Eurobitume TF Data Collection

Position Paper on Test Methods used during the Data Collection

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Executive Summary

Eurobitume's Task Force Data Collection has created a comprehensive database of bitumen properties for many commercially available bitumens in Europe. The database includes paving grades, hard paving grades, PMB and special binders that were commercially available at the time of the project.

TF Data Collection was established in response to a request from CEN to collect test data on bituminous binders for the purpose of development of new performance related specifications. This Data Collection is not giving any statements on how performance related specification should look like but is addressing binder tests only.

The purpose of the project was to allow assessment of the various test methods proposed as candidates to measure properties which may be suitable for future bitumen specifications. Many of the traditional test methods were developed for paving grade and hard paving grade bitumen, but have demonstrated deficiencies when measuring the characteristics of PMB.

The outcome of the data collection supports positions taken earlier by bitumen industry representatives in the CEN specification discussions with hard evidence, e.g. on the applicability and limits of test methods.

Simple tests can and should be used for simple binders i.e. Normal paving grades and Hard grade bitumens. The introduction of more complex tests for simple binders should not be considered unless they provide substantial improvement versus today. Needle penetration is a suitable test for all types of binders, but the interpretation of ring and ball softening point is different for paving grade and hard paving grades than for PMB.

More complex binders require more tests. Dynamic Shear Rheometer (DSR) equipment offers the possibility to replace several traditional test methods for PMB, e.g. ring and ball softening point, or dynamic viscosity at 60°C, but parameters and limits still need to be agreed on. DSR also offers opportunities for testing simple binders, but this is not the recommended route as simple tests are satisfactory.

For PMB there is a need to measure high temperature properties in a better way than traditional tests allow. The determination of EVT1 from LSV-testing in DSR might offer a solution, but because the method and the standard are relatively new, further evaluation is necessary.

The Dynamic Shear Rheometer is considered to be an important tool for testing in future, especially for modified binders. Some test methods do not appear to add value: e.g. Zero Shear Viscosity.

None of the test methods measuring low temperature properties (Fraass Breaking Point and BBR) are completely satisfactory the way they are currently used.

Some of the correlations between properties reported in the BiTVal Phase 1 Report have not been supported by the data collection project. The database will help discussions on these issues.

Introduction

Eurobitume's Task Force Data Collection has created a comprehensive database of bitumen properties for many of the commercially available bitumens in Europe. The database includes paving grades, hard paving grades, PMB and special binders that were commercially available at the time of the project.

The purpose of the project is to allow assessment of the various test methods proposed to measure properties which may be suitable for future bitumen specifications. It is recognised that the binder properties alone do not determine pavement, or even mixture performance. Other parameters, such as aggregate characteristics, mix design, manufacture and laying are also considered important. Future specifications must ensure that bitumens which are currently available and performing satisfactorily are not excluded and also that innovation is not discouraged.

Eurobitume has compiled a database comprising 146 unique datasets representing commercially available binders including 69 paving grades, 58 Polymer Modified Binders (PMB), 15 hard paving grades and 4 special binders, i.e. those not supplied to an existing European Specification. The datasets were collected between 2006 and 2007.

Background

TF Data Collection was established in response to a request from CEN to collect data on bituminous binders for the purpose of development of new performance related specifications. This report does not provide any statements on how performance related specifications should be structured, but is addressing binder tests only.

The European Standards EN 12591, EN 13924 and EN 14023 are based on various national standards which were in existence when the process of harmonisation started, i.e. in 1990. These standards are based on so-called "empirical" specifications and are, by common consent, reasonably successful and adequate for use in Europe for most binders. The relationship of many of the test methods to performance is fairly well understood, but they cannot be said to be directly performance related.

The next step in the process of European Standardisation is to progress to the development of new specifications which are more-directly performance-related. It will require agreement to be reached on the properties to be specified and the test methods to measure them. It will also require agreement to be reached on how best to differentiate the specifications to allow for differing climate and traffic.

What was done, issues encountered?

Eurobitume members used the template developed by CEN TC336 WG1 *(Annex 1)* and carried out tests on binders from their portfolio. The process was complicated by the need to ensure that members could submit the data confidentially, to ensure that the identity of the binders submitted could not be determined once data were in the database. This was achieved by using an independent contractor to receive the data and enter it into the database.

Data integrity was identified as an issue from early in the process. Due to the necessity to maintain confidentiality, correction of erroneous data once entered into the database was problematic. Participants were able to identify their own data within the database, but the requirement to maintain confidentiality meant that they needed to request the contractor to delete erroneous datasets and replace them with corrected versions. This led to strict version control being necessary to ensure that the numerous corrections necessary were incorporated properly. Examples of commonly encountered problems were;

- Wrong units being used, e.g. g/ml instead of kg/m³.
- Incorrect binder type being selected, e.g. paving grade bitumen rather than PMB.
- Typographical errors, e.g. omission of decimal point.

For future versions Eurobitume concluded that the template should incorporate limit values for data entries in order to limit a number of the most commonly encountered problems.

At the time of writing it is considered that the data contained within the database are correct in respect of reported values and that no transcription errors are included. However, the issue of data integrity is likely to be encountered for future versions of the database and in future data collection projects and has the potential to significantly diminish the usefulness/applicability of the database.

Use of the database

As previously stated, the data collection project was devised to assess candidate test methods for the development of performance related specifications. The present position paper has been developed to present the views of Eurobitume on the suitability of test methods for future specifications, taking into account the interpretation of the results and the experience obtained with carrying out the tests.

Caveat

During the analysis of the database a number of good correlations were identified between properties that are not fundamentally related, e.g. for paving grade bitumen a strong correlation was identified between Needle Penetration and Softening Point. The binders tested during this project constitute a reasonable subset of commercially available products, but certain classes, e.g. paving grade bitumen can be assumed to comply with existing European Specifications. Consequently, for the population in the database, the fact that certain properties are required to comply with specification limits means that the samples were not random. Therefore correlations can be anticipated that are artifacts of the sample population, rather than genuine correlations between test methods. In this paper we have commented under each test method whether we believe a correlation to be a consequence of the specification, or a genuine relationship between a given property and another property.

European Test Methods are subject to revision on a regular basis. During the period of this project a number of the test methods are known to have undergone revision, some of which will make comparison of test results that use different versions of a test invalid. The list of test methods used and the version of the standard are listed in *Annex 2.* Developments on test methods or on new tests in Europe, or in other areas, were not assessed during this project.

The Eurobitume project only considered binder test methods, therefore the database should only be used to evaluate properties of binders and cannot, at this time, provide information on mixture or pavement properties.

Statistical analysis

In order to provide the statements on test methods used during the data collection, practical experience with test methods was collected in a systemic approach. Additionally, correlation checks were undertaken for all available test methods. The results are listed in tables in the respective chapters. In order to avoid duplication, correlations are only stated for one of the two test methods reviewed.

The statistics were carried out based on 90% reliability and only where at least ten data points were available to be checked. Correlation between test results was checked for each of the four different types of binder and for groups of them. However, only those results are given where most of the binders are included: e.g. if a correlation was determined for all binders but also for paving grade bitumen only, only the correlation result for all binders is given.

TF Data Collection considered that a correlation existed if $R^2 \geq 0.9$ for the regression graph determined.

Glossary

BiTVal	Bitumen Test Validation
BBR	Bending Beam Rheometer
CEN	Comité Européen de Normalisation (European Committee for Standardization)
CPD	Construction Products Directive
DSR	Dynamic Shear Rheometer
EN	European Standard
EVT	Equi-Viscous Temperature
FD	Force Ductility
G*	Complex Shear Modulus
LSV	Low Shear Viscosity
LTA	Long Term Ageing
NPG	Normal Paving Grades
PAV	Pressure Ageing Vessel
Pen	Penetration
PMB	Polymer Modified Bitumen
R&B	Ring and Ball
RCAT	Rotating Cylinder Ageing Test
RTFOT	Rolling Thin Film Oven Test
SP R&B	Softening Point Ring and Ball
STA	Short Term Ageing
TS	Technical Specification
WI	Work Item

Position towards individual Test / Calculation Methods or Procedures

Part 1 - Conventional Characteristics

1.1. Nominal Penetration Range

General Comments: It is not a test but an indicator for conventional bitumen (paving grades) and currently used to differentiate between and name the grades.

