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Bitumen and bituminous binders — Accelerated long-term ageing conditioning by a Pressure Ageing Vessel (PAV)

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National foreword

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May 2012

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Bitumen and bituminous binders - Accelerated long-term ageing conditioning by a Pressure Ageing Vessel (PAV)

Bitumes et liants bitumineux - Vieillissement long-terme accéléré réalisé dans un récipient de vieillissement sous pression (PAV)

Bitumen und bitumenhaltige Bindemittel - Beschleunigte Langzeit-Alterung mit einem Druckalterungsbehälter (PAV)

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Foreword

This document (EN 14769:2012) has been prepared by Technical Committee CEN/TC 336 "Bituminous binders", the secretariat of which is held by AFNOR/BNPé.

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of an identical text or by endorsement, at the latest by November 2012, and conflicting national standards shall be withdrawn at the latest by November 2012.

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

This document supersedes EN 14769:2005.

Compared with EN 14769:2005, the following changes have been made:

- a) terms and definition: update of point 3.2, 3.3 and addition of 3.4 plus Notes;
- b) Note added in 5.1 and sub-clauses 5.5, 5.7 and 5.8 restructured;
- c) Note 4 introduced, Note 5 improved and new paragraph added about conditioning temperatures and warning added in Clause 6;
- d) references to EN 14895 withdrawn.

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1 Scope

This European Standard specifies an accelerated ageing/conditioning procedure for bituminous binders. The procedure involves ageing trays of binder at elevated temperatures under pressurised conditions in a pressure ageing vessel (PAV).

NOTE For binders to be used in hot asphalt applications, the pre-conditioning of the sample would typically be done using one of the methods in the EN 12607 series. For binders to be used in bituminous emulsion and cut-back or fluxed applications, the stabilising of the sample should be such that there are no volatiles remaining.

WARNING — The use of this European Standard can involve hazardous materials, operations and equipment, in particular, the use of a high pressure ageing vessel. This European Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this European Standard to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use. If there is the likelihood of volatile components being present in a binder, this procedure shall not be used.

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.

EN 12607-1:2007, *Bitumen and bituminous binders — Determination of the resistance to hardening under the influence of heat and air — Part 1: RTFOT method*

EN 12607-2:2007, *Bitumen and bituminous binders — Determination of the resistance to hardening under the influence of heat and air — Part 2: TFOT method*

EN 12607-3:2007, *Bitumen and bituminous binders — Determination of the resistance to hardening under the influence of heat and air — Part 3: RFT method*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

short-term ageing

short-term conditioning

conditioning that the binder experiences during the method described in EN 12607-1:2007, EN 12607-2:2007 and EN 12607-3:2007

3.2

stabilisation

conditioning of the binders from bituminous emulsions, cut-backs or fluxed bitumens to produce residual binders for further determination of their characteristics

EXAMPLE The procedure described in EN 13074-2:2007.

3.3

long-term ageing

ageing that the binder experiences during its service life

3.4

accelerated long-term ageing

accelerated long-term conditioning

simulated long-term ageing that the binder goes through during the accelerated pressure ageing procedure

Note 1 to entry: In the case of hot mix asphalt binders, the long-term ageing is carried out on binders that have already been conditioned through short-term ageing/conditioning.

Note 2 to entry: In the case of bituminous emulsion and cut-back, the long-term ageing is carried out on binders after a stabilisation procedure.

4 Principle

A static film of binder is heated to a specified temperature under a specified air pressure for a given period of time. This is done in order to simulate the changes occurring to the binder within the pavement during use.

The effects of this ageing procedure are evaluated on the residual binder after the test.

NOTE The ageing of binders during service is affected by ambient temperature and air pressure as well as by a mixture of associated variables such as volumetric mixture proportions, mixture permeability, aggregate properties and other factors. This test is intended to provide an evaluation of the relative ageing behaviour of binders under specified conditions, but it cannot account entirely for bituminous application variables or provide relative resistance to ageing at in-service conditions.

5 Apparatus

Usual laboratory apparatus and glassware, together with the following:

5.1 Pressure/temperature vessel, designed to operate at $(2,1 \pm 0,1)$ MPa between 80 °C and 115 °C. Either 5.1.1 and 5.1.2 or 5.1.3 is applicable (see Figure 1).

