



Transport
Roads & Maritime
Services

Test method T514

Diethyl ether asphaltenes in bituminous material

NOVEMBER 2012



Revision Summary

| Ed/Rev Number | Clause Number | Description of Revision | Authorisation | Date |
|---------------|---------------|--|---------------|---------------|
| | | Reformatted and Revision Summary Added. Safety Note Added- 4(j),6(a)(d), 8(a) altered | D.Dash | Sep 1999 |
| | | Generally Revised | D.Dash | Sept 2003 |
| | | Section 6 Clause (b) and (c) Revised Clause2 Safety note altered. 4(k) altered | G.Hall | Mar 2004 |
| | | ASTM method added to scope, Section 4 (o) added, Sections 3, 5&6 revised, additions made to Section 7. | | Aug 2008 |
| Ed 1/Rev 0 | All | New Issue. Based on “Austroads” TM and the MRWA Test Method WA 326.2. Title Altered | D. Hazell | Sept 2008 |
| Ed 2/ Rev 0 | All | Reformatted RMS template | J Friedrich | November 2012 |
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Note that Roads and Maritime Services is hereafter referred to as ‘RMS’.

The most recent revision to Test method T514 (other than minor editorial changes) are indicated by a vertical line in the margin as shown here.

Test method T514

Diethyl ether asphaltenes in bituminous material

1. Scope

This test method sets out the procedure for determining the asphaltene content of bituminous material having a penetration (25°C, 100 g and 5 s) greater than 5 (see note in part 5. Preparation of Sample). It applies particularly to residual bitumen. The procedure is derived DC Broome "The Testing of Bituminous Mixtures" E. Arnold, London 1934 pp 152-3 and ASTM 3279 - 73. A reference method is published as IP 143/90.

2. Safety Notes

A poster describing the action to be taken in the event of bitumen burns must be displayed in the laboratory in the vicinity of the bitumen pouring area(s).

Use either tongs or heat resisting gloves when handling hot bitumen. Loosen or puncture lids before heating containers. Examine cold samples for signs of water. Remove all visible water. Wear spectacles when heating samples. Cleaning solvents such as toluene may be toxic, handle such solvents in a fume cupboard, and consult safety data sheet. Care must be taken when handling diethyl ether as it is highly flammable and also has a narcotic /anaesthetic effect.

3. Definition

The asphaltene content of a petroleum product is the percentage by weight excluding mineral matter, which is insoluble in diethyl ether but soluble in toluene.

4. Apparatus

- (a) A thermostatically controlled oven with good air circulation, capable of maintaining a temperature within the range of 100°C to 110°C.
- (b) A heating device, such as a hotplate.
- (c) A metal container of about one half litre capacity.
- (d) Conical flasks of 200 mL capacity.
- (e) Sintered glass crucible as described in AS 2341.8 "Determination of matter insoluble in toluene".
- (f) Reflux condensers to fit the conical flasks.
- (g) Glass rod tipped with polythene tubing, a "policeman".
- (h) A desiccator.
- (i) A balance of 100 g capacity, accurate and readable to 0.2 mg.
- (j) A thermometer reading to 110°C calibrated in 1°C divisions.
- (k) Tongs or suitable heat resistant oven gloves to handle containers of hot and molten material.
- (l) Diethyl ether Analytical Reagent (AR) grade.
- (m) Methylated spirits (commercial grade)
- (n) Steam bath
- (o) Optional – Low aromatic kerosene or decalin.

5. Preparation

Melt the sample in its original container by means of the air oven at 100°C to 110°C avoiding unnecessarily long heating. Stir thoroughly and pour off a portion of about 100 g into a metal or glass container. Heat this portion on the hotplate until sufficiently fluid to pour into the tared flask.

Note: If the material has a penetration of 5 or less or a viscosity greater than 40,000 Pa.s at 60°C, it should be cut back with 10-15% low aromatic kerosene or decalin, sub sampling temperature up to 180°C is permissible. If so it will be necessary to verify actual non-volatile content at the point of 6(b) by doing a T511 test and scaling the mass of bitumen A to be the actual bitumen present in the sub sample taken.

6. Procedure

- Carry out the determination in duplicate.
- Weigh to the nearest 1 mg, 2 ± 0.1 g of a representative portion of the sample into a 200 mL conical flask. Then heat the flasks on a hotplate to melt the bitumen and spread it evenly over the bottom of the flask. Use the minimum amount of time and heating to achieve this.
- Add 100 mL of Diethyl Ether to the flask. Warm the flask and contents under a reflux condenser on top of a warm steam bath with gentle swirling, until the bitumen has dispersed. Allow the flask to cool and stand tightly corked for 18 hours to 24 hours in the dark.
- Filter the contents of the flask through a prepared and weighed sintered glass crucible (prepare by washing and a final rinse with Methylated spirit and drying and weighing the same as (f)), dislodging mechanically any material adhering to the flask with the "policeman" and wash it into the crucible with diethyl ether.
- Wash the filter with further 20 mL portions of diethyl ether until the filtrate is a pale straw colour, and then wash with 20 mL portions of boiling Methylated spirits until the last 20 mL of filtrate on evaporation of a small volume, say 5 mL, leaves no perceptible residue on a watch glass. (When dried for 30 minutes at 100 - 110°C leaves less than 5 mg of residue.)
- Dry the crucible in the oven at 100°C to 110°C for 1 hour. Cool in a desiccator and weigh to the nearest 1 mg.

7. Calculation and Reporting

The mass of material retained in the crucible, calculated as a percentage of that part of the sample soluble in toluene as determined by AS 2341.8 "Determination of matter insoluble in toluene", is reported as the diethyl ether asphaltenes content.

Calculate the percentage diethyl ether asphaltene present from the following equation.

$$\% \text{ diethyl ether asphaltenes} = \frac{B - \frac{AC}{100}}{A \left(1 - \frac{C}{100}\right)} \times 100$$

- Where
- A = mass of bitumen taken at 6(a)
 - B = apparent mass of asphaltenes at 6(c)
 - C = the % insoluble in toluene as determined by AS 2341.8
 - D = 100 – mass loss from T511 test
 - E = mass of hard bitumen present in the cut-back sub sample

If the material has been cut back with low aromatic kerosene or decalin then A should be replaced with E

$$E = A D$$

Where D = 100-mass loss on T511 test.

Report the result rounded to the nearest 0.1%