



Designation: D 1856 – 95a

## Standard Test Method for Recovery of Asphalt From Solution by Abson Method<sup>1</sup>

This standard is issued under the fixed designation D 1856; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This method covers the recovery by the Abson method of asphalt from a solution from a previously conducted extraction. The asphalt is recovered with properties substantially the same as those it possessed in the bituminous mixture and in quantities sufficient for further testing.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials<sup>2</sup>
- D 96 Test Methods for Water and Sediment in Crude Oil by Centrifuge Method (Field Procedure)<sup>3</sup>
- D 2172 Test Methods for Quantitative Extraction of Bitumen from Bituminous Paving Mixtures<sup>4</sup>
- D 2939 Test Methods for Emulsified Bitumens Used as Protective Coatings<sup>5</sup>
- E 1 Specification for ASTM Thermometers<sup>6</sup>

### 3. Summary of Test Method

3.1 The solution of solvent and asphalt from a prior extraction is distilled under prescribed conditions to a point where most of the solvent has been distilled, at which time carbon dioxide gas is introduced into the distillation process to remove all traces of the extraction solvent. The recovered asphalt (distillation residue) can then be subjected to further testing as required.

<sup>1</sup> This method is under the jurisdiction of ASTM Committee D-4 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.25 on Analysis of Bituminous Mixtures.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 04.02.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 05.01.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 04.03.

<sup>5</sup> *Annual Book of ASTM Standards*, Vol 04.04.

<sup>6</sup> *Annual Book of ASTM Standards*, Vol 14.03.

### 4. Significance and Use

4.1 The asphalt should be extracted from the aggregate-asphalt mixture in accordance with Method A of Test Methods D 2172 (centrifuge method) as there is some experimental evidence that the recovered asphalt may have slightly lower penetration values when recovered from solutions obtained from hot extraction methods.

### 5. Apparatus

5.1 *Centrifuge*, batch unit capable of exerting a minimum centrifugal force of 770 times gravity<sup>7</sup> or continuous unit capable of exerting a minimum force of 3000 times gravity. (The apparatus specified in Test Methods D 96 may also be used.)

5.2 *Centrifuge Tubes*—A supply of wide-mouth bottles or centrifuge tubes as shown in Fig. 1 or Fig. 2 of Test Methods D 96.

5.3 *Distillation Assembly*, as shown in Fig. 1, and consisting of the following items:

5.3.1 *Extraction Flasks*—Two 250-ml, wide-mouth, heat-resistant flasks, one for distillation and the other for the receiver.

5.3.2 *Glass Tubing*—Heat-resistant glass tubing, having 10-mm inside diameter and gooseneck shaped (as shown in Fig. 1) for connecting the flask to the condenser.

5.3.3 *Inlet Aeration Tube*,<sup>8</sup> at least 180 mm in length, having a 6-mm outside diameter with a 10-mm bulb carrying six staggered side holes approximately 1.5 mm in diameter.

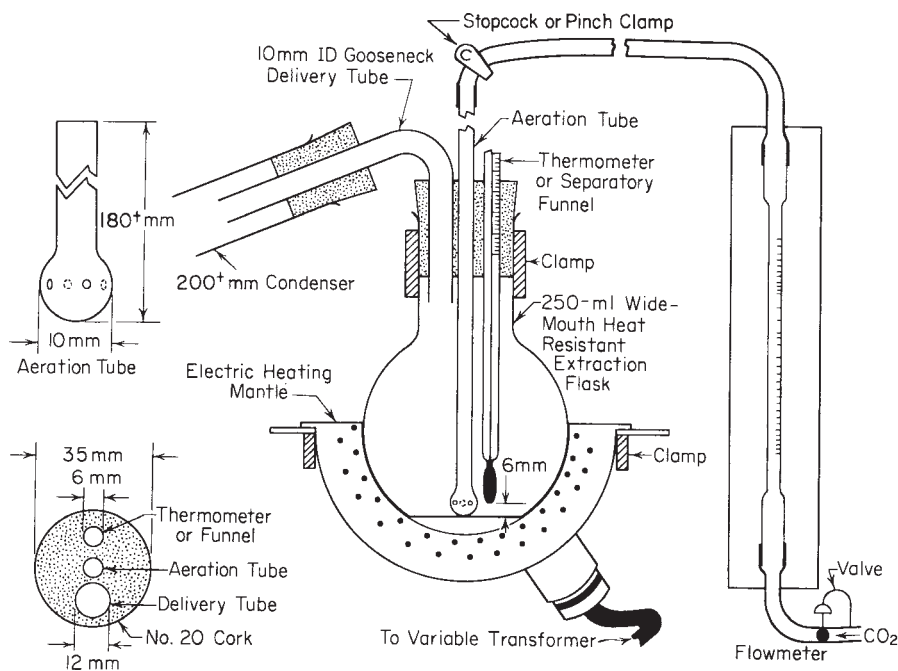
5.3.4 *Electric Heating Mantle*, with variable transformer, oil bath or fluidized sand bath, to fit a 250-ml flask.

