BitVal - Analysis of Available Data for Validation of Bitumen Tests

FEHRL Overview

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Under the day-to-day management of the Executive Committee, FEHRL is engaged in research topics including road safety, materials, environmental issues, telematics and economic evaluation.

Research capacity is provided by the national institutes and makes use of the wide range of test facilities available.

Aims and Objectives

The mission of FEHRL is to promote and facilitate collaboration between its institutes and provide high quality information and advice to governments, the European Commission, the road industry and road users on technologies and policies related to roads.

The objectives of collaborative research are:

- to provide input to EU and national government policy on highway infrastructure
- to create and maintain an efficient and safe road network in Europe
- to increase the competitiveness of European road construction and road-using industries
- to improve the energy efficiency of highway construction and maintenance
- to protect the environment and improve quality of life

Analysis of Available Data for Validation of Bitumen Tests

Report on Phase 1 of the BiTVal Project

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Contents

Summary

The Comité Européen de Normalisation / European Committee for Standardisation was formed in the early sixties and was charged with preparing common rules and standards to be used in all member countries to ensure no barriers to trade. As part of that programme, new harmonised European specifications for paving grade bitumens are being developed. Bitumen specifications have remained relatively unchanged over the last forty years until a European Standard was published in 1999, whose development involved contributors such as producers, contractors and administrations all over Europe.

In order to take a systematic approach to next generation binder specifications, the European bitumen/asphalt industry has made major efforts in recent years to define the "performance-related" (P-R) requirements for paving binders. It is, of course, recognised that the binder properties alone do not determine pavement performance. Other parameters, such as aggregate characteristics, mixture design, manufacture and laying are also considered as important. A process is being followed to ensure that, for the second generation standards, the performance relationships of a binder property are assessed before a specification is developed. The BiTVal project has been developed to assist that process.

The FEHRL Board agreed that the first phase of the project, named BiTVal, should go ahead. Many FEHRL members, who had to arrange their financing for the project, from around Europe agreed to participate in Phase 1, which is a desk study, analysing information gathered from all sources, and additional work needed would be identified during this process. The BiTVal project is expected to:

- Deliver the appropriate answers for assessing the suitability of test methods for characterising the relevant performance of related bitumen properties.
- Establish their relevance and correlation to the asphalt pavement performance.
- Give the required level of confidence in the future European specification system.

The key outputs of Phase 1 of the BiTVal project will be a database, covering publications of the identified bitumen properties and their relationship to asphalt properties and/or road performance, and a FEHRL report to TC336 WG1 summarising the P-R aspects for each test method, together with recommendations for their use in the next generation of standards.

In the BiTVal project, all the binder tests that might be used by TC336 WG1 have been reviewed together with the conditioning/ageing procedures that might be used to assess binder durability.

The BiTVal database has been searched for correlations between all these tests and the following critical performance characteristics, in asphalt mixture tests and/or pavement performance assessments:

- Permanent Deformation.
- Stiffness.
- Low Temperature Cracking.
- Fatigue Cracking.
- Adhesion.

The overall conclusions in terms of recommendations for a bitumen test to assess the potential asphalt properties are:

- The oscillation ZSV test, which is relatively simple and provides good correlations for permanent deformation.
- The DSR binder stiffness and/or penetration for stiffness, with the DSR test preferred because the penetration test is not suitable for PMBs.

- Either BBR limiting temperature or a DTT parameter for low temperature cracking, with the concept of critical cracking temperature, combining both the BBR and the DTT results, as an alternative for the future.
- The empirical characteristics (such as penetration, softening point and viscosity) and some rheological characteristics before and after ageing (such as RCAT or RTFOT and PAV) remain the best criteria to assess the fatigue behaviour of asphalt.
- The approach of using surface energies of materials enables some fundamental insights about adhesion to be gained, although equilibrium in an asphalt mixture is probably never realised.

However, the preferences are conditional and further research is required. These research needs are prioritised as:

- Essential – Oscillation ZSV test (for deformation resistance), critical cracking temperature (for low temperature cracking) and control aggregates (for adhesion).
- Important – Direct tensile test (for low temperature cracking), fracture toughness test (for low temperature cracking), bitumen fatigue test (for fatigue) and relationship with site data (for adhesion).
- Desirable – Creep ZSV/repeated creep tests (for deformation resistance), DSR relationship with site data (for stiffness) and relationship with site data (for low temperature cracking).

With regard to the database and library developed as part of this project, it is proposed that:

- Access becomes unrestricted to the BiTVal database on the FEHRL website.
- The BiTVal library remains on a password-protected area of the FEHRL website with access for participants in the BiTVal project plus other FEHRL members.
- The use of the BiTVal database and library for any future project is conditional on the institutes involved on that project updating them.

1. Introduction

1.1 CEN TC336/WG1 – Bituminous Binders for Paving

The Comité Européen de Normalisation / European Committee for Standardisation (CEN) was formed in the early sixties and was charged with preparing common rules and standards to be used in all member countries to ensure no barriers to trade.

As part of that programme, new harmonised European specifications for paving grade bitumens are being developed in two stages:

- First Generation (CEN TC19/SC1, since 1990) – The working groups have completed their work to produce specifications and test methods for paving bitumens, for use throughout Europe, which were based on existing national standards. Such specifications (already published in 1999 as EN 12591, and now under revision after 5 years in use) are well known as empirical test based specifications.

  The first generation standards include EN 12591 [1.01] for paving grade bitumens, prEN 13924 [1.02] for hard paving grades and EN 14023 [1.03] for polymer-modified binders.
- Second Generation (CEN TC336, since 2000) – The next task of the working groups, in order to meet the requirements of the Construction Products Directive, is to produce "harmonised technical specifications that are performance based". The new standards should reflect the binder contribution to the performance of the asphalt pavement, with the inclusion of existing or new properties and test methods, as appropriate.

Bitumen specifications have remained relatively unchanged over the last forty years and the specifications now being developed represent a very significant step forward for all involved contributors such as producers, contractors and administrations all over Europe.

The second generation of standards can be more 'market-driven'. The aim is good quality asphalt roads that perform well throughout their lifetime. Specifications and standards should meet both the technical and commercial needs of the asphalt industry and its customers, and it is important these needs are properly identified and understood.

The overall purpose of the specification system will be to ensure that binders can be evaluated on a fair and comparable basis, that the appropriate binder can easily be selected for a particular application and that the binder can be used with confidence in its quality. Ideally, the system should be suitable throughout Europe, for all types of climate and traffic conditions, for a large variety of pavement applications, and applicable to all categories of binders: conventional, multigrade and modified bitumens.

1.2 Eurobitume Workshop and BiTSpec Seminars

1.2.1 Performance-related requirements

In order to take a systematic approach to 2nd Generation binder specifications, the European bitumen/asphalt industry has made major efforts in recent years to define the "performance-related" (P-R) requirements for paving binders.

In 1999 Eurobitume organised a workshop with global attendance representing all sides of the asphalt industry and delegates collectively identified the principle performance requirements of asphalt pavements and then related these to the appropriate binder properties and then identified possible tests required to measure these properties. The outcome from that workshop was the starting point for much of the subsequent work [1.04].

In 2000, CEN TC336 was established and the 'new' WG1 started work on development of a new performance-related specification for paving binders. This specification includes PMBs as well as unmodified binders, and so amalgamates the work of the old WG1 (Paving Grades) and WG4 (Modified Bitumens) committees. Progress on the development of these second generation specifications has been reported in a CEN Technical Report [1.05].

There have also been two major exercises to update and refresh the thinking on the outcomes of the 1999 Eurobitume Workshop.

1.2.2 BiTSpec Project

In 2002/03 a series of regional seminars, supported by Eurobitume and the European Asphalt Pavement Association (EAPA), were held around Europe, on the subject of "Bituminous Binder Testing and Specifications", culminating in the BiTSpec Seminar in Brussels, in June 2003. The seminars were well attended by over 1000 representatives from all parts of the industry, and the outputs from that project were made available to WG1 and given in the Eurobitume BiTSpec Proceedings [1.06].

1.2.3 TC336 Advisory Group

The CEN TC336 Advisory Group (which is constituted by the five key European Road Industry Stakeholder Groups) has provided a comprehensive report in 2003 on the subject of "Binder Requirements", compiled from individual position papers from Eurobitume, EAPA, the Forum of European National Highway Research laboratories (FEHRL), the Western European Road Directorate (WERD) which is now renamed the Conference of European Directors of Roads (CEDR), the International Institute of Synthetic Rubber Producers (IISRP). The final report is being used by WG1 [1.07].

It is important that the market needs are correctly identified and addressed, hence the involvement of key stakeholders in the process: road owners and authorities, specifiers, road contractors, asphalt and binder producers. A good and effective communication between all these players was necessary for satisfactory development of the WG1 programme.

1.3 BiTVal Project

It is, of course, recognised that the binder properties alone do not determine pavement performance. Other parameters, such as aggregate characteristics, mixture design, manufacture and laying are also considered as important. A process is being followed to ensure that, for the second generation standards, the performance relationships of a binder property are assessed before a specification is developed. The basic sequential steps are as follows:

(1) Identify the binder properties linked to the performance requirements of asphalt pavements.
(2) Select and standardise appropriate (new) test methods to measure these properties.
(3) Collect data and ensure field validation for establishing (new) binder specifications.
(4) Review the grading system according to the (new) specification.

The CEN TC336 working groups have addressed steps (1) and (2). To address step (3), FEHRL proposed to organise a European project on validation of the new EN test methods, and this was presented and discussed at the BiTSpec seminar in June 2003.

The FEHRL Board agreed that the first phase of the project, named BiTVal, should go ahead. Phase 1 is a desk study, analysing information gathered from all sources, and additional work needed would be identified during this process.

This BiTVal project represents a significant effort, requiring support and participation from industry and authorities in many countries. It is expected to:

- Deliver the appropriate answers for assessing the suitability of test methods.
- Establish their relevance and correlation to the asphalt pavement performance.
- Give the required level of confidence in the future specification system to be used during many years in the whole of Europe.

For the convenience of readers, many acronyms that have had to be used in the report have been gathered together in Annex A.

The key outputs of Phase 1 of the BiTVal project will be a database, covering publications of the identified bitumen properties and their relationship to asphalt properties and/or road performance, and a FEHRL report to TC336 WG1 summarising the P-R aspects for each test method, together with recommendations for their use in 2nd generation standards.

The project plan for Phase 1 is given in Annex B.

For tests that are assessed as potentially useful, but for which there are insufficient data to confirm validity, the project report will include a proposals for obtaining the missing data.

1.4 Properties Reviewed

CEN TC336 Working Group 1 decided from the beginning to establish Task groups to work on the three key properties that were identified at Eurobitume Workshop ’99 as essential parts of any new specifications for paving binders:

- High (service) Temperature properties
- Low (service) Temperature properties
- Durability (approached through procedures for binder Ageing & Conditioning)

For these topics there was considerable existing background information and test methods from the USA, particularly from the Strategic Highways Research Program (SHRP) project developments. These were taken into account together with published development work from around the world.

Other properties and test methods have been subsequently added to the list in an ongoing process to address the identified performance requirements, in particular:

- Adhesion
- Cohesion
- Fatigue

In principle, the goal of the validation study is to determine the relation between a certain bitumen characteristic and the pavement behaviour. In practice, it is usually difficult to find strict correlations between the bitumen characteristics and the pavement behaviour; it is easier to relate a bitumen characteristic to tests on the asphalt mixture, and correlate those tests on the asphalt mixture with the behaviour of the mixture in the road.

In the BiTVal project, all the binder tests that might be used by TC336 WG1 have been reviewed in Chapter 2 (where they are reported in alphabetical order) whilst the conditioning/ageing procedures that might be used by TC336 WG1 to assess binder durability have been reviewed in Chapter 3. The BiTVal database has been searched for correlations between all these tests and the following critical performance characteristics, in asphalt mixture tests and/or pavement performance assessments:

- Permanent Deformation (Chapter 4)
- Stiffness (Chapter 5)
- Low Temperature Cracking (Chapter 6)
- Fatigue Cracking (Chapter 7)
- Adhesion (Chapter 8)

The relationships between the various tests and properties are shown schematically in Annex C.

1.5 BiTVal Database

A database has been developed containing information from published, unpublished and ongoing research projects relating bitumen properties and/or test methods to asphalt mixture properties and/or pavement performance.

The database contains the relevant papers from 35 recent global bitumen/asphalt conferences and has been used to search all reported correlation work between the selected binder properties and asphalt/pavement performance, as given in Chapters 4 to 8, using the proformas in Annex D. All of these references are included in the database and are given in Annex E of this report.

The database, together with a library of all the references used in the project, has been stored on a dedicated FEHRL website [1.08].

1.6 References

[1.01] **Comité Européen de Normalisation.** *Bitumen and bituminous binders – Specifications for paving-grade bitumens.* EN 12591: 2000.

[1.02] **Comité Européen de Normalisation.** *Bitumen and bituminous binders – Specifications for hard paving-grade bitumens.* prEN 13924: 2005.

[1.03] **Comité Européen de Normalisation.** *Bitumen and bituminous binders – Framework specification for polymer modified bitumens.* EN 14023: 2005.

[1.04] **Eurobitume Workshop 99**. *Performance Related Properties for Bituminous binders,* Luxembourg, 6th May 1999.

[1.05] **CEN TC336 WG1 Technical Report.** *Bitumen and bituminous binders – Development of performance-related specifications: status report 2005,* CEN/TR 15352.

[1.06] **Eurobitume BiTSpec Seminar**. *Bituminous binder testing and specifications*, Brussels, 12th & 13th June 2003.

[1.07] **CEN TC336 Advisory Group, Ad Hoc Report**. *Future binder specification system – Synthesis of stakeholders' needs and expectations*, CEN/TC 336/ AG N9, 2002.

[1.08] **FEHRL Website**. http://bitval.fehrl.org

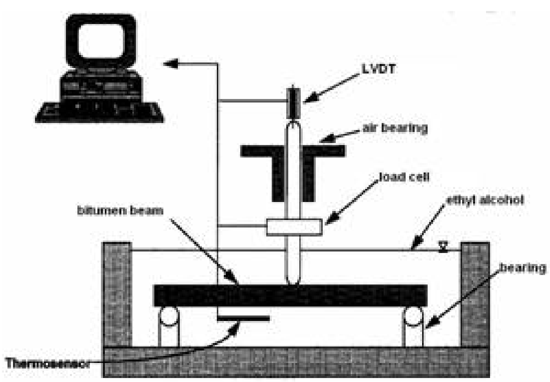
2. Bitumen tests

2.1 Bending Beam Rheometer (BBR) Test

2.1.1 Description

The BBR was developed from the SHRP project in the USA, where it has been used for at least 10 years and for over 6 years elsewhere. The European Standard for the BBR is EN 14771 [2.01].

The BBR is a three-point bending-beam test, designed to characterise the low-temperature behaviour of bituminous binders. The test determines the flexural creep stiffness of bituminous binders in the range of 30 MPa to 1 GPa by means of the bending beam rheometer. The bending beam rheometer is used to measure the mid-point deflection, in three-point bending, of a beam of bituminous binder. A constant load is applied to the mid-point of the test specimen for a defined loading time and the deflection is measured as a function of time. A low temperature liquid bath (ethyl alcohol) is used to control the temperature (Figure 2.1). The creep stiffness *S* of the test specimen for the specific loading times is calculated from the bending stress and strain. In addition to the creep stiffness, the logarithmic creep rate, generally known as the *m*-value, is determined. The *m*-value represents the slope of tangent to log *S* – log *t* graph at *t* = 60 s (Figure 2.2).



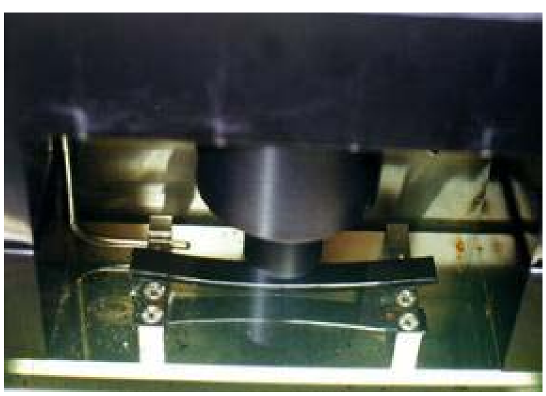


Figure 2.1 – BBR experimental setup

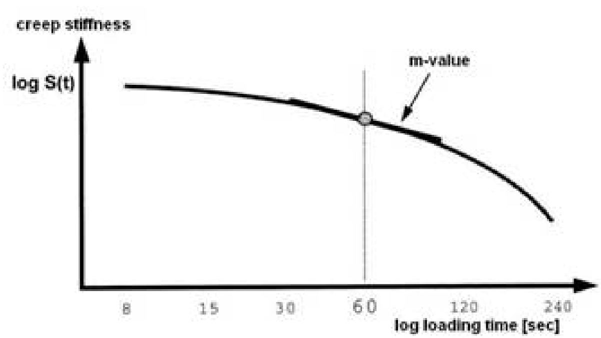


Figure 2.2 – BBR definition of the *m*-value [2.01]

2.1.2 Equivalent Standardised Tests

The test has been standardised in the USA as ASTM D 6648-01 [2.02].

