INTERNATIONAL STANDARD

ISO 12460-5

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Wood-based panels — Determination of formaldehyde release —

Part 5:
Extraction method (called the perforator method)

Panneaux à base de bois — Détermination du dégagement de formaldéhyde —

Partie 5: Méthode d'extraction (dite méthode au perforateur)

Reference number
ISO 12460-5:2011(E)

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Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 12460-5 was prepared by Technical Committee ISO/TC 89, *Wood-based panels*.

ISO 12460 consists of the following parts, under the general title *Wood-based panels — Determination of formaldehyde release*:

— *Part 1: Formaldehyde emission by the 1-cubic-metre chamber method*

— *Part 3: Gas analysis method*

— *Part 4: Desiccator method*

— *Part 5: Extraction method (called the perforator method)*

Introduction

ISO 12460-1 specifies the 1-cubic-metre chamber as the reference method for the determination of formaldehyde release. Derived test methods specified in ISO 12460-3, ISO 12460-4 and ISO 12460-5 are intended for factory production control.

The “perforator value”, as determined by the method laid down in this part of ISO 12460, is considered to be the “formaldehyde content” of the tested board.

The test results are 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.

Wood-based panels — Determination of formaldehyde release —

Part 5: Extraction method (called the perforator method)

1 Scope

This part of ISO 12460 specifies an extraction method, known as the "perforator method", for the determination of the formaldehyde content of unlaminated and uncoated wood-based panels.

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.

ISO 16979, *Wood-based panels — Determination of moisture content*

ISO 16999, *Wood-based panels — Sampling and cutting of test pieces*

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.

Commercially prepared solutions may be used, provided they can be shown to give results equivalent to those obtained with solutions prepared in the laboratory.

5 Apparatus

5.1 Precision balance, with 0,001 g scale intervals.

5.2 Oven, well-ventilated and capable of maintaining a temperature of (103 ± 2) °C.

5.3 Spectrophotometer, with cells of optical path length 50 mm, capable of measuring absorbance at 412 nm.

5.4 Extraction apparatus, consisting of the following:

- spiral condenser, with a total length of approximately 400 mm, cone 45/40, socket 29/32 (see Figure 1, item 2);
- conical adaptor, socket 45/40, cone 71/51 (see Figure 1, item 3);
- filter insert, porosity P 160 (100 pm to 160 pm), with bowl and filter diameters of 60 mm (see Figure 1, item 4);
- perforator attachment, 1 000 ml, with stopcock (4 mm bore), socket 71/51, cone 29/32 (see Figure 1, item 5);
- conical adaptor, socket 29/32, cone 45/40 (see Figure 1, item 8);
- 1 000 ml round bottom flask, socket 45/40 (see Figure 1, item 9);
- (double) bulbed tube, cone 29/32 (length approximately 380 mm), with external diameter of 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) (see Figure 1, item 6);
- absorption bulb (e.g. conical flask 250 ml) (see Figure 1, item 7).

5.5 Laboratory equipment, consisting of the following:

- volumetric flask, 2 000 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, 1 000 ml, calibrated at 20 °C;
- six volumetric flasks, 100 ml, calibrated at 20 °C;
- bulb pipette or automatic/digital pipette, 100 ml, calibrated at 20 °C;
- bulb pipette or automatic/digital pipette, 25 ml, calibrated at 20 °C;
- three bulb pipettes or automatic/digital pipettes, 10 ml, calibrated at 20 °C;
- stoppered flasks, each 50 ml;
- two volumetric cylinders, 250 ml;
- water bath;
- desiccator.

6 Test pieces

Sampling and cutting shall be carried out in accordance with ISO 16999.

6.1 Sampling

6.1.1 The test pieces shall 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 × 25 mm × 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 shall be immediately cut up after cooling. The test pieces taken from the board shall be stored, hermetically sealed, at room temperature.