Suitability for future specification: Not relevant, because nominal penetration range is a designation and not a test. However, this is historically used for designation of conventional binders.

1.2. Needle penetration [EN 1426]

General Comments: Needle penetration is a well known and established test which is generally applicable.

Precision: Precision data are stated in the standards. However, a number of Round Robin tests suggest that, in practice, the precision is not as good as stated [Ref. *ESSAIS CIRCULAIRES 2007 PROGRAMME N° 86, « BITUMES »: BNPé – Département Produits et Méthodes, Septembre 2007*].

Timing: Including preparation approximately 3 h.

Comments from BiTVal: For unmodified bitumens, the penetration test correlates well with the stiffness of the bitumen measured, using the DSR, at the same temperature (25°C) and at a frequency of 0.4 Hz, with the equivalent loading time. In rheological terms, a good correlation has been identified between log (G*), the complex shear modulus and log(pen).

It is generally considered that, for paving grade bitumens, the R&B softening point is equivalent to a penetration of 800 x 0.1 mm.

Correlation with other test methods from this Data Collection:

Penetration on fresh binders

Correlation with	**All binders**	**Paving grades, hard paving grades and special binders**	**Paving grades and hard paving grades**	**Paving grades only**	**PMB only**
LSV – EVT1 (fresh)		R^2 = 0.91, log			
Penetration (short term aged)	R^2 = 0.94, lin				
R&B Softening Point (fresh)		R^2 = 0.91, log			

Penetration on short term aged binders

Correlation with	**All binders**	**Paving grades, hard paving grades and special binders**	**Paving grades and hard paving grades**	**Paving grades only**	**PMB only**
R&B Softening Point (fresh)			R^2 = 0.90, log		

The correlation recorded between Penetration and R&B softening point is most probably forced by the fact that the binders studied comply with existing specifications.

Observation / remarks: BiTVal stated that correlation has been found in the papers with DSR measurements at 25°C. During the Data Collection tests were performed at 20° and 30°C and no correlation was found at any frequency between 0.1 and 10 Hz. TF Data Collection believes that R^2 values of 0.8 do not show a correlation while literature sometimes regards this value as a proof for correlation.

Suitability for production / quality control: Yes, but the relatively long time needed until results are obtained should be taken into account.

Suitability for future specification: Yes, but the limited precision for some binders should be taken into account. Though the method is well established, it is not performed accurately by all users.

1.3. Softening Point Ring and Ball [EN 1427]

General Comments: It is a well known and established test applicable for conventional binders. The significance of the test is uncertain for PMB, in particular highly modified PMB.

Precision: The precision data are provided in the test method standards having been established in several international round robin tests. However, some round robin tests have shown borderline (and higher) results [CEN TC 336 WG 1 TG 9].

Timing: 45 minutes.

Comments from BiTVal: It is generally considered that, for paving grade bitumen, the R&B softening point is equivalent to a penetration of 800 x 0.1 mm. Although many test methods measure related properties and therefore there will be some relationship, no formal correlation has been found in the papers reviewed between the softening point test and other bitumen tests.

Correlation with other test methods from this Data Collection:

Softening Point Ring and Ball on fresh binders

Correlation with	All binders	Paving grades, hard paving grades and special binders	Paving grades and hard paving grades	Paving grades only	PMB only
Dynamic Viscosity @ 60°C (fresh)		R^2 = 0.91, exp			
LSV – EVT1 (fresh)		R^2 = 0.95, exp			
LSV – EVT2 (fresh)		R^2 = 0.94, lin			
ZSV (fresh)		R^2 = 0.96, exp			
G* @ 60°C and 1,6 Hz (fresh)		R^2 = 0.94, exp			
Temperature @ G*/sin δ = 1 kPa (fresh)		R^2 = 0.96, exp			
R&B softening point (short term aged)	R^2 = 0.94, exp				
Dynamic Viscosity @ 60°C (short term aged)		R^2 = 0.93, exp			
LSV – EVT1 (short term aged)		R^2 = 0.94, lin			
ZSV (short term aged)		R^2 = 0.94, exp			
G* @ 60°C and 1,6 Hz (short term aged)		R^2 = 0.91, exp			
Temperature @ G*/sin δ = 2.2 kPa (short term aged)		R^2 = 0.90, exp			

The correlation recorded between Penetration and R&B softening point is most probably forced by the fact that studied binders comply with existing specifications.

Softening Point Ring and Ball on short term aged binders

Correlation with	All binders	Paving grades, hard paving grades and special binders	Paving grades and hard paving grades	Paving grades only	PMB only
LSV – EVT1 (fresh)		R^2 = 0.91, lin			
LSV – EVT2 (fresh)		R^2 = 0.91, lin			
ZSV (fresh)		R^2 = 0.95, exp			
G* @ 60°C and 1,6 Hz (fresh)			R^2 = 0.90, exp		
Temperature @ G*/sin δ = 1 kPa (fresh)		R^2 = 0.91, exp			
Def. Energy FD 10°C (fresh)		R^2 = 0.95, lin			
Dynamic Viscosity @ 60°C (short term aged)		R^2 = 0.96, exp			
LSV – EVT1 (short term aged)		R^2 = 0.95, lin			
LSV – EVT2 (short term aged)		R^2 = 0.93, lin			
ZSV (short term aged)		R^2 = 0.97, exp			
G* @ 60°C and 1,6 Hz (short term aged)		R^2 = 0.92, exp			
Temperature @ G*/sin δ = 2.2 kPa (short term aged)		R^2 = 0.95, exp			
Def. Energy FD 10°C (long term aged)				R^2 = 0.99, lin	

Observation / remarks: BiTVal stated that no formal correlation has been found in the papers reviewed between the softening point test and other bitumen tests. However, the Data Collection results proved clearly that the softening point ring and ball is at least for some groups of binders correlating very well with other tests.

Suitability for production / quality control: Yes, useful for production and quality control, because the method is well known, relatively quick, and correlated to other properties.

Suitability for future specification: Yes, suitable for specification purposes for paving grade and hard paving grade binders, but not necessarily for PMB.

1.4. Penetration Index [EN 1427]

General Comments: Penetration index is obtained by calculation.

The calculation formula is based on the assumption that the penetration of the binder is 800 0.1 mm at its softening point ring and ball. This is correct for most conventional binders but not for PMB and special binders. The result of the calculation could be improved if it is based on penetration values at different temperatures.

Precision: It is related to the precision of penetration and softening point ring and ball tests. The variability can be +/- 0.5 units.

Timing: To determine the penetration index, a calculation is carried out. The determination of the necessary characteristics takes approximately 3 hours.

Comments from BiTVal: Although many test methods measure related properties and therefore there will be some relationship, no formal correlation has been found in the papers reviewed between the penetration index and other bitumen tests.

Suitability for production / quality control: Yes, for paving grade bitumen. Penetration index gives a fair approximation of the temperature susceptibility between 25°C and the R&B SP temperature of these binders.

Suitability for future specification: Because penetration index gives an approximation of the temperature susceptibility of normal paving grade binders it could be used for future specifications for these binders only.

1.5. Dynamic Viscosity at 60°C [EN 12596]

General Comments: The applicability is limited to binder with softening point R&B below 60°C.

The standard requires a vacuum of "40 000 Pa +/- 67 Pa": It is not stated if this is a relative vacuum (in comparison with atmospheric conditions or not) and the recommended precision of (+/- 67 Pa) cannot be met with any equipment.

It is essential to use a suitable capillary and cleaning of the capillaries must be carried out properly.

Precision: Good for Newtonian liquids.

Timing: Less than two hours.

Comments from BiTVal: The dynamic viscosity at 60°C measured with the capillary viscometer test to the Australian standard AS 2341.02 was correlated with other binder properties for multigrade binders. A good correlation is reported with G*/sin δ at a frequency of 10 rad/sec and 60°C before and after RTFO-ageing. The correlation with the Ring and Ball softening point was good after RTFO-ageing, but not before.