2.1.3 Precision

The precision given in EN 14771 is a repeatability, *r*, of 9 % of the mean value and a reproducibility, *R*, of 27 % of the mean value for the creep stiffness and a repeatability, *r*, of 4 % of the mean value and a reproducibility, *R*, of 13 % of the mean value for the m-value.

2.1.4 Relationship with Other Bitumen Tests

There is a broad correlation with the Fraass breaking point (Section 2.10) for paving grade bitumens and also some suggestion with PMBs. However, the interpretation of the *m*-values found for some multigrade binders and for certain types of PMBs is not necessarily consistent with that for paving grade bitumen.

2.2 Binder Fatigue Test

2.2.1 Description

There is currently no European standard with only some laboratory test methods existing in a few laboratories around the world. In the test, a fatigue crack is induced by applying continuous oscillatory shear loading with a rheometer, as describe in Section 2.8. It has been shown [2.03, 2.04] that the DSR can only be used to evaluate fatigue properties in a narrow stiffness or temperature region. The fatigue phenomena due to the repeated of traffic loads imposed on binders is reputed to produce large deformations. Therefore, binders have to be tested in the non-linear region in order to accumulate significant damage [2.03].

All the tests are made with a parallel plate geometry [2.05] in order to compare the fatigue responses of binders for:

- The linear region of strain for one temperature and frequency, with the number of cycles needed to reduce the initial value of G* by 50 % being recorded.
- The non-linear region for one temperature and frequency, the change of G* versus the strain level between 5 % and 50 % being recorded.
- The non-linear region for one temperature and the same initial stress level, the change of G* versus the cycle number being recorded.

However, the failure mechanism inside the film of bitumen depends on the temperature at which the test is carried out and on the size of the gap [2.03] (Figure 2.3).

In order to observe fatigue cracking when the stiffness of the binder is high (10 MPa to 50 MPa with frequencies between 10 Hz and 50 Hz), it is necessary to ensure that the measurements are not biased by the compliance of the equipment. In this stiffness range, repeated sinusoidal oscillations, with controlled-stress as well as controlled-strain deformations, lead to an abrupt decrease in modulus after a certain number of loadings (Figure 2.4).

The different penetration grades cannot always be compared at a constant temperature because the stiffness of the binders needs to be high. For this situation, the test can be adapted [2.04] by comparing binders at a constant value of G*.

The test can also be adapted by conducting the test at the same temperature but with rest periods [2.05, 2.06]. In this case, the criterion is to retain the cumulative dissipated energy ratio for each binder.

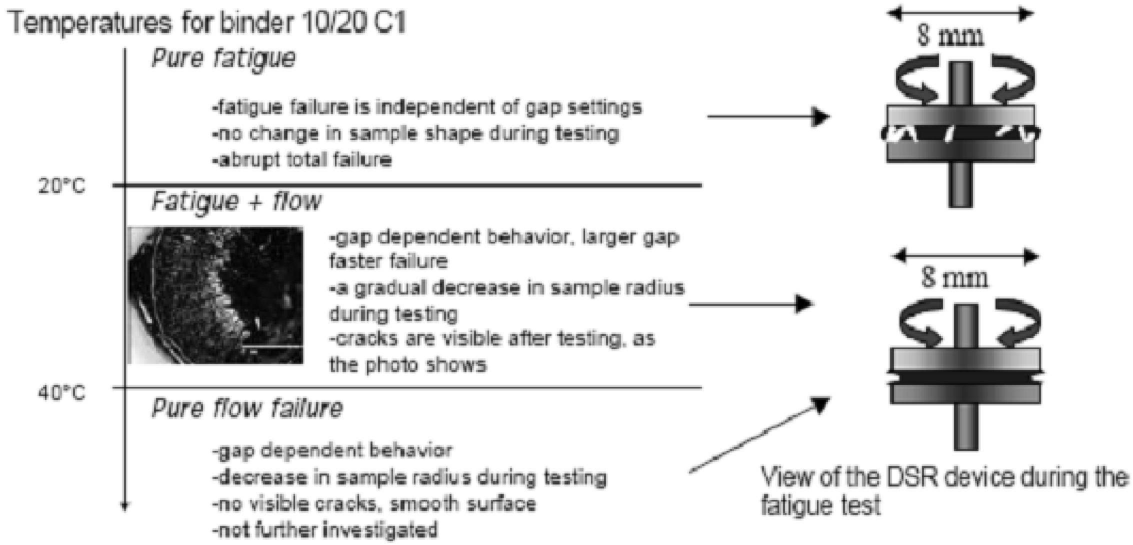


Figure 2.3 – Schematic phenomena taking place in the DSR during fatigue testing [2.03]

2.2.2 Equivalent Standardised Tests

There is no known equivalent standardised method for the binder fatigue test.

2.2.3 Precision

The repeatability and reproducibility of the tests and the confidence are not known to have been determined.

2.2.4 Relationship with Other Bitumen Tests

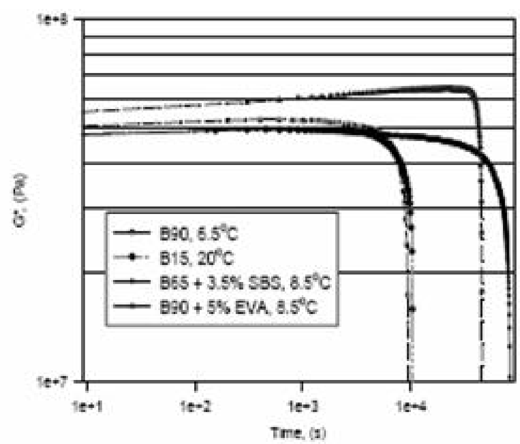


Figure 2.4 – Different binders fatigue results at the same initial strain level

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 binder fatigue test and other bitumen tests.

2.3 Capillary Viscometer Test

2.3.1 Description

There are two European Standard tests for measuring viscosity with capillaries.

EN 12595 [2.07] specifies a method for the determination of kinematic viscosity of bituminous binders at 60 ºC and 135 ºC, in the range from 6 mm²/s to 300 000 mm²/s. Results from the method can be used to calculate dynamic viscosity when the density of the test material is known or can be determined. This method is usually used to determine the viscosity of unmodified bitumen at 135 ºC.

Standard EN 12596 [2.08] specifies a method for the determination of dynamic viscosity by vacuum capillary of bituminous binders at 60 ºC, in the range from 0,0036 Pa.s to 580 000 Pa.s. This method is usually used to determine viscosity of unmodified bitumen at 60 ºC.

Preparation of samples and cleaning the tubes takes a lot of effort. These methods are not suitable for measuring modified bitumen.

2.3.2 Equivalent Standardised Tests

EN 12595 has been standardised elsewhere in the world as ASTM D 2170-95 [2.09] and EN 12596 as ASTM D 2171-94 [2.10].

2.3.3 Precision

The precision values are given in Table 2.1.

Table 2.1 – Precision values in EN 12595 [2.07] and EN 12596 [2.08]

Standard	Temperature (ºC)	Range	Repeatability, *r* (% of mean)	Reproducibility, *R* (% of mean)
EN 12595	135	< 600 mm²/s	4	6
		≥ 600 mm²/s	4	9
EN 12596	60	< 2000 Pa.s	6	12
		≥ 2000 Pa.s	6	10

2.3.4 Relationship with Other Bitumen Tests

The dynamic viscosity at 60 °C measured with the capillary viscometer test to the Australian standard AS 2341.02 [2.11] was correlated with other binder properties for multigrade binders [2.12]. A good correlation is reported with $G^*/\sin\delta$ 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.

2.4 Coaxial Cylinder Viscosity Test

2.4.1 Description

The test method described in European Standard EN 13702-2 [2.13] has been developed for modified binders, but it is suitable for all types of bituminous binders. In EN 13702-2, recommended test conditions are:

- temperature 60 ºC with shear rate 1 s^{-1},
- temperature 100 ºC with shear rate 100 s^{-1} and
- temperature 150 ºC with shear rate 100 s^{-1}.

2.4.2 Equivalent Standardised Tests

There is no known equivalent standardised method for the coaxial cylinder viscosity test. However, the cone and plate viscosity test to EN 13702-1 (Section 0) can be used as an alternative.

2.4.3 Precision

The European standard EN 13702-2 proposes the following precision data, at least until results of further round robin tests are available:

- Difference between two results under repeatability conditions > 5 % in one case in twenty.
- Difference between two results under reproducibility conditions > 15 % in one case in twenty.

2.4.4 Relationship with Other Bitumen Tests

It has been stated [2.14] that "Originally, the penetration was related to the steady state dynamic viscosity, which is difficult to measure below the 'Ring and Ball' temperature. The correlation between the penetration and the dynamic viscosity has been reconfirmed repeatedly".

The rheological property changes of two different paving grade binders (D 70 and D 200) modified by two different elastomers (SBS and SBR) during the laboratory-ageing test (TFOT) have been measured [2.15]. The results are given in Table 2.2. It is not specified which test was used to measure the dynamic viscosity. However, linear regression on the results in Table 2.2 gives a good correlation between dynamic viscosity at 60 ºC and R&B softening point (R^2 = 0,95) , but the number of binders considered is too small to derive a meaningful correlation coefficient.

Table 2.2 – Test results of two binders with modifications [2.15]

Binder	D 70			D 200		
	Base	+ 4 % SBR	+ 4 % SBS	Base	+ 4 % SBR	+ 4 % SBS
Penetration at 25°C (0,1 mm)	80	67,5	57,5	173	136	104
Softening Point R&B (°C)	43,8	47,8	50,7	36,2	41,8	50,4
Dynamic viscosity at 60°C (Pa.s)	216	260	334	93,2	216	296
After TFOT						
Change in weight (%)	0,046	0,015	0,012	-0,143	-0,176	-0,269
Penetration at 25°C	60	61,5	46,5	102	85,5	89
Softening Point R&B (°C)	48,1	50,7	53,3	43,3	49,1	54,4
Dynamic viscosity at 60°C (Pa.s)	298	470	534	202	370	389
Stiffening Indexes						
dPen25 (%)	25	8,9	19,1	41	37,1	14,4
dTR&B (°C)	4,3	2,9	2,6	7,2	7,3	4,1
Hardening index	1,4	1,8	1,6	2,2	1,7	1,3

Experimental data on the classical tests for a larger number of binders, including PMBs, has been found [2.16]. The data, reproduced in Table 2.3, show the dynamic viscosity in combination with penetration and R&B softening point. Simple regression of these data gives a weak correlation between R&B softening point and logarithm of dynamic viscosity at 60 °C with $R^2 = 0,48$.

Table 2.3 – Classical test results from several binders [2.16]

Binder	Binder Penetration (0,1 mm)	Softening Point (°C)	Dynamic Viscosity (Pa.s)
35 Pen grade	31	56,4	1 000
50 Pen grade	52	51,2	381
70 Pen grade	59	49,4	255
PMB 1	49	59,6	2 850
PMB 2	44	64,0	300
PMB 3	46	58,4	1 370
PMB 4	42	61,6	2 080
PMB 5	51	77,6	42 200
PMB 6	47	59,4	1 480
PMB 7	70	79,2	6 760
PMB 8	105	83,4	60 000
PMB 9	193	78,8	2 270
MG	40	61,8	2 680
WAX 1	46	67,4	335
WAX 2	44	65,6	144
WAX 3	51	59,4	1 140

The properties of multigrade bitumen have also been studied [2.17]. Although the method of viscosity measurement is not specified, the correlations in Table 2.4 were found. The correlations show that the binder property that is most closely correlated to the dynamic viscosity is the Ring and Ball softening point (after RTFOT).

Other references [2.18 to 2.22] all contain some data on the dynamic viscosity in combination with other rheological properties (although, in most of these references, the dynamic viscosity is measured with a Brookfield viscometer rather than the coaxial cylinder method). A general conclusion from these references is that a correlation is observed between dynamic viscosity and R&B softening point temperature, but the number of binders considered in each paper is too small to draw quantitative conclusions on the degree of correlation.

To summarise, the most significant study of the relation between the dynamic viscosity and other rheological properties is the one discussed in reference [2.16]. This study, which involves a high number of PMBs, shows that the correlation between the dynamic viscosity and the R&B softening point is rather weak.

Table 2.4 – Correlations for multigrade bitumen

Property	Correlated with	Correlation coefficient >0,9
Viscosity at 60 °C after RTFOT	Softening point (after RTFOT) Penetration (after RTFOT) Penetration index (after RTFOT) G*/sin δ (after RTFOT) δ (after RTFOT) G* (after RTFOT) Viscosity at 60°C (before RTFOT)	Yes No No No (0,58) No No Yes
Viscosity at 60 °C before RTFOT	Softening point (before RTFOT) Penetration (before RTFOT) Penetration index (before RTFOT) G*/sin δ (before RTFOT) δ (before RTFOT) G* (before RTFOT)	No No No No (0,68) No No

2.5 Cone and Plate Viscosity Test

2.5.1 Description

The test method described in European Standard EN 13702-1 [2.23] has been developed for modified binders, but it is suitable for all types of bituminous binders. In EN 13702-1, recommended test temperatures are 60 ºC, 100 ºC and 150 ºC and the shear rate is set at 0,05 s^{-1}, but the diameter and the angle of the cone are not prescribed.

2.5.2 Equivalent Standardised Tests

There is no known equivalent standardised method for the cone and plate viscosity test. However, the coaxial cylinder viscosity test to EN 13702-2 (Section 2.4) can be used as an alternative.

2.5.3 Precision

The European standard EN 13702-1 proposes the following precision data, at least until results of further round robin tests are available:

- Difference between two results under repeatability conditions > 5 % in one case in twenty.
- Difference between two results under reproducibility conditions > 15 % in one case in twenty.

2.5.4 Relationship with Other Bitumen Tests

The coaxial cylinder viscosity test and cone and plate viscosity test are similar tests [2.24] with the conclusions already given in Section 2.4.4.

2.6 Creep Zero Shear Viscosity Test

2.6.1 Description

The test is a binder creep test, designed to measure zero shear viscosity (ZSV, notated as η_0). ZSV is also referred to as the first Newtonian viscosity and is believed to be a suitable indicator to evaluate the partial contribution of the bituminous binder (including polymer modified binders) to the rutting resistance of asphalt. The test is conducted at elevated service temperatures, these being significant for rutting.

In a low shear creep test, ZSV is the inverse of the slope of the compliance curve in the steady state flow regime, where the slope becomes constant (Figure 2.5) according to Equation (2.1).

$$\frac{dJ(t)}{dt} = \frac{1}{\eta_0} \tag{2.1}$$

A procedure to perform the test in a shear rheometer has been defined [2.25]. The reproducibility of the results when following the test protocol was investigated. It was concluded that the test is suitable for conventional, multigrade and lightly modified binders. For highly modified binders, it was concluded that the steady state creep flow cannot be attained within a reasonable creep period and, hence, ZSV cannot be measured.

A draft test method was prepared by CEN TC 336/WG1/TG1, "High Temperature Performance", that, at the time of writing, was at CEN Enquiry stage. In this test method, the parallel plate geometry is recommended with a diameter of 20 mm or greater, a 2 mm gap and the conditions given in Table 2.5. The cone and plate geometry is also appropriate. The draft also specifies a range of the viscosity (100 to 50 000 Pa.s) beyond which the test is not applicable. The upper limit is in accordance with the conclusion discussed above [2.25].

Figure 2.5 – Compliance curve measured in a low shear creep test

Table 2.5 – Test conditions recommended by the draft European test method

Type of binder	Stress (Pa)	Time (h)	Temp. (°C)
Non modified	50	1	60
Polymer modified	10 – 50	4	60

The average viscosity over the last 15 min (900 s) is derived from the compliance curve according to Equation (2.2).

$$\eta_i = \frac{\Delta t}{\Delta J} = \frac{900}{(J_{end} - J_{15\,\min\,before\,end})} \tag{2.2}$$

If, at the end of the recommended creep time, the viscosity is still increasing by more than 5 % per 15 min, the creep time should be extended to a maximum of 8 h. If the viscosity increases by less than 5 % per 15 min, it is recorded as the steady state viscosity. If the test results are independent of the stress level (linear range), this steady state viscosity is accepted as the ZSV.

2.6.2 Equivalent Standardised Tests

There is no equivalent standardised test.

