ADDRESSING AGEING CHARACTERISTICS OF BITUMINOUS BINDERS IN EUROPE

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CEN TC336 WG1

ABSTRACT

Simulating the ageing of bituminous binders in European laboratories is harmonised by the introduction of European standards.

For short term ageing, the EN 12607 series describes three different conditioning methods using the RTFOT, the TFOT and the RFT. For long term ageing, the conditioning is described in EN 14769 for PAV and EN 15323 for RCAT. Other short and long term ageing protocols have been proposed, but the methods are not yet standardised in Europe.

Task Group 3 of CEN/TC336 WG1 has been re-convened to review the existing harmonised conditioning methods and evaluate other protocols. The aim is to produce a recommendation of which method (or methods) should be considered for European Standardisation and possible introduction into a new European performance-related binder specification.

It is recognised that different types of binder will need different approaches. Short term ageing protocols for PMBs and warm mix binders have to be considered and long term ageing protocols for all binders need to be established.

TG3 is gathering all the available knowledge to enable the review of existing standards and the evaluation of other conditioning protocols. This paper will describe the background of the process and provide information on the methods under consideration and the discussion regarding their suitability for different binders.

Keywords: Ageing, Best Available Technology, Comité Européen de Normalisation, Standardisation
1. BACKGROUND

The European Committee for Standardisation is responsible for the development of harmonised European Standards and technical specifications. Product standards and test methods should all be European harmonised.

For this purpose the European Committee introduced Technical Committees for each subject. Bituminous Binders are handled by TC 336 and Asphalt is handled by TC 227. The word Asphalt in Europe means the product consisting of bituminous binder, aggregate and filler. Bituminous binders are (unmodified or modified) products from the refining of crude oil and also include bitumen emulsions.

There are two Working Groups associated with TC 336. WG1 is working on binders used in hot mix paving applications and WG2 is handling all the emulsions and cut-backs. CEN TC336 WG1 carries out its work through a number of small Task Groups. TG3 is working on short term and long term ageing protocols for bituminous binders.

Figure 1. CEN TC 336 structure with Working Groups and Task Groups.

This paper describes the current work of TC 336, WG1, TG3 and the issues concerning the ageing of bituminous binders are discussed.

2. INTRODUCTION

The first European standard for paving grades (EN 12591) was published in 1999. In this product specification, paving grade bitumen was addressed by characteristics for each type of penetration bitumen grade. The product standard was a harmonisation of all the European countries represented in CEN and some compromises were necessary. All the characteristics in the EN product standard are measured according to an EN for a test method. CEN is responsible for the normalisation of all the standards about bituminous binders and members represent the standardisation bodies of each country. The scope for CEN TC336 WG1 TG3 is to evaluate and update procedures for short term and long term ageing/conditioning on bituminous binders. The aim is to produce a recommendation of which method (or methods) should be considered for European Normalisation and possible introduction into a new European Performance-Related binder specification. Table 1 shows the current ENs for conditioning of bituminous binders.
Table 1. European standards for conditioning bituminous binders

<table>
<thead>
<tr>
<th>Description</th>
<th>Standard</th>
</tr>
</thead>
<tbody>
<tr>
<td>Short Term Ageing conditioning</td>
<td></td>
</tr>
<tr>
<td>Rolling Thin Film Oven Test (RTFOT)</td>
<td>EN 12607-1</td>
</tr>
<tr>
<td>Thin Film Oven Test (TFOT)</td>
<td>EN 12607-2</td>
</tr>
<tr>
<td>Rotating Flask Test (RFT)</td>
<td>EN 12607-3</td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td>Long Term Ageing conditioning</td>
<td></td>
</tr>
<tr>
<td>Pressure Ageing Vessel (PAV)</td>
<td>EN 14769</td>
</tr>
<tr>
<td>Rotating Cylinder Ageing Test (RCAT)</td>
<td>EN 15323</td>
</tr>
</tbody>
</table>

The work performed in USA under the Strategic Highway Research Program (SHRP) was of excellent quality and was referenced when preparing European product and test standards. The well-known Rolling Thin Film Oven Test (RTFOT) was developed in the USA in 1962 [1] and introduced as a standard, number ASTM D 2872. The method was developed using paving grade bitumen and a relationship was established between bitumen recovered from asphalt produced in the asphalt plant and bitumen conditioned in the RTFOT in the laboratory.

