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1. **Confirmation of the Binder Class and Type**
   - Binder conformance to the required PG specification

2. **Binder Ageing**
   - Determining the rate of short term and long term ageing

3. **Forensic Investigations**
   - The extent of binder influence on premature failure of an asphalt surfacing (Deformation or rutting, cracking, ravelling)

4. **Determination of binder stiffness**
   - Can be used in pavement performance prediction models

*In other words – the test is crucial to the pavement engineering industry!*
The results from three random national laboratories using identical samples but different recovery methods:

<table>
<thead>
<tr>
<th>Property</th>
<th>Lab 1</th>
<th>Lab 2</th>
<th>Lab 3</th>
<th>Test Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ash Content (%)</td>
<td>15.5</td>
<td>0.22</td>
<td>1.7</td>
<td>ASTM D482</td>
</tr>
<tr>
<td>GC Area Counts (solvent)</td>
<td>&gt;74000</td>
<td>0</td>
<td>22 900</td>
<td>BE-TM-BINDER-5-2010</td>
</tr>
<tr>
<td>Recovered Penetration (dmm)</td>
<td>55</td>
<td>28</td>
<td>35</td>
<td>EN 1426</td>
</tr>
</tbody>
</table>

The variation between laboratories is unacceptable and underlines the need for a single national recovery method.
Binder Recovery Stages

3-Step process

• Binder Extraction – binder is dissolved with a suitable solvent

• Removal of Aggregate Fines – attained using centrifuging, filtration or a combination of both

• Solvent Removal – distillation (Abson) or rotary Evaporation
Technical Performance of the Binder Recovery Process

Defined as:
- A measure of the accuracy – how close do the recovered binder properties reflect the binder properties prior to recovery
- A measure of the repeatability of the process (Use the COV as a measure)

Factors affecting Technical Performance
- The type and age of the binder – (e.g. aromatic high molecular weight bitumen vs a waxy-type bitumen vs highly aged binder)
- The type of aggregate – Some absorptive limestone aggregates can affect the technical performance of a particular solvent.
- The specific binder property used to define the technical performance, e.g. softening point vs G*
- The extraction and recovery method used
Sources of Poor Technical Performance

- The hardening effect of the solvent on the recovered binder
- Incomplete removal of the solvent from the recovered binder
- Incomplete removal of all the binder from the aggregate
- Incomplete separation of aggregate fines from the binder
**Extraction Process**

**Historical Development**

- In 1903, Dow performed the first reported extraction and recovery using \((\text{CS}_2)\)

- Abson method in the 1930’s was the first method to gain widespread acceptance - Benzene.

- Once the toxicity of benzene was understood, it was replaced in the 1950’s and 1960’s by a range of mostly chlorinated solvents still widely used today.
  - Trichloroethylene (TCE)
  - 1,1,1-trichloroethane (TCA)
  - Methylene chloride (or dichloromethane (DCM))
  - Chloroform (or trichloromethane (TCM))
  - Carbon tetrachloride
  - Toluene (methylbenzene)
Most Popular Extraction Test Methods Internationally

- ASTM D2172 (Method A) – A cold extraction process followed by centrifuge - trichloroethylene, n-Propyl Bromide or methylene chloride

- ASTM D2172 (Method B) – A hot reflux (Soxhlet) extraction process – as above

- California Test 362 – Method of determining asphalt content of bituminous mixtures by vacuum extraction – 1,1,1 trichloroethane

- AASHTO T319 – (developed as part of SUPERPAVE) - n-Propyl Bromide or 85/15 toluene/ethanol

- EN 12697-3 –Bitumen recovery: Rotary evaporator - dichloromethane (methylene chloride) or other suitable solvents including toluene, tetrachloroethylene, trichloroethylene, xylene, 1,1,1-trichloroethane and benzene.
Extraction Process – Solvent choice

To choose an effective solvent, Consider:
  • Technical Performance (esp Solvent Hardening, extent of binder dissolution)
  • Safety (chlorinated solvents, n-propylbromide)
  • Environment (chlorinated solvents)

Binder Dissolution (Incomplete removal of binder from the aggregate):
  • A number of researchers have shown that the binder is never completely removed from the aggregate, regardless of the solvent used for extraction.
  • Incomplete extraction causes the binder content of hot mix asphalt to be underestimated by up to 0.5%.
  • The solvent having the highest solvency power (especially for aged binder) is pyridine, which is highly toxic. To determine the extent of binder dissolution, compare your solvent results with pyridine
Extraction Process – Solvent choice

Solvent hardening:
• Increases of 5 – 300% in binder stiffness has been reported
• Solvent type (Carbon tet > Other chorinated > Toluene > Benzene)
• Time
• Temperature (ASTM D2172 (Method B) and California Test 362 were found to employ higher temperatures)
• Light
• Oxygen
• Binder-aggregate types

Taking all these factors into consideration – CSIR has found 85/15 toluene/ethanol to be the best choice. Recommended time = 8 hours
Aggregate Fines Removal

Methods:
- ASTM D 1856 specifies centrifuging the extracted binder solution prior to distillation.
- California Test 362 specifies the addition of Celite to the extracted binder solution prior to distillation in order to bind the fines.
- AASHTO T319 specifies two filtration steps of the extracted binder solution prior to centrifuge and distillation.
- EN 12697-3 gives the option of separation by centrifuging or filtration

Extent of Fines Removal:
- In general, a solvent with a higher density would tend to retain more fines
- A higher fines content within the recovered binder – results in an increase in the stiffness of the recovered binder
- The fines content of the recovered binder can be measured via the ash content (ASTM D482)

Recommended extraction method - ASTM D2172 (Method A)
Solvent removal

Required Principle:
• Recover in least amount of time and at lowest temperature practical to achieve the required technical performance.

Most Popular Recovery Test Methods Internationally
• ASTM D1856 – The Abson distillation recovery at atmospheric pressure and using a carbon dioxide carrier gas
• ASTM D5404 – Distillation recovery under reduced pressure using the Rotary Evaporator Apparatus.
• AASHTO T319 – (developed as part of SUPERPAVE)
• EN 12697-3 – Bitumen recovery: Rotary evaporator
Incomplete removal of the solvent from the recovered binder:

- Residual solvent remaining in the recovered binder after solvent recovery results in a reduced stiffness for the recovered binder.
- A solvent with a higher boiling point tends to result in the less efficient removal of the solvent from the binder.

Detection of Residual Solvent

- Infrared Spectroscopy
- Gas chromatography
- High performance liquid chromatography
- Gel permeation chromatography
Abson vs Rotavapour – lack of consensus

- Residual solvent - Mixed research results
- Time - Abson takes considerably longer (2-5 times)
- Ease of execution – The Abson method is complex; requiring skill and technical expertise. The method remains widely used because it can be more accurate given the recovery conditions can be adjusted for the binder type in question. Consequently, it is ideally suited for research or forensic investigations, especially when the type of asphalt binder is unknown.

Recommended that both methods be adopted nationally – Rotavapor suited to QC labs and Abson suited to research labs
Maintain quality of results by:

- Internal periodic evaluation of standard samples. ie manufacture a mix with a known bitumen at low temperature to minimise ageing (Mixing temperature 100°C) using minimum mixing time.

- External Participation in a national proficiency scheme (round robin testing)

- Report the recovered binder results along with the residual solvent content and ash content
Presentation is based on Sabita Manual 29, which was authored by myself and Georges Mturi – consult for more information

Manual 29: Guide to the safe use of solvents in a bituminous products laboratory

The recommended national test method can be requested from Georges Mturi.
Thank you