An alternative to the creep test for measuring the ZSV is the oscillation ZSV test (Section 2.13). However, the draft test method for the oscillation test has been designed to determine the equi-viscous temperature for a low shear viscosity (LSV) of 2000 Pa.s, while the creep ZSV test determines the ZSV (which is also actually a low shear viscosity) at a given temperature.

2.6.3 Precision

Precision was estimated in a round-robin exercise conducted by CEN TC336 WG1/TG1 involving 9 laboratories. Five binders were tested (2 pure bitumens and 3 PMBs). The resulting precision is given in Table 2.6.

Table 2.6 – Precision data of the ZSV by the creep test method

Statistic	Bit A	Bit B	PMB 1	PMB 2	PMB 3
Overall mean (Pa.s)	190	10481	3355	11908	904788
Repeatability coefficient of variation (%)	5,3	11,7	6,1	7,7	36,6
Reproducibility coefficient of variation (%)	15,1	17,4	12,3	17,3	91,4

PMB3 should not be considered because its overall mean ZSV value is outside the range of applicability mentioned in the draft test method (ZSV range from 100 to 50000 Pa.s).

The round robin established the limit of viscosity outside which the test is not applicable. This limit is reflected in the standard.

2.6.4 Relationship with Other Bitumen Tests

ZSV by the creep test and ZSV by the oscillation test theoretically determine the same binder property, so the results from both tests have been compared by various researchers [2.26 to 2.29]. Both tests give the same results for unmodified binders as well as for some binders with low polymer content. However, the results often differ for highly modified binders because either. The reason for this is that either:

- the steady state is not reached within a reasonable time of testing in the creep test (hence the draft standard for the test specifies an upper limit for the measurable ZSV); or
- the frequency is not sufficiently low to obtain the low frequency plateau in the viscosity curve in the oscillation test.

However, at the high concentration of polymer (high viscosity) when the two methods diverge, the standard for the creep test excludes its use.

Correlations between ZSV by the creep test and other binder properties related to permanent deformation were investigated for a set of 15 binders [2.27]. The creep period was one hour, regardless of whether or not the creep flow had reached steady state. Table 2.7 shows the correlations obtained for the unmodified binders. ZSV by the oscillation method, the SHRP parameter of G*/sinδ, the repeated creep test and the static creep test have all been found to correlate closely to the traditional rheological properties of penetration and R&B softening point.

Table 2.7 – Correlations between various bitumen tests for unmodified binders [2.27]

Linear correlation coefficients	Logarithmic (tests after 1 day storage)						Temperature	
	ZSV oscillation	G*/sin(δ)		RCT		Static Creep Test	PG grading	R&B
	0,001 Hz	0,001 Hz	1,59 Hz	25 Pa	300 Pa	25 Pa	(°C)	(°C)
Log(pen @ 25°C)	0,94	0,94	0,96	0,94	0,95	0,94	0,94	0,96
R&B (°C)	0,98	0,98	0,98	0,98	0,98	0,98	0,95	1,00
PG grading (°C)	0,97	0,97	0,99	0,98	1,00	1,00	1,00	0,95

Table 2.8 shows the correlation coefficients obtained for modified binders. For these binders, the static creep test has a good correlation only with the ZSV by the oscillation method, with the SHRP parameter of G*/sinδ when measured at a very low oscillation frequency of 0,001 Hz, and with the repeated creep test.

Table 2.8 – Correlations between various bitumen tests for modified binders [2.27]

Rheological test, after 1 day (logarithmic) at 50 °C	Logarithmic					
	ZSV oscillation 0,001 Hz	G*/sin(δ) 0,001 Hz	G*/sin(δ) 10 rad/s	RCT 25 Pa	RCT 300 Pa	Static creep
ZSV oscillation @ 0,001 Hz	1,00	0,94	0,02	0,99	0,77	0,94
G*/sin(δ) @ 0,001 Hz	–	1,00	0,03	0,94	0,75	0,92
G*/sin(δ) @ 10 rad/s	–	–	1,00	0,02	0,00	0,01
RCT @ 25 Pa	–	–	–	1,00	0,75	0,96
RCT @ 300 Pa	–	–	–	–	1,00	0,80
Static creep	–	–	–	–	–	1,00
SHRP grading	–	–	–	–	–	–
High PG temperature	0,46	0,49	0,21	0,50	0,09	0,31
Traditional tests:						
Log(pen @ 25 °C)	0,00	0,00	0,85	0,00	0,00	0,10
R&B (°C)	0,53	0,51	0,25	0,59	0,19	0,47

2.7 Direct Tensile Test (DTT)

2.7.1 Description

The Direct Tensile Test (DTT) is a procedure used to measure the strain at failure and stress at failure in an asphalt binder test specimen pulled at a constant rate of elongation. It can be used with unaged or aged material (Figure 2.6). The test apparatus is designed for testing within the temperature range from -36 °C to +6 °C. This test method was developed for binders at temperatures where they exhibit brittle or brittle-ductile failure. This failure will result in a fracture of the test specimen as opposed to a ductile failure in which the specimen stretches without fracturing. The test is not applicable at temperatures where failure is by ductile flow. Strain at failure is used as the criterion for specifying the low temperature properties of asphalt binders in accordance with the SHRP binder classification in conjunction with the BBR test (Section 2.1).

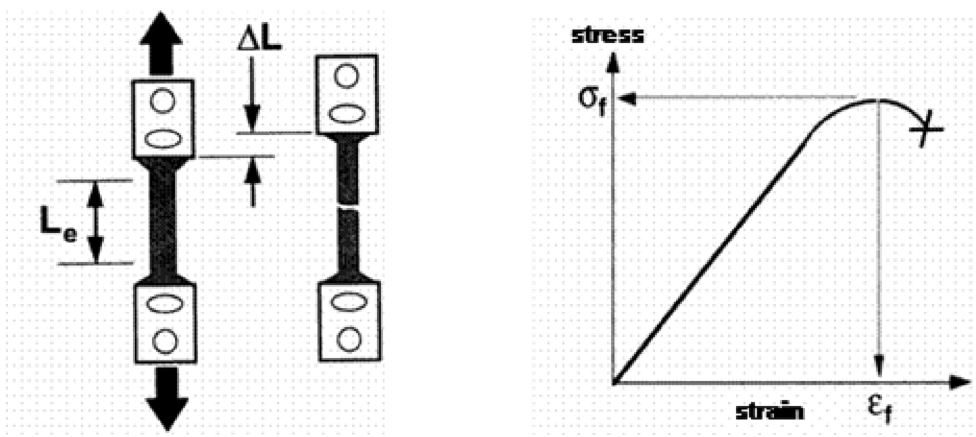


Figure 2.6 – DTT measurement principle [2.16]

2.7.2 Equivalent Standardised Tests

The direct tensile test is not yet standardised in Europe. However, the test has been standardised in AASHTO standards TP3-98 [2.28] and T314 [2.29].

2.7.3 Precision

The precision is not known to have been determined.

2.7.4 Relationship with Other Bitumen Tests

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 direct tensile test and other bitumen tests. Nevertheless, the combination of the BBR (Section 2.1) and DTT results can be used to determine a critical thermal cracking temperature.

2.8 Dynamic Shear Rheometer (DSR) Test

2.8.1 Description

EN 14770 [2.30], the European Standard for the DSR-test, describes a procedure for the determination of the complex shear modulus and phase angle using a dynamic shear rheometer (Figure 2.7). The test is performed in oscillatory shear, in stress or in strain controlled mode, over a range of temperatures and frequencies (Figure 2.8). The rheometer is fitted with parallel plates, with a constant gap. Temperature control encompasses both plates. Parallel plates with a diameter between 8 mm and 25 mm and gap settings from 0,5 mm to 2,0 mm are recommended.

The test consists of performing isothermal frequency sweeps at discrete temperature steps. The time between two frequency sweeps shall be sufficient to allow for thermal equilibrium in the sample. Isotherms of G* (Pa) and δ (º) against frequency (Hz) are the basic test results.

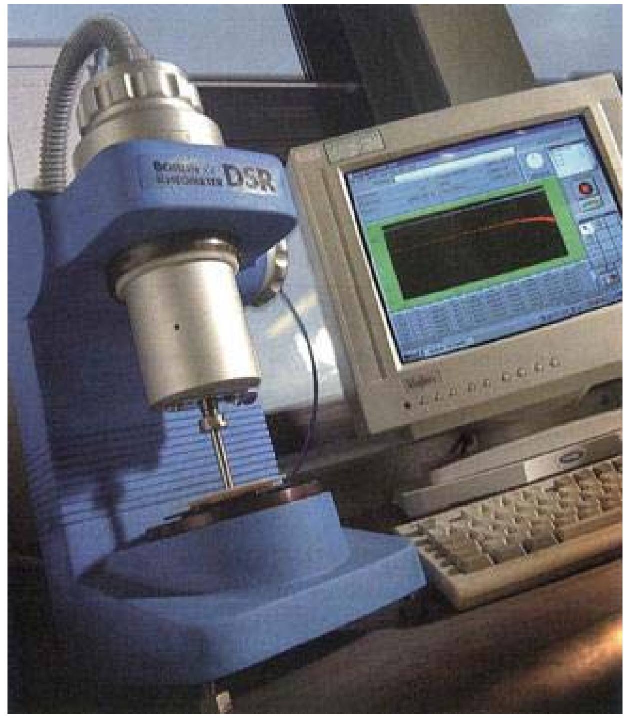


Figure 2.7 – Dynamic Shear Rheometer
(reproduced with kind permission from The Shell Bitumen Handbook [2.31])

2.8.2 Equivalent Standardised Tests

The test has been standardised in the USA as AASHTO TP5-97 [2.32].

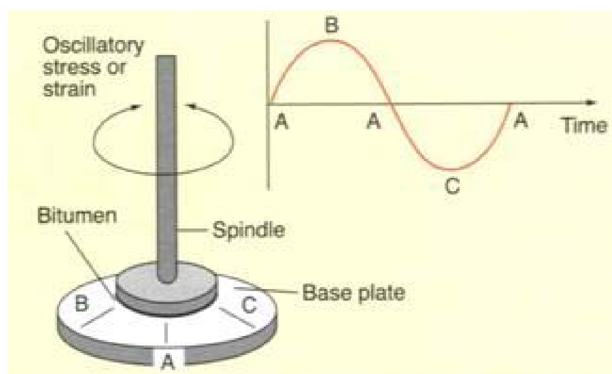


Figure 2.8 – Schematic of DSR mode of test
(reproduced with kind permission from The Shell Bitumen Handbook [2.31])

2.8.3 Precision

The precision statement from EN 14770 indicates that tests under reproducibility conditions have been carried out using the AASHTO test according to SHRP protocols, which are similar to EN 14770, and also by RILEM using nominally the same method on a range of binders. The test is approximately as precise as the softening point test (Section 2.17.3). In particular, the results from the RILEM exercise on the measurement of complex modulus and phase angle for rotational DSRs with parallel plate sample geometries of 25 mm, 1 mm gap and 8 mm, 2 mm gap, indicated that:

- Reproducibility of G* may be practically achieved in the range below 10 %, independent of the type of binder (pure or modified) and its state (original or aged by RTFOT or PAV).
- Reproducibility of phase angle may be practically achieved in the range below 5 %, independent of the type of binder (pure or modified) and its state (original or aged by RTFOT or PAV).

2.8.4 Relationship with Other Bitumen Tests

Penetration has been correlated with DSR measurements, as described in Section 2.15.4.

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. 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 [2.33] that the stiffness of the binder can be predicted from the penetration index and R&B softening point for paving grade bitumens.

At very low testing frequency, the ratio *G**/sinδ is related to the oscillation ZSV because ZSV is defined by equation (2.3).

$$\eta_0 = \frac{1}{\omega J''} = \frac{G^*}{\omega \sin \delta} \tag{2.3}$$

Hence, there is also a relation with creep ZSV.

2.9 Force Ductility Test

2.9.1 Description

The method and calculation of the deformation energy are described in EN 13589 [2.34] and EN 13703 [2.35]. The device consists of a water bath with temperature control and a traction device. The bitumen specimen is fixed onto the traction device (Figure 2.9). The specimen is elongated with a constant speed of (50 ± 2,5) mm/min. Usually, the test temperature is 5 °C but, depending on the brittleness of the specimen, the test temperature can be increased in steps of 5 °C. The maximum testing time is determined by the length of the water bath. The testing time is about 20 min for a water bath of a length of 1000 mm. Preparation time for the specimen is approximately 4 h (including the cooling). The primary use of the force ductility test is to distinguish between modified and unmodified bitumen. Polymer-modified bitumen is specified in EN 14023 [2.36]. The deformation energy between 200 mm and 400 mm is the specification criterion (Figure 2.10).

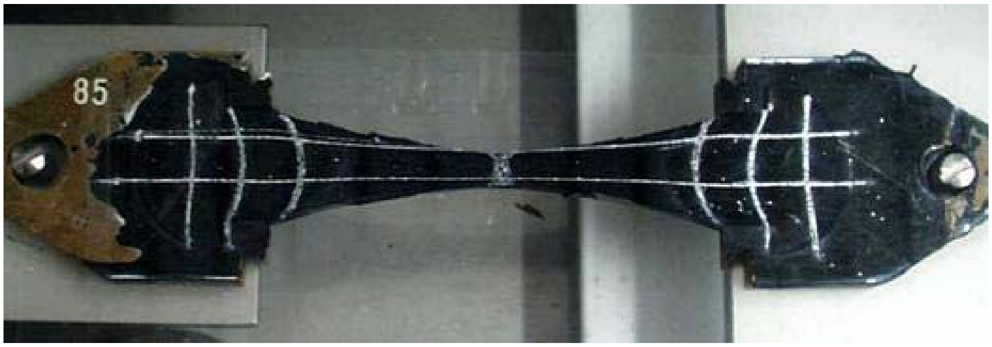


Figure 12.9 – Force ductility specimen

2.9.2 Equivalent Standardised Tests

There is no known equivalent standardised method for the force ductility test. However, the tensile test, EN 13587 [2.37], and the Vialit test, EN 13588 [2.38], are also used for assessing the cohesive properties of bitumen. Some correlation has been found [2.39] between the tensile test and the force ductility test. Generally, deformation energies measured by the tensile test are lower than values measured by force ductility at 5 °C. The sequence of the tested bitumen is almost the same for both methods. However, a systematic study comparing the three methods is missing.

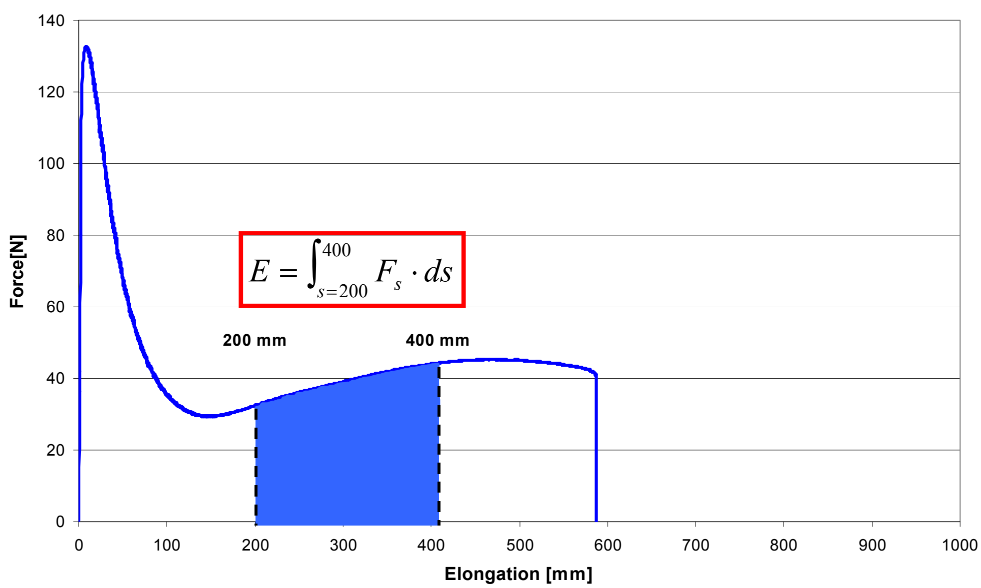


Figure 2.10 – Deformation energy (EN 14023)

2.9.3 Precision

Precision data was determined by a European Round-Robin-test under participation of 18 laboratories in 2002. The data were in accordance with EN 13703 [2.35]. The repeatability is 0,11 J/cm² for $E_{0.2\text{-}0.4} < 1$ J/cm² and 8 % for $E_{0.2\text{-}0.4} > 1$ J/cm² while the reproducibility is 0,39 J/cm² for $E_{0.2\text{-}0.4} < 1$ J/cm² and 33 % for $E_{0.2\text{-}0.4} > 1$ J/cm².

2.9.4 Relationship with Other Bitumen Tests

Correlation between the maximum energy of the force ductility curve and penetration has been found [2.40]. 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 (Figure 12.11).