In the current edition of ASTM D 2872 this relationship is still described in the paragraph **Significance and use** but there is no reference to the research (remember US word for bitumen is asphalt):

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4. **Significance and Use**

4.1 This test method indicates approximate change in properties of asphalt during conventional hot-mixing at about 302°F (150°C) as indicated by viscosity and other rheological measurements. It yields a residue which approximates the asphalt condition as incorporated in the pavement. If the mixing temperature differs appreciably from the 302°F (150°C) level, more or less effect on properties will occur. This test method also can be used to determine mass change, which is a measure of asphalt volatility.

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European test method EN 12607 consists of three parts. Part 1 is the RTFOT as shown in table 1. The relationship of the RTFOT conditioning to the binder hardening in the asphalt plant is also included in the scope of the European standard. Again no reference to the original work or other evidence is given in the standard.

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1 **Scope**

This European Standard specifies a method for measuring the combined effects of heat and air on a thin moving film of bitumen or bituminous binder simulating the hardening that a bituminous binder undergoes during mixing in an asphalt mixing plant.

The method described is not applicable to some modified binders or to those where the viscosity is too high to provide a moving film. The sample may creep out of the glass container and flow on the heating elements of the oven during testing.

The method is referred to as RTFOT, i.e. Rolling Thin Film Oven Test.

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The scopes of all three parts of EN 12607 include the statement that the procedure simulates ageing during mixing of asphalt. Laboratory conditioning and characterisation of a binder is not necessarily the same as the performance of the binder in different applications. This is particularly relevant to warm and cold mix binders processed in asphalt plants at a temperature of 130 °C or lower where the relationship to RTFOT conditioning at 163 °C is not established. Research shows that aggregate and filler influence the ageing process [2] and this is not taken into account in the laboratory binder conditioning.
In the current 5-year review of EN 12607, the relation between binder ageing conditioning and the binder ageing during asphalt production should be carefully considered.

The use of a standardised conditioning protocol for both short term and long term ageing for all types of binder (paving grade, Polymer Modified Bitumen (PMB), wax modified binders, etc.) should be evaluated. The relationship between laboratory conditioning and asphalt production and service is not thoroughly investigated or proven for all binders and performance. However, the number of binders requiring an alternative may be very small.

3. AGEING CONDITIONING TEST METHODS

In the European bitumen product standards, only short term conditioning is described. At national level, conditioning for long term ageing is prescribed in some countries, usually informative for the purpose of gathering information. The protocols vary and RTFOT +PAV, TFOT + PAV, RCAT, 3 x RTFOT and modified RTFOT are all used in different countries. These protocols are usually called up for specific binders and work has been done to demonstrate equivalence with RTFOT + PAV[1] and RCAT + RCAT[3] for the particular binders.

Short and long term conditioning should be part of a type testing regime and separate from the normal day to day quality control. Most binder characteristics can be determined from the properties after short term ageing and long term ageing is appropriate only if it introduces new information. The Eurobitume/CEN Data Collection [4] showed that normal paving grade un-aged characteristics correlate with short term aged and long term aged characteristics. For modified binders this correlation is not proven and therefore it can be concluded that characterisation of special binders after long term conditioning reveals relevant new information. Bahia [5] introduced an upgrade of simple and complex binders for PG grading in 1998 based on the gaps identified in the PG system used in the US.

3.1 Short term conditioning

TG 3 of TC 336 WG1 has identified short term conditioning methods available in Europe. The RTFOT is the most commonly used protocol and is called up in the bitumen product standards. Other conditioning protocols are available around the world, such as the MRTFO used in Australia, described in AG-PT T104, but these are is not listed because there is little experience with European binders.

The advantages and disadvantages of the short term conditioning protocols relevant for Europe are listed in the tables 2 and 3.
Table 2. Advantages of European conditioning for short term binder ageing.