5.1.1 Pressure vessel, which shall be made from stainless steel and shall have internal dimensions adequate to contain a pan holder capable of holding a number of containers (as per requirements and dimensions given in EN 12607-2:2007).

The bottom of the pressure vessel shall be such that the containers are held in a horizontal position with the binder film thickness evenly distributed across the diameter of the container. A diagram showing a possible configuration of the vessel pan holder and containers and specifying dimensional requirements is shown in Figure 2.

NOTE 1 Most pan holder assemblies hold up to 10 containers.

NOTE 2 Other pressure vessels of different internal dimensions may be used provided that the operating conditions can be satisfied. In such cases, the containers used may differ from the standard dimensions given in EN 12607-2:2007.

5.1.2 Forced-draft oven, to be used with 5.1.1 and which is capable of

- e) bringing the loaded pressurised vessel to the desired conditioning temperature $\pm 0,5$ °C, as recorded by a suitable thermometer inside the vessel, within two hours after loading the pressure vessel into the oven,
- f) maintaining the temperature at all points within the vessel at the ageing temperature $\pm 0,5$ °C.

The oven shall have sufficiently large interior dimensions to allow forced air to freely circulate within the oven and around the pressure vessel. The oven shall contain a stand or a shelf, which supports the loaded pressure vessel in a level position above the lower surface of the oven.

5.1.3 Pressure vessel, having the same dimensions described in 5.2.1, with an integral temperature control system that is capable of

- a) bringing the loaded pressure vessel to the desired conditioning temperature $\pm 0,5$ °C, as recorded by the thermometer inside the vessel, within two hours,
- b) maintaining the temperature at all points within the vessel at the ageing temperature $\pm 0,5$ °C.

The vessel may be a separate unit to be placed in a forced draft oven or an integral part of a temperature control system.

SAFETY PRECAUTIONS — The pressure ageing vessel operates at high temperatures and high pressures. All safety guidelines issued by equipment manufacturers shall be adhered to.

5.2 Pressure controlling devices, which include the following:

5.2.1 Pressure release valve, which prevents pressure in the vessel from exceeding 2,5 MPa during the ageing procedure.

5.2.2 Pressure regulator, capable of controlling the pressure within the vessel to $\pm 0,1$ MPa and with a capacity sufficient to reduce the pressure from the source of compressed air so that the pressure within the vessel is maintained at the operating pressure of $(2,1 \pm 0,1)$ MPa.

5.2.3 Slow release bleed valve, which allows the pressure in the vessel at the completion of the test to be reduced from 2,1 MPa (the operating pressure) to atmospheric pressure within 8 min to 15 min.

5.2.4 Pressure gauge, capable of measuring the pressure within the vessel to within 0,1 MPa during the test, and which shall be calibrated to an accuracy of $\pm 0,1$ MPa at appropriate intervals.

5.3 Thermometer, accurate to 0,1 °C for measuring the temperature inside the pressure vessel.

NOTE A resistance thermal detector (RTD) has been found to be suitable.

5.4 Temperature recording device, which is a data acquisition system capable of recording the temperature throughout the test to 0,1 °C.

NOTE The current method of monitoring temperature is via a computerised log of time and temperature. It is assumed that the temperature recorded is the temperature at every area within the ageing vessel.

5.5 Metal containers, with diameters of (140 ± 1) mm, the standard for which is defined in EN 12607-2:2007, 4.3.

Similar containers with other diameters can be used if their diameters are determined with the same tolerance and the amount of binder is adjusted as given in Formula (1). Weigh $(50,0 \pm 0,5)$ g in the container (nominal diameter 140 mm Ø). If a container with another diameter is used weigh the amount given in Formula (1):

$$M = 50,0 \text{ g} \times (d_2 \times d_2) / (d_1 \times d_1) \pm 0,5 \text{ g} \quad (1)$$

where

d_1 is the normal container diameter (140 ± 1) mm,

d_2 is the actual container diameter in millimetres with a tolerance of ± 1 mm, and

M is the mass of binder at ambient temperature for the actual container.