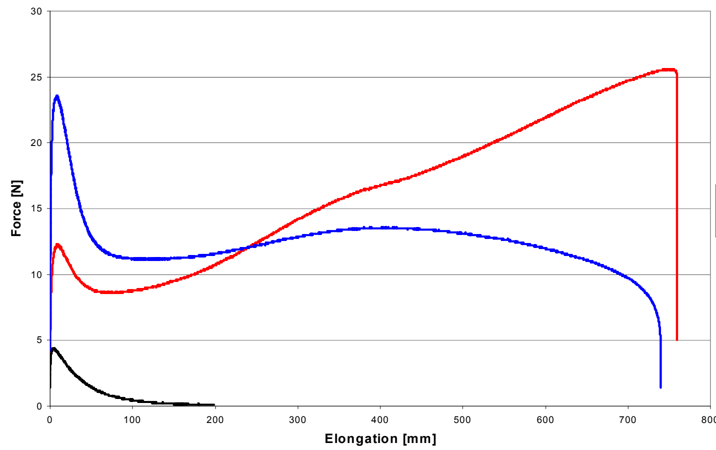


Figure 12.11 – Force ductility curves for three different PMB binders

The force ductility device can also be used for the determination of the elastic recovery, EN 13398 [2.41].

2.10 Fraass Breaking Point Test

2.10.1 Description

The Fraass breaking point test provides a measure of the brittleness of bitumen and bituminous binders at low temperatures. A sample of bituminous binder is applied to a metal plate at an even thickness. This plate is submitted to a constant cooling rate and flexed repeatedly until the binder layer breaks (Figure 2.12). The temperature at which the first crack appears is reported as the Fraass breaking point.

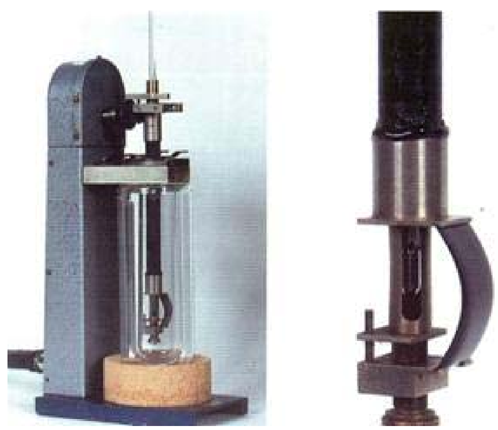


Figure 2.12 – Fraass breaking point (reproduced with kind permission from The Shell Bitumen Handbook [2.41])

2.10.2 Equivalent Standardised Tests

The European Standard for Fraass breaking point is EN 12593 [2.42]. This standard replaced several national versions, including BS 2000-80 [2.43] in the UK.

2.10.3 Precision

The precision given in EN 12593 [2.42] is a repeatability, *r*, of 3 ºC and a reproducibility, *R*, of 6 ºC.

2.10.4 Relationship with Other Bitumen Tests

There is a broad correlation of the Fraass breaking point test with the BBR (Section 2.1) for unmodified bitumen and also some suggestion with PMBs.

2.11 Fracture Toughness Test (FTT)

2.11.1 Description

The resistance to fracture of a material is known as its fracture toughness. Fracture toughness generally depends on temperature, environment, loading rate, the composition of the material and its microstructure, together with geometric effects (constraint). Fracture toughness is a critical input parameter for fracture-mechanics based on fitness-for-purpose assessments.

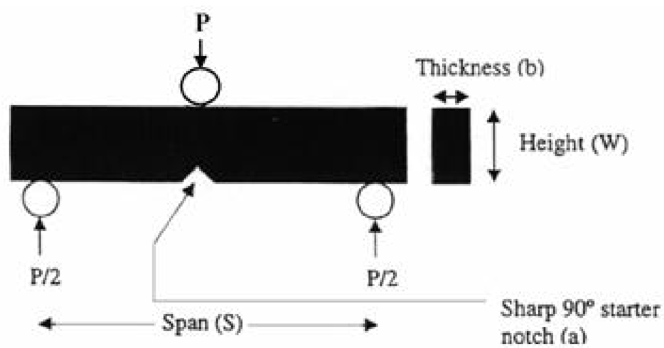
Various measures of ‘toughness' exist, including the widely used but qualitative Charpy impact test. Although it is possible to correlate Charpy energy with fracture toughness, a large degree of uncertainty is associated with correlations. It is preferable to determine fracture toughness in a rigorous fashion, in terms of *K* (stress intensity factor), CTOD (crack tip opening displacement), or *J* (the *J* integral).

Standards exist for performing fracture mechanics tests, with the most common specimen configuration shown in Figure 2.13 (the single-edge notch bend, SENB, specimen). A sharp fatigue notch is inserted in the specimen, which is loaded to failure. The crack driving force is calculated for the failure condition, giving the fracture toughness.

2.11.2 Equivalent Standardised Tests

There are no standards for the fracture toughness of bitumen. However, National Standards have been developed for fracture toughness testing of metals. In particular:

- The UK BS 7448 [2.44] includes four parts, for testing of metallic materials, including parent materials, weldments, high strain rates (dynamic fracture toughness testing, still in preparation) and *R*-curves (for ductile tearing). BS 7448: Part 2 is the first Standard worldwide to apply specifically to weldments.
- A series of American ASTM Standards [2.45] cover *K*, CTOD, *J* testing (including *R*-curves), together with a



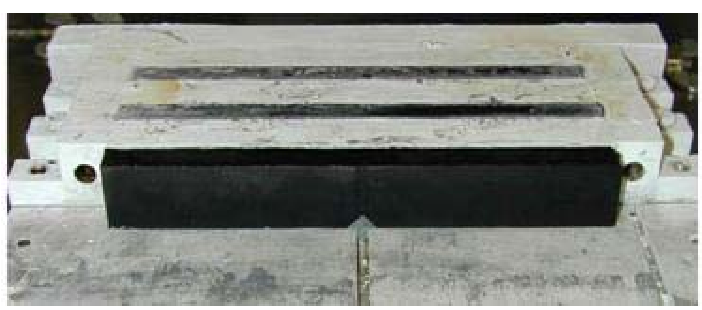


Figure 2.13 – FTT test layout and specimen preparation

summary of applicable terminology.

- The European Structural Integrity Society (ESIS) [2.46] has published procedures for *R*-curve and standard fracture toughness testing of metallic materials.

Although different Standards have historically been published for determining *K*, CTOD and *J*, the tests are very similar, and generally all three values can be established from one test. None of these standards specifies tests on bitumen specifically.

2.11.3 Precision

No precision data are published for FTT on bitumen yet.

2.11.4 Relationship with Other Bitumen Tests

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 fracture toughness test and other bitumen tests.

2.12 Oscillatory Squeeze Flow Rheometer

2.12.1 Description

The compressional rheometer is able to measure complex shear modulus (G^*), storage modulus (G') and loss modulus (G'') without the need for delicate air bearings and motor necessary for a typical controlled stress rheometer [2.47].

The sample is loaded between two parallel plates, the upper of which is driven by an oscillatory force in the axis normal to the plate surfaces as shown in the schematic in Figure 2.14. The force is generated by a linear motor and the displacement measured by a linear transducer. Temperature control is through Peltier elements placed in thermal contact with the lower plate. In addition, the sample and upper plate are covered by an insulated cover and the internal space is filled with water (c. 3 ml) to minimise thermal gradients. There is a Pt 100 in good thermal contact with the lower plate to sense the temperature, and another sensing the water temperature above the upper plate to ensure that the whole system is at thermal equilibrium.

This is a new technique for measuring bitumen properties and is less expensive and more robust than the dynamic shear rheometer because of its mechanical simplicity. It also does not need a water bath, computer or air supply.

This test is currently not included in any CEN standard test method because it has only recently been introduced.

It has been concluded [2.47] that, on the basis of both the quality of the data and the speed with which measurements can be made, this instrument combines the measurement of the rheological properties of bitumens, with the ability to rapidly predict traditional properties. As such, it offers potential as a quality assurance tool.

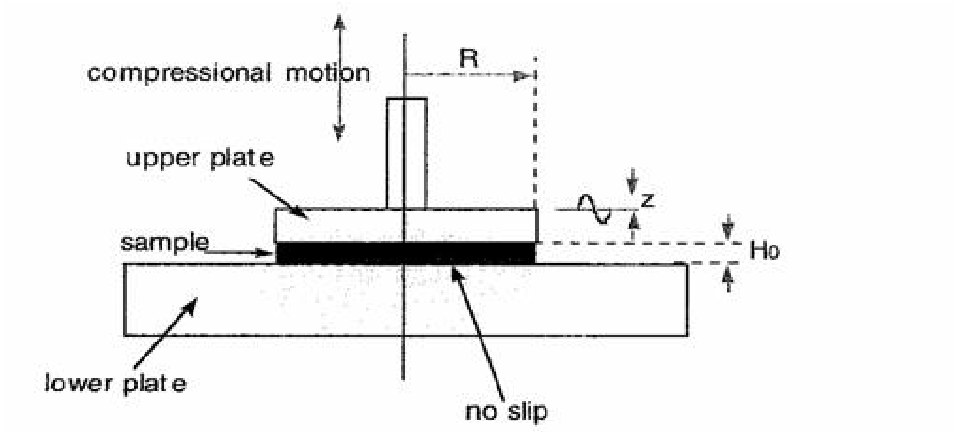


Figure 2.14 – Schematic of operation of compressional rheometer

2.12.2 Equivalent Standardised Tests

There are no current standards for the oscillatory squeeze flow rheometer, although it could be incorporated into the European Standard for the DSR, EN 14770 [2.30] (Section 2.8).

2.12.3 Precision

Of 58 measurements on 21 bitumens [2.47], only 3 failed to meet the most rigorous comparison between measured and predicted values of penetration. That is, the difference between them should be within the reproducibility of the penetration test itself. For bitumens which met CEN specifications in terms of penetration and softening point gradings, the softening point could be predicted accurately to within the reproducibility of the softening point test.

2.12.4 Relationship with other bitumen tests

The oscillatory squeeze flow rheometer was designed as an alternative to the dynamic shear rheometer (DSR).

Penetration has been correlated with DSR measurements, as described in Section 2.15.4. Measurements have been made using the compressional rheometer at 25 °C and 0,4 Hz to evaluate this relationship for the oscillatory squeeze flow rheometer test. Similarly, the R&B softening point, an equiviscous temperature, can be calculated using the DSR test (Section 2.8). It has been concluded [2.47] that measurement of *G** at 25 °C and 0,4 Hz can be used to predict bitumen penetration in approximately 12 min, and both penetration and softening point may be predicted in approximately 22 min if an additional measurement of G* at 60 °C and 0,4 Hz is made.

2.13 Oscillation Zero Shear Viscosity Test

2.13.1 Description

It is possible to perform an oscillation test as an alternative to the creep zero shear viscosity test (Section 2.6) in order to determine the zero shear viscosity (ZSV, again notated as η_0).

In the frequency domain, ZSV is related to the loss compliance $J''(\omega)$ by [2.48] according to Equation (2.4).

$$J''(\omega) - \int_0^{\infty} \omega \left[J_{de}(\infty) - J_{de}(t) \right] \cos \omega t \, dt = \frac{1}{\omega \eta_0} \tag{2.4}$$

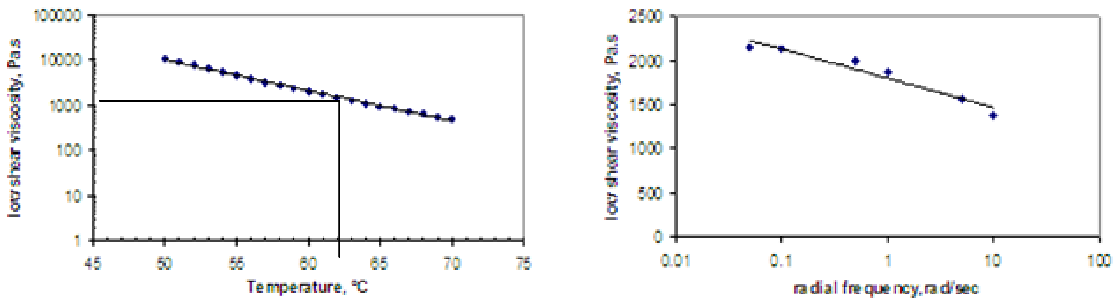
Consequently, when the oscillation frequency tends to zero, the zero shear viscosity can be found in accordance with Equation (2.5).

$$\eta_0 = \frac{1}{\omega \, J''(\omega)} = \frac{G^*}{\omega \sin \delta} \tag{2.5}$$

The test is performed by means of a dynamic shear rheometer in oscillation mode.

In 2004, a draft test method was prepared by Technical Committee CEN TC 336 WG1/TG1, "High Temperature Performance". At the time of writing, the draft was at CEN Enquiry stage. In this draft, the term ZSV is replaced by Low Shear Viscosity (LSV) because it is practically not possible to measure at zero shear. The same comment is also applicable to the creep ZSV. This test is also not performed at zero shear, but at a low shear rate. Instead of measuring the LSV at a given temperature, an equi-viscous temperature based on LSV is determined (temperature at which LSV equals 2000 Pa.s).

The test is performed in two steps (Figure 2.15):



Step 1: Temperature sweep at 0,1 rad/s

Step 2: Frequency sweep at EVT1

Figure 2.15 – Principle of the measurement of the equi-viscous temperature for LSV equal to 2000 Pa.s

- Step 1 consists of a temperature sweep at a frequency of 0,1 rad/s. The temperature at which the viscosity attains the value of 2000 Pa.s is a first approximation of the equi-viscous temperature (EVT1)
- In step 2, a frequency sweep is performed at the temperature EVT1, down to a very low frequency (e.g. from 10 down to 0,01 rad/s) to obtain the LSV at EVT1. The difference between this LSV and 2000 Pa.s allows the increase in temperature with respect to EVT1, required in order to obtain a LSV of 2000 Pa.s, to be determined. The correction of EVT1 by this increase in temperature leads to equi-viscous temperature EVT2.

Step 2 may not be necessary for pure binders because the viscosity of these binders reaches a plateau at sufficiently low frequencies. Therefore, EVT2 ≈ EVT1. For heavily modified binders, EVT2 can be significantly higher than EVT1.

2.13.2 Equivalent Standardised Tests

No equivalent standardised tests are currently known to exist. However, the test is related to the creep ZSV test (Section 2.6).

2.13.3 Precision

Two round robin tests have been carried out by CEN TC 336/WG1/TG1, in which 15 laboratories participated. Five binders were studied, including two pure bitumen and three PMBs. The outcome of these tests is given in Table 2.9.

2.13.4 Relationship with Other Bitumen Tests

Oscillation ZSV is related to $G^*/\sin\delta$, as shown in Equation (2.6), when measured at the same low frequency, because both properties are theoretically interrelated and measured using the same type of test equipment (i.e. DSR).

$$\eta_0 = \frac{1}{\omega J''} = \frac{G^*}{\omega \sin\delta} \tag{2.6}$$

Table 2.9 – Precision values for the oscillation zero shear viscosity test

Parameter	Statistic	Bit A	Bit B	PMB 1	PMB 2	PMB 3
EVT1 (at 2000 Pa.s)	Overall mean (°C)	44,7	69,3	60,8	67,5	60,6
	Repeatability std. dev. (°C)	0,7	0,7	0,7	1,0	0,6
	Reproducibility std. dev. (°C)	0,7	1,0	1,5	1,6	1,9
EVT2	Overall mean (°C)	45,4	70,9	63,1	71,6	66,7
	Repeatability std. dev. (°C)	0,6	0,8	0,7	0,7	0,7
	Reproducibility std. dev. (°C)	1,0	1,8	2,2	2,5	2,3

The relation with creep ZSV was already discussed in Section 2.6.4.

Correlations of ZSV by oscillation at a temperature of 50 °C with other binder properties have been reported [2.27]. Tables 2.7 and 2.8 in Section 2.6.4 show the linear correlation coefficients obtained. It is observed that oscillation ZSV (or LSV) at 0,001 Hz correlates closely with the results from the repeated creep test. The correlations with $G^*/\sin\delta$ at 0,001 Hz and with the creep ZSV were also good.

Correlation data between the equi-viscous temperatures by the oscillation zero shear viscosity test and other bitumen tests have not yet been reported.

2.14 Penetration Test

2.14.1 Description

The penetration of a standard needle into a conditioned test sample is measured (Figure 2.16). For penetrations up to 500 x 0,1 mm, the operating parameters are a test temperature of 25 °C, an applied load of 100 g, and a duration of loading of 5 s.

For penetrations above 500 x 0,1 mm, the test temperature is reduced to 15 °C but the operating parameters of the applied load and the duration of loading remain unchanged.

There is also a penetration test at 15 °C with a higher load and longer loading time.