5.3.5 *Water-Jacketed Condenser*, Allihn type, with 200-mm minimum jacket length, or equivalent.

5.3.6 *Thermometer*—An ASTM Low Distillation Thermometer 7E or 7F, as specified, having a range from – 2 to 300°C or 30 to 580°F, respectively, and conforming to the requirements in Specification E 1.

<sup>7</sup> An “International” No. 2 centrifuge operating at 1900 rpm or an “SMM Continuous Centrifuge” exerting a force of 3000 times gravity at 9000 rpm, have been found satisfactory for this purpose.

<sup>8</sup> Inlet Aeration Tube, Part No. 226, available from Wm. A. Sales, Ltd., 419 Harvester Court, Wheeling, Ill. 60090; request Part No. 226.



**FIG. 1 Distillation Assembly for Bitumen Recovery**

5.3.7 *Gas Flowmeter*,<sup>9</sup> as shown in Fig. 1, or any type capable of indicating a gas flow of up to 1000 ml/min.

5.3.8 *Corks, No. 20*, drilled as shown in Fig. 1.

5.3.9 *Flexible Elastomeric Tubing*, resistant to chlorinated solvents having sufficient length and size to connect the aeration tube to flowmeter, and equipped with a pinch clamp or stopcock to close aeration tube prior to introducing carbon dioxide.

5.3.10 *Separatory Funnel*,<sup>10</sup> (*Alternative Procedure*, see 9.3.1) 125-ml capacity.

## 6. Reagents and Materials

6.1 *Carbon Dioxide Gas*—A pressurized tank, with pressure-reducing valve or other convenient source.

6.2 The solvent for extracting the asphalt from mixtures should be reagent grade trichloroethylene.<sup>11</sup>

## 7. Precautions

7.1 **Caution**—The solvent listed in 6.2 should be used only under a hood or with an effective surface exhaust system in a well-ventilated area, since it is toxic to some degree as indicated below:

Solvent	TLV, ppm <sup>A</sup>	STEL, ppm <sup>B</sup>
Trichloroethylene	50	200

<sup>A</sup> Threshold limit value (TLV) as established by the American Conference of Governmental Industrial Hygienists (ACGIH), Bldg. D-7, 6500 Glenway Ave.,

<sup>9</sup> The Monostat Corp. "Flowmeter" No. 9144 has been found satisfactory for this purpose.

<sup>10</sup> Kimball separatory funnel No. 29028 has been found satisfactory for this purpose.

<sup>11</sup> Trichloroethylene, Technical Grade, Type I, Federal Specification O-T-634, latest revision may be used, but it is recommended that for each new supply of the solvent a blank should be run on an asphalt of known properties.

Cincinnati, OH 45211-4438. 1990/1991 values. The TLV is a time weighted average for an exposure period of 8 h per day, 5 days per week.

<sup>B</sup> Short term exposure limit (STEL) as established by ACGIH. 1990/1991 values.

## 8. Sample

8.1 The sample shall consist of the solution from a previous extraction by Method A of Test Methods D 2172 of asphalt-aggregate mixture of sufficient quantity to result in 75 to 100 g of recovered bitumen. More or less quantities of bitumen may be recovered; however, the properties of the recovered bitumen may not be in agreement with those recovered of the aforementioned quantities, and in case of a disagreement, 75 to 100 g should be recovered.

8.2 During the extraction process, it is important that all of the asphalt in the mixture be extracted as there could be some selective solvency of the asphalt and the harder, more viscous components of the asphalt might be left in the mixture if extraction is not carried to completion.

8.3 Since heavy petroleum distillates such as mineral spirits or kerosine will affect the properties of the recovered asphalt, it is important to avoid the use of such solvents in cleaning the extraction and recovery apparatus and use only trichloroethylene for cleaning. Residues of heavy petroleum solvents on the equipment may contaminate the recovered asphalt and affect its test properties. It is also necessary to use new filter rings, clean felt pads, or other uncontaminated filtering media in the extraction process to avoid contamination from a previous extraction.

8.4 Generally, the bitumen in mixtures will progressively harden when exposed to air, particularly if the mixtures are in a loose condition. Therefore, it is important to protect bituminous mixtures from exposure to air and preferably to store them in airtight containers at a temperature below 0°C (32°F) until they can be tested. When samples of bituminous mixture

are warmed for preparing representative proportions for extraction tests in accordance with Method A of Test Methods D 2172, they should be placed in an oven in covered containers and heated to a maximum temperature of 110°C (230°F) for the minimum time to obtain workability, but no longer than 30 min. If the samples have been stored at a low temperature, they should be allowed to reach room temperature before placing them in the oven.

## 9. Procedure

9.1 The entire procedure, from the start of the extraction to the final recovery, must be completed within 8 h.

9.2 Centrifuge the solution from the previous extraction for a minimum of 30 min at 770 times gravity in either bottles or centrifuge tubes in batch apparatus specified. If a continuous centrifuge is used, the extract solution shall be charged at a rate not to exceed 150 ml/min, while the unit is operating at a speed calculated to produce a centrifugal force of not less than 3000 times gravity.

9.3 Concentrate the solution to about 200 ml by any primary distillation operation using a flask large enough to hold all the solution from the extraction. Transfer the residue from the primary distillation flask, using several washes of solvent to rinse all of the residue into the 250-ml distillation flask. Assemble the apparatus as shown in Fig. 1, except position the bottom of the aeration tube above the surface of the solution. Continue distillation until the temperature reaches 135°C (275°F), at which time lower the aeration tube so that the bulb is in contact with the bottom of the flask, and introduce carbon dioxide gas at a low rate (approximately 100 ml/min) to provide agitation and prevent foaming. If foaming or bumping occurs, introduce carbon dioxide intermittently at the beginning of the distillation at a rate of 100 ml/min to prevent this. When the temperature reaches 157 to 160°C (315 to 320°F), increase the carbon dioxide gas flow to approximately 900 mL/min. Maintain this gas flow rate for 10 min while also maintaining the temperature of the residue in the flask at 160 to 166°C (320 to 330°F). If, after 10 min, dripping of condensed solvent from the delivery tube is still occurring, maintain the gas flow and temperature until 5 min after the dripping ceases in order to flush solvent vapors from the flask. In no case shall the time of flow of carbon dioxide gas be less than 15 min. At the end of this period, discontinue gas flow and heat.

9.3.1 *Alternative Procedure*—Assemble the apparatus as shown in Fig. 1 with the separatory funnel in the thermometer hole in the cork. (It may be advantageous to insert the separatory funnel in a separate hole drilled in the cork stopper.) Raise the aeration tube so that the bulb is above the surface of the solution. Fill the separatory funnel with the centrifuged solution and open the stopcock to fill the flask approximately one half full of solvent mixture. Apply low heat to the flask and start distillation. Adjust the funnel stopcock to introduce fresh solvent at a rate that will keep the flask approximately one half full during distillation, adding additional solvent mixture to the funnel until all solvent has been introduced into the distillation flask. Wash the solvent mixture container and funnel with fresh solvent to transfer all asphalt into the distillation flask. Continue distillation until the temperature reaches 135°C (275°F), at which time lower the aeration tube so that the bulb is in

contact with the bottom of the flask and introduce carbon dioxide gas at a low rate (approximately 100 ml/min) to provide agitation and prevent foaming. If foaming or bumping occurs, introduce carbon dioxide intermittently at the beginning of the distillation at a rate of 100 ml/min to prevent this. When the temperature reaches 157 to 160°C (315 to 320°F), increase the carbon dioxide gas flow to approximately 900 mL/min. Maintain this gas flow rate for 10 min while also maintaining the temperature of the residue in the flask at 160 to 166°C (320 to 330°F). If, after 10 min, dripping of condensed solvent from the delivery tube is still occurring, maintain the gas flow and temperature until 5 min after the dripping ceases in order to flush solvent vapors from the flask. In no case shall the time of flow of carbon dioxide gas be less than 15 min. At the end of this period, discontinue the gas flow and heat.

9.4 If the residue in the flask is highly viscous at 163°C (325°F), so that dispersion of the carbon dioxide in the residue is restricted and the recovered asphalt is expected to have a penetration at 25°C (77°F) of less than 30, maintain the carbon dioxide gas flow and temperature for 20 to 22 min.

9.5 The recovered asphalt can be heated to reliquefy and portions taken for penetration, softening point, ductility, ash content, and kinematic and absolute viscosity determinations as required. Ash content determinations shall be conducted on all recovered bitumens in accordance with Test Methods D 2939 and reported with other test data on the recovered asphalt. Ash contents of recovered asphalts greater than 1 % may affect the accuracy of the penetration, ductility, softening point, or viscosity tests.

NOTE 1—Determination of the weight of asphalt recovered serves as a check to assure that all of the solvent has been removed when this weight is compared with the weight of asphalt extracted as determined by Test Methods D 2172. The weight of the recovered asphalt should be corrected for the ash content determined.

## 10. Precision and Bias

10.1 *Precision*—Data from the AMRL<sup>12</sup> proficiency sample database from 1974 to 1992 was analyzed to develop estimates of test precision.<sup>13</sup> The results of the analysis are summarized below. The grades of asphalt binders included in the analysis consisted of AC-5, AC-10, AC-15, AC-20, AC-30, AC-40, AR-2000, and AR-4000. The precision estimates provided apply to unaged asphalt binder having physical test properties within the following ranges:

Penetration at 25°C (77°F)	29 to 181
Kinematic viscosity at 135°C (275°F), cSt	200 to 720
Viscosity at 60°C (140°F), P	520 to 5320

10.1.1 *Single-Operator Precision*—The figures given in Column 2 are the coefficients of variation that have been found to be appropriate for the tests and conditions described in Column 1. When performed by the same operator on the same sample in the same laboratory using the same apparatus, the difference in the results of two properly conducted tests,

<sup>12</sup> These numbers represent, respectively, the (1s %) and (d2s %) limits as described in ASTM Practice C 670.

<sup>13</sup> The data is included in a research report available from ASTM Headquarters. Request RR: D04-1008.



expressed as a percent of their mean, shall not exceed the figures given in Column 3.

Test Index	Coefficient of Variation (percent of mean) <sup>12</sup>	Acceptable Range of Two Results (percent of mean) <sup>12</sup>
<i>Single-Operator Precision:</i>		
Penetration at 25°C (77°F)	11	30
Kinematic viscosity at 135°C (275°F), cSt	9	26
Viscosity at 60°C (140°F), P	18	51

10.1.2 *Multilaboratory Precision*—The figures given in Column 2 are the coefficients of variation that have been found to be appropriate for the tests and conditions described in Column 1. When performed by different operators in the different laboratories, the difference in the results of two

properly conducted tests, expressed as a percent of their mean, shall not exceed the figures given in Column 3.

Test Index	Coefficient of Variation (percent of mean) <sup>12</sup>	Acceptable Range of Two Results (percent of mean) <sup>12</sup>
<i>Multilaboratory Precision:</i>		
Penetration at 25°C (77°F)	21	58
Kinematic viscosity at 135°C (275°F), cSt	16	46
Viscosity at 60°C (140°F), P	33	93

10.2 *Bias*—Since there is no data at this time to prepare a bias statement, no statement on bias is made.

**11. Keywords**

11.1 asphalt; bituminous mixture; distillation; solution

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