The formaldehyde determination should be carried out not more than 72 h 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 the conditioning — all of 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 constant mass at a temperature of (23 ± 1) °C and a relative humidity of (50 ± 5) %.

Constant mass is considered to have been reached when the results of two successive weightings, carried out at intervals of not less than 24 h, 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 conditioning shall be avoided.

7 Procedure

7.1 Number of extractions

The extractions shall be carried out in duplicate.

NOTE For internal routine control, a single extraction could 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 shall be carried out.

7.2 Determination of moisture content

Determine the moisture content in accordance with ISO 16979.

Determine the moisture content in duplicate on a sample of at least four test pieces (25 mm × 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 weightings, carried out at intervals of not less than 6 h, do not differ by more than 0,1 % of the mass of the test pieces.

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

7.3 Extraction in the perforator

Before the apparatus is used, the sidearm of the perforator attachment (see Figure 2) shall be provided with thermal insulation, in order to achieve circulation of the toluene.

Weigh about 110 g of test pieces to 0,01 g and place in the round bottom flask (5.4). Add 600 ml of toluene (4.1). Pour about 1 000 ml of distilled water into the perforator attachment, leaving a space of 20 mm to 30 mm between the surface of the water and the siphon outlet. Subsequently, connect the round bottom flask to the perforator. Then connect the condenser and the gas absorption equipment. Fill the absorption bulb of the gas absorption equipment (5.4) with about 100 ml of distilled water and connect to the apparatus.

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

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 (see Figure 1, item 7) into other parts of the equipment either during or after the extraction process.

The extraction shall continue for (120 ± 5) min, starting at the moment that the first bubbles pass through the filter insert (see Figure 3). The heating shall be such that the onset of bubbling occurs between 20 min and 30 min after turning on the heating device.

After this (120 ± 5) min period, switch off the heating and remove the gas absorption bulb.

After cooling to room temperature, transfer the water contained in the perforator into the volumetric flask by means of the stopcock. Rinse the perforator twice, each time with 200 ml of distilled water. Pour the rinsing water into the volumetric flask and discard the toluene. Pour the water contained in the absorption bulb of the gas absorption equipment into the flask. Then make up the volume of the water contained in the flask to 2 000 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.

The quality of toluene is checked by this test. If the blank value of formaldehyde is higher than 0,2 mg/600 ml of toluene, the toluene shall be rejected.

7.5 Determination of formaldehyde in the extract

7.5.1 General

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

7.5.2 Principle

The determination is based on the Hantzsch 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.

Other suitable photometric procedures may also be used.

7.5.3 Reagents

7.5.3.1 Acetylacetone solution

Transfer 4 ml acetylacetone to a 1 000 ml volumetric flask (5.5), dissolve in distilled water and fill to volume with distilled water.

7.5.3.2 Ammonium acetate solution

Transfer 200 g ammonium acetate to a 1 000 ml volumetric flask (5.5), dissolve in distilled water and fill to volume with distilled water.

7.5.4 Procedure

Take 10 ml from the aqueous solution (see 7.3) with a pipette and add to 10 ml acetylacetone solution (see 7.5.3.1) and 10 ml ammonium acetate solution (see 7.5.3.2) in a 50 ml flask. Stopper, shake and warm the flask for 10 min in a water bath of 60 °C. Cool the greenish-yellow solution to room temperature, protecting against the influence of light (for about 1 h). Determine the absorbance of this solution at a wavelength of 412 nm against distilled water using a spectrophotometer (5.3). A blank value is determined in parallel with distilled water and taken into consideration in the determination of the perforator value (see 8.2).

When smaller volumes of solutions with the same ratio of reagents are used, e.g. applying automatic/digital pipettes (5.5), it should be ensured that enough analytical solution is available for the spectophotometric determination. Furthermore, it should be taken into consideration that measurement errors could have increasing influence on the result.

7.5.5 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.