<table>
<thead>
<tr>
<th>Short term ageing</th>
<th>Standard</th>
<th>Advantage</th>
</tr>
</thead>
<tbody>
<tr>
<td>RTFOT</td>
<td>EN 12607-1</td>
<td>Equipment widely available</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Conditioning takes 1 ¼ hours</td>
</tr>
<tr>
<td>TFOT</td>
<td>EN 12607-2</td>
<td>Equipment is simple and available in most laboratories</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Same sample discs as PAV</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Method allows different temperatures for different binders</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Binder recovery after conditioning is 90%</td>
</tr>
<tr>
<td>RFT</td>
<td>EN 12607-3</td>
<td>Equipment is standard laboratory equipment</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Lots of experience with the conditioning method</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Dynamic conditioning method</td>
</tr>
<tr>
<td></td>
<td></td>
<td>100 g bitumen aged in one flask</td>
</tr>
<tr>
<td>RCAT</td>
<td>EN 15323</td>
<td>Same equipment as long term ageing RCAT</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Dynamic conditioning method</td>
</tr>
<tr>
<td></td>
<td></td>
<td>500 g bitumen aged in one flask</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Sample after short term ageing RCAT can be used directly for long term ageing</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Binder recovery after conditioning is 90%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Due to steel rod less viscosity dependence</td>
</tr>
<tr>
<td>modified ageing</td>
<td>UK DfT,</td>
<td>Same basic equipment as RTFOT</td>
</tr>
<tr>
<td>RTFOT</td>
<td>Specification</td>
<td>Suitable for all grades, hard and PmB</td>
</tr>
<tr>
<td></td>
<td>for Highway</td>
<td>Conditioning 45 minutes</td>
</tr>
<tr>
<td></td>
<td>Works</td>
<td>Dynamic conditioning method continually mixed</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Less viscosity dependence due to steel screw</td>
</tr>
</tbody>
</table>
Table 3. Disadvantages of European conditioning for short term binder ageing.

<table>
<thead>
<tr>
<th>Short term ageing</th>
<th>Standard</th>
<th>Disadvantage</th>
</tr>
</thead>
<tbody>
<tr>
<td>RTFOT</td>
<td>EN 12607-1</td>
<td>Representative for 90% of asphalt production (10% excepted e.g. binders for low temperature asphalt and highly viscous binders)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>280 g bitumen divided into 8 flasks</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Loses almost 25 m% of bitumen due to 8 different flasks</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Cleaning of bottles is time consuming</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Viscosity depending conditioning</td>
</tr>
<tr>
<td>TFOT</td>
<td>EN 12607-2</td>
<td>Static conditioning method and therefore surface hardening and doubt on ageing regime</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Only three discs in every test, 150 g binder</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Conditioning takes 5 hours</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Only experience with soft grades and industrial bitumen</td>
</tr>
<tr>
<td>RFT</td>
<td>EN 12607-3</td>
<td>Conditioning takes 2.5 hours</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Less than 100 g bitumen for testing</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Viscosity depending conditioning</td>
</tr>
<tr>
<td>RCAT</td>
<td>EN 15323</td>
<td>Conditioning takes 4 hours</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Sample holder is heavy and needs special attention</td>
</tr>
<tr>
<td>modified ageing</td>
<td></td>
<td>Equipment is not widely spread in Europe</td>
</tr>
<tr>
<td>RTFOT</td>
<td>UK DfT, Specification for Highway Works</td>
<td>Uses other flasks than regular RTFOT</td>
</tr>
</tbody>
</table>

Short term ageing protocols all have advantages and disadvantages, but RTFOT is established as suitable for characterisation of most binders and there are many years of experience. RCAT produces a greater amount of binder and can also be used for long term ageing.

An alternative may be needed for a few highly viscous binders, and warm and cold mix binders respectively for warm and low temperature asphalt will probably need to be evaluated as part of the asphalt mix.

3.2 Long term conditioning

No description is made in any of the current bitumen product standards for long term ageing for paving applications in Europe. For paving grade binders, the characteristics of the binder can be determined satisfactorily after short term ageing but for complex modified binders, more information may be needed. The SHRP in the US introduced characterisation after RTFOT + PAV in the PG grading system several years ago and there is now a lot of experience. Some European countries have introduced binder testing after long term ageing and several different protocols are in use. The question remains open as to whether this is for characterisation of binders or whether this is required for a particular application. Generally, before a protocol has been introduced work has been done to compare the ageing with ageing after RTFOT + PAV.