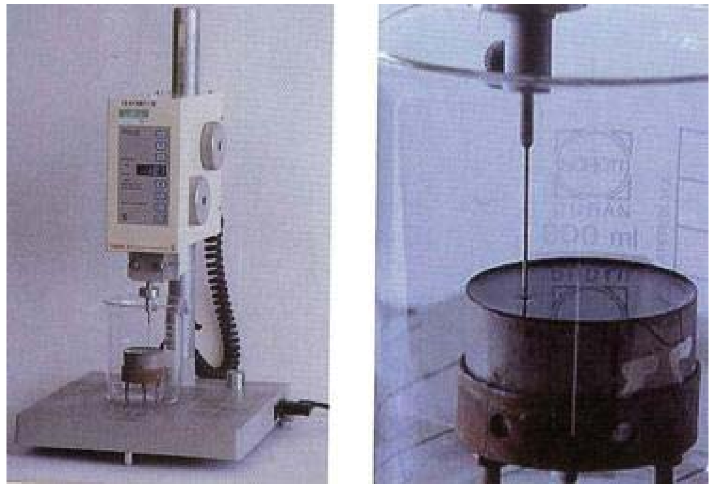


Figure 2.16 – Penetration test
(reproduced with kind permission from The Shell Bitumen Handbook [2.31])

2.14.2 Equivalent Standardised Tests

EN 1426 [2.49] is the European Standard for the test, but the same conditions are standardised elsewhere in the world and, previously, throughout the countries in Europe.

2.14.3 Precision

The precision for paving grade bitumen is given in Table 2.10. These precision data are not necessarily applicable at other conditions or for modified bitumen.

Table 2.10 – Precision for penetration test

Operating conditions			Penetration in 0,1 mm	Repeatability, *r*	Reproducibility, *R*
Temperature	Load	Duration			
25 °C	100 g	5 s	< 50 ≥ 50	2 4 % of mean	3 6 % of mean
15 °C	100 g	5 s	≥ 50	5 % of mean	8 % of mean
5°C	200 g	60 s	< 50 ≥ 50	2 9 % of mean	4 13 % of mean

2.14.4 Relationship with Other Bitumen Tests

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) [2.50].

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.

Although many other 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.

2.15 Penetration Index

2.15.1 Description

The penetration index, *PI*, [2.31] is a measure for the temperature susceptibility of a bitumen that can be derived mathematically either from the penetration values at two temperatures or from the standard penetration and softening point values, as given in Equations (2.7) and (2.8) respectively.

$$PI = \frac{\log(pen@T_1) - \log(pen@T_2)}{T_1 - T_2} \tag{2.7}$$

$$PI = \frac{1952 - 500\log(pen) - 20\,SP}{50\log(pen) - SP - 120} \tag{2.8}$$

However, *SP* in Equation (2.8) is the ASTM (unstirred) softening point which will generally be 1.5 °C higher for unmodified bitumen than the EN 1427 (stirred) value.

The values of *PI* range from around -3 for highly temperature susceptible bitumens to around +7 for highly blown bitumens with low temperature susceptibility [2.31]. For paving grade bitumen used for highways, the typical range is -1,5 to +1,0.

2.15.2 Equivalent Standardised Tests

The calculation of the penetration index has not been standardised.

2.15.3 Precision

The precision of the measure is dependant on the precision of the measurement of the penetration and softening point or on the two values of penetration.

2.15.4 Relationship with Other Bitumen Tests

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.

2.16 Repeated Creep Test

2.16.1 Description

The repeated creep test was designed to determine the resistance of the binder to permanent deformation under conditions of repeated loading and unloading cycles. The test is conducted at elevated service temperatures, which are significant for rutting.

An AASHTO test protocol has been published in NCHRP report 459 [2.51]. According to this protocol, the test is performed using a dynamic shear rheometer at low stress level (between 25 and 300 Pa at the outer edge of the plates). The loading time is typically 1 s, but 2 s or 3 s could also be used. The ratio between loading time and unloading time has to be 1:9 (e.g. 9 s unloading for 1 s loading). The sample is subjected to 100 cycles and the strain is measured as a function of time. The test data of cycles 50 and 51 are fitted using the four-element Burgers model (Figure 2.17). This model yields the value of the viscosity η_0 of the serial dashpot of Burger's model, which is responsible for the permanent deformation component. The creep stiffness G_v, calculated from Equation (2.9), is proposed as an indicator for the resistance to permanent deformation.

$$G_v(t) = \frac{\eta_0}{t} \tag{2.9}$$

where t is the total loading time

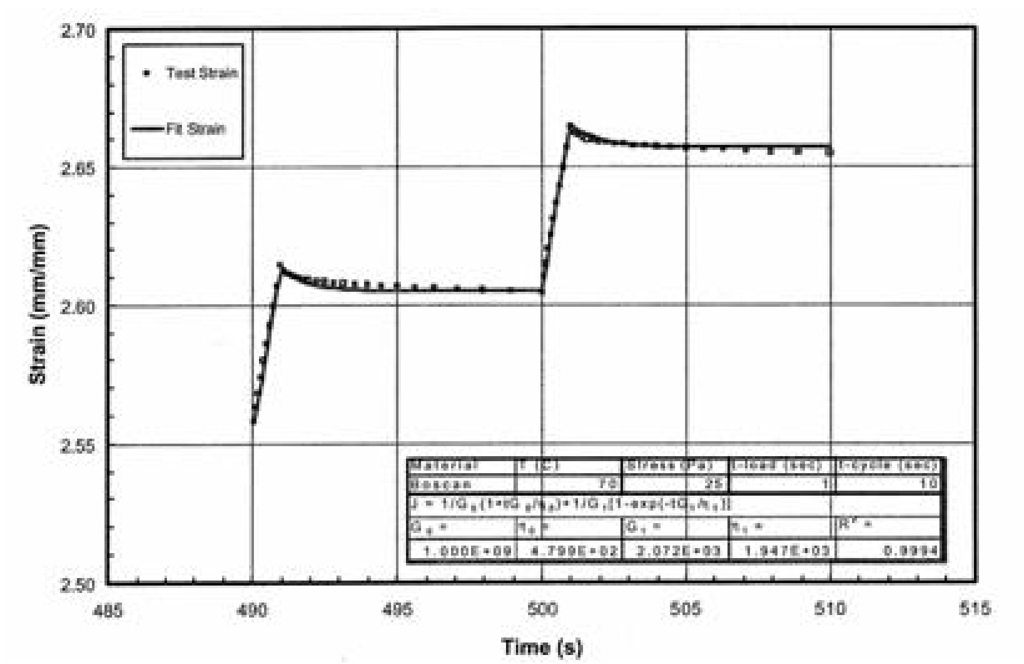


Figure 2.17 – Determination of the viscosity η_0 using Burger's model [2.51]

2.16.2 Equivalent Standardised Tests

The Repeated Creep Test has not yet been standardised in Europe.

2.16.3 Precision

The precision is not known to have been determined.

2.16.4 Relationship with Other Bitumen Tests

The data of the repeated creep test are fitted using Burger's four-parameter model. The viscosity of the serial dashpot in Burger's model theoretically equals the ZSV (Sections 2.6 and 2.13). A comparison was made between the ZSV derived from fitting the repeated creep test with Burger's model and the results from both the creep ZSV and oscillation ZSV tests [2.26]. The observed systematic under-estimation of ZSV with the repeated creep test was explained by the creep cycles being too short to attain steady state shear flow. The same conclusion was drawn in a comparison of the results from oscillation ZSV to the ZSV derived from the repeated creep test [2.52].

The results of the repeated creep test have been compared with the oscillation ZSV test and the creep ZSV test for a total of 13 pure and modified binders [2.53]. Figure 2.18 shows the range of accumulated strain. The accumulated strain correlated reasonably well with ZSV.

Correlations between the repeated creep test and other bitumen tests have also been reported in [2.27] (see Tables 2.8 and 2.9 in Section 2.6.4). A good correlation is reported with ZSV by the oscillation test, G*/sinδ at 0.001 Hz and with the creep ZSV.

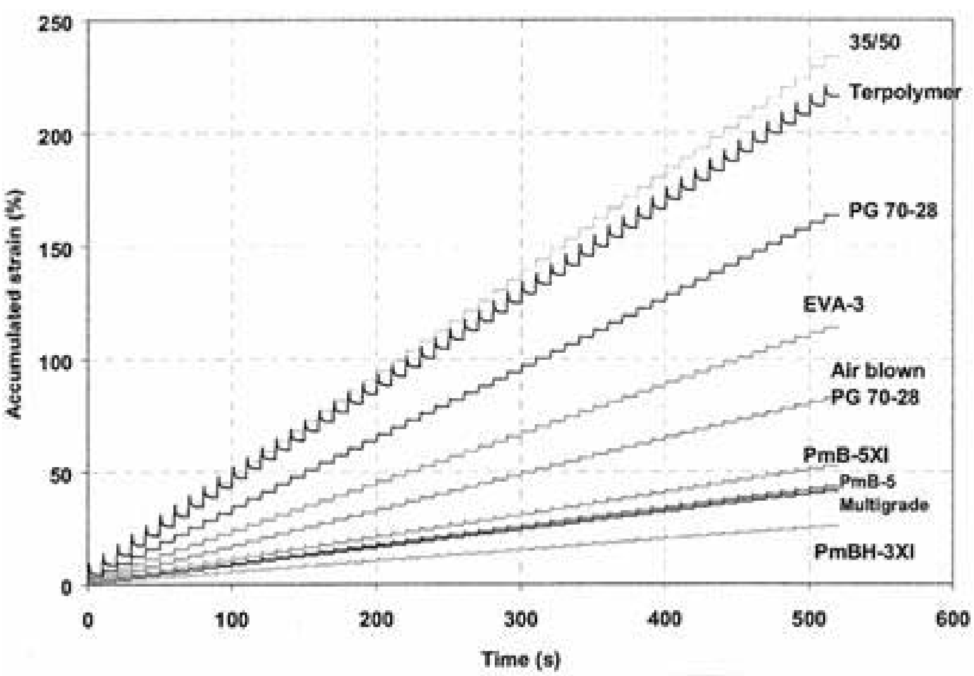


Figure 2.18 – Repeated creep tests at 60 °C and a stress level of 50 Pa [2.53]

2.17 Softening Point (Ring and Ball) Test

2.17.1 Description

Softening Point (Ring and Ball) Test is a method for the determination of the softening point of bitumen and bituminous binders, in the range 30 °C to 150 °C. Two horizontal discs of bituminous binder, cast in shouldered brass rings, are heated at a controlled rate in a liquid bath while each supports a steel ball (Figure 2.19). The softening point is reported as the mean of the temperatures at which the two discs soften enough to allow each ball, enveloped in bituminous binder, to fall a distance of (25,0 ± 0,4) mm.

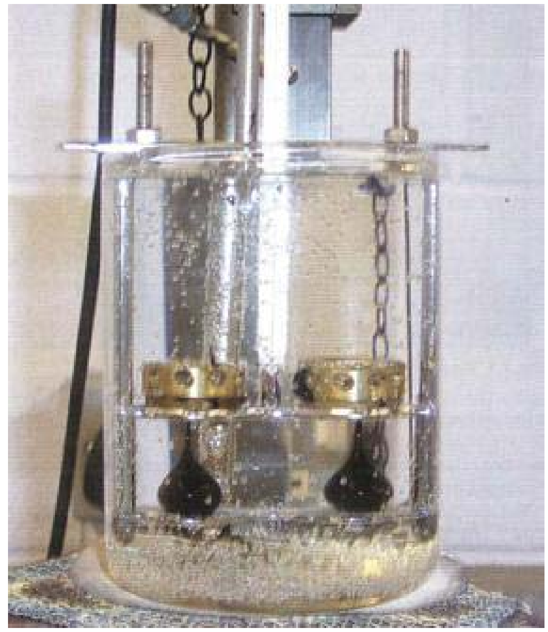


Figure 2.19 – Softening point test
(reproduced with kind permission from The Shell Bitumen Handbook [2.41])

2.17.2 Equivalent Standardised Tests

The European Standard for R&B softening point test is EN 1427 [2.54]. Prior to 1999, most countries had their own versions of the test which were very similar to the harmonised test. The principal difference was that some standards, including ASTM D36-95 [2.55], do not include stirring the liquid bath, as in EN 1427, without which the result will generally be 1.5 °C higher for unmodified bitumen; this difference may not apply to modified binders.

2.17.3 Precision

The precision quoted in EN 1427 is:

- Repeatability, *r*, of 1 °C and reproducibility, *R*, of 2 °C for unmodified bitumen in water.
- Repeatability, *r*, of 1.5 °C and reproducibility, *R*, of 3.5 °C for modified bitumen in water.
- Repeatability, *r*, of 1.5 °C and reproducibility, *R*, of 5.5 °C for oxidised bitumen in glycerol.

2.17.4 Relationship with Other Bitumen Tests

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.

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.

2.18 Tensile Test

2.18.1 Description

The tensile test is performed at a constant stretching speed and temperature and was originally intended for polymer modified bitumens. Test specimens are elongated until failure or up to a given proportional elongation over their initial length. The European standard for the test is EN 13587 [2.37].

The procedure is based on similar methods used on other materials such as rubber or plastics. The tensile properties measured are useful as indicators for quality assessment of the materials. One of them, the conventional energy (calculated in accordance with EN 13703 [2.35]), has been chosen as the specification criterion to evaluate the cohesion characteristics of polymer modified bitumens.

Different test temperatures (ranging from –20 ºC to +20 ºC) and speeds (1, 10, 50, 100 and 500 mm/min) can be used. The test temperature is kept within ± 0,5 C by means of a temperature chamber. Test equipment also includes appropriate attachment jaws for a correct clamping of the specimens, force and elongation measurement devices.

Binder specimens are cast using dumbbell-shaped moulds of fixed dimensions (H2 type). However, other geometries are allowed given the difficulties found when preparing and working with this type of specimen. The results obtained from different geometries are not equivalent.

The tensile force applied and the elongation of each specimen are recorded during the test, so that the force against elongation curves can be obtained. The tensile properties normally reported are stress and proportional elongation at:

- the flowing threshold;
- fracture;
- 400 % elongation; and
- the maximum elongation if fracture is not reached.

Further calculations from the curves include the conventional (or cohesion) energy at 400 % elongation, which is the quotient of the deformation energy at this point and the initial cross section of the specimen (EN 13703 [2.35]).

2.18.2 Equivalent Standardised Tests

There are no known equivalent standardised methods.

2.18.3 Precision

In accordance with EN 13703 [2.35], the repeatability for the conventional energy, in J/cm², corresponding to an elongation of 0,2 m (400 %), $E'_{0,2}$ is 10 % and the reproducibility is 30 %.

2.18.4 Relationship with Other Bitumen Tests

The tensile test has several common features with the direct tensile test (DTT) (Section 2.7) 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 [2.37], 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 [2.34] (Section 2.9). 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 drawbacks of a fixed stretching speed of 50 mm/min and a narrower test temperature range of 5 ºC to 25 ºC.

EN 13703 for deformation energy [2.37] states two calculation procedures for the conventional energy, depending on the method followed. Different specification limits have to be fixed for each test result.

2.19 Vialit Pendulum Test

2.19.1 Description

The Vialit pendulum test assesses the degree of cohesion of the bituminous binder. The general layout is shown in Figure 2.20 and the apparatus used for carrying out the test is shown in Figure 2.21. The procedure, which is standardised in EN 13588 [2.56], involves placing a thin film of binder between two steel cubes and measuring the energy required to remove the upper block. The cracks appear in the bitumen film and not at the steel-bitumen interface. It is of greatest significance in situations where aggregate is placed in direct contact with traffic stresses, for example in surface dressings and the chippings in hot rolled asphalt surface courses. The maximum impact energy is usually increased by polymer modification, as is the overall energy across the entire temperature range. Its significance for other materials has yet to be fully evaluated [2.58].

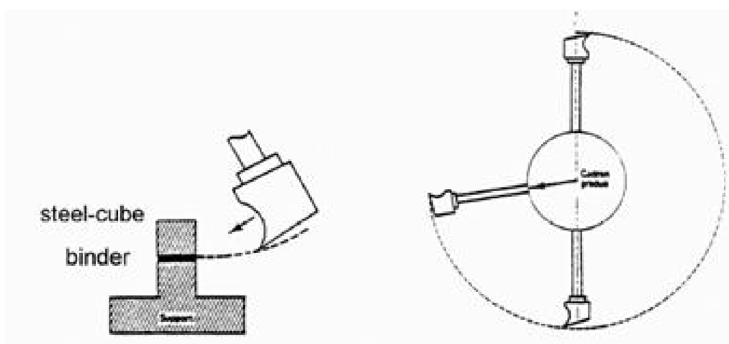


Figure 2.20 – Schematic of the Pendulum test [2.57]

2.19.2 Equivalent Standardised Tests

No equivalent identified.