7.5.5.1 Formaldehyde standard solution

Reagents:

- standard iodine solution, $c_{(I2)}$ = 0,05 mol/l;
- standard sodium thiosulfate 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 %.

The solutions shall be standardized before use.

Dilute about 2,5 g formaldehyde solution (35 % to 40 % concentration) in a 1 000 ml volumetric flask with distilled water and make up to the mark. Determine the exact formaldehyde concentration as follows.

Mix 20 ml of the formaldehyde standard solution with 25 ml iodine solution and 10 ml sodium hydroxide solution. After 15 min standing, protected from light, add 15 ml of sulfuric acid solution to the mixture. The excess iodine is back-titrated with the thiosulfate solution. At the end of the titration, add some drops of starch solution as an indicator. Carry out a blank test with 20 ml distilled water in parallel.

The formaldehyde content is calculated using Equation (1):

$$\rho_{(\mathrm{HCHO})} = (V_0 - V) \times 15 \left[c_{(\mathrm{Na_2S_2O_3})} \right] \times 1\,000/20 \qquad (1)$$

where

$\rho_{(\mathrm{HCHO})}$ is the formaldehyde concentration, in milligrams per litre (mg/l);

V_0 is the volume of thiosulfate solution for the blank test, in millilitres;

V is the volume of thiosulfate titration solution for the test, in millilitres;

$c_{(\mathrm{Na_2S_2O_3})}$ is the concentration of the thiosulfate solution, in mol/l.

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

7.5.5.2 Formaldehyde calibration solution

Using the concentration determined in 7.5.5.1, calculate the volume which will contain 15 mg formaldehyde. Transfer this volume, using a microburette, to a 1 000 ml volumetric flask and make up to the mark with distilled water; 1 ml of this calibration solution contains 15 µg formaldehyde.

7.5.5.3 Determination of the standard curve

Pipette (5.5) either 0, 5, 10, 20, 50 or 100 ml of formaldehyde calibration solution (see 7.5.5.2) into a 100 ml volumetric flask and make up to the mark with distilled water; 10 ml of each dilution is analysed photometrically by the same procedure as described above (see 7.5.4). Plot the absorbance values against the formaldehyde concentrations (between 0 and 0,015 mg/ml) on graph paper. Determine the slope either graphically or by calculation.

8 Expression of results

8.1 Moisture content

The moisture content, w_{H}, as a percentage by mass, of the wood-based panel is given by Equation (2):

$$w_{\mathrm{H}} = \frac{m_1 - m_0}{m_0} \cdot 100 \qquad (2)$$

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*, $w_{(\mathrm{HCHO})}$, is expressed in milligrams of formaldehyde per 100 g of oven-dry board, and is calculated using Equation (3):

$$w_{(\mathrm{HCHO})} = \frac{(A_\mathrm{s} - A_\mathrm{b}) \cdot f \cdot (100 + w_\mathrm{H}) \cdot V}{m_\mathrm{H}} \tag{3}$$

where

A_s is the absorbance of the analysed extraction solution;

A_b is the absorbance of an analysis with distilled or demineralized water;

f is the slope of the standard curve, in milligrams per millilitre (mg/ml);

w_H is the moisture content of the wood-based panel, as a percentage;

m_H is the mass of test pieces, in grams;

V is the volume of the volumetric flask (2 000 ml).

The perforator value of a wood-based panel is considered to be the mean value from the results of the two or three extractions.

Results shall be expressed to one decimal place.

The perforator values for particleboards, oriented strand boards (OSBs) and dry process fibreboards (MDFs) shall be applied to wood-based panels conditioned to a moisture content of 6,5 %. In the case of particleboards, OSBs or MDFs with different moisture contents, the perforator value shall be multiplied by a factor, F.

In the case of particleboards and OSBs with different moisture content (in the range of $3\ \% \le w_\mathrm{H} \le 10\ \%$), the perforator value shall be multiplied by a factor, F, calculated from Equation (4):

$$F = -\,0{,}133\ w_\mathrm{H} + 1{,}86 \tag{4}$$

In the case of MDFs with different moisture contents, the perforator value shall be multiplied by a factor, F, calculated from Equations (5) and (6):

- For panels with moisture contents in the range of $4\ \% \le w_\mathrm{H} \le 9\ \%$:

$$F = -\,0{,}133\ w_\mathrm{H} + 1{,}86 \tag{5}$$

- For panels with moisture contents $w_\mathrm{H} < 4\ \%$ and $w_\mathrm{H} > 9\ \%$:

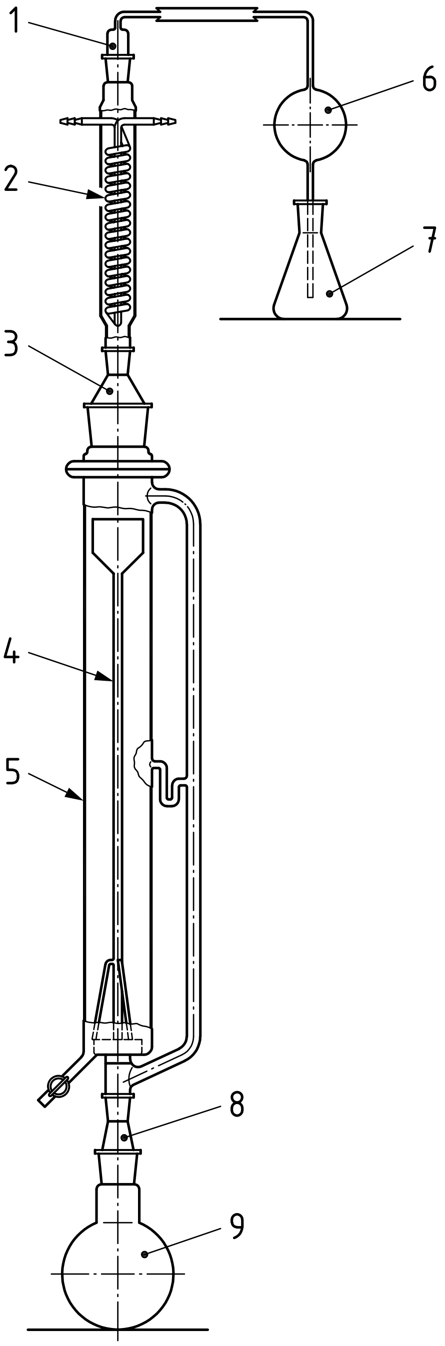
$$F = 0{,}636 + 3{,}12\mathrm{e}_{(-0{,}346 w_\mathrm{H})} \tag{6}$$

9 Test report

The test report shall contain, if known, the following information, together with a reference to this part of ISO 12460:

- origin of the board;
- place, location (e.g. factory, in the case of installed boards: ceiling, floor, wall, etc.) and state (e.g. moisture content, surface coating, finishing) of the board at the time of sampling, in particular, the moisture content;
- type of 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.

In respect of the final point, report all the information about the operations which are not in accordance with this part of ISO 12460 (sampling of test pieces, conditioning, etc.).