Correlation with other test methods from this Data Collection:

Dynamic Viscosity on fresh binders

Correlation with	All binders	Paving grades, hard paving grades and special binders	Paving grades and hard paving grades	Paving grades only	PMB only
LSV – EVT1 (fresh)		R^2 = 0.91, log			
LSV – EVT2 (fresh)		R^2 = 0.91, log			
G* @ 60°C and 1,6 Hz (fresh)			R^2 = 0.92, lin		
Temperature @ G*/sin δ = 1 kPa (fresh)	R^2 = 0.94, log				
Def. Energy FD 25°C (fresh)	R^2 = 0.92, lin				
Dynamic Viscosity @ 60°C (short term aged)			R^2 = 0.94, lin		

Dynamic Viscosity on short term aged binders

Correlation with	**All binders**	**Paving grades, hard paving grades and special binders**	**Paving grades and hard paving grades**	**Paving grades only**	**PMB only**
ZSV (fresh)				R^2 = 0.96, lin	
Temperature @ G*/sin δ = 1 kPa (fresh)			R^2 = 0.91, log		
LSV – EVT1 (short term aged)	R^2 = 0.92, log				
ZSV (short term aged)			R^2 = 0.99, lin		
Temperature @ G*/sin δ = 2.2 kPa (short term aged)		R^2 = 0.93, log			

Observation / remarks: The statements from BiTVal can be confirmed only in some respects. It cannot be seen from the Data Collection that very good correlations exist with softening point ring and ball whatever the binder type: this is true only for the group of paving grades, hard paving grades and special binders but not for PMBs. For this group, a good correlation exists before and after short term ageing. The good correlation between viscosity at 60°C and G* (60°C, 10 rad/s) is confirmed only for paving grade and hard paving grades, while the correlation with Temp @ G*/sin (δ) =1.0 kPa (fresh) is good whatever the binder type. It can be seen that correlations also exist with LSV - EVT1 (fresh), LSV - EVT2 (fresh), ZSV (STA) and with the Temp @ G*/sin (δ) = 2.2 kPa (aged) for certain groups of binders.

Suitability for production / quality control: Yes, useful for production and quality control, because the method is well known, relatively quick, and correlated to other properties.

Suitability for future specification: No. Due to the limitations on applicability (only Newtonian liquids), this test cannot be recommended for inclusion in a general specification. However, dynamic viscosity is a useful indicator for soft binders and could be part of specifications for binders that show Newtonian behaviour.

1.6. Kinematic Viscosity at 135°C [EN 12595]

General Comments: The test is applicable for most grades and on fresh as well as on aged binders. However, only Newtonian liquids can be tested properly.

Precision: Good for fresh binders and Newtonian liquids, becomes worse for non-Newtonian liquids.

Timing: Less than two hours.

Comments from BiTVal: No comments to the kinematic viscosity at 135°C in the BiTVal report.

Correlation with other test methods from this Data Collection:

Kinematic Viscosity on fresh binders

Correlation with	**All binders**	**Paving grades, hard paving grades and special binders**	**Paving grades and hard paving grades**	**Paving grades only**	**PMB only**
Def. Energy FD 20°C (long term aged)	R^2 = 0,93, lin				

Kinematic Viscosity on short term aged binders

Correlation with	All binders	Paving grades, hard paving grades and special binders	Paving grades and hard paving grades	Paving grades only	PMB only
G* @ 60°C and 1,6 Hz (fresh)			R^2 = 0.92, lin		
Temperature @ G*/sin δ = 1 kPa (fresh)		R^2 = 0.94, log			
LSV – EVT1 (fresh)		R^2 = 0.92, log			
LSV – EVT2 (fresh)		R^2 = 0.91, log			
Def. Energy FD 20°C (long term aged)	R^2 = 0,99, lin				

Observation / remarks: There are no comments in the BiTVal report. The Data Collection shows that there are some very good correlations between LSV - EVT1 and the Temp @ G*/sin (δ) = 1kPa on short term aged paving grade and hard paving grade binders.

Suitability for production / quality control: Yes, useful for production and quality control.

Suitability for future specification: No. Kinematic viscosity is needed for information on handling only and is not related to the performance of the binder.

1.7. Fraass Breaking Point [EN 12593]

General Comments: Relatively easy on routine testing for conventional grades, but more difficult for PMB. In the case of PMB the temperature specified for sample preparation (80°C) is not always sufficient, the viscosity being too high.

The test shows a high variability on all binders. With automatic apparatus, an improvement of the precision was encountered.

Precision: The precision of results varies depending on the equipment used, the binder tested and the experience of the person performing the test. Precision data are stated in the standards. However, a number of Round Robin tests suggest that, in practice, the precision is not as good as stated, with Reproducibility value around twice the value stated in the method, which is already quite large (6°C) [Ref. *ESSAIS CIRCULAIRES 2007 PROGRAMME N° 86, « BITUMES »: BNPé – Département Produits et Méthodes, Septembre 2007*].

Timing: Up to three hours.

Comments from BiTVal: There is a broad correlation of the Fraass breaking point test with the BBR for unmodified bitumen and also some suggestion with PMB.

Correlation with other test methods from this Data Collection: No correlation between Fraass Breaking Point and other test methods was determined on the base of $R^2 \geq 0.9$.

Observation / remarks: The statements from BiTVal cannot be confirmed.

Suitability for future specification: No. The difficulties with sample preparation and poor precision make this test unsuitable for future specifications.

1.8. Elastic Recovery (for PMB only) [EN 13398]

General Comments: Test method provides indication of the elastic behaviour of the product and is only applicable to elastomer modified PMB.

The results allow a screening of the status of the binder for the presence of an elastomeric polymer and therefore some indication of the polymer modification level in case of elastomer modified PMB. It also gives some indication of the binder type and can help to differentiate between an elastomer modified binder and others.

The requested elongation of 200 mm cannot be met with all binders at all temperatures (e.g. hard binders or aged binders). This shortcoming could be overcome if different temperatures than 10 and 25°C were allowed.

Precision: Acceptable but impact of thermal history of the binder on precision.

Timing: Approximately 3 hours.

Correlation with other test methods from this Data Collection: No correlation between Elastic Recovery and other test methods was determined on the base of $R^2 \geq 0.9$.

Suitability for production / quality control: Yes, to indicate the presence of an elastomer in PMB.

Suitability for future specification: Because elastic recovery gives an indication of the presence of an elastomer in PMB it could be used as such for future specifications for elastomer modified PMB.

1.9. Storage Stability (for PMB only) [EN 13399]

General Comments: The test evaluates the storage stability of polymer modified binders, which relates to the handling/storage characteristics of a binder.

Depending of the type of modification, softening point ring and ball is not always a good parameter to judge storage stability; sometimes penetration is giving differences not observed by softening point ring and ball.

It is uncertain if sample size and duration of the conditioning period are representative of real storage conditions. The LAST test [Ref. Evaluation of the Laboratory Asphalt Stability Test, Publication No. FHWA-HRT-04-111] may be a better tool to compare with actual storage conditions.

Precision: The test uses a difference in properties between the top and bottom sections of a sample and therefore the precision of the softening point and the needle penetration apply here.

Timing: Three days of conditioning and 3 hours of testing.

Suitability for future specification: This property is critically important for handling and storage and therefore must be addressed by manufacturers of PMB; however, the test results obtained may not necessarily indicate actual behaviour in practice.

1.10. Flash Point [EN ISO 2592]

General Comments: A well known test needed for HSE and handling purposes.

Precision: Good.

Timing: Less than 2 hours.

Suitability for future specification: Yes, as it is linked to essential requirement 4 of CPD: Safety in use.

Flash point is not related to the performance of the binder in the pavement but is needed for handling information and HSE issues. It can be an indicator of the overall quality of a binder.

1.11. Solubility [EN 12592]

General Comments: Filtration can be very time consuming (up to 2 days), but the method does not specify any time limit and it is not clear if this affects the measurements. In some countries filtering membranes are used which may lead to better results. Problems can occur when too fine glass powder is used. Problems can also occur with some modified binders.

Some bitumens need to be heated to be dissolved properly in xylene, but the method does not specify anything on this (e.g. effects on the measurements).

The solubility of a binder is determined as an indicator of the purity of the binder only and not for performance purposes.

Precision: Good.

Timing: Normally less than 3 hours, but can be up to two days due to filtration.