2.19.3 Precision

The following precision data are the best currently estimated and are proposed until results of further round robin tests are available.

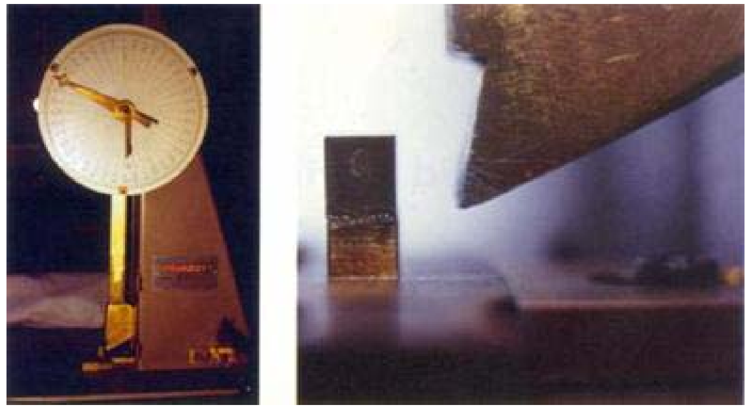


Figure 2.21 – Vialit pendulum test

For unmodified bitumens:

- Repeatability: difference between 2 successive results < 0,06 J/cm^2.
- Reproducibility: difference between 2 single results < 0,18 J/cm^2.

For modified bitumens:

- Repeatability: difference between 2 successive results < 0,10 J/cm^2.
- Reproducibility: difference between 2 single results < 0,36 J/cm^2.

2.19.4 Relationship with Other Bitumen Tests

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.

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3. Binder Conditioning Regimes

3.1 Rolling Thin-Film Oven Test (RTFOT)

3.1.1 Description

The rolling thin film oven test (RTFOT) is one of the most commonly used standardised tests to simulate the short-term ageing of binders. This test is used to measure the combined effects of heat and the air on a thin film of bitumen or bituminous binder in permanent renewal. It simulates the hardening which a bituminous binder undergoes during the mixing, transporting and compacting processes, referred to as short-term ageing.

The RTFOT in accordance with EN 12607-1 [3.01] involves rotating eight glass bottles, each containing 35 g of bitumen, in a vertically rotating shelf, while blowing hot air into each sample bottle (Figure 3.1). During the test, the bitumen flows continuously around the inner surface of each container in relatively thin films at a temperature of 163 °C for 75 min. The vertical circular carriage rotates at a rate of 15 revs/min and the air flow is set at a rate of 4000 ml/min. The method ensures that all the bitumen is exposed to heat and air and the continuous movement ensures that no skin develops to protect the bitumen. The effects of this treatment are determined from measurements of the properties of the binder before and after the test and from determining the change in mass [3.02, 3.03].

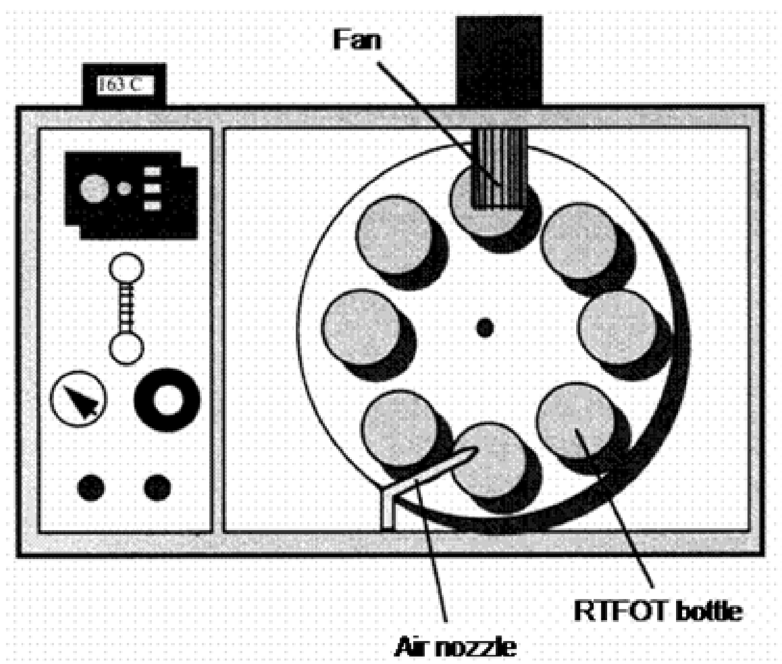

Figure 3.1 – RTFOT, schematic drawing [3.02]

3.1.2 Equivalent Standardised Tests

The RTFOT test has been standardised in the USA as ASTM D 2872 [3.04]. Two other ageing procedures that used in a similar manner are the thin film oven test (TFOT), EN 12607-2 [3.05], and the rotating flask test (RFT), EN 12607-3 [3.06].

3.1.3 Precision

The precision quoted in EN 12607-1 [3.01] is repeated in Table 3.1.

Table 3.1 – RTFOT precision [3.01]

		Repeatability, *r*	reproducibility, *R*,
Change of mass (> 0,3 % & < 0,8 %)	(% absolute)	0,15	0,20
Penetration	(% absolute)	7	10
∆R&B < 6,5 °C > 6,5 °C		1,5 2,0	2,0 4,0
Dynamic viscosity ratio @ 60°C	(relative %)	10 %	20 %

3.1.4 Predictive capacity of the RTFOT

Four bitumens were used to make a hundred samples of bitumen and corresponding asphalt mixtures, manufactured with the three main types of plants (discontinuous, continues, 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 statistics 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 is a good method to indicate the risk of premature hardening of asphalt mixtures.

3.2 Thin-Film Oven Test (TFOT)

3.2.1 Description

Another test to simulate the short-term ageing of binders is the thin film oven test (TFOT). In the TFOT, a 50 ml sample of bitumen is placed in a flat 140 mm diameter container resulting in a film thickness of 3,2 mm. Two or more of these containers are then positioned on a shelf rotating at 5 rpm to 6 rpm in an oven for 5 h at 163 °C. The TFOT was adopted by AASHTO in 1959 and by ASTM in 1969 (ASTM D1754 [3.08]) as a means of evaluating the hardening of bitumen during plant mixing. However, a major criticism of the TFOT is that the thick binder film results in a large volume to the exposed surface area of the binder. There is a concern that ageing (primarily volatile loss) may be limited to the 'skin' of the bitumen sample because the bitumen is not agitated or rotated during the test [3.02].

3.2.2 Equivalent Standardised Tests

The European Standard for the TFOT is EN 12607-2 [3.05]. The test has also been standardised in the USA as ASTM D 1754 [3.08].

3.2.3 Precision

The precision quoted in EN 12607-2 [3.04] is repeated in Table 3.2.

Table 3.2 – TFOT precision [3.04]

	Repeatability, *r*	reproducibility, *R*,
Change of mass TFOT 120 °C ≤0,1 (% absolute) >0,1 (% absolute)	 0,02 % 8 % of mean value	 0,14 % 38 % of mean value
Dynamic Viscosity ratio @ 60 °C Ratio <1,5	 6 % of mean value	 16 % of mean value

3.2.4 Relationship with Other Conditioning Regimes

Although many conditioning methods modify the binder properties by similar means and therefore there will be some relationship, no formal correlation has been found in the papers reviewed between the thin film oven test and other bitumen conditioning methods.

3.3 Rotating Flask Test (RFT)

3.3.1 Description

The RFT method consists of ageing a 100 g sample of bitumen in the flask of the rotary evaporator for a period of 150 min at a temperature of 165 °C. The material forming the surface of the specimen is constantly replaced because the flask is rotated at 20 rpm, thus preventing the formation of a skin on the surface of the bitumen [3.02].

3.3.2 Equivalent Standardised Tests

The European Standard for the RFT is EN 12607-3 [3.06].

3.3.3 Precision

The precision quoted in EN 12607-3 [3.06] is repeated in Table 3.3.

3.3.4 Relationship with Other Conditioning Regimes

Although many conditioning methods modify the binder properties by similar means and therefore there will be some relationship, no formal correlation has been found in the papers reviewed between the rotating flask test and other bitumen conditioning methods.

Table 3.3 – RFT precision [3.06]

		Repeatability, *r*	reproducibility, *R*,
Change of mass (> 0,3 % & < 0,8 %)	(% absolute)	0,15	0,20
Penetration	(% absolute)	7	10
ΔR&B < 6,5 °C > 6,5 °C		1,5 3,0	2,0 4,0
Dynamic Viscosity ratio @ 60°C	(relative %)	10 %	20 %

3.4 Modified RTFOT

3.4.1 Description

One of the main problems with using the RTFOT for modified bitumens is that these binders, because of their high viscosity, will not roll inside the glass bottles during the test. In addition, some binders have a tendency to roll out of the bottles. To overcome these problems, the modified rolling thin film oven test (MRTFOT) was developed. The test is identical to the standard RTFOT except that a set of 127 mm long by 6.4 mm diameter steel rods are positioned inside the glass bottles during oven ageing. The principle is that the steel rods create shearing forces to spread the binder into thin films, thereby overcoming the problem of ageing high viscosity binders. Initial trials of the MRTFOT indicate that the rods do not have any significant effect on the ageing of conventional penetration grade bitumens. However, recent research work has indicated that using the metal rods in the MRTFOT does not solve the problem of roll-out of modified binder and further validation work is required before the technique can be accepted [3.02].

3.4.2 Equivalent Standardised Tests

None found.

3.4.3 Precision

No data found.

3.4.4 Relationship with Other Conditioning Regimes

Although many conditioning methods modify the binder properties by similar means and therefore there will be some relationship, no formal correlation has been found in the papers reviewed between the modified rotating flask test and other bitumen conditioning methods.

3.5 Pressure Ageing Test (PAV)

3.5.1 Description

The Pressure Ageing Vessel (PAV) was developed in the SHRP project to simulate long-term, in-service oxidative ageing of bitumen in the field. The method involves hardening of bitumen in the RTFOT or TFOT followed by oxidation of the residue in a pressurised ageing vessel. In the USA, the PAV procedure entails ageing 50 g of bitumen in a 140 mm diameter container (giving a binder film that is approximately 3,2 mm thick) within the heated vessel, pressurised with air to 2,07 MPa for 20 h at temperatures between 90 °C and 110 °C (Figure 3.2) [3.02]. Elsewhere, other conditions have been used such as reducing the temperature to 85 °C for an extended 65 h in the UK, when the test is known as PAV_{85} or the high pressure ageing test (HiPAT).

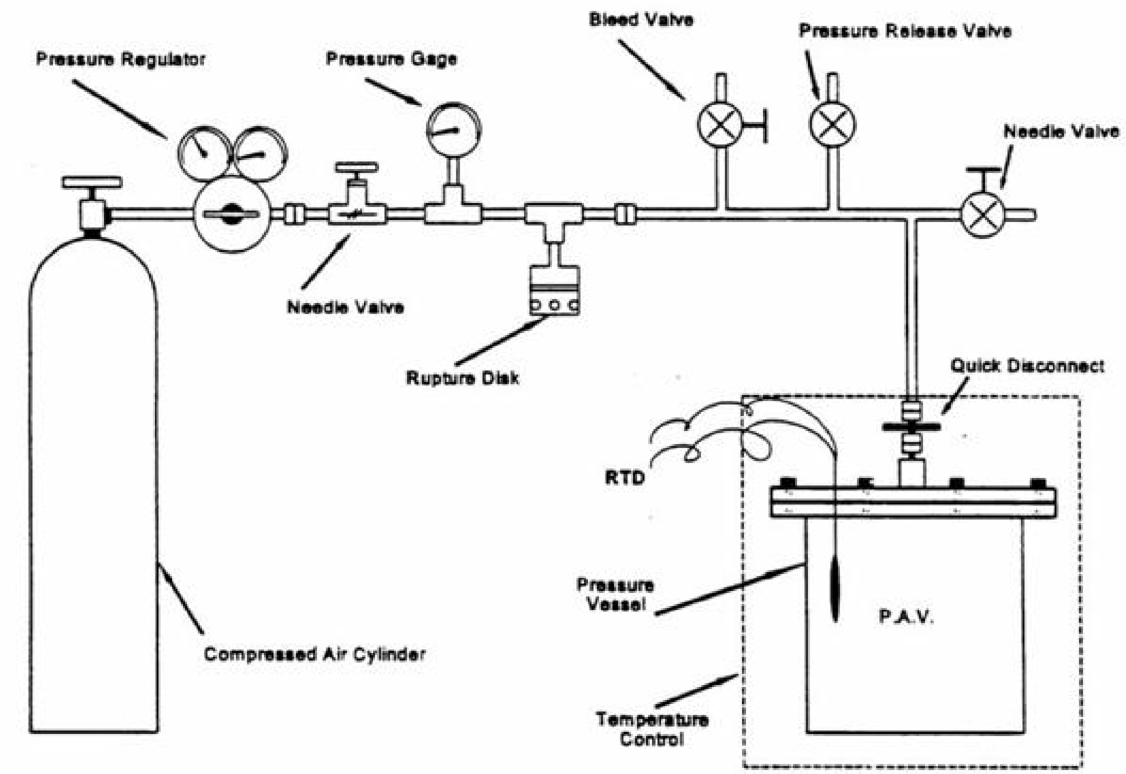

Figure 3.2 – Pressure Ageing Vessel [3.02]

3.5.2 Equivalent Standardised Tests

The European Standard for the PAV is still in draft form as prEN 14769 [3.09]. The test has also been standardised in the USA as AASHTO PP1-98 [3.10].

3.5.3 Precision

No precision data are published for PAV yet.

3.5.4 Relationship with Other Conditioning Regimes

20 h of PAV ageing at 100 °C was found to correspond to 178 h of RCAT ageing [3.02]. However, subsequent comparisons found the value was (176 ± 16) h with RCAT at 85 °C (Section 3.6).

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 [3.02].

3.6 Rotating Cylinder Ageing Test (RCAT)

3.6.1 Description

RCAT is a device for ageing/conditioning bituminous binders [3.11]. Tests simulating short-term ageing (STA) and/or long-term ageing (LTA) of paving grade or modified bitumen or of mastics can be performed. At the time of writing, a European standard for the test has been submitted for CEN enquiry as prEN 15323 [3.12].

The test consists of a closed testing cylinder in stainless steel with a central opening which allows taking small test portions at predetermined exposure times (Figure 3.3) [3.12]. During the test, this opening is fitted with a Teflon plug through which a stainless steel tube penetrates almost to the rear of the device in order to feed the cylinder with a constant flow of oxygen (LTA) or air (STA). For temperature calibration purposes, a thermocouple can be fixed on this tube. During test, the testing cylinder rotates at 1 revs/min (LTA) or 5 revs/min (STA) on two round drive bars in a ventilated oven. A grooved solid stainless steel roller is also introduced inside the rotating cylinder and makes a gravity-induced rotating movement about its axis to press and distributes the binder in the cylinder into an even film against the inner wall of the cylinder [3.13].

(a) (b)

Figure 3.3 –The RCAT device (a) and its scheme (b)

The parameters for short and long-term ageing of bitumen (or mastics) with RCAT are summarised in Table 3.4 [3.11, 3.14].

The RCAT method is a conditioning method but measurement of the characteristics at different time allows a kinetic approach of the ageing phenomenon. This approach is based on the fact that the development of some characteristics of the binder [3.15, 3.16] can be described in a linear manner using Equation (3.1).

$$S_t = S_0 + \sqrt[n]{K.t} \quad \text{with} \quad K = k \cdot \left(S_0 - S_f\right)^n \tag{3.1}$$

S_o, S_t and S_f represent the reaction indicator S at time t = 0, t = t and t = time at the end of its functional life (that is, when the binder can no longer fulfil its intended role in the asphalt mixture), K the overall reaction constant, k the rate constant. R&B softening point, reciprocal of the logarithm of the penetration, asphaltenes content A7 and rheological characteristics (viscosity, complex modulus [3.17], phase angle, etc.) can serve as reaction indicators. Some infrared absorption intensities can also be used.

Table 3.4 – Experimental parameters for short- and long-term ageing of binders

RCAT	STA: RCAT163*	LTA: RCAT90*
Sample	Bitumen or mastic	
Quantity	500 - 550 g	
Duration	235 min	140 h
Rotation speed (min-1)	5	1
Gas	air	oxygen
Flow rate (l/min)	4,0	0,075
Temperature (°C)	163	90
Pressure (MPa)	0,1	
Film thickness (mm)	± 2,5	
Characteristics measurement	At the end	After 17, 65, 140 h

* Value represents testing temperature (°C) during the RCAT conditioning.

3.6.2 Equivalent Standardised Tests

The European standard for the RCAT is still in draft form as prEN 15323 [3.12]. There are no known equivalent standardised methods.

3.6.3 Precision

For the short-term conditioning, no data are published.