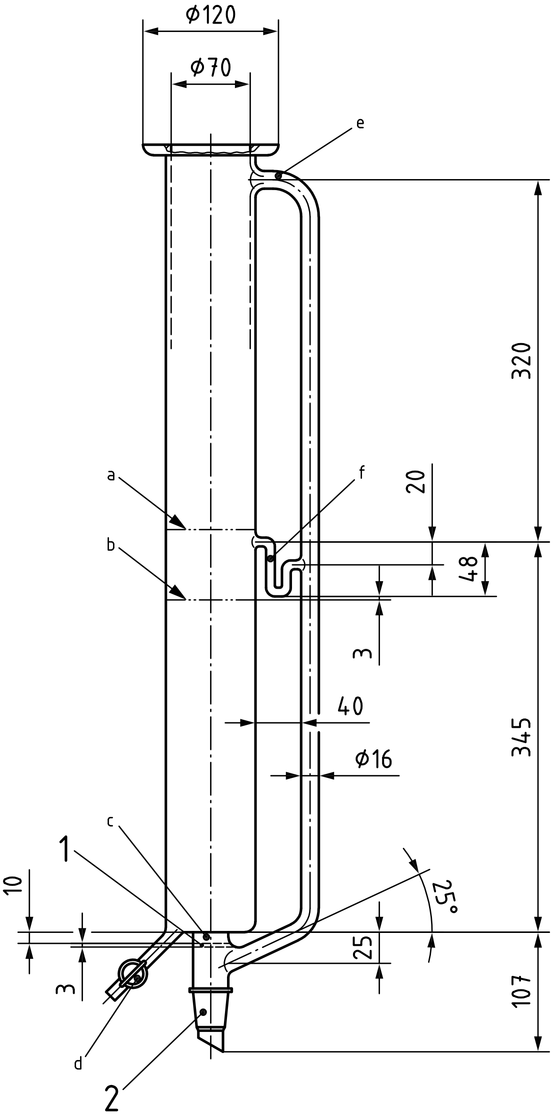


Key

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, 1 000 ml with socket 45/40

Figure 1 — Extraction apparatus

Dimensions in millimetres



Key

1 blanking plate

2 std. 29/32

[a] Toluene level.

[b] Maximum water level.

[c] 4 dia. hole each side.

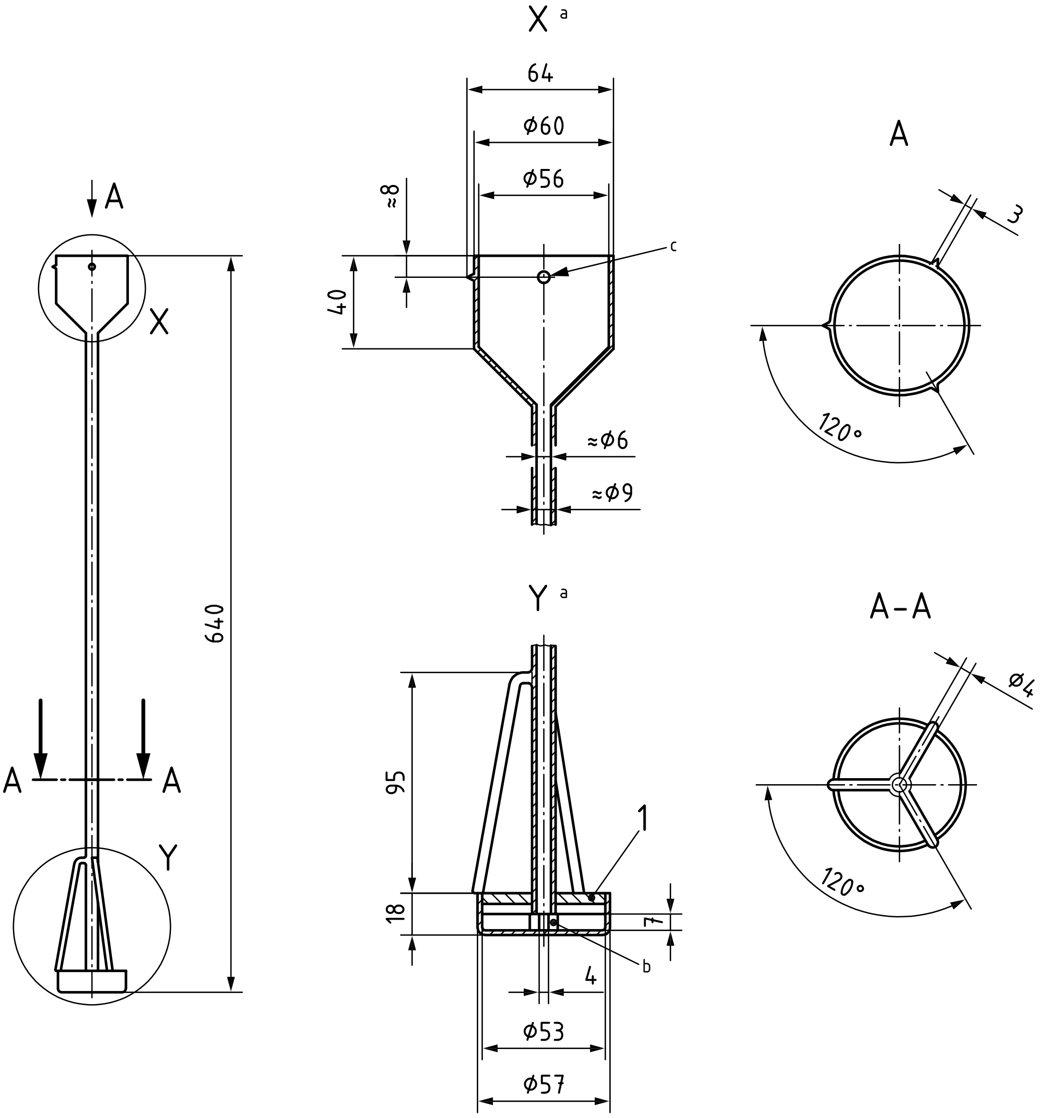
[d] Stopcock 4 dia. bore.