Long term ageing is related to the asphalt application and is also influenced by the aggregate and filler. Due to a higher void content, open graded asphalt where voids are interconnected will show different behaviour in time (binder will age faster and through all the thickness of the layer) than a dense asphalt application (binder aging only on the surface).

Another issue is the combination of short term and long term conditioning. Asfaltos Españoles S.A. presented research [6] on the differences between RTFOT + PAV and TFOT + PAV for penetration grade bitumen. They showed that the long term conditioning in the PAV over-ruled the difference in the short term conditioning between RTFOT and TFOT. The advantage was the use of the same sample collectors for TFOT and PAV, so no extra handling is necessary to perform the long term ageing after short term ageing.

The report on phase 1 of the BiTVal project [7] gives an overview of all the binder aging conditioning method (description, precision and relationship with other binder regimes) and a state of the art of the correlation of laboratory...
ageing with field performance. In this report PAV test and RCAT were compared (advantages, disadvantages) and in terms of long-term ageing, no one test seems to be satisfactory for all cases. Other researchers [8] investigated the conditioning of PAV for 25 hours instead of RTFOT + PAV for 20 hours for penetration grade bitumen and concluded that results are comparable.

### Table 4. Advantages of European conditioning for long term binder ageing.

<table>
<thead>
<tr>
<th>Long term ageing</th>
<th>Standard</th>
<th>Advantage</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>PAV</strong></td>
<td>EN 14769</td>
<td>Equipment widely used</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Same sample discs as TFOT</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Variability of conditioning</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Static conditioning method reflecting field conditioning for most binders</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Using all discs ends up with almost 500 g bitumen</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Binder recovery after conditioning is 90%</td>
</tr>
<tr>
<td><strong>RCAT</strong></td>
<td>EN 15323</td>
<td>Same equipment used for short term ageing</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Suitable conditioning reflecting field performance for most binders and suitable for mastic conditioning</td>
</tr>
<tr>
<td></td>
<td></td>
<td>500 g bitumen aged in one flask</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Binder recovery after conditioning is 90%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Dynamic conditioning method under oxygen</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Less viscosity dependence due to steel rod</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Variability of conditioning</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Small round robin has been carried out</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Risk assessment report available</td>
</tr>
<tr>
<td><strong>3x RTFOT</strong></td>
<td>Austrian regulations</td>
<td>Used in Austria for paving grades</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Demonstrated equivalence with PAV for 70/100 paving grade</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Test performed in one day</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Equipment is known and available</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Dynamic conditioning method</td>
</tr>
<tr>
<td><strong>HiPAT</strong></td>
<td>EN 14769 (PAV at 85 °C and 65 hours)</td>
<td>Equipment is widely used</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Low temperature static conditioning</td>
</tr>
<tr>
<td><strong>LT-RFT</strong></td>
<td>Description available</td>
<td>Same equipment used for short term ageing</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Equipment is known and available</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Dynamic conditioning method under oxygen</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Less viscosity dependence due to steel balls</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Variability of conditioning</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Small round robin has been carried out</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Risk assessment report available</td>
</tr>
<tr>
<td><strong>Modified ageing RTFOT</strong></td>
<td>UK DfT, Specification for Highway Works</td>
<td>Equipment is known and available</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Dynamic conditioning continually mixed</td>
</tr>
</tbody>
</table>
Table 5. Disadvantages of European conditioning for long term binder ageing.

