboards - Formaldehyde" and it was only intented for determining the formaldehyde content of particleboards. The basis was an extraction method developed by the European Federation of Associations of Particleboard Manufacturers, FESYP, and known as the "Perforator Method". This method was intented for testing particularly particleboards at the manufacturing stage, and was later adapted for testing all wood-based panels.

For this European standard a mandate "Timber Structures" has been given to CEN and EFTA in the framework of the Directive on the approximation of laws, regulations and administrative provisions of the Member States relating to construction products (89/106/EEC).

National Standards identical to this European Standard shall be published at the latest by 93-01-31 and conflicting national standards shall be withdrawn at the latest by 93-01-31.

In accordance with the Common CEN/CENELEC Rules the following countries are bound to implement this European Standard : Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Introduction

The "perforator value" as determined by the method laid down in this standard is considered to be the "formaldehyde content" of the tested board.

The test results shall be considered in relation to the specific board conditions at the time of testing.

The emission of formaldehyde from wood-based panels (e.g. particleboard, plywood, fibreboard) is a complex process.

For a given board, the test result depends upon the age, ageing conditions, moisture content etc., at the time of testing.

In addition, the correlation between the perforator value and the formaldehyde emission of the board depends on the type of board.

1 Scope

This European Standard specifies an extraction method, known as the "Perforator Method". It is used for the determination of the formaldehyde content of unlaminated and uncoated wood-based panels.

2 Normative references

This European Standard incorporates by dated or undated reference, provision from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated reference, 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.

EN 322 Wood-based panels - Determination of moisture content

3 Principle

The formaldehyde is extracted from test pieces by means of boiling toluene and then transferred into distilled or demineralized water. The formaldehyde content of this aqueous solution is determined photometrically by the acetylacetone method.

4 Reagents

For the analysis, only reagents of analytical quality and distilled or demineralized water shall be used.

4.1 Toluene, which is free from water and from impurities which may interfere with the test.

4.2 Acetylacetone of analytical grade

4.3 Ammonium acetate of analytical grade

NOTE: Commercially prepared solutions may be used, provided it can be shown that they give an equivalent result.

5 Apparatus

5.1 Precision balance, scale interval 0,001 g.

5.2 Well ventilated drying oven, capable of maintaining a temperature of (103 ± 2) °C.

5.3 Spectrophotometer

5.4 Extraction apparatus

The apparatus consists of:

- Spiral Condenser, total length approximately 400 mm, cone 45/40, socket 29/32 (item reference 2)
- Conical adaptor, socket 45/40, cone 71/51 (item reference 3)
- Filter insert, porosity P 160 (100 µm to 160 µm), bowl and filter diameter 60 mm (item reference 4)
- Perforator attachment 1000 ml with stopcock (4 mm bore), socket 71/51, cone 29/32 (item reference 5)
- Conical adaptor, socket 29/32, cone 45/40 (item reference 8)
- 1000 ml round-bottomed flask, socket 45/40 (item reference 9)

- (Double) bulbed tube, cone 29/32 (length approximately 380 mm), external diameter approximately 10 mm, bulb diameter approximately 50 mm, distance between bulb and bottom end of tube approximately 200 mm (distance between the bulbs approximately 50 mm) (item reference 6)

- Absorption bulb (e. g. conical flask 250 ml) (item reference 7).

NOTE: The item references are given in figure 1.

5.5 Laboratory equipment

- Volumetric flask 2000 ml, calibrated at 20 °C
- Conical flask, 250 ml
- Precision burette, 50 ml, calibrated at 20 °C
- Watch glass with diameter of about 120 mm
- Two volumetric flasks, 1000 ml, calibrated at 20 °C
- Six volumetric flasks, 100 ml, calibrated at 20 °C
- Bulb pipette, 100 ml, calibrated at 20 °C
- Bulb pipette, 25 ml, calibrated at 20 °C
- Three bulb pipettes, 10 ml, calibrated at 20 °C
- Stoppered flasks each 50 ml
- Two volumetric cylinders, 250 ml
- Water bath
- Desiccator

6 Test pieces

6.1 Sampling

6.1.1 The test pieces are to be taken, evenly distributed, over the width of the (cooled) board but excluding a 500 mm wide strip at either end of the board.

6.1.2 Take 12 test pieces of 25 mm x 25 mm x thickness of the board for the determination of the moisture content and a sufficient number of test pieces of the same dimensions to obtain approximately 500 g of the board for the extraction by perforator.