[e] Side arm.

[f] 8 dia. siphon tube.

Figure 2 — Perforator attachment

Dimensions in millimetres



Key

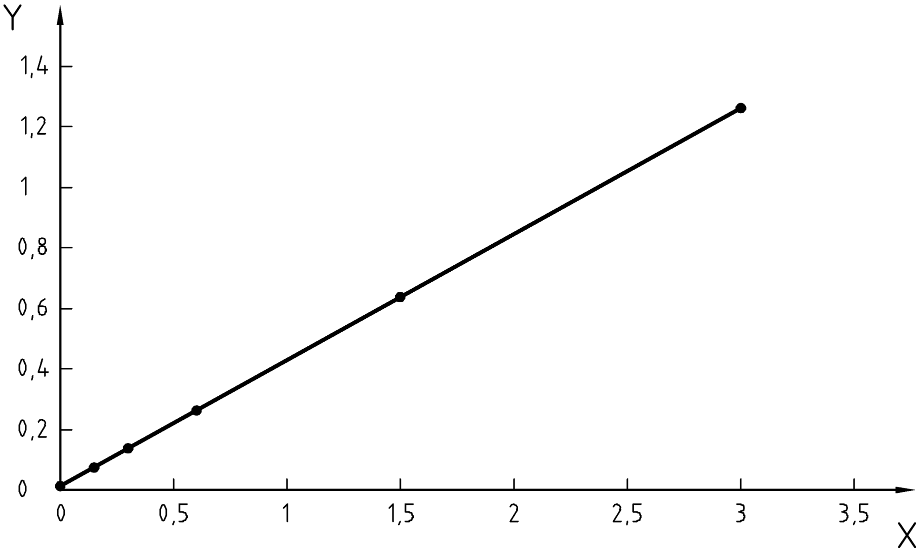
1 sintered glass filter (porosity P 160)

[a] Detail (sectional view).

[b] Two 4 dia. spacing rods.

[c] Holes 5 dia. on each side.

Figure 3 — Filter insert



$$c = f \cdot (A_s - A_b)$$

Key

Y absorbance, $A_s - A_b$

X concentration, c, of the diluted calibration solution, 10^{-3} mg/ml

Figure 4 — Example of a calibration curve for formaldehyde determined by acetylacetone method (path length 50 mm**)**

ICS 79.060.01

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