5.6 Balance, which shall be capable of weighing to an accuracy of $\pm 0,1$ g.

5.7 Vacuum oven (optional), which shall be capable of maintaining a temperature up to 180 °C with an accuracy of ± 5 °C.

The vacuum system shall be capable of maintaining pressures below $(15,0 \pm 2,5)$ kPa absolute. Alternatively, the pressure vessel itself may be used in lieu of the vacuum oven if it is capable of maintaining a temperature of 180 °C and a vacuum of $(15,0 \pm 2,5)$ kPa.

5.8 Commercial bottled air, which is suitable for the purpose of providing a sufficient supply of pressurized air to carry out the test. (Only bottled air is suitable.)

6 Procedure

SAFETY PRECAUTIONS — Use laboratory safety procedures when handling the hot bituminous binders and preparing the specimens and when removing the residue from the pressure vessel. Take particular care when lifting the lid of the pressure vessel.

Pre-heat the PAV vessel or forced draft oven to the ageing temperature. The binder is firstly pre-conditioned as necessary, prior to the long-term ageing procedure. The procedure used shall be stated in the report.

If it is necessary to prepare more than one set of samples in the short-term ageing/conditioning procedure (using either EN 12607 series or a stabilisation procedure) in order to obtain sufficient quantity for this procedure, sub-samples shall be combined and homogenised before the test. The stabilisation procedure shall be stated in the report.

NOTE 1 The hot residue may be poured immediately into the container or allowed to cool for PAV ageing later. If allowed to cool, the container with the binder should be covered and stored at ambient temperature. When it is to be used, the sample should be re-heated in accordance with EN 12594 for the minimum possible time and stirred prior to pouring into the container.

NOTE 2 Pre-heating the vessel up to 15 °C above the conditioning temperature may be used to reduce the drop in PAV temperature during the loading process and minimise the time required for stabilisation of the set temperature after loading. However, pressurising the vessel causes an increase in temperature and the vessel may cool slowly, making it difficult to achieve the specified temperature at the start of the test.

Pre-heat the container to the PAV conditioning temperature. Place the heated container on a balance and pour $(50,0 \pm 0,5)$ g into the container. Ensure that the binder is distributed over the entire base of the container by tilting the filled container if necessary.

NOTE 3 The binder film thickness will be about 3,2 mm. The number of containers per binder will depend on the amount of binder required for subsequent testing. The heating of the container should be sufficient to enable the flow of the binder.

NOTE 4 $(50,0 \pm 0,5)$ g refers to containers conforming to EN 12607-2. When using containers of a different diameter, the requirement is that sufficient binder shall be poured such that the film thickness will be about 3,2 mm (see 5.5).

Place the pan holder inside the heated pressure vessel and set the ageing temperature to the specified temperature.

NOTE 5 Typical conditioning temperatures are 85 °C, 90 °C, 100 °C and 110 °C. Typical ageing times found to be suitable are 20 h \pm 10 min for the 90 °C, 100 °C and 110 °C temperature conditions, and 65 h \pm 30 min for the 85 °C temperature condition.

The chemistry of bituminous binder ageing is complex and the mechanism changes with increasing temperature. For this reason, the conditioning temperature should be kept as low as possible. High temperatures may make this ageing procedure unrepresentative of field ageing. The 110 °C condition should be limited to conditioning of binders for use in high ambient temperature (desert) areas.

Binders containing polymers could exhibit separation and/or degradation in a way that does not occur during natural ageing.

NOTE 6 Several temperatures are currently used for the accelerated long-term ageing method. No correlation of a particular temperature with performance has been established at this time.

Place the filled containers in the pan holder. Place the pan holder complete with containers into the pressure vessel. Close the pressure vessel, ensuring that all bolts, clamps etc are secure and tighten the pressure vessel lid according to the manufacturer's instructions.

NOTE 7 Containers containing binders from different sources may be used in the same ageing test. Less than the full set of 10 containers may also be used.

NOTE 8 If desired, the change in mass after the long-term ageing may be determined by following the procedure given in EN 12607-2:2007, 6.2, the only difference being the heating period is that which is described in this method.

The loading process into the pressure vessel shall be carried out as quickly as possible to minimise temperature loss.

WARNING: When the temperature within the vessel is 5 °C to 20 °C below the ageing temperature, apply an air pressure in accordance with the manufacturer's instructions of $(2,1 \pm 0,1)$ MPa and commence the test. If the temperature within the PAV fails to reach the ageing temperature within two hours after application of pressure, the test shall be discontinued and the samples discarded.

Maintain the temperature and pressure for a specified ageing time.

If the PAV ageing vessel has reached the ageing temperature within the first two hours and the following conditions occur during the ageing procedure:

- the temperature indicated by the temperature recording device is above or below the set ageing temperature by more than 1,0 °C for more than 60 min, and
- the pressure is higher than $(2,1 + 0,2)$ MPa or lower than $(2,1 - 0,2)$ MPa of the set pressure for more than 30 min,

the test shall be terminated and the samples discarded.

At the completion of the ageing time, slowly release the internal pressure in the PAV using the air pressure bleed valve. Adjust the bleed valve as required so that the pressure is reduced smoothly to atmospheric within a period of 8 min to 15 min. The pressure release time does not form part of the 20 h or 65 h ageing time.

NOTE 9 If the pressure is reduced too rapidly, the binder samples may foam.

Open the pressure vessel and carefully remove the pan holder complete with the containers. Place the filled containers in a pre-heated oven set to (170 ± 5) °C for (30 ± 1) min. When fluid, stir each container to remove any air bubbles. If there is more than one container of any one binder, combine the contents of these containers and pour the hot residue into a suitable, larger, single container.

In some instances, air bubbles may still be entrapped within the binder. If this is the case, transfer the containers with the binder into a vacuum oven set at (170 ± 5) °C. Allow the containers to stand in the oven for (10 ± 1) min without the vacuum applied. After 10 min of equilibration, open the vacuum valve and rapidly reduce the pressure to $(15 \pm 2,5)$ kPa and maintain this pressure for (30 ± 1) min. At the end of the 30 min, release the vacuum and remove the container from the oven. If a vacuum oven is used, the fact shall be recorded.

NOTE 10 The specified heating times should not be exceeded.

If testing of the PAV residue is not to take place immediately after the ageing procedure, the sample containers shall be allowed to cool, and then sealed and stored at ambient temperature.

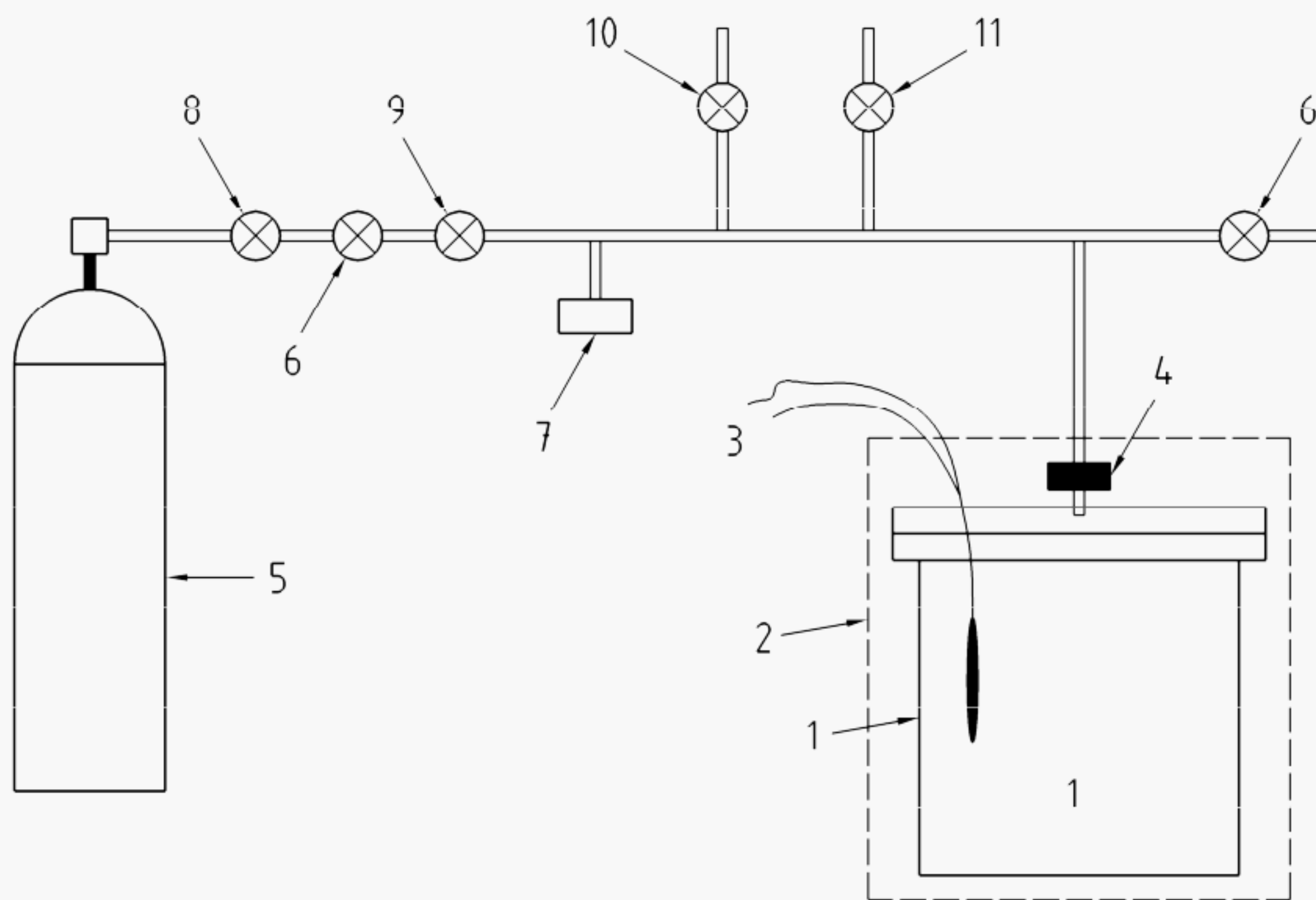
7 Precision

This European Standard is a conditioning procedure and has no result in itself. No precision data has been established.

8 Report

The ageing/conditioning procedure report shall contain at least the following information:

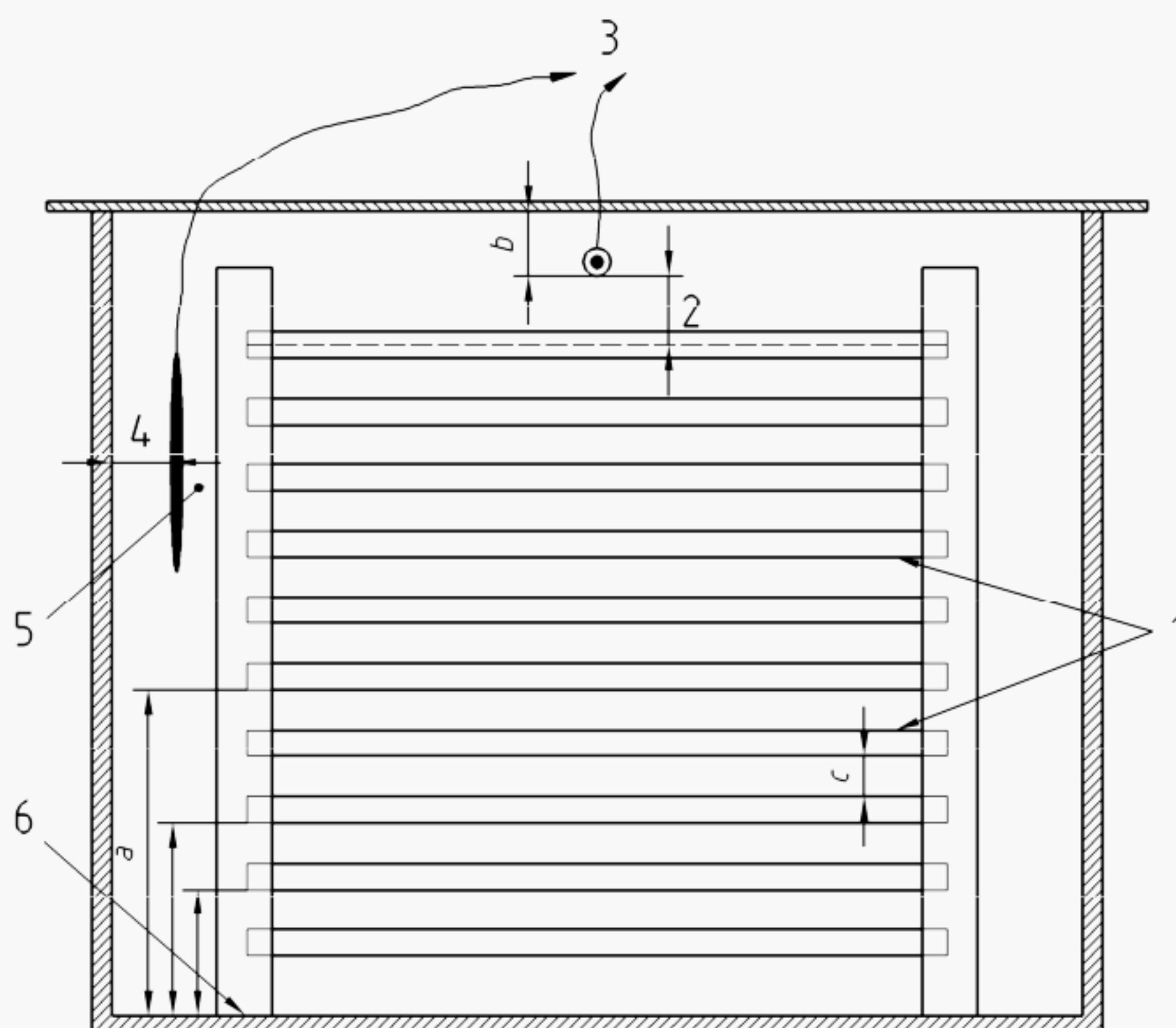
- a) reference to this European Standard;
- b) type and complete identification of the sample under this procedure;
- c) short-term ageing/conditioning method, or stabilisation procedure, if used;
- d) ageing procedure conditions, including temperature, pressure, ageing time and vacuum oven, if used;
- e) diameter of the container used;
- f) any deviation, by agreement or otherwise, from the procedure specified;
- g) date of the ageing procedure.



Key

- | | | |
|--------------------------|---------------------------|---------------------------|
| 1 pressure ageing vessel | 5 compressed air cylinder | 9 pressure gauge |
| 2 temperature control | 6 needle valve | 10 bleed valve |
| 3 thermometer | 7 rupture disc | 11 pressure release valve |
| 4 quick disconnect | 8 pressure regulator | |

Figure 1 — Example of PAV test system



Key

- 1 TFOT containers according to EN 12607-2:2007 supported on shelves: the entire assembly shall be removed as an integral unit. Assembly is to include containers and supports.
- 2 ≥ 10 mm from top of the binder
- 3 to temperature transducer and readout
- 4 wall clearance ≥ 10 mm
- 5 provide at least 5 mm to nearest surface
- 6 assembly support point
- a* distance *a* controls the level of the containers. The assembly shall be supported at three or more points. Distance *a* measured from each assembly support point to the bottom of the container shall be controlled to $\pm 0,5$ mm. Provision shall be made to ensure that the bottom of the PAV is levelled such that the thickness of the binder in each container varies by no more than $\pm 0,5$ mm across the diameter of any container.
- b* distance *b* shall be such that any active portion of the temperature transducer is ≥ 10 mm from the top surface of the PAV.
- c* distance *c* shall be ≥ 12 mm

Figure 2 — Diagram of pan assembly within the PAV

Bibliography

- [1] AASHTO D6521-03a, Standard practice for accelerated ageing of asphalt binder using a pressurized ageing vessel (PAV).
- [2] EN 12594, *Bitumen and bituminous binders – Preparation of test samples*.
- [3] EN 13074-2, *Bitumen and bituminous binders — Recovery of binder from bituminous emulsion or cut-back or fluxed bituminous binders — Part 2: Stabilisation after recovery by evaporation*

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