6.2 For production control

If this method is used for production control, the board selected for sampling is immediately cut up after cooling. The test pieces taken from the board are to be stored hermetically sealed at room temperature.

The formaldehyde determination should be carried out not more than 72 hours after sampling.

6.3 For other purposes

If this method is used for other purposes, e. g. for boards already installed, the method chosen for sampling, preparation of test pieces and conditioning, all which influence the final result, shall be agreed between the parties and indicated in the test report.

Unless otherwise agreed, the test pieces shall be conditioned to a constant mass at a temperature of (23 ± 1) °C and a relative humidity of (45 ± 5) %.

Constant mass is considered to have been reached when the results of two successive weighings, carried out at intervals of not less than 24 hours, do not differ by more than 0,1 % of the mass of the test pieces.

Contamination of test pieces from other sources of formaldehyde during the conditioning shall be avoided.

> NOTE: The climate for conditioning of test pieces is the test climate recommended by the Concerted Action Committee "Indoor air quality and its impact on man" (COST 613) of the European Community given in the report "Formaldehyde emission from wood-based materials: Guideline for the determination of steady state concentrations in test chambers".

7 Procedure

7.1 Number of extractions

The extractions are to be carried out in duplicate.

> NOTE: For internal routine control, a single extraction may be sufficient.

The individual values of a duplicate extraction may not deviate by more than 20 % related to the greater of the two single values; otherwise a third extraction is to be carried out.

7.2 Determination of moisture content

Determine the moisture content in accordance with EN 322.

Determine the moisture content in duplicate on a sample of at least 4 test pieces (25 mm x 25 mm).

Weigh the sample (5.1) to an accuracy of 0,1 % onto the watch glass (5.5) and dry in the oven (5.2) at a temperature of (103 ± 2) °C to constant mass (about 12 h).

Constant mass is considered to have been reached when the results of two successive weighings, carried out at intervals of not less than 6 hours, do not differ by more than 0,1 % of the mass of the test pieces.

After the removal from the drying oven, the test pieces shall be allowed to cool in a desiccator before weighing.

7.3 Extraction in the perforator

> NOTE: Before the apparatus is used the sidearm of the perforator attachment shall be provided with thermal insulation, in order to achieve circulation of the toluene.

About 110 g of test pieces are weighed to an accuracy of 0,1 g and put into the round bottom flask (5.4). 600 ml of toluene (4.1) are added. Subsequently, the round bottom flask is connected to the perforator. About 1000 ml of distilled water is poured into the perforator attachment, leaving a space of 20 mm to 30 mm between the surface of the water and the siphon outlet. The condenser and the gas absorption equipment are then connected. The absorption bulb of the gas absorption equipment (5.4) is filled with about 100 ml of distilled water and connected to the apparatus.

When the apparatus has been assembled, the cooling water and heating are turned on.

Toluene shall flow back regularly throughout the whole period of perforation, with a reflux rate of 70 drops to 90 drops per minute.

Care shall be taken that no water flows back from the absorption bulb (item reference 7) into other parts of the equipment during and after the extraction process.

The extraction is carried on for two hours, starting at the moment that the first bubbles pass through the filter insert. The heating shall be such that the onset of bubbling occurs between 20 minutes and 30 minutes after turning on the heating device.

After these two hours the heating is switched off and the gas absorption bulb is removed.

The water contained in the perforator is, after cooling to room temperature, transferred into the volumetric flask by means of the stop cock. The perforator is rinsed twice, each time with 200 ml of distilled water. The rinsing water is poured into the volumetric flask and the toluene discarded. The water contained in the absorption bulb of the gas absorption equipment is poured into the flask. The volume of the water contained in the flask is then made up to 2000 ml with distilled water.

7.4 Blank test

Repeat the test without test pieces using new toluene from the same batch as used for the extraction.

NOTE: The quality of toluene is checked by this test. If the blank value of formaldehyde is higher than 1 mg/600 ml of toluene the toluene shall be rejected.

7.5 Determination of formaldehyde in the extract

The formaldehyde content of the aqueous extract is determined photometrically, using the acetylacetone method.

7.5.1 Principle

The determination is based on the Handtzsch reaction in which aqueous formaldehyde reacts with ammonium ions and acetylacetone to yield diacetyldihydrolutidine (DDL). DDL has an absorbance maximum at 412 nm. The reaction is highly specific to formaldehyde.

NOTE: Other suitable photometric procedures may also be used.

7.5.2 Reagents

7.5.2.1 Acetylacetone solution

4 ml acetylacetone is added to a 1000 ml volumetric flask, dissolved in distilled water and made up to the mark with distilled water.

7.5.2.2 Ammonium acetate solution

200 g ammonium acetate is dissolved in distilled water in a 1000 ml volumetric flask and made up to the mark.

7.5.3 Procedure

10 ml is taken from the aqueous solution (7.3) with a pipette and added to 10 ml acetylacetone solution (7.5.2.1) and 10 ml ammonium acetate solution (7.5.2.2) in a 50 ml flask. The flask is stoppered, shaken and warmed for 15 minutes in a water bath of 40 °C. The now greenish-yellow solution is cooled to room temperature protected against the influence of light (about 1 hour). The absorbance of this solution is determined at a wavelength of 412 nm against distilled water using a spectrophotometer (5.3). A blank value is determined parallel with destilled water and taken into consideration in the determination of the perforator value (8.2).

7.5.4 Calibration curve

The calibration curve (see figure 4) is produced from a standard formaldehyde solution, the concentration of which has been determined by iodometric titration. The calibration curve shall be checked at least once a week.

7.5.4.1 Formaldehyde standard solution

Reagents:

- Standard iodine solution $c(I_2) = 0{,}05$ mol/l
- Standard sodium thiosulphate solution $c(Na_2S_2O_3) = 0{,}1$ mol/l
- Standard sodium hydroxide solution $c(NaOH) = 1$ mol/l
- Standard sulphuric acid solution $c(H_2SO_4) = 1$ mol/l
- starch solution = 1 % m/m

The solutions shall be standardized before use.

About 2,5 g formaldehyde solution (concentration 35 % to 40 %) are diluted in a 1000 ml volumetric flask with distilled water and made up to the mark. The exact formaldehyde concentration is determined as follows:

20 ml of the formaldehyde standard solution are mixed with 25 ml iodine solution and 10 ml sodium hydroxide solution. After 15 minutes standing, protected from light, 15 ml of sulphuric acid solution are added to the mixture. The surplus of iodine is back-titrated with the thiosulphate solution. At the end of the titration some drops of starch solution are added as an indicator. A blank test with 20 ml distilled water is carried out in parallel.

The formaldehyde content is calculated by the following formula:

$$c(HCHO) = (V_0 - V) \times 15\ [c(Na_2S_2O_3)] \times 1000/20$$

where:

$c(HCHO)$ is the formaldehyde concentration in mg/l

V_0 is the volume of thiosulphate solution for the blank test in millilitres

V is the volume of thiosulphate titration solution for the test in millilitres

$c(Na_2S_2O_3)$ is the concentration of the thiosulphate solution in mol/l

NOTE: 1 ml 0,1 mol/l thiosulphate solution corresponds to 1 ml 0,05 mol/l iodine solution and 1,5 mg formaldehyde.

7.5.4.2 Formaldehyde calibration solution

Using the concentration determined in 7.5.4.1, calculate the volume which will contain 15 mg formaldehyde. Transfer this volume, using a microburette, to a 1000 ml volumetric flask and make up to the mark with distilled water. 1 ml of this calibration solution contains 15 µg formaldehyde.

7.5.4.3 Determination of the standard curve

Pipette either zero, 5, 10, 20, 50 or 100 ml of formaldehyde calibration solution (7.5.4.2) into a 100 ml volumetric flask each and make up to the mark with distilled water. 10 ml of each dilution is analysed photometrically by the same procedure as described above (7.5.3). The absorbance values are plotted against the formaldehyde concentrations (between 0 and 0,015 mg/ml) on the millimeter graph paper. The slope is either determined graphically, or calculated.

8 Expression of results

8.1 Moisture content

The moisture content H (in mass % m/m) of the wood based panel is given by the formula:

$$H = \frac{m_1 - m_0}{m_0} \cdot 100$$

where:

m_1 is the mass of the test pieces before drying, in grams

m_0 is the mass of the test pieces after drying, in grams

8.2 Perforator value

The formaldehyde content, known as the "Perforator value", is expressed in mg formaldehyde per 100 g of oven-dry board, and is calculated by the following formula:

$$\text{Perforator value} = \frac{(A_S - A_B) \cdot f \cdot (100 + H) \cdot V}{m_H} \text{ mg/100 g ovendry board}$$

where:

A_S is the absorbance of the analyzed extraction solution

A_B is the absorbance of an analysis with distilled or demineralized water

f is the slope of the standard curve (in mg/ml)

H is the moisture content of the wood-based panel in percent

m_H is the mass of test pieces, in grams

V is the volume of the volumetric flask (2000 ml)

The "Perforator value" of a wood based panel is considered to be the mean value of the results of the two or three extractions. Results shall be expressed to one decimal place.

9 Test report

The test report shall contain if known the following informations with reference to this standard:

- Origin of the boards
- Place, location [1] and state [2] of the board at the time of sampling (in particular the moisture content)
- Type of the board
- Thickness of the board (mm)
- Density of the board (kg/m^3)
- Date of manufacture of the board
- Date of sampling
- Information referring to the board regarding finishing, sanding etc.
- Date of formaldehyde determination
- Moisture content (%), at the time of testing (according to 7.2)
- Perforator value (mg formaldehyde/100 g dry board)
(Individual values of each extraction and mean value)
- Description of further details [3]

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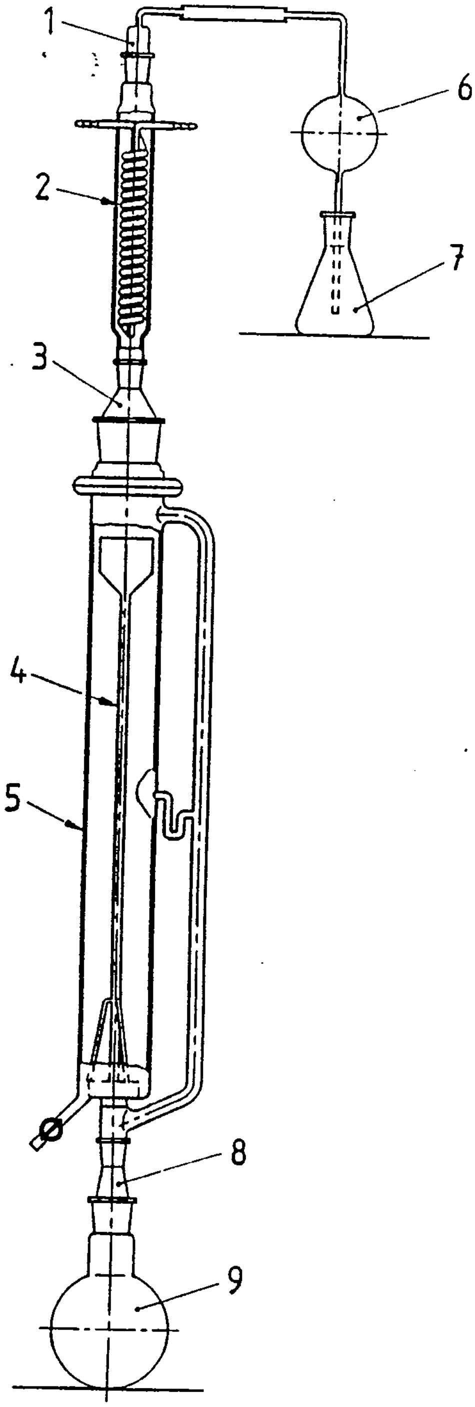
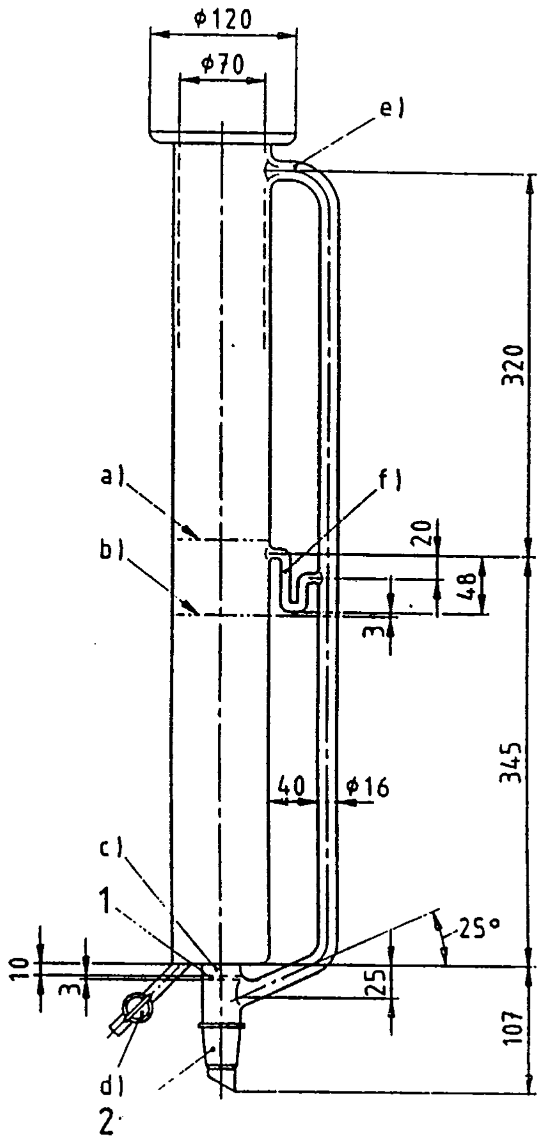
1) For example: factory etc.; in the case of installed boards: ceiling, floor, wall etc.
2) For example: moisture content, surface coating, finishing
3) Report all the information about the operations which are not in accordance with the standard (sampling of test pieces, conditioning etc.)