For the long-term ageing, a limited round robin test [3.12 3.18] involving 5 laboratories and 3 binders (50/70 bitumen, 50/85 SBS PMB and 50/85 EVA PMB) was undertaken to determine the reproducibility levels. The monitored characteristics were penetration and R&B softening point for different exposure times at 90 °C. The conclusion was that the standard deviation of reproducibility (s_R) for penetration and R&B softening point was almost unaffected by the ageing procedure, except in the case of penetration for an EVA PMB. Table 3.5 gives the reproducibility values found in this experiment.

Table 3.5 – Value of reproducibility, *R*, obtained for penetration and R&B softening point from the round robin test with the RCAT
(both non-aged and aged cases taken into account)

Test	Bitumen	PMB average (SBS and EVA resp.)
Penetration (mm/10)	5 (or less)	6 (4 – 8)
R&B softening point (°C)	2.6	3.4 (2.4 – 4.4)

3.6.4 Relationship with Other Conditioning Regimes

The RCAT method has been compared to other bitumen conditioning regimes such as RTFOT (EN 12607-1 [3.01], Section 3.1) and PAV (prEN 14769 [3.08], Section 3.5) because it can be used to simulate short- and/or long-term ageing. In Table 3.6, the characteristics of each method are reviewed.

The two major advantages of RCAT method are the use of the same device to simulate short-term and long-term ageing separately or in combination and the large amount of aged binder or mastic prepared. (It is possible to age mastics with a filler content of up to 30 % by volume without any major problem [3.19] - more information about ageing mastics can be found [3.20, 3.21].) The latter advantage allows a great reduction in the number of handling operations that are useless and/or detrimental to the repeatability of characteristics determination [3.11, 3.19, 3.22].

In the case of short-term ageing, RTFOT and RCAT163 ageing are approximately equivalent [3.11]. For equivalent ageing, the RCAT163 method takes longer than the RTFOT procedure but has the advantage that a larger amount of aged material is prepared (about 12 times ignoring that 8 bottles are conditioned together in RTFOT).

In the case of long-term ageing, literature gives more details about the advantages and the disadvantages of RCAT method compared to the PAV100 conditioning [3.11, 3.22], where the value represents testing temperature (°C) during the PAV conditioning. The most important are:

- The ageing of the sample is homogeneous because RCAT test is dynamic. With the PAV test, due to the thickness of the binder film and to the static test, the concentration of oxygen will not be distributed homogeneously during the reaction. In spite of the pressure applied, the diffusion phenomenon will lead to differences in ageing between the surface and the bulk of the exposed sample. Furthermore, in the case of the static test (PAV), the ageing of PMBs also presents some scatter because of the migration of certain polymers to the surface [3.11].
- The amount of aged binder is sufficient for further tests, and test portions (25 g to 30 g) can easily be taken at various intervals to monitor the development of characteristics and properties with reaction time (kinetic approach).
- The temperature of RCAT90 test is more appropriate because 100 °C appears as a transition temperature between ageing mechanisms [3.13] (Section 3.8.1).
- The pressure condition is safer because of some risks of accident with a pressurised cabinet exposed to a temperature of 100 °C.
- The duration of the RCAT test is its major disadvantage: 240 h for RCAT85 and 140 h for RCAT90. These run times exceed largely the 20 h needed for PAV100.

The correlation between PAV100 and RCAT90 ageing methods was estimated by the comparison of results for the increase in R&B softening point. For eight pairs of values, the correlation factor, R^2, was 0,80 [3.23].

Furthermore, 20 h of PAV100 was established to be equivalent to (176 ± 16) h of RCAT85 and to 125 ± 11 h of RCAT90. However, for certain PMB ageing, a larger variability can be observed which can be due to the segregation of polymer during PAV test [3.11, 3.22].

In the literature, there is no comparison between RCAT and PAV85 (HiPAT) available.

Table 3.6 – Comparison of the experimental parameters of short- and long-term ageing/conditioning tests

Method	STA		LTA		
	RTFOT	RCAT163	PAV100	RCAT85	RCAT90†
Test	Dynamic		Static	Dynamic	
Sample	Binder		Binder	Binder or mastic	
Quantity (g)	8 x 35	500 – 550	10 x 50	500 – 550	
Duration	75 min	235 min	20 h	240 h	140 h
Rotation speed (rev/min)	15	5	/	1	
Gas	air		air	oxygen	
Gas flow (l/min)	4		/	0,075	
Temperature (°C)	163		100	85	90
Pressure (MPa)	0,1		2,1	0,1	
Approx. film thickness (mm)	1,25	2,5	3,2	2,5	
Characteristics measurement	At the end		At the end	After 17, 65, 140 h	

† Currently the RCAT90 condition is used but a lot of previous articles were published with RCAT85. A temperature of 90 °C is preferred for reasons of test duration.

3.7 Weatherometer

No papers were found in the database that made reference to weatherometers, the use of which has never been standardised. Therefore, no description of weatherometers is offered here.

3.8 Correlation of Laboratory Ageing with Field Performance

3.8.1 Review of Durability Test Methods

There are many conditioning regimes that have been designed to simulate the relative hardening that occurs during the mixing and laying process by accelerated ageing of a thin film of the binder in the presence of oxygen at elevated temperature. Some regimes also subject the binder to pressure oxidative ageing in order to simulate the longer-term process of ageing in the field. These conditioning regimes are described in the previous sections.

An extensive list of the various conditioning regimes, with their principal parameters, has been compiled [3.02] and is reproduced here as Table 3.7.

Table 3.7 – Bitumen ageing methods [3.02]

Test Method	Temp. (ºC)	Time (h)	Size (g)	Film (mm)	Extra Features
Thin film oven test (TFOT)	163	5	50	3,2	–
Modified TFOT (MTFOT)	163	24	–	0,10	–
Rolling TFOT (RTFOT)	163	1,25	35	1,25	Air flow 4000 ml/min
Extended RTFOT (ETFOT)	163	8	35	1,25	Air flow 4000 ml/min
Nitrogen RTFOT (NRTFOT)	163	1,25	35	1,25	N_2 flow 4000 ml/min
Modified RTFOT (MRTFOT)	163	1,25	35	1,25	Steel rods
Rotating flask test (RFT)	165	2,5	100	–	Flask rotation 20 rpm
Shell microfilm test	107	2	–	0,005	–
Modified Shell microfilm test	99	24	–	0,020	–
Modified Shell microfilm test 2	107	2	–	0,015	–
Rolling microfilm oven test (RMFOT)	99	24	0,5	0,020	Benzene solvent
Modified RMFOT	99	48	0,5	0,020	1,04 mm dia. opening
Tilt-oven durability test (TODT)	113	168	35	1,25	–
Alternative TODT	115	100	35	1,25	–
Thin film accelerated ageing test (TFAAT)	130 or 113	24 or 48	4	0,160	3 mm dia. opening
Iowa durability test	65	1 000	50 *	3,2	2,07 MPa pure oxygen
Pressure oxidation bomb	65	96	– †	0,030	2,07 MPa pure oxygen
Rotating cylinder ageing test (RCAT)	70 to 110	144	500	2,0	4 to 5 l/h pure oxygen
Pressure ageing vessel (PAV)	90 to 110	20	50 * or ‡	3,2	2,07 MPa pure oxygen
High pressure ageing test (HiPAT)	85	65	50 ‡	3,2	2,07 MPa pure oxygen

* TFOT residue † ERTFOT residue ‡ RTFOT residue

The conclusions from the review of these tests [3.02] found that short-term ageing conditioning methods fall into two categories: oven heating and extended mixing. The most commonly used binder ageing tests are the high temperature TFOT and RTFOT, developed to simulate the hardening that occurs during the mixing, transporting and compacting processes of asphalt. However, bitumen aged in the TFOT or RTFOT experience higher volatile loss during testing relative to that experienced during low temperature field ageing of pavement mixtures. It is accepted that RTFOT and similar tests are probably adequate for short-term ageing because there is good evidence that they simulate it fairly well. Nevertheless, the regimes may need to overcome operational difficulties when testing PMBs.

However, the mechanism of oxidative ageing in these high-temperature oven ageing tests differs from field ageing. Based on the inability of these high-temperature oven ageing tests to predict field ageing, tests were introduced with reduced temperatures and increased ageing times. The most commonly used tests to simulate long-term ageing are currently the

PAV and RCAT. Nevertheless, none of the ageing regimes of TFOT, RTFOT and PAV are able to simulate bitumen field ageing of porous asphalt mixtures.

The changes in rheological properties, IR spectra and reaction mechanisms of a range of unmodified and polymer-modified binders between the PAV at 100 ºC and the RCAT at 85 ºC are quite similar. However, the higher temperature of the PAV does result in some segregation of the polymer in some PMBs. Therefore, in terms of long-term ageing, although no one test seems to be satisfactory for all cases, the RCAT method, based on a kinetic approach to ageing, is probably the most acceptable or, possibly, the PAV method at 85 ºC (HiPAT).

3.8.2 *In situ* ageing of the bituminous binders in road pavements

The *in situ* ageing of the bituminous binders in road pavement is generally assumed to be dependent on the air voids content of the surface layer. The higher the air voids content, the deeper the oxidation in the exposed layer. The ageing in the surface layer occurs only in the upper 0.5 to 1 centimetre of the layer in contact with the air in dense bituminous layers (voids <5 %) and over the full thickness of the layer in porous asphalt mixtures.

By comparison with changes induced by field ageing, it has been established [3.24, 3.25, 3.26, 3.27] that binder ageing is characterised by:

- Changes in the generic composition: appearance of oxidised functions (development of ester, acid and sulfoxide functions) and transformation of the cyclic compounds into resins and then into asphaltenes with preferential formation and accumulation of resins during field ageing, contrarily to the “in construction” ageing with preferential formation of asphaltenes.
- Modifications of the properties: an increase of R&B softening point and a decrease of penetration.
- Variations in rheological properties: viscosity, complex modulus and phase angle.

3.8.3 Correlation of laboratory ageing with field performance

Different comparisons between some binder characteristics obtained after a complete RCAT cycle of binder ageing and those measured on binder recovered from road section with correspondent binders are reported in the literature:

- For dense asphalt mixtures (unmodified bitumen), ageing binders during 144 h at 85 °C with RCAT is consistent with 20 years of Belgian field ageing of the surface layer in the case of the asphaltenes content [3.25, 3.26].
- For porous asphalt (pure bitumen, SBS or EVA modified bitumen), 240 h at 83 °C was determined to be equivalent to 12 years on the road considering the asphaltene content A_7. However, when looking at values of penetration, R&B softening point, stiffness and phase angles, a more severe ageing was observed on the road than with RCAT [3.17].

The correlation of laboratory ageing with the field performance can also be realised by a kinetic study based on the measurement of reaction indicators on the samples recovered during the ageing-process. More information can be found [3.13, 3.15, 3.25, 3.28, 3.29].

3.8.4 Field correlations: ageing/durability

Nearly 25 trafficked sections were monitored during seven years to evaluate the ageing of four bitumens and the cracking behaviour of the asphalt mixture [3.30]. The physico-chemical data of the new, artificially ageing with the RTFOT and the PAV and of the binders extracted from the asphalt after seven years of age, were given. The various results of this programme of work make it possible to consider evaluating the influence of the rheological characteristics of the bitumens on the phenomenon of ageing and cracking by the top of the asphalt.

A model of kinetics of ageing of the bitumens has been proposed [3.30] based on the data of RTOT and PAV together with the physico-chemical and rheological characteristics, mostly correlated with the degree of cracking. Deduction from the critical points making it possible to predict the phenomenon of cracking. This work highlights that, for a given type of asphalt (same void content, binder content, aggregate gradation, etc), the thresholds are reached after variable periods depending on the quality of the bitumen.

Table 3.8 shows the most significant parameters with respect to the degree of cracking according to the analysed bitumens and to the differences between the characteristics of the artificially aged bitumens and the original bitumen, both initially after time in service.

Table 3.8 – Importance of the correlation between the physico-chemical characteristics and the ageing of the bitumen

Parameters	Original Bitumen	RTFOT + PAV	Delta PAV/original	Road @ 7 years	Delta road/original
R&B Softening Point	★★	★★	★	★★★	★★
% asphaltenes	★★★	★★★		★★★	
T °C, δ = 27 °				★★★★★	★★★★★
T °C, δ = 45 °	★	★★	★★	★★★★	★★★★
T °C, S = 300 MPa					★★★★
m60 @ T °C, S = 300 MPa	★	★★		★★★★	★★★
T °C @ m = 0,3			★★★	★★★★★	★★★★★

The number of stars indicates the strength of the correlation, with more stars equating to stronger correlation, as shown in Table 3.9.

Table 3.9 – Star rating for correlation coefficient values

Value of r	Numbers stars
$0,5 < r \leq 0,6$	★
$0,6 < r \leq 0,7$	★★
$0,7 < r \leq 0,8$	★★★
$0,8 < r \leq 0,9$	★★★★
$0,9 < r \leq 1$	★★★★★

The parameters that have the strongest correlations with cracking are the asphaltene content, the R&B softening point temperature and the rheological parameters of the temperature giving a phase angle of 27° and the *m*-value for *S* = 300 MPa. These correlations are more marked if the analysis is undertaken on the ratio of property to that of the original bitumen.

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4. Permanent Deformation

4.1 Asphalt tests

Permanent deformation is the irreversible structural change in an asphalt layer caused by high pressure on the surface at elevated service temperatures. The relevant European asphalt tests are EN 12697-22 Wheel tracking [4.01] and EN 12697-25 Cyclic compression test [4.02]. In the USA, the SUPERPAVE shear tester (SST) [4.03] is used to evaluate the resistance to permanent deformation.

The wheel tracking test, described in EN 12697-22, is used to measure the rut formed by repeated passes of a loaded wheel at constant elevated service temperature. The standard describes three types of test devices (extra-large size device, large size device and small size device).

- Extra-large size device: The specimens have dimensions (700 x 500) mm and a height of either 30 mm, 50 mm, 60 mm, 75 mm or 100 mm. A wheel fitted with a pneumatic tyre without a tread pattern and having a track width of 110 mm travels 700 mm over the specimen in 2,5 s. The rolling load applied to the test specimen is 10 kN at the centre of the test specimen.
- Large size device: The specimens have dimensions (500 x 180 x 50) mm or (500 x 180 x 50) mm. A wheel fitted with a 400 x 8 pneumatic tyre without a tread pattern and having a track width of 80 mm travels 410 mm over the specimen with a frequency of 1 Hz. The rolling load applied to the test specimen is 5 kN at the centre of the test specimen.
- Small size device: The block specimens have minimum dimensions of (260 x 300) mm or a diameter of at least 200 mm for cylindrical specimens. A treadless tyre with an external diameter between 200 and 205 mm and a rectangular cross profile with a track width of 50 mm travels 230 mm over the specimen with a frequency of 26,5 load cycles/min. The rolling load applied to the test specimen is 700 N.

The number of load cycles (i.e. two load passes) with extra-large size device is between 14 000 and 30 000 while with large size devices it is between 30 000 and 100 000. Two procedures are available for small size devices: Procedure A with 1000 load cycles and Procedure B with 10 000 load cycles. The conditioning and testing of samples is in air unless using Procedure B with small size devices, when the conditioning and testing of samples can be in either air or water.

The results are represented as proportional rut depth against number of load cycles except for Procedure A with small devices, which gives the results as wheel tracking rate and wheel tracking depth.

EN 12697-25 describes two test methods for determining the resistance to permanent deformation of asphalt: the uniaxial cyclic compression test and the triaxial cyclic compression test. The specimens may be either prepared in the laboratory or cored from a pavement.

- In the uniaxial cyclic compression test, a cylindrical test specimen with height of 60 mm and diameter of 150 mm, maintained at elevated conditioning temperature, is placed between two parallel loading platens. The upper platen has a diameter of 100 mm (due to inclination the contact area with the specimen has a diameter of 96 mm) and the lower platen is larger than the specimen. The specimen is subjected to a cyclic axial block-pulse pressure, with frequency 0,5 Hz (1 s loading and 1 s rest period) and a stress of (100 ± 1) kPa. There is no additional lateral confinement pressure applied.

- In the triaxial cyclic compression test, a cylindrical test specimen, maintained at elevated conditioning temperature (typical between 30 ºC and 60 ºC), is placed between two parallel loading platens. When the nominal maximum aggregate size is less than or equal to 16 mm, the specimen has a minimum height of 50 mm and a minimum diameter of 50 mm. A specimen with nominal maximum aggregate size greater than 16 mm has a minimum height of 80 mm and a minimum diameter of 80 mm. The specimen is subjected to a cyclic axial pressure (σ_A) and a static lateral confinement pressure (σ_C). The cyclic axial pressure can be a block-pulse or haversine with a frequency from 1 Hz to 5 Hz.