<table>
<thead>
<tr>
<th>Long term ageing</th>
<th>Standard</th>
<th>Disadvantage</th>
</tr>
</thead>
<tbody>
<tr>
<td>PAV</td>
<td>EN 14769</td>
<td>With current procedure several RTFOT short term ageing runs are required</td>
</tr>
<tr>
<td></td>
<td></td>
<td>High pressure procedure, so a compressor or compressed air is needed and careful attention are required</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Precautions need to be taken for overnight runs</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Currently recommended after RTFOT short term ageing</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Static test may not be suitable for some PMBs</td>
</tr>
<tr>
<td>RCAT</td>
<td>EN 15323</td>
<td>Only a few laboratories have equipment</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Conditioning takes 140 hours, precautions need to be taken for overnight/weekend testing</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Need of oxygen devices</td>
</tr>
<tr>
<td>3x RTFOT</td>
<td>Austrian</td>
<td>Austrian regulations, Not an EN</td>
</tr>
<tr>
<td></td>
<td></td>
<td>280 g bitumen divided into 8 flasks</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Loses almost 25 m% of bitumen due to 8 different flasks</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Cleaning of bottles is time consuming</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Viscosity depending conditioning</td>
</tr>
<tr>
<td>HiPAT</td>
<td>EN 14769</td>
<td>Only used in UK</td>
</tr>
<tr>
<td></td>
<td>85 °C and 65 hours PAV</td>
<td>Only used in UK</td>
</tr>
<tr>
<td>LT-RFT</td>
<td>Description available</td>
<td>Not an EN standard</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Less than 100 g bitumen after conditioning</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Limited experience available</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Precautions need to be taken for overnight runs</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Due to use of silicon oil and pure oxygen the risk is high (Risk assessment report)</td>
</tr>
<tr>
<td>Modified ageing RTFOT</td>
<td>UK DfT, Specification for Highway Works</td>
<td>Uses other flasks than regular RTFOT</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Conditioning takes 22 hours at 135 °C, precautions need to be taken for overnight testing.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Limited amount of bitumen after conditioning</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Cleaning of bottles is time consuming</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Limited experience available</td>
</tr>
</tbody>
</table>

4. CONCLUSIONS

The scope for CEN TC336 WG1 Task Group 3 is to evaluate and update procedures for short term and long term ageing/conditioning of bituminous binders in the laboratory.

The aim is to review European standards for the methods in use, and to produce a recommendation of which regime (or regimes) should be considered for possible introduction into a new European Performance-Related binder specification.

It is recognised that different types of binder will need different approaches. Short Term Ageing protocols for PMBs and warm mix binders need to be considered and Long Term Ageing protocols for all binders need to be established.

4.1 Short term ageing
The EN 12607 series describes three different conditioning methods using the RTFOT, the TFOT and the RFT. Short term ageing commonly uses the RTFOT in European bitumen specifications and, in theory, the protocol could be carried out at different (equivalent viscous) temperatures to accommodate all binders. In the scopes of all three parts of EN 12607 there is a statement that the procedure simulates ageing during mixing of asphalt. RTFOT may simulate mixing of asphalt with regular paving grade bitumen, but not necessarily when using e.g. binders with high or low viscosity, as the temperature of RTFOT as given in EN 12607-1 is fixed at 163 °C.

It is considered that existing protocols are good enough for paving grade binders, but work is needed for high viscous binders, e.g. hard grades and PmB, and warm mix binders.

4.2 Long term ageing

TG3 has gathered information and reviewed the advantages and disadvantages of the available Long Term Ageing protocols used in Europe and conditioning is described in EN 14769 for PAV and EN 15323 for RCAT. Other long term ageing protocols have been proposed, but the methods are not yet standardised in Europe.

There has been discussion about the practicality and the relevance of the different long term ageing protocols used in Europe. It is recognised that some methods are simpler and produce more binder for further testing, but the protocol should give results representative of ageing in the field.

TG3 will now work on a recommendation for a reference method.

4.3 Ageing protocols for warm mix binders

There are several different techniques for the production of warm mix binders. In some cases the binder is pre-blended and can be tested as supplied. In other techniques, modifiers are added at the asphalt mixer or a foaming process is used and so the binder cannot be tested except as recovered from the mix. This introduces a further variable because the recovery method may influence the properties. It may be more appropriate to evaluate the asphalt mixture for asphalt constructions produced by modification/foaming of the binder in the mixer.
LITERATURE

7] Nicholls et al., BiTVal – Analysis of available data for validation of bitumen tests, report on phase 1 of the BiTVal project, Fehrl, download from http://bitval.fehrl.org/