1 conical adaptor 29/32
2 Dimroth cooler
3 conical adaptor 45/40 to 71/51
4 filter insert
5 perforator attachment
6 (double) bulbed tube
7 conical flask 250 ml
8 conical adaptor 29/32 to 45/40
9 round bottom flask 1000 ml with socket 45/40

Figure 1: Extraction apparatus

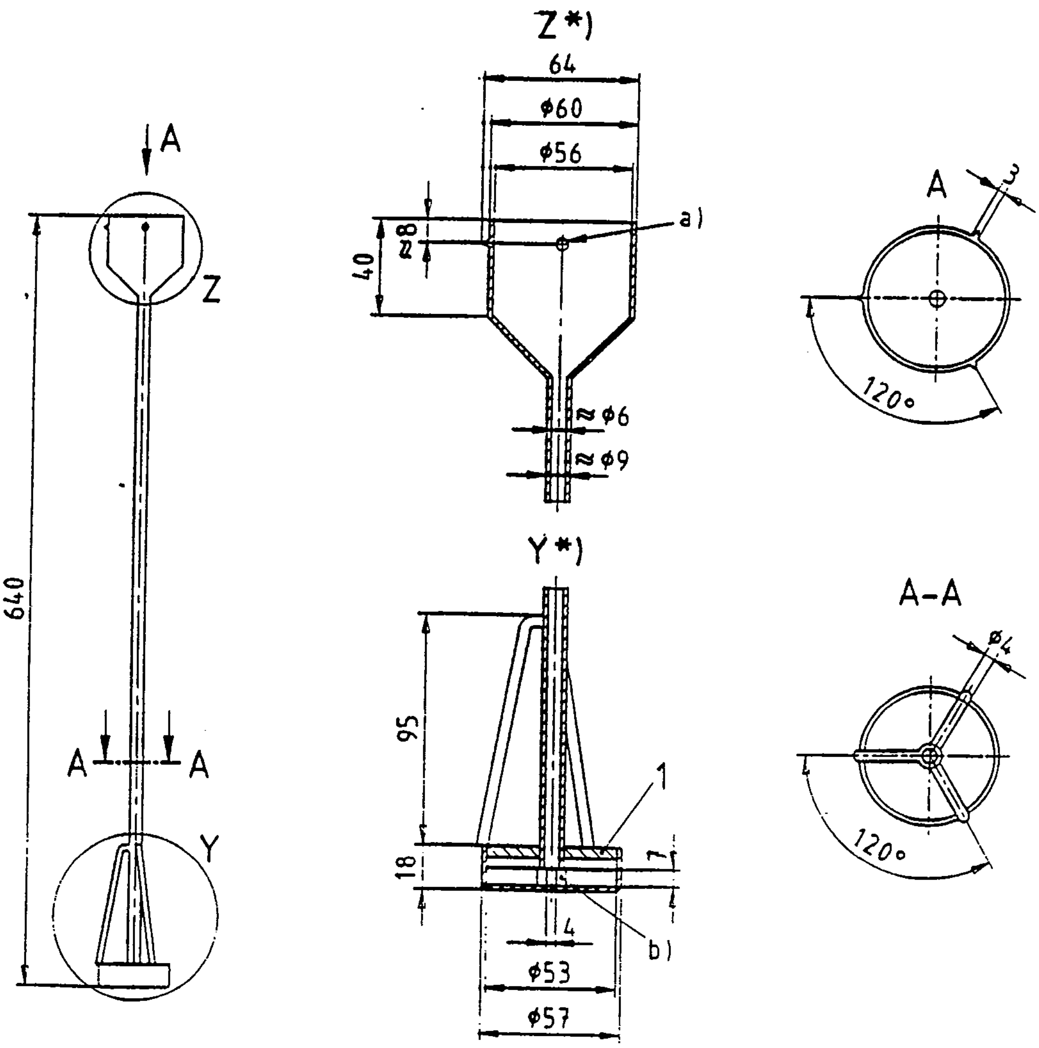
dimensions in mm



1 blanking plate	c) 4 dia. hole each side
2 std. 29/32	d) stopcock 4 dia. bore
a) toluene level	e) side arm
b) maximum water level	f) 8 dia. siphon tube

Figure 2: Perforator attachment

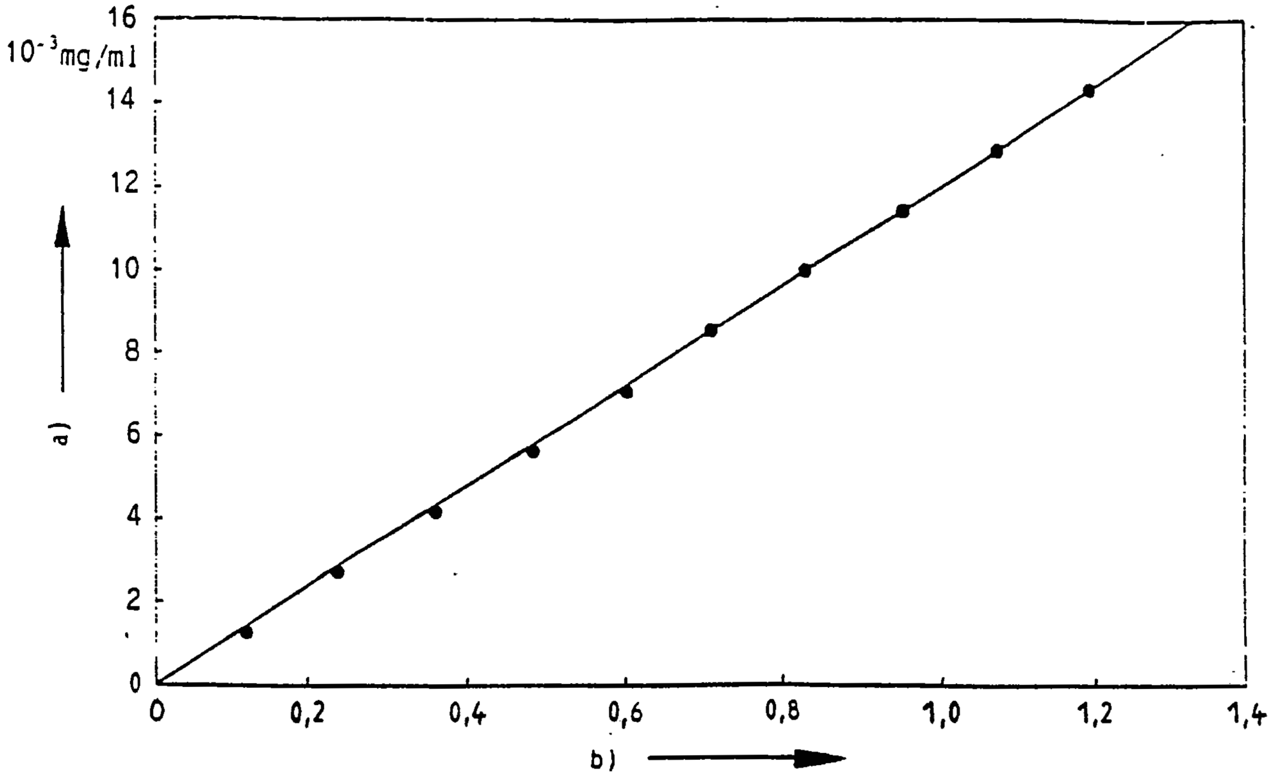
dimensions in mm



1 sintered glass filter (porosity P 160)
a) holes 5 dia. on each side

b) two 4 dia. spacing rods
*) detail (sectional view)

Figure 3: Filter insert



$c = f \cdot A_S$

a) Concentration of the diluted calibration solution (c)

b) Absorbance ($A_S - A_B$)

Figure 4: Example of a calibration curve