Suitability for future specification: Solubility is deemed to be an indicator of the overall quality of a binder. It is not related to the performance of the binder in the pavement but requested for information only.

1.12. Density [EN ISO 3838]

General Comments: The density of a binder is determined for information only and not for performance purposes.

Precision: Good.

Timing: Less than 2 hours.

Suitability for future specification: Density is not related to the performance of the binder in the pavement but needed for information only.

Part 2: Ageing Procedures

2.1. Resistance to Hardening – RTFOT- Method [EN 12607-1]

General Comments: The procedure is typically carried out at a single temperature, 163°C. This might be too high for soft paving grades or for binders used in warm mixes and too low for hard grades and some PMB. For future specifications it may be better to perform the procedure at a defined EVT.

For paving grade binders a good correlation with the bitumen ageing during asphalt production in a discontinuous (batch pugmill) asphalt plant is stated and the procedure also gives information on the ageing susceptibility of the binder.

It may be difficult to obtain sufficient sample for further tests, especially when the product is viscous (PMB and hard paving grade bitumen).

To ensure accurate test results it is essential that representative samples of binder are obtained from the ageing flasks.

Precision: As it is a conditioning procedure and not a test in itself, no precision data can be given on it. Nevertheless, the procedure is considered to be good.

Timing: Approximately 4 hours: The procedure can be rather long when several repetitions are needed to obtain enough sample for further testing.

Comments from BiTVal: Four bitumens were used to make a hundred samples of bitumen and corresponding asphalt mixtures, manufactured with the three main types of asphalt plants (discontinuous, continuous, rotary dryer mixer) [3.07]. The main properties of the bitumens (penetration at 25°C, R&B softening point, ductility at 17°C) were determined before and after RTFOT and on the corresponding bitumens taken from the coated materials. A comparative statistical analysis of the results after RTFOT and coating was carried out.

The study showed that:

- The bitumen source and the grade of the bitumen have a major role on the thermal susceptibility to hardening with coating.
- The manufacturing process and the composition of the asphalt mixture did not have a significant effect, on average, on the hardening of the bitumen in the experiment.
- The predictive capacity of the RTFOT method is satisfactory. In particular, it makes it possible to assess the change of R&B softening point with an acceptable precision.

The RTFOT was a little more severe overall than mixing asphalt for the experiment conducted. Therefore, the RTFOT would appear to be a good method to indicate the degree of hardening of asphalt mixtures during asphalt production for the binders tested in the experiment.

Suitability for future specification: This is a conditioning procedure only and not a test. Good correlation is found between individual properties on short term aged and fresh binder in most cases (for details, see results on individual properties). This good correlation is probably a consequence of the use of this test in the present bitumen specifications. To have a short term ageing procedure and the related parameters in the specifications is recommended.

2.2. Pressure Ageing Vessel – PAV [EN 14769]

General Comments: The reliability of the equipment (maintenance, spare parts) is currently under question.

Potentially this is a good procedure, but test temperature depends on the binder type. It is uncertain whether the current test method for PAV ageing is the best procedure, as RTFOT-ageing is required prior to testing, which leads to a relatively small sample volume being available despite the long test time.

PAV is intended to give information of long term ageing susceptibility, but at present there are limited data to correlate the whole spectrum of European binders against their ageing in asphalt pavements.

It is questioned whether PAV is suitable in all cases of PMB.

There are still questions about the applicability of this ageing procedure as simulation of actual long term ageing due to the prolonged high temperature experienced by the binder.

Precision: As it is a conditioning procedure and not a test in itself, no precision data can be given on it.

Timing: All in all two working days are needed to be able to test the long-term aged binders. For some participants it is a relatively long procedure to be performed in quality control, but it might be used in a type testing scheme.

Comments from BiTVal: 20 h of PAV ageing at 100°C was found to correspond to 178 h of RCAT ageing. However, subsequent comparisons found the value was (176 ± 16) h with RCAT at 85°C. 5 h of PAV ageing at 100°C and 2.07 MPa was found to be equivalent to standard RTFOT ageing, and 25 h of PAV ageing at 100°C and 2.07 MPa was found to be equivalent to standard RTFOT plus PAV ageing.

Suitability for future specification: This is a conditioning procedure only and not a test.

PAV is currently the only widely used long term ageing procedure for bituminous binders.

2.3. Rotating Cylinder Ageing Test – RCAT [EN 15323]

General Comments: RCAT equipment is only in very limited use in Europe.

RCAT could be considered as a more promising procedure than PAV as short and long term ageing are performed in the procedure. Ageing kinetics of PMB can be very different in PAV and RCAT and there is uncertainty which method is the better predictor of ageing in the field. It seems to be easier to perform short and long term ageing in RCAT than in RTFOT plus PAV, but it takes much longer.

RCAT was developed as an easier conditioning procedure than PAV and was indirectly checked against PAV [Ref.: A. Verhasselt and A. Vanelstraete - Long-term ageing – Comparison between PAV and RCAT ageing tests. Second Eurasphalt & Eurobitume Congress, Sessions 1 and 4, pp. 897-905, Barcelona 2000].

There are still questions about the applicability of this ageing procedure as simulation of actual long term ageing due to the prolonged high temperature experienced by the binder.

Precision: As it is a conditioning procedure and not a test in itself, no precision data can be given on it.

Timing: 5 hours are needed for short term ageing and 6 working days to be able to condition the long-term aged binders. The actual manual handling time needed is less than that needed for carrying out RTFOT plus PAV.

Suitability for future specification: This is a conditioning procedure only and not a test. Insufficient data are available to conclude on the usefulness of the procedure for future specifications. Some practical issues exist which may make other ageing regimes preferable.

Part 3 - Resistance to Hardening

3.1. Change in mass [EN 12607-1]

General Comments: Change in mass is obtained by weighing and calculation.

Precision: Good.

Timing: After finishing the RTFOT procedure, another 2.5 hours are necessary to have the results of change in mass as the hot binder needs to cool down.

Comments from BiTVal: see item 2.1

Suitability for future specification: The test may provide some information on loss of volatile material, which may be HSE related.

3.2. Retained Penetration [EN 12607-1 in combination with EN 1426]

General Comments: The retained penetration is obtained by calculation. After RTFOT, the penetration of the aged binder is tested and compared with the value of the fresh binder. In addition, see the general comments given in 2.1.

The change of the binder property due to the impact of heat and air is observed which may be relevant to evaluate the binder quality.

Precision: The precision depends on the precision of the penetration test.

Timing: Between 1.5 and 4 hours for RTFOT and 3 hours for penetration testing.

Suitability for future specification: For paving grade binder the test appears to be useful. However, for hard paving grade bitumen penetration values close to the precision limits are measured, which may limit the usefulness of this parameter for future specifications.

3.3. Change in Softening Point Ring and Ball [EN 12607-1 in combination with EN 1427]

General Comments: This parameter is also based on calculation. As the softening point ring and ball of some PMB may decrease after RTFOT, the wording should be "change" in softening point ring and ball instead of "increase", which is currently used in EN 14023. In addition, see the general comments given in 2.1.

The change of the binder property due to the impact of heat and air is observed which may be relevant to evaluate the binder quality.

Precision: The precision depends on the precision of the softening point test.

Timing: Between 1.5 and 4 hours for RTFOT and 45 minutes for softening point testing.

Suitability for future specification: For conventional binders the test appears to be useful. However, the issues mentioned above concerning PMB may limit the usefulness of the parameter for future specifications.

Part 4: High Service Temperature Characteristics

4.1. Complex Modulus in DSR [EN 14770]: temperature sweep between 40 and 80°C (at given frequencies) and frequency sweep between 0.1 and 10 Hz (at given temperatures)

General Comments: The test can be applied on most binders, including highly modified PMB. Frequency sweep interval may need to be extended to lower frequencies for further use of data for EVT1 calculations.

The test is performed within the linear visco-elastic range, while permanent deformation in pavements can be linked to non-linearity at high stresses.

The test enables discrimination between elastomer modified binders and paving grade binders by analysis of the phase angle curve.

The results can be dependent on the apparatus used. The standard is imprecise, allowing different interpretations of the data. Consequently, analysis is difficult and experience is needed for meaningful interpretation.