During the test, the change in height of the specimen is measured at specified numbers of load applications. The results are represented in a creep curve. The test does not allow quantitative prediction of rutting, but makes it possible to rank various mixtures in terms of resistance to permanent deformation.

The SUPERPAVE shear tester (SST) is a servo-hydraulic machine that can apply both axial and shear loads at constant temperatures to a cylindrical (150 x 150) mm specimen using closed-loop control. The current SST protocols consist of different modes of operation. Two of the commonly used modes are the frequency sweep at constant height (FSCH) and the repeated shear at constant height (RSCH). In each mode, different types of information are available. The FSCH test involves the application of a sinusoidal shear strain with a certain peak amplitude (e.g. 0,4 µm/mm) at a fixed temperature of interest at each of the following frequencies: 10; 5; 2; 1; 0,5; 0,2; 0,1; 0,05; 0,02; and 0,01 Hz. The generated response parameters are the complex shear modulus (G^*), the phase angle (δ), the recoverable shear modulus (G'), and the loss shear modulus (G''). The RSCH test consists of applying 5000 cycles of a haversine shear load with a shear stress level of (68 $\pm$ 5) kPa: the axial load is varied automatically during each cycle to maintain constant height of the specimen to within 0,0013 mm. The test involves the repeated application of a 0,1 s load pulse followed by a 0,9 s rest period during which the permanent deformation is recorded and used for comparisons. The protocol followed is in accordance with AASHTO provisional standard TP7–94 [4.03], which contains a detailed description of the SST test in the different modes of operation. The information obtained from the SST using different modes of operation is used conventionally by researchers to compare generated data for any proposed mixture of unknown performance with another mixture with known performance under the same conditions at identical temperatures [4.04].

The Carleton in-situ shear strength test (CISST) facility is a device for measuring the in-situ shear strength. A torque is applied to a loading platen bonded to the asphalt surface with a strong epoxy resin. The torque and the angle of twist at failure are measured [4.05].

The following paragraphs discuss the correlations between the binder tests and asphalt permanent deformation. The reported correlation coefficients are only indicative because they depend on many factors including:

- the number of bitumen samples
- the type of bitumen (e.g. only lightly modified or highly modified)
- the range of binder properties that is covered
- the type of regression law used to establish the correlation
- the exact test conditions applied in both the binders tests and the asphalt tests
- the precision of the tests.

The asphalt mixture composition also has an impact on the degree of correlation. The permanent deformation of the mixture is less sensitive to the binder properties with a rut resistant aggregate skeleton.

4.2 Capillary Viscometer Test

4.2.1 Description

For description, equivalent standardised tests and precision, see Section 2.3.

4.2.2 Relationship with Asphalt Tests

Wheel tracking rates to BS 598-110 [4.06] of mixtures with 16 binders, including 9 PMBs, have been reported [4.07] with fairly good correlation to dynamic viscosity at 60 ºC, as shown in Figure 4.1. The dynamic viscosity was measured using the vacuum capillary method to ASTM D 2171-94 [4.08].

The rut resistant performance of multigrade binder is reported [4.09] to have a good correlation with viscosity at 60 °C after RTFOT, as measured with a capillary viscometer according to the Australian standard AS 2341-2 [4.10]. Unfortunately, the wheel tracking tests on which this statement is based are not given.

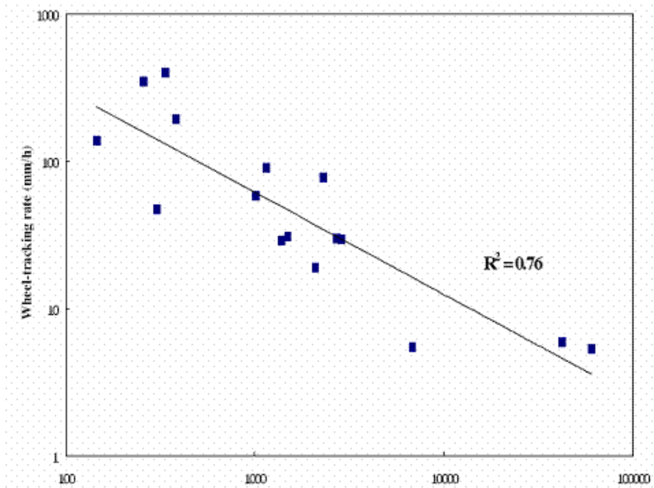


Figure 4.1 – Relationship between wheel-tracking rate and dynamic viscosity

4.2.3 Relationship with Site Experience

No data were found that relate the viscosity measured with the capillary viscometer to site experience.

4.2.4 Durability

No conclusions can be obtained with respect to durability. However, it is generally accepted that resistance to permanent deformation and viscosity of unmodified binder both increase with ageing. A decrease of viscosity has also been observed after ageing for some modified bitumens, but no data have been found about permanent deformation of asphalt mixtures that contain such modified bitumens.

4.2.5 Overview of Capillary Viscometer Test as Descriptor for Permanent Deformation

A single paper [4.07] has shown a fairly good correlation between resistance to permanent deformation and capillary viscosity.

4.3 Coaxial Cylinder Viscosity Test

4.3.1 Description

For description, equivalent standardised tests and precision, see Section 2.4.

4.3.2 Relationship with Asphalt Tests

A few papers have tried to correlate the binder viscosity to the permanent deformation of asphalt mixes. The conclusions are not always in agreement and are summarised as follows:

- There are good correlations between viscosity and permanent deformation for unmodified bitumen, but there are not enough experiments for validation [4.11]. This paper considered measurements of dynamic viscosity at 135 °C.
- There was no correlation between permanent deformation and viscosity for PMBs, measured with the Brookefield viscometer at 135 °C [4.12].
- Wheel tracking tests to BS 598-110 [4.06] were correlated with dynamic viscosity at 60 °C, 90 °C and 135 °C with unmodified and SBS-modified bitumens [4.13]. "No significant correlation was found between rutting resistance of asphalt concrete and bitumen properties. Nevertheless, the tendency was observed that viscosity, softening point and penetration index affect to some extent resistance to rutting."

The general conclusion is that there is a correlation between binder viscosity and asphalt permanent deformation, but the correlation coefficient is not very high, as shown in Figure 4.1. Generally, as with other tests, the correlation is better with unmodified bitumen. However, when the number of experiments is small, both good and bad correlations can be found, depending on the binders and asphalt mixture considered in the experiments.

4.3.3 Relationship with Site Experience

Average field rut depths of different asphalt mixtures, with three different binders, were correlated to binder viscosity at 135 °C (measured with Brookefield viscometer) [4.11]. The conclusion is that correlations are poor, even between the laboratory asphalt tests and site experience.

4.3.4 Durability

As for the capillary viscometer test (Section 4.2.4), no conclusions can be obtained with respect to durability.

4.3.5 Overview of Coaxial Cylinder Viscosity Test as Descriptor for Permanent Deformation

Resistance to permanent deformation and the dynamic viscosity are correlated, but the correlation coefficients are not very high [4.09, 4.11 to 4.15].

4.4 Cone and Plate Viscosity Test

4.4.1 Description

For description, equivalent standardised tests and precision, see Section 0.

4.4.2 Relationship with Asphalt Tests

Assuming that the coaxial cylinder and the cone and plate geometry lead to similar results, the conclusions can be generalised. Therefore, the relationship between the dynamic viscosity and asphalt tests for permanent deformation are the same as for the coaxial cylinder viscosity test (Section 4.3.2).

4.4.3 Relationship with Site Experience

Assuming that the coaxial cylinder and the plate and cone geometry lead to similar results, the conclusions can be generalised. Therefore, the relationship between the dynamic viscosity and site experience of permanent deformation are the same as for the coaxial cylinder viscosity test (Section 4.3.3).

4.4.4 Durability

As for the capillary viscometer test (Section 4.2.4) and the coaxial cylinder viscosity test (Section 4.3.4), no conclusions can be obtained with respect to durability.

4.4.5 Overview of Cone and Plate Viscosity Test as Descriptor for Permanent Deformation

The overview for the cone and plate viscosity is the same as for the coaxial cylinder viscosity test (Section 4.3.5).

4.5 Creep Zero Shear Viscosity Test

4.5.1 Description

For description, equivalent standardised tests and precision, see Section 2.6.

4.5.2 Relationship with Asphalt Tests

Correlations were observed between creep ZSV and rutting in a laboratory test track [4.16, 4.17] and between creep ZSV and uniaxial dynamic creep tests [4.18, 4.19]. Table 4.1 shows the reported correlation coefficients.

Table 4.1 – Correlation data for creep shear viscosity test

Ref.	Temp. (°C)	No. of binders	Binder types	Range of Creep ZSV (Pa.s)	Correlation coefficient
[4.16]	52 – 76	7	Pure and PMB, fresh	$200 - 5 \times 10^5$	0,54
[4.17]	40	14	Pure and PMB, fresh & after RTFOT	$500 - 10^7$	0,88 before RTFOT 0,92 after RTFOT (in log-log scale)
[4.18]	40 & 60	4	Pure and PMB, fresh	$300 - 3 \times 10^4$	0,81 – 0,98
[4.19]	60	12	Pure and PMB, fresh & after RTFOT	$500 - 2 \times 10^5$	0,73 after RTFOT

Although the reported correlations are generally good, all the authors found that the reliability of the binder test results is less for the highly modified binders due to the fact that steady state flow is not attained in the creep phase. Increasing the test temperature partly improves the situation, but the test temperature may then no longer be relevant.

A more rigorous but time-consuming version of the creep ZSV test has been used [4.16]. The test was performed at various stress levels and the Carreau model was used to extrapolate the viscosity to zero shear stress. Although good results are obtained, this procedure was recognised to be too complicated and time-consuming for specification purposes.

4.5.3 Relationship with Site Experience

The results from the creep ZSV were correlated with rut depth in a laboratory test track [4.17], which is a surrogate for site experience. Of the seven binders considered in the more rigorous version of the creep ZSV test [4.16], the test results of five binders were correlated with rutting data in an accelerated load facility and the test results of two binders were correlated with in-field rutting data. The findings are discussed in Section 4.5.2.

4.5.4 Durability

It is commonly known that the resistance to permanent deformation improves as a result of ageing (not taking into account rutting induced by excessive ravelling).

As seen in Table 4.1, creep ZSV was measured on both fresh and RTFOT aged binders. A slightly better correlation has been observed [4.17, 4.19] with the creep ZSV measured after RTFOT-ageing.

4.5.5 Overview of Creep Zero Shear Viscosity Test as Descriptor for Permanent Deformation

Four different sources have reported [4.16 to 4.19] on the correlation between creep ZSV and permanent deformation of asphalt. All test data confirm that creep ZSV affects permanent deformation. The sensitivity of asphalt rutting to the creep ZSV depends on the type of mixture because the binder only partially contributes to asphalt rutting - the aggregate skeleton and binder-aggregate interaction also have an important impact. Therefore, the creep ZSV test can, at this moment, not be considered as a surrogate for asphalt testing but could be used to rank binders for use with a particular aggregate skeleton.

The reproducibility of the test results is reported to be good for pure and lightly modified PMBs but rather poor for highly modified PMBs, as expected because the creep method is not suitable for highly modified binders. The test results for creep ZSV at elevated service temperatures are in a range covering several decades (from approximately 10^2 Pa.s to 10^7 Pa.s). However, the draft European standard specifies an upper limit of 50 000 Pa.s because, otherwise, the test period becomes too long and the reproducibility of the test result deteriorates.

For unmodified binders, the test is quite simple because the steady state regime is quickly reached. The practicality of the test is less for modified binders because the measuring times needed to attain steady state flow may become very long. Also, criteria need to be defined for deciding when the steady state regime has been reached. If not, the result will depend on how long the creep test is continued.

Care should be taken that the test is performed in the linear domain where the results are independent of the applied stress level. Some researchers suggest doing the test at several stress levels (at least three different) and fitting the ZSV as a function of the shear rate using the Carreau model [4.16, 4.20]. With this variant of the test, it is ensured that the result is really the ZSV in the linear domain. However, this procedure is considered to be too complex and time-consuming for specification purposes.

It is reported that creep ZSV of the binder after short term ageing correlates slightly better with permanent deformation of the asphalt mixture than the creep ZSV of the fresh binder [4.17, 4.19].

Although the validity of the creep ZSV test as an indicator for permanent deformation is commonly accepted, questions remain concerning the precision of the results for PMBs with a high polymer content. This concern was also the conclusion of the round robin test conducted by CEN TC336 WG1/TG1. A few possible approaches that require further investigation are:

- Improving the precision by refining the test protocol (e.g. by fixing criteria for stopping the creep test).
- Defining a range of binders for which the test can be applied (e.g. polymer content below a given level).
- Defining a range of viscosity values to which the test applies (this approach is followed in the draft European Standard).

4.6 Dynamic Shear Rheometer (DSR) Test

4.6.1 Description

For description, equivalent standardised tests and precision, see Section 2.8.

As an indicator for permanent deformation, the complex modulus at elevated service temperature and low frequency is most relevant.

SUPERPAVE introduced the ratio $G^*/\sin\delta$ (or the inverse of the loss compliance J'') at a test frequency of 1,59 Hz as a rutting indicator. This parameter, commonly known as the “SHRP rutting parameter”, accounts for the combined beneficial effect of a high stiffness G^* and a small phase angle δ. Other combinations of rheological data are $G^*/\sin\delta$ at 7.8 Hz [4.21, 4.22] and G^* at a low frequency.

4.6.2 Relationship with Asphalt Tests

A large amount of data are available on the correlation between G^* or $G^*/\sin\delta$ and permanent deformation of asphalt [4.07, 4.13, 4.15, 4.21 to 4.38]. Only the common conclusions are summarised here because the number of references is too large to discuss separately and because most of the findings are in agreement.

For unmodified bitumen, a generally good correlation of G^* or $G^*/\sin\delta$ with asphalt tests is reported by all authors, although not significantly better than for softening point.

For PMBs, the reported correlations range from acceptable to poor or even bad [4.07, 4.21, 4.24 to 4.28]. As an example, Figure 4.2 shows the wheel tracking rate as a function of

$G^*/\sin\delta$ for a wide variety of binders. Considering just the unmodified bitumen (circles), R^2 is reported to be as high as 0,99.

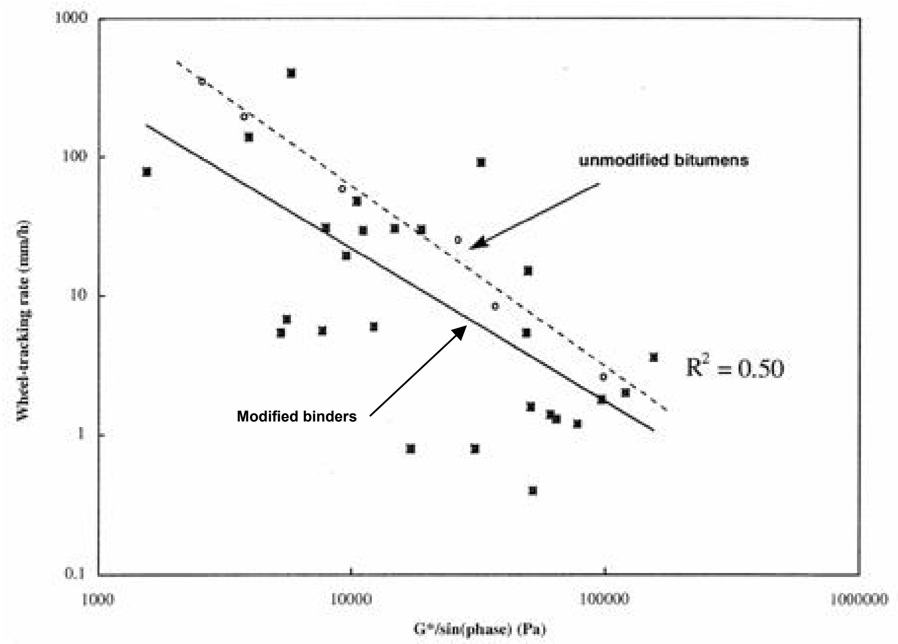


Figure 4.2 – Example of a correlation between wheel tracking rate and $G^*/\sin\delta$ at ω = 10 rad/sec [4.06]

The factors that explain why differences in the correlation coefficients may exist are discussed in 4.1. Particularly for DSR, differences in test conditions (test temperature and frequency) are key factors to explain the degree of correlation. For example, it has been shown [4.29] that the correlation between the SHRP parameter and the rut rate in wheel tracking tests depends on the frequency of the wheel tracking test. It has also been shown [4.25, 4.30] that decreasing the frequency at which $G^*/\sin\delta$ is measured results in a better correlation with the asphalt rutting tests.

$G^*/\sin\delta$ at 7,8 Hz is reported [4.21] to correlate with the wheel tracking test for unmodified or lightly modified binders, but this indicator underestimates the resistance to permanent deformation of highly modified PMