
**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)



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#)

The committee responsible for this document is ISO/TC 89, *Wood-based panels*.

This second edition cancels and replaces the first edition (ISO 12460-5:2011), which has been technically revised.

The objective of the revision was to improve the detection limit and the reproducibility of the method with regard to boards with low formaldehyde content.

Compared to ISO 12460-5:2011, the following modifications have been made:

- a) in [5.3](#) recommendation of cell length for low emitting samples is added;
- b) in [6.4](#) relative humidity for conditioning climate is increased to $(65 \pm 5) \%$;
- c) in [7.1](#) and [8.2](#) procedure and evaluation of third extraction are modified;
- d) in [7.3](#) accuracy of weighing of test pieces enhanced and higher mass of the test pieces is added as an option for low emitting boards;
- e) in [7.4](#) precision of blank test is improved;
- f) in [7.5.1](#) fluorimetric determination of formaldehyde is added an option;
- g) in [7.5.4](#) temperature of water bath is increased to 60 °C;
- h) in [8.2](#) modification factors for moisture content is included;
- i) in [Figure 4](#) calibration curve is modified;
- j) [Annex A](#) for calibration and quality assurance is added.

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)*

Additional parts dealing with small-scale chamber method is planned.

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Introduction

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 dependent on the specific board conditions at the time of testing.

The emission of formaldehyde from wood-based panels (e.g. particleboard, plywood, fibreboard, OSB) 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.

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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”. It is used for the determination of the formaldehyde content of unlaminated and uncoated wood-based panels.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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.

4.4 Formaldehyde solution.

Commercially available formaldehyde solution (concentration typically between 35 % mass fraction to 40 % mass fraction).

5 Apparatus

5.1 Precision balance, with 0,01 g scale intervals.

5.2 Well-ventilated oven, capable of maintaining a temperature of $(103 \pm 2) ^\circ\text{C}$.

5.3 Spectrophotometer, capable of measuring absorbance at 412 nm. A cell of 50 mm path length is recommended, especially for low emitting samples.

5.4 Extraction apparatus.

The apparatus consists of the following:

- spiral condenser (Dimroth cooler), 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 diameters: 60 mm (item reference 4);
- perforator attachment, 1 000 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);
- 1 000 ml round bottom 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);
- heating mantle (item reference 10).

The item references are given in [Figure 1](#). Details of the apparatus are shown in [Figure 2](#) and [Figure 3](#).

Permanent PTFE seals should be used rather than paraffin or silicone grease sealants.

5.5 Laboratory equipment.

The apparatus consists 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;
- volumetric pipette, 100 ml, calibrated at 20 °C;
- volumetric pipette, 25 ml, calibrated at 20 °C;
- volumetric pipettes (calibrated at 20 °C), 1 ml, 2 ml, 5 ml, 10 ml, 15 ml, 20 ml, 25 ml, 50 ml, 100 ml;
- three volumetric pipettes, 10 ml, calibrated at 20 °C;
- stoppered flasks, each 50 ml;
- two volumetric cylinders, 250 ml;
- water bath, capable of maintaining a temperature of (60 ± 1) °C;
- water bath, capable of maintaining a temperature in the range of 20 °C to 25 °C;

— desiccator.

6 Test pieces

6.1 General

Sampling and cutting are done in accordance with ISO 16999.

6.2 Sampling

6.2.1 The test pieces are to be taken and evenly distributed over the width of the (cooled) board, but excluding a 250 mm wide strip from the end of each board.

6.2.2 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.

6.3 Sampling for factory 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 h after sampling.

6.4 Sampling 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 constant mass at a temperature of $(20 \pm 2) ^\circ\text{C}$ and a relative humidity of $(65 \pm 5) \%$.

Constant mass is considered to have been reached when the results of two successive weighings carried out at intervals of not less than 24 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 are to be carried out in duplicate.

For factory production control, a single extraction can be sufficient.

The individual values of a duplicate extraction shall not deviate by more than 0,5 mg/100 g, otherwise, a third extraction has to 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) with a minimum mass of 20 g.

Weigh the sample (5.1) to an accuracy of 0,1 g onto the watch glass (5.5) and dry in the oven (5.2) at a temperature of $(103 \pm 2) ^\circ\text{C}$ 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 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

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 0,1 g and put into the round bottom flask (5.4). 600 ml of toluene (4.1) are added. About 1 000 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. Subsequently, the round bottom flask is connected to the perforator. 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.

For boards with low formaldehyde content, the mass of the test pieces can be extended up to 200 g provided that the density of the samples enables such an extension.

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 out for (120 ± 5) min 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 min and 30 min after turning on the heating device.

After these 120 min, the heating is switched off and the gas absorption bulb removed.

The water contained in the perforator is, after cooling to room temperature, transferred into the volumetric flask by means of the stopcock. 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 2 000 ml with distilled water.

7.4 Blank test

When using toluene from a new batch, carry out an extraction without test pieces applying the procedure as in 7.3.

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.

A fluorimetric determination may be used optionally.

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 can also be used.

7.5.3 Reagents

7.5.3.1 Acetylacetone solution

4 ml acetylacetone is transferred to a 1 000 ml volumetric flask, dissolved in distilled water, and filled to volume with distilled water.

7.5.3.2 Ammonium acetate solution

200 g ammonium acetate is transferred to a 1 000 ml volumetric flask, dissolved in distilled water, and filled to volume with distilled water.

Optionally, a premixed reagent of acetylacetone and ammonium acetate as described in ISO 12460-4:2008, 4.1 can be used.

7.5.4 Analytical procedure

10 ml is taken from the aqueous solution (7.3) with a pipette and added to 10 ml acetylacetone solution (7.5.3.1) and 10 ml ammonium acetate solution (7.5.3.2) in a 50 ml flask. The flask is stoppered, shaken, and warmed for 10 min in a water bath of 60 °C. The heated flask is then cooled in a water bath operated in a temperature range between 20 °C and 25 °C for at least 15 min protected from sunlight or is cooled to room temperature, protected against the influence of light (for about 1 h). 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 in parallel with distilled water and taken into consideration in the determination of the perforator value (8.2).

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

7.5.5 Calibration curve

7.5.5.1 General

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. It is possible to modify this frequency if it is proved that the slope of the standard curve does not deviate. In this case, the checking shall be made at least once a month and for each change of reagents.

7.5.5.2 Formaldehyde standard solution

Reagents:

—	Standard iodine solution	$c(\text{I}_2)$	= 0,05 mol/l
—	Standard sodium thiosulfate solution	$c(\text{Na}_2\text{S}_2\text{O}_3)$	= 0,1 mol/l
—	Standard sodium hydroxide solution	$c(\text{NaOH})$	= 1 mol/l

- Standard sulphuric acid solution $c(\text{H}_2\text{SO}_4)$ = 1 mol/l
- Starch solution = 1 %

The solutions shall be standardized before use.

About 2,5 g formaldehyde solution (concentration 35 % to 40 %) are diluted in a 1 000 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 min standing protected from light, 15 ml of sulfuric acid solution are added to the mixture. The excess iodine is back-titrated with the thiosulfate 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 using Formula (1):

$$c(\text{HCHO}) = (V_0 - V) \times 15 \times c(\text{Na}_2\text{S}_2\text{O}_3) \times 1\,000 / 20 \quad (1)$$

where

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

$c(\text{Na}_2\text{S}_2\text{O}_3)$ is the concentration of the thiosulfate solution, in mols per litre (mol/l);

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

V is the volume of thiosulfate titration solution for the test, in millilitres (ml).

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

Other titration procedures such as the method described by ISO 2227 or ISO 14184-1 can be used for the determination of the formaldehyde concentration.

7.5.5.3 Formaldehyde calibration solution

Using the concentration determined in 7.5.5.2, 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.

7.5.5.4 Determination of the calibration curve

Pipette 0 ml, 1 ml, 2 ml, 5 ml, 10 ml, 20 ml, 50 ml, and 100 ml of formaldehyde calibration solution (see 7.5.5.3) in 100 ml volumetric flasks and fill to volume with distilled water. 10 ml of each dilution are analysed photometrically by the same procedure as described above (see 7.5.4). The absorbance values are plotted against the formaldehyde concentrations c (between 0 mg/ml and 0,015 mg/ml) and the slope, f , is calculated (see example in Figure 4).

For low emitting boards, a cell with a path length of 50 mm, instead of 10 mm, with a calibration curve as shown in ISO 12460-3 is recommended.

7.6 Quality assurance

It is recommended to run a perforator extraction efficiency test at least twice a year or when the perforator set-up has been completely disassembled. A procedure is given in Annex A.

8 Expression of results

8.1 Moisture content

The moisture content, H , as a mass fraction of the wood-based panel is given by using Formula (2):

$$H = \frac{m_1 - m_o}{m_o} \times 100 \quad (2)$$

where

m_1 is the mass of the test pieces before drying, in grams (g);

m_o is the mass of the test pieces after drying, in grams (g).

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 using Formula (3):

$$\text{perforator value} = \frac{(A_s - A_b) \times f \times (100 + H) \times V}{m_H} \text{ mg/100 g} \quad (3)$$

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);

H is the moisture content of the wood-based panel as mass fraction, in percent (%);

m_H is the mass of test pieces, in grams (g);

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 extractions.

If three replicates are carried out (see 7.1), the most deviating result is discarded.

Results shall be expressed to one decimal place.

The perforator values for particleboards, OSB, and MDF shall be applied to wood-based panels conditioned to a moisture content of 6,5 %. In the case of particleboards, OSB, or MDF with different moisture contents, the perforator value shall be multiplied by a factor, F .

In the case of particleboards and oriented strand boards (OSB) with different moisture content (in the range of $3 \% \leq H \leq 10 \%$), the perforator value shall be multiplied by a factor, F , which can be calculated by using Formula (4):

$$F = -0,133 H + 1,86 \quad (4)$$

In the case of dry process fibreboards (MDF) with different moisture contents, the perforator value shall be multiplied by a factor, F , calculated by using Formula (5) and Formula (6):

For panels with moisture contents in the range of $4 \% \leq H \leq 9 \%$:

$$F = -0,133 H + 1,86 \quad (5)$$

For panels with moisture contents $H < 4 \%$ and $H > 9 \%$:

$$F = 0,636 + 3,12 e^{(-0,346 H)} \quad (6)$$

9 Test report

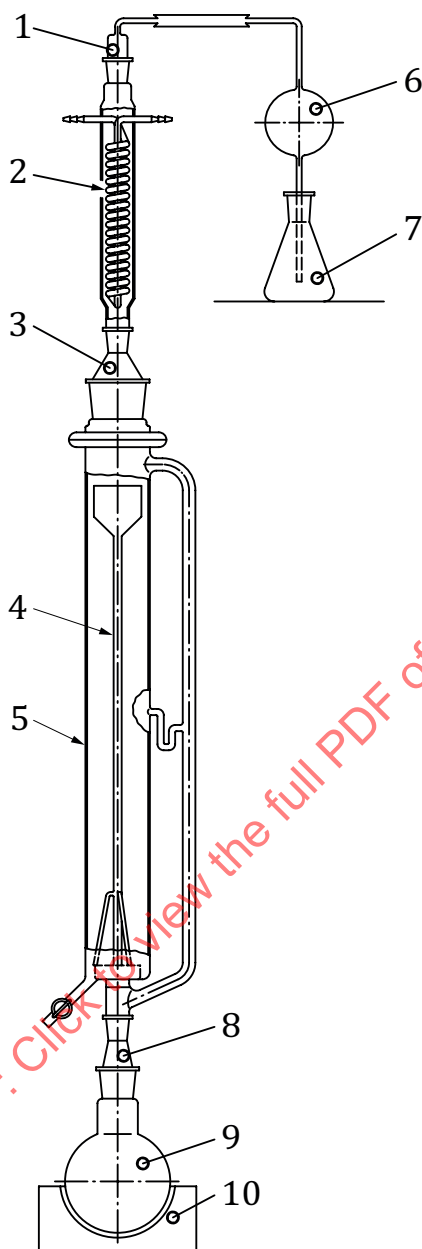
The test report shall contain, if known, the following information with a reference to this part of ISO 12460:

- a) a reference to this part of ISO 12460, i.e. ISO 12460-5;
- b) origin of the board;
- c) place, location¹⁾, and state²⁾ of the board at the time of sampling (in particular, the moisture content);
- d) type of board;
- e) thickness of board (mm);
- f) density of board (kg/m³);
- g) date of manufacture of the board;
- h) date of sampling;
- i) information referring to the board regarding finishing, sanding, etc.;
- j) date of formaldehyde determination;
- k) moisture content (%) at the time of testing (according to 7.2);
- l) perforator value in milligrams formaldehyde per 100 g of oven dry board (individual values of each extraction and mean value);
- m) descriptions of further details³⁾.
- n) limit of quantification in milligrams formaldehyde per 100 g of oven dry board and the estimated uncertainty of measurement in percent (%) or in in milligrams formaldehyde per 100 g of oven dry board.

1) For example, factory, etc. In the case of installed boards, ceiling, floor, wall, etc.

2) For example, moisture content, surface coating, finishing.

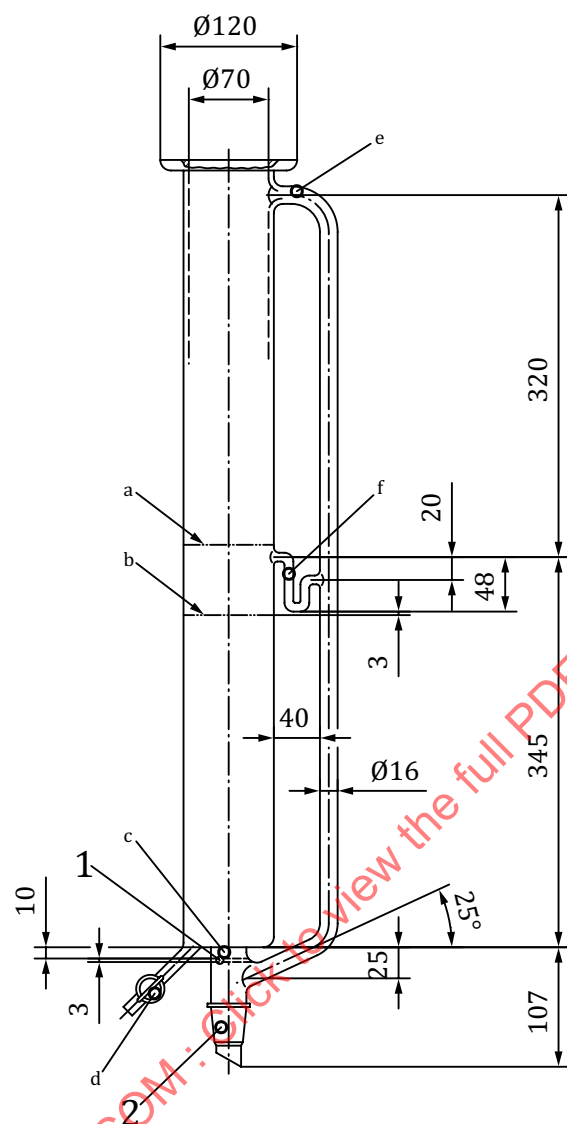
3) Report all the information about the operations which are not in accordance with this part of ISO 12460 (sampling of test pieces, conditioning, etc.).

**Key**

- | | | | |
|---|--------------------------------|----|---|
| 1 | conical adaptor 29/32 | 6 | (double) bulbed tube |
| 2 | dimroth cooler | 7 | conical flask 250 ml |
| 3 | conical adaptor 45/40 to 71/51 | 8 | conical adaptor 29/32 to 45/40 |
| 4 | filter insert | 9 | round bottom flask 1 000 ml with socket 45/40 |
| 5 | perforator attachment | 10 | heating mantle |

Figure 1 — Extraction apparatus

Dimensions in millimetres

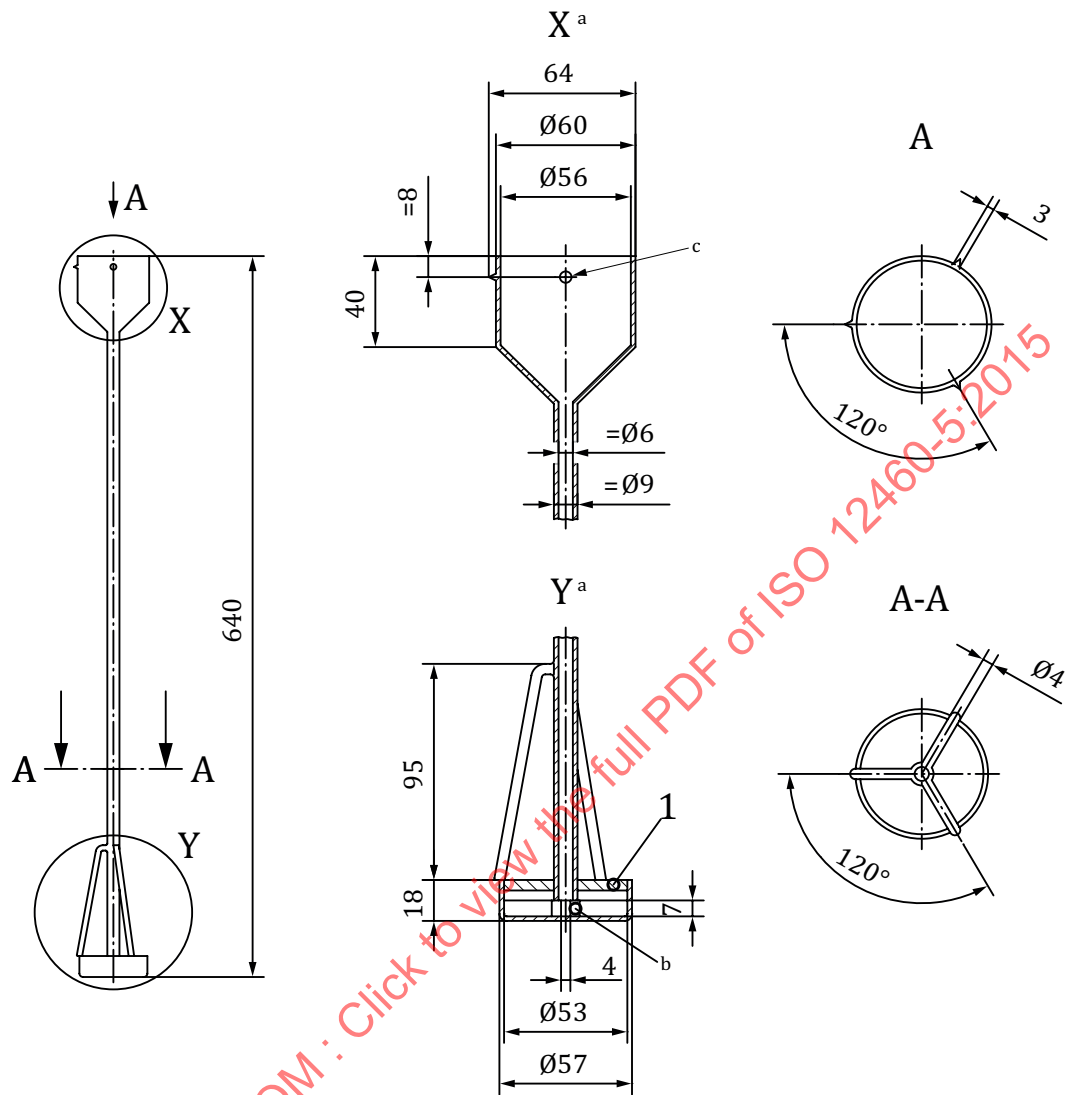


Key

- | | | | |
|---|----------------------|---|------------------------|
| 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 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