The test evaluates visco-elastic properties of binder, but it is necessary to solve practical aspects related to the definition of a final numerical result (curve, reference values).

Maybe more repetitions are necessary for good accuracy. Care should be taken with sample preparation and thermal history.

Precision: The repeatability can be quite high, up to 20-30% on G* [Ref. "Complex Modulus of Bituminous Binders: Results of the Round Robin Test of the GE1 working group (France)" - B. Eckmann et al, Eurasphalt & Eurobitume 2008 Congress, Copenhagen 21-23 May 2008].

In general, the standard sets a rather wide precision range; this needs to be assessed.

Timing: Full testing may take some time (temperatures and frequencies), but can be fully automatic with modern DSR equipment. Testing over a full sweep is not considered as a quality control test. Temperature sweep at a specific frequency could be a faster option and in general faster than e.g. R&B or penetration test.

The test takes 2 hours to one day per sample, depending on the sample preparation and testing conditions. To obtain results this test is faster than LSV and ZSV.

Comments from BiTVal: Penetration has been correlated with DSR measurements. It is generally considered that, for paving grade bitumen, the R&B softening point is equivalent to a penetration of 800 x 0.1 mm. From the relationship between log(G*) and log(pen), it is possible to calculate the value of G* which equates to 800 pen (G*800 pen). Measurement of G* at more than one temperature enables a relationship between G* and temperature to be established and it is then possible to determine the temperature which corresponds to G*800 pen, nominally the R&B softening point. It has been found that the stiffness of the binder can be predicted from the penetration index and R&B softening point for paving grade bitumen. At very low testing frequency, the ratio G*/sin δ is related to the oscillation ZSV. Hence, there is also a relation with creep ZSV.

Correlation with other test methods from this Data Collection:

Complex Modulus G at 60°C and 1.59 Hz (10 rad/s) on fresh binders*

Correlation with	**All binders**	**Paving grades, hard paving grades and special binders**	**Paving grades and hard paving grades**	**Paving grades only**	**PMB only**
LSV – EVT1 (fresh)		R^2 = 0.96, log			
LSV – EVT2 (fresh)		R^2 = 0.92, log			
Temperature @ G*/sin δ = 1 kPa (fresh)	R^2 = 0.96, log				
LSV – EVT1 (short term aged)		R^2 = 0.91, log			
Def. Energy FD 20°C (long term aged)	R^2 = 0.95, lin				

Complex Modulus G at 60°C and 1.59 Hz on short term aged binders*

Correlation with	**All binders**	**Paving grades, hard paving grades and special binders**	**Paving grades and hard paving grades**	**Paving grades only**	**PMB only**
LSV – EVT1 (fresh)		R^2 = 0.92, log			
LSV – EVT2 (fresh)		R^2 = 0.91, exp			
ZSV (fresh)				R^2 = 0.96, lin	
LSV – EVT1 (short term aged)		R^2 = 0.93, log			
ZSV (short term aged)				R^2 = 0.94, lin	
Temperature @ G*/sin δ = 2.2 kPa (short term aged)	R^2 = 0.92, log				
Def. Energy FD 20°C (long term aged)	R^2 = 0.94, lin				

Temperature at which G/sin δ = 1 kPa (fresh binders)*

Correlation with	**All binders**	**Paving grades, hard paving grades and special binders**	**Paving grades and hard paving grades**	**Paving grades only**	**PMB only**
LSV – EVT1 (fresh)		R^2 = 0.98, lin			
LSV – EVT2 (fresh)		R^2 = 0.96, lin			
ZSV (fresh)		R^2 = 0.97, exp			
LSV – EVT1 (short term aged)		R^2 = 0.92, lin			
LSV – EVT2 (short term aged)		R^2 = 0.90, lin			
ZSV (short term aged)		R^2 = 0.92, exp			

Temperature at which G/sin δ = 2.2 kPa (short term aged binders)*

Correlation with	All binders	Paving grades, hard paving grades and special binders	Paving grades and hard paving grades	Paving grades only	PMB only
LSV – EVT1 (fresh)		R^2 = 0.91, log			
LSV – EVT2 (fresh)		R^2 = 0.94, lin			
ZSV (fresh)		R^2 = 0.94, exp			
LSV – EVT1 (short term aged)		R^2 = 0.95, lin			
LSV – EVT2 (short term aged)		R^2 = 0.93, lin			
ZSV (short term aged)		R^2 = 0.98, exp			

Observation/remarks: No correlation was found between DSR results at high temperature and penetration. A good correlation is confirmed between G* and R&B temperature but only if PMB are not taken into account.

A mathematical correlation is found between shear modulus and FD results on long term aged binder, but no physical reason is seen for that correlation.

Suitability for future specification: Temperature and frequency susceptibilities are fundamental properties of binders and, as such, may be useful for future specifications.

However the standard, as such, provides too much data to be used in specifications. Suitable parameters and limits would need to be determined from mixture properties and field performance.

A combination with ageing procedures could be considered.

4.2. Low Shear Viscosity in DSR – LSV [WI 336067, later prTS 15324]: EVT1 and EVT2

General Comments: The procedure can be used for all binders, but the protocol could be improved:

- No need is seen to carry out the temperature sweep at each temperature degree in order to generate acceptable EVT1 data.
- Indication of a starting temperature (lowest temperature) or range of temperatures would be useful for the temperature sweep (only a viscosity range is recommended).
- The calculation of EVT2 is not straight-forward, which can easily lead to mistakes. The extrapolation supposes that there is a plateau at low frequencies, which may not be the case for all binders, especially PMB.

Note that data for EVT1 calculations might also be obtained by frequency sweep in DSR, if the frequency interval is extended to include lower frequency values (see 4.1). Doing this would avoid performing several tests on the same binder and thus save time.

Precision: According to the precision data given in the project standard (Round Robin test performed in 2003), it is estimated that R (paving grade bitumen) = 2.0 to 2.8°C and R (PMB) = 4.3 to 5.3°C. This is comparable or worse than Reproducibility indicated for R&B softening point (resp. 2.0°C for paving grade bitumen and 3.5°C for PMB).

Note that this Round Robin test was performed on an earlier version of the protocol.

Timing: Between half a day and a full working day. The testing is shorter than that of ZSV, but can be longer than a frequency sweep at high temperature (complex modulus see 4.1). EVT2 results need long testing time because of low frequency.

Comments from BiTVal: Probably there is an acceptable link with rutting for EVT1, but in case of EVT2, the frequency may be too low to be linked with rutting.

Correlation with:

LSV – EVT1 (fresh binders)

Correlation with	All binders	Paving grades, hard paving grades and special binders	Paving grades and hard paving grades	Paving grades only	PMB only
LSV – EVT2 (fresh)	R^2 = 0.96, exp				
ZSV (fresh)		R^2 = 0.97, exp			
LSV – EVT1 (short term aged)	R^2 = 0.90, lin				
LSV – EVT2 (short term aged)	R^2 = 0.91, lin				
ZSV (short term aged)		R^2 = 0.93, exp			
Def. Energy FD 20°C (long term aged)	R^2 = 0.95, log				

LSV – EVT1 (short term aged binders)

Correlation with	All binders	Paving grades, hard paving grades and special binders	Paving grades and hard paving grades	Paving grades only	PMB only
LSV – EVT2 (fresh)		R^2 = 0.93, exp			
ZSV (fresh)		R^2 = 0.94, exp			
LSV – EVT2 (short term aged)	R^2 = 0.96, exp				
ZSV (short term aged binder)		R^2 = 0.96, exp			
Vialit pendulum maximum (short term aged)				R^2 = 0.923, exp	

LSV – EVT2 (fresh binders):

Correlation with	All binders	Paving grades, hard paving grades and special binders	Paving grades and hard paving grades	Paving grades only	PMB only
ZSV (fresh)		R^2 = 0.98, exp			
SP R&B (short term aged)		R^2 = 0.91, lin			
LSV – EVT2 (short term aged)	R^2 = 0.93, lin				
ZSV (short term aged)		R^2 = 0.95, exp			
BBR Temperature at which m = 0.3 (long term aged)		R^2 = 0.93, log			
Def. Energy FD 20°C (long term aged)	R^2 = 0.99, log				

LSV – EVT2 (short term aged binders)

Correlation with	All binders	Paving grades, hard paving grades and special binders	Paving grades and hard paving grades	Paving grades only	PMB only
ZSV (fresh)		R^2 = 0.93, exp			
Def. Energy FD 25 (fresh)	R^2 = 0.97, log				
G* @ 60°C and 1.6 Hz (short term aged)			R^2 = 0.90, exp		
ZSV (short term aged)		R^2 = 0.96, exp			

Observation / remarks: EVT1 is well correlated with dynamic viscosity at 60°C and is measurable on more datasets than dynamic viscosity at 60°C: EVT1 was measured on 131 binders (and no 'non measurable' values were recorded) while dynamic viscosity at 60°C was measured only on 97 binders and was non measurable for 26 binders (23 PMB and 3 hard grades). EVT1 could possibly be proposed instead of dynamic viscosity at 60°C.

A mathematical correlation is seen between EVT1 (fresh binder) and FD at 20°C (long term aged binder), but no physical reason is seen for that correlation. The same remark applies for the correlation seen between EVT2 and BBR results.

Suitability for future specification: This test may be useful for future specifications, especially for PMB. EVT1 is more preferred than EVT2 as there is a very good correlation between EVT1 and EVT2. In addition, it is less time consuming to measure only EVT1 and this parameter could also be deduced from a frequency sweep which would avoid performing several tests on the same binder and thus save time.

4.3. Zero Shear Viscosity [WI 336068, later prTS 15325]

General Comments: The test method is based on coherent theoretical concepts and fundamental rheological properties that can be addressed with the latest generations of DSRs.

However, the relevance of the test can be questioned for PMB due to the following reasons:

- A long time is needed to reach the testing plateau. The standard sets 4 h to reach the plateau, which is not always sufficient. For most PMB the final result does not provide the ZSV, but an approximation of the ZSV due to the fact that the viscosity depends on the shear. A very poor repeatability is observed for PMB (see "precision" below).
- Very often acceptable results cannot be obtained, ZSV being higher than the recommended measurement range (100-50 000 Pa·s); independent of polymer content or ageing conditioning.

For soft binders characterised in this Data Collection (Penetration>150 0.1mm), the ZSV measured is most often below the recommended measurement range.

Precision: The repeatability is reasonably good for unmodified bitumen, but poor for PMB with variations up to more than 50% for the same binder tested in the same lab [Ref. Rheometrical quantification of Bituminous Binders for specification purposes by S. Nigen-Chaidron, E&E 2008 congress].

In the provisional standard the results of a Round Robin test are displayed. Standard deviation for reproducibility is as high as 17% meaning that reproducibility could reach a level of ~50%.

Timing: For unmodified bitumen, the test can be performed within a half working day while for PMB it takes between 12 hours and two days to obtain results. For PMB it is often necessary to perform 3 test repeats. If two values are close to each other and the 3rd is different it is not clear which of them is valid.

Comments from BiTVal: None.

Correlation with:

ZSV (fresh binders)

Correlation with	All binders	Paving grades, hard paving grades and special binders	Paving grades and hard paving grades	Paving grades only	PMB only
ZSV (short term aged)	R^2 = 0.97, lin				
Def. Energy FD 20°C (long term aged)	R^2 = 0.96, lin				

ZSV (short term aged binders)

Correlation with	All binders	Paving grades, hard paving grades and special binders	Paving grades and hard paving grades	Paving grades only	PMB only
Def. Energy FD 25°C (fresh)	R^2 = 0.91, lin				
Def. Energy FD 20°C (long term aged)	R^2 = 0.94, lin				

Suitability for future specification: The shortcomings of this test make it unsuitable for inclusion in a future specification. Ring and ball softening point is simpler to test and gives similar interpretation for conventional binder. Complex modulus and phase angle or EVT1 are preferred, especially for PMB (better precision). The determination of ZSV takes too much time for specification purposes.

Part 5: Intermediate Service Temperature Characteristics

5.1. Complex Modulus G* and phase angle in DSR [EN 14770]: temperature sweep between 10 and 40°C (at given frequencies) and frequency sweep between 0.1 and 10 Hz (at given temperatures)

General Comments: Problems occurred in some cases when measuring hard paving grade bitumen (penetration ≤ 20 0.1 mm) at T=10ºC, due to the limitations of the DSR equipment. Some older rheometers may have torque limitations in case of testing hard paving grades at low temperatures. However, the procedure seems to be satisfactory for most binders when suitable DSRs are used.

Measurement system compliance may be an issue when testing hard paving grade binders at low temperatures.

Maybe more repetitions are necessary for good accuracy. Care should be taken with sample preparation and thermal history.

Precision: The repeatability can be quite high, up to 20-30% on G* [Ref. "Complex Modulus of Bituminous Binders: Results of the Round Robin Test of the GE1 working group (France)" B. Eckmann et al, Eurasphalt & Eurobitume 2008 Congress, Copenhagen 21-23 May 2008].

In general, the standard sets a rather wide precision range, this needs to be assessed.

Timing: Full testing may take some time (temperatures and frequencies), but normally fully automatic with recent DSRs. Testing over a full sweep is not considered as a quality control test. A temperature sweep at a specific frequency could be a faster option and in general faster than e.g. R&B or penetration test.

The test takes 2 hours to one day per sample, depending on the sample preparation and testing conditions.

Comments from BiTVal: see item 4.1.

Correlation with:

Complex Modulus G at 20°C on fresh binders*

Correlation with	All binders	Paving grades, hard paving grades and special binders	Paving grades and hard paving grades	Paving grades only	PMB only
BBR Temperature at which m = 0.3 (fresh)		R^2= 0.91, log			

Observation / remarks: BiTVal stated that correlation has been found in the papers between penetration and DSR measurements at 25°C. TF Data Collection performed tests at 20°C and 30°C and no such correlation was found at any frequency between 0.1 and 10 Hz. TF DC believes that R^2 values of 0.8 do not show a correlation while literature sometimes regards this value as a proof for correlation.

Suitability for future specification: Temperature and frequency susceptibilities are fundamental properties of binders and, as such, may be useful for future specifications. However the standard, as such, provides too much data to be used in specifications. Suitable parameters and limits would need to be determined from mixture properties and field performance.

A combination with ageing procedures could be considered.

Part 6: Low Service Temperature Characteristics

6.1. Bending Beam Rheometer BBR [EN 14771]: Determination of Stiffness and m-value at -16°C as well as determination of temperature at which stiffness S = 300 MPa and m-value = 0.3 respectively

General Comments: Stiffness is a fundamental property of a binder, whereas m-value is not. Low service temperature properties of binder are generally important in a pavement and low temperature failure typically occurs late in service life. Therefore it is sensible to address the low temperature behaviour of long term aged binders. However, because very good correlation was found between some BBR properties on fresh and on long term aged binder it is under discussion if there is a need to carry out the long term ageing step.

To determine the temperature at which S = 300 MPa or m = 0.3 respectively, the testing is carried out at two different temperatures. However, for the most commonly used paving grade bitumens in Europe (50/70 and 70/100) stiffness is approximately 300 MPa at -16°C, so for these binders no additional information is gained when carrying out this test protocol at different temperatures.

For modified binders, some trends can be observed but there is an issue that the low temperature performance of PMB may be underestimated due to the m-value being the limiting factor. Evidence from field performance suggests that PMBs resist fracture propagation better than unmodified bitumen but this phenomenon could not be determined by this study. Some field experience suggests better low temperature behaviour of PMB compared to unmodified binders, but this is not always evident from BBR results. Nevertheless, BBR is probably less prone to problems than Fraass breaking point, but the test method as currently written still has deficiencies.

Sample preparation is important and de-moulding of the specimen is critical, as such problems occur when working with very hard and soft binders. Alternative moulds should be allowed in the standard, provided that the beam conforms to the size and tolerances specified in the standard.

Precision: Results confirm a good repeatability, but it depends on the specimen preparation and the final geometry (of the prepared specimen). The precision is considered to be better than that of Fraass breaking point.

Timing: The test takes 2 to 4 hours per sample and can be carried out faster if it is only performed at one temperature, but that will also lead to some loss of information (temperature susceptibility ...).

Correlation with:

BBR S @ -16°C on fresh binders

Correlation with	All binders	Paving grades, hard paving grades and special binders	Paving grades and hard paving grades	Paving grades only	PMB only
BBR Temp. @ S = 300 (fresh)	R^2 = 0.93, log				
BBR Temp. @ m = 0.3 (fresh)		R^2 = 0.91, log			
BBR S @ -16°C (long term aged)	R^2 = 0.94, lin				
BBR Temp. @ S = 300 (long term aged)	R^2 = 0.92, log				

BBR S @ -16°C on long term aged binders

Correlation with	All binders	Paving grades, hard paving grades and special binders	Paving grades and hard paving grades	Paving grades only	PMB only
BBR Temp. @ S = 300 (fresh)	R^2 = 0.92, log				
BBR Temp. @ m = 0.3 (fresh)			R^2 = 0.91, log		
BBR Temp. @ S = 300 (long term aged)	R^2 = 0.94, log				

BBR m @ -16°C on fresh binders

Correlation with	All binders	Paving grades, hard paving grades and special binders	Paving grades and hard paving grades	Paving grades only	PMB only
BBR Temp. @ m = 0.3 (fresh)		R^2 = 0.91, log			

BBR m @ -16°C on long term aged binders

Correlation with	All binders	Paving grades, hard paving grades and special binders	Paving grades and hard paving grades	Paving grades only	PMB only
BBR Temp. @ m = 0.3 (long term aged)				R^2 = 0.91, log	

BBR Temperature at which S = 300 on fresh binders

Correlation with	All binders	Paving grades, hard paving grades and special binders	Paving grades and hard paving grades	Paving grades only	PMB only
BBR Temp. @ m = 0.3 (fresh)		R^2 = 0.93, lin			
BBR Temp. @ S = 300 (long term aged)	R^2 = 0.96, lin				

BBR Temperature at which S = 300 on long term aged binders

Correlation with	All binders	Paving grades, hard paving grades and special binders	Paving grades and hard paving grades	Paving grades only	PMB only
BBR Temp. @ m = 0.3 (fresh)		R^2 = 0.93, lin			

BBR Temperature at which m = 0.3 on fresh binders

Correlation with	All binders	Paving grades, hard paving grades and special binders	Paving grades and hard paving grades	Paving grades only	PMB only
DSR G* at 20°C, 1.6 Hz (fresh)		R^2 = 0.91, exp			

Suitability for future specification: BBR is potentially suitable for specification purposes, but the issue of a possible under-estimation of the performance of PMB needs to be resolved. Stiffness related data are preferred over m-value data. If used for specifications, a temperature result, e.g. the temperature at which a particular stiffness is reached, is more preferred than a stiffness value as it is easier to understand.

In general, no other test but BBR and Fraass breaking point used during this data collection measured the low temperature properties of a binder. Unless other low temperature tests are proposed and evaluated one of these two tests may be necessary part of a specification.

Part 7: Cohesion

7.1. Deformation Energy with Force Ductility Method [EN 13589 and EN 13703]

General Comments: When carried out according to the present specification for PMB, the application is limited as not all products reach elongations between 20-40 cm (as required) at the currently specified temperatures.

The force ductility curve gives some information on the polymer modification, which may be interesting for the producer and the user of the binder. Focusing on pure numeric figures and not interpreting the full curve of testing means that useful information may be lost.

Sample preparation is crucial, problems may occur when working with very hard binders. The de-moulding of the specimen is critical, because they may break or be damaged easily.

As the equipment and the sample preparation is similar or identical to that used for testing elastic recovery and, in some countries ductility, both the equipment and the experience gained with it are widely spread over Europe.

The procedure described in the standard allows the use of too many temperatures.

The standard requires the measurement to be started at 5°C and the temperature increased by 5°C steps (up to 25°C), but for example, measurements are not possible on hard paving grades or PMB at 5 or 10°C. Furthermore, after PAV the test cannot always be carried out due to the hardness of the aged binder. Therefore the temperature should be indicated for each type of binder according to type (conventional/PMB) and penetration range. Other options are to start at 25°C and decrease temperature or do the testing only at a given temperature (e.g. 20°C) to be able to compare all bitumen under similar conditions.

Precision: The precision data of the method were evaluated in an European round robin test and are given in the standard. They are deemed to be satisfactory, especially if the test is performed on 3 samples, taking 2 of the results into account.

Timing: The time needed to prepare and perform the test is acceptable, with 2 to 4 hours needed per sample and temperature.

Comments from BiTVal: Correlation between the maximum energy of the force ductility curve and penetration has been found. The force ductility curve gives qualitative information on the cohesive and elastic properties of polymer modified bitumen. These properties are determined by the polymers in terms of their type, distribution, concentration and network. The force ductility device can also be used for the determination of the elastic recovery, EN 13398.

Correlation with:

Force ductility on fresh binders

Correlation with	All binders	Paving grades, hard paving grades and special binders	Paving grades and hard paving grades	Paving grades only	PMB only
FD 10: Def. Energy FD 10 (short term aged)	R^2 = 0.92, lin				
FD 5: Def. Energy FD 15 (long term aged)	R^2 = 0.91, exp				

Force ductility on short term aged binders

Correlation with	All binders	Paving grades, hard paving grades and special binders	Paving grades and hard paving grades	Paving grades only	PMB only
FD 15: Def. Energy FD 10 (fresh)					R^2 = 0.93, log
FD 5: Def. Energy FD 5 (fresh)				R^2 = 0.99, lin	

Suitability for production / quality control: Yes, under clearly specified testing conditions, i.e. temperature. The data curves give useful information on the binder properties and the testing procedure is easy to perform. Additionally the testing equipment is already widely used.

Suitability for future specification: The test may be suitable for future specification, but it is recommended that the test temperature should be clearly specified for each binder type, or a temperature relating to the binder properties should be chosen. The test is not recommended for specification purposes after long term ageing, because either not all binders can be tested when long term aged or a good correlation exists between deformation energy on fresh and on long term aged binders. Furthermore, as the procedure is limited to one specific speed it is therefore also limited in performance prediction.

The results allow a differentiation between polymer modified and unmodified binders.

7.2. Deformation Energy with Tensile Test Method [EN 13587 and EN 13703]

General Comments: The equipment to perform the tensile test is not widely spread throughout laboratories. Within this data collection, only 3 datasets contained results from testing the fresh and the short term aged binder with tensile test and the test was carried out at 5°C only. The limited use of this test must be taken into account whenever discussing future specification schemes.

For the data collection project the test was limited to a temperature range between 5 and 25°C. The test does not add new information compared to the Force Ductility Test. If performed at lower temperatures and with low speed, additional information on the low temperature properties of a binder might be obtained.

Precision: The repeatability of the test is deemed to be fair. When working at low temperatures, the sample preparation is difficult.

Timing: Approximately half a working day is needed to carry out the test.

Comments from BiTVal: The tensile test has several common features with the direct tensile test (DTT) despite the procedures being intended for different purposes. In the DTT, a ductile-brittle transition temperature is sought whereas, in the tensile test to EN 13587, a sample is rejected if a brittle break occurs. The tensile test is similar to the force-ductility test where the elastic/rubbery properties are tested with the elongation in these tests normally being >100 %. DTT is a low temperature test and the result is a temperature whereas the tensile test is an elongation test and the result is an elongation and force. Nevertheless, the specification for the main components of the equipment for both tests, except the attachment devices, are compatible, making it possible to use the same basic equipment with most of the test machines and temperature chambers available on the market for both tests with the necessary modifications. A single stretching speed of 1 mm/min is required for both tests and the test temperature range is common to both procedures.

Similarly defined, but not equivalent, tensile properties can be measured through the force ductility test to EN 13589. Although readily available and less costly than the tensile test equipment (providing the ductilometer has not also to be purchased), the force ductility apparatus has the drawback of a narrower test temperature range of 5ºC to 25ºC.

EN 13703 for deformation energy states two calculation procedures for the conventional energy, depending on the method followed. Different specification limits have to be fixed for each test result.

Suitability for production / quality control: Not enough data are available to give a statement.

Suitability for future specification: The test is not widely used and there is little experience at European level.

7.3. Vialit Pendulum [EN 13588]

General Comments: The test is only applicable to surface dressing binders and should not be used in other areas.

The choice of the test temperatures depends on the analyst. This leads to a limited comparability between results from different sources.

Within this data collection, only 22 datasets contained results from testing the fresh binder with Vialit Pendulum (16 on short term aged binders, 3 on long term aged binders). The limited use and applicability of this test must be taken into account whenever discussing future specification schemes.

Precision: A limited study on the repeatability was carried out by Energy Institute in UK; all in all the repeatability of the test is rather poor.

Timing: The test is time consuming; it takes nearly a week to carry out the test under all circumstances to be taken into account.

Comments from BiTVal: Although many test methods measure related properties and therefore there will be some relationship, no formal correlation has been found in the papers reviewed between the Vialit pendulum test and other bitumen tests.

Correlation with:

Vialit pendulum maximum on fresh binders

Correlation with	All binders	Paving grades, hard paving grades and special binders	Paving grades and hard paving grades	Paving grades only	PMB only
Vialit maximum (short term aged)	R^2 = 0.967, log				

Suitability for production / quality control: Limited to binders for surface dressing purposes.

Suitability for future specification: It must be kept in mind that this test is not appropriate for all binders in general but for a limited application (surface dressings) only.

Annex 1 – Data Collection template

PART 1: PRODUCT DESCRIPTION ACCORDING TO CONVENTIONAL SPECIFICATIONS

EN 12591 Paving Grades - prEN 13924 Hard Grades - EN 14023 Polymer Modified Bitumens

Product type	Normal Paving Grade	
	Hard Grade	
	PMB	
	Special Bitumen	

Characteristic	Test method	Unit	Reported value		
			Binder condition		
			Fresh	STA	LTA
Nominal penetration range	EN 1426	0.1 mm			
Penetration @ 25 °C	EN 1426	0.1 mm			
Softening point R&B	EN 1427	°C			
Penetration index	(2)	-			
Dynamic viscosity @ 60 °C	EN 12596	Pa.s			
Kinematic viscosity @ 135 °C	EN 12595	mm^2/s			
Fraass breaking point	EN 12593	°C			
Elastic recovery @ 10 °C (1)	EN 13398	%			
Elastic recovery @ 25 °C (1)	EN 13398	%			
Storage stability (1)	EN 13399	°C			
Resistance to hardening	EN 12607-1				
Change in mass	EN 12607-1	%			
Retained penetration @ 25 °C	EN 1426	%			
Increase in softening point R&B	EN 1427	°C			
Flash point	EN ISO 2592	°C			
Solubility	EN 12592	%			
Density	EN ISO 3838	-			

(1) to be reported for PMB's only
(2) Normative Annex B of EN 12591:1999 Calculation of the penetration index (PI Pfeiffer)

PART 2: PERFORMANCE-RELATED PROPERTIES

Indicate the Long Term Ageing Procedure tyemperature	
PAV at ...°C [pr EN 14769]	11
RCAT [WI 336...]	5

Characteristics	Test method	Unit	Reported value – Binder condition: Fresh	STA	LTA
At high service temperature					
Complex modulus (DSR)					
G* and phase angle - for temperature sweep 40 - 80 °C (at certain frequencies) - for frequency sweep 0.1 - 10 Hz (at certain temperatures)	prEN 14770	kPa / deg	use separate input sheet		
Low Shear Viscosity (DSR)					
Equiviscous temperature EVT1 @ LSV = 2,0 kPa.s and 0,1 rad/s	WI 336067	°C			
Equiviscous temperature EVT2 @ LSV = 2,0 kPa.s and 0,001 rad/s	WI 336067	°C			
Zero Shear Viscosity (DSR)					
Zero shear viscosity (creep mode) @ 60°C	WI 336068	kPa.s			
At intermediate service temperature					
Complex Modulus (DSR):					
G* and phase angle - for temperature sweep 10 - 40 °C (at certain frequencies) - for frequency sweep 0.1 - 10 Hz (at certain temperatures)	prEN 14770	kPa / deg	use separate input sheet		
At low service temperature					
Bending Beam Rheometer (BBR)	prEN 14771				
Stiffness @ -16°C		MPa			
m-value @ -16°C		-			
Temperature Stiffness @ 300 MPa		°C			
Temperature m-value @ 0.300		°C			
Cohesion (choice of test)					
Force ductility @ 5 °C	EN 13589 -13703	J/cm^2			
Force ductility @ 10 °C	EN 13589 -13703	J/cm^2			
Force ductility @ 15 °C	EN 13589 -13703	J/cm^2			
Force ductility @ 20 °C	EN 13589 -13703	J/cm^2			
Force ductility @ 25 °C	EN 13589 -13703	J/cm^2			
Tensile test @ 5 °C	EN 13589 -13703	J/cm^2			
Tensile test @ 10 °C	EN 13589 -13703	J/cm^2			
Tensile test @ 15 °C	EN 13589 -13703	J/cm^2			
Tensile test @ 20 °C	EN 13589 -13703	J/cm^2			
Tensile test @ 25 °C	EN 13589 -13703	J/cm^2			
Vialit pendulum: maximum	EN 13588	J/cm^2			

Annex 2 – List of test methods used during the project

In order to assure that the Eurobitume Data Collection project, which is a contribution to the CEN Data Collection project, will be carried out according to similar editions of published test methods, this document provides an overview of the standards which may be of relevance for this project.

Standard	Title of document	Edition
EN 1426	Bitumen and bituminous binders – Determination of needle penetration	October 1999
EN 1427	Bitumen and bituminous binders – Determination of softening point – Ring and Ball method	October 1999
EN 12591	Bitumen and bituminous binders – Specifications for paving grade bitumen	November 1999
EN 12592	Bitumen and bituminous binders – Determination of solubility	November 1999
EN 12593	Bitumen and bituminous binders – Determination of the Fraass breaking point	November 1999
EN 12594	Bitumen and bituminous binders – Preparation of test samples	December 1999
EN 12595	Bitumen and bituminous binders – Determination of kinematic viscosity	November 1999
EN 12596	Bitumen and bituminous binders – Determination of dynamic viscosity by vacuum capillary	November 1999
EN 12607-1	Bitumen and bituminous binders – Determination of the resistance to hardening under influence of heat and air – Part 1: RTFOT method	October 1999
EN 13398	Bitumen and bituminous binders – Determination of the elastic recovery of modified binders	December 2003
EN 13399	Bitumen and bituminous binders – Determination of storage stability of modified binders	December 2003
EN 13588	Bitumen and bituminous binders – Determination of cohesion of bituminous binders with pendulum test	February 2004
EN 13589	Bitumen and bituminous binders – Determination of the tensile properties of modified bitumen by the force ductility method	December 2003
EN 13703	Bitumen and bituminous binders – Determination of deformation energy	December 2003
prEN 13924	Bitumen and bituminous binders – Specifications for hard paving grade bitumens	December 2005

Standard	Title of document	Edition
EN 14023	Bitumen and bituminous binders – Framework specification for polymer modified bitumens	October 2005
EN 14769	Bitumen and bituminous binders – Accelerated long-term ageing conditioning by a Pressure Ageing Vessel (PAV)	October 2005
EN 14770	Bitumen and bituminous binders – Determination of complex shear modulus and phase angle - Dynamic Shear Rheometer (DSR)	October 2005
EN 14771	Bitumen and bituminous binders – Determination of the flexural creep stiffness – Bending Beam Rheometer (BBR)	May 2005
prEN 15323	Bitumen and bituminous binders – Accelerated long-term ageing conditioning by the rotating cylinder method (RCAT)	August 2005
prEN 15324	Bitumen and bituminous binders – Determination of equiviscous temperature based on Low Shear Viscosity using a Dynamic Shear Rheometer in low frequency oscillation mode	August 2005
prEN 15325	Bitumen and bituminous binders – Determination of Zero Shear Viscosity (ZSV) using a Dynamic Shear Rheometer in creep mode	August 2005
EN ISO 2592	Petroleum products – Determination of flash and fire points – Cleveland open cup method	August 2001
EN ISO 3838	Crude petroleum and liquid or solid products, Determination of density or relative density – Capillary-stoppered pyknometer and graduated bicapillary pyknometer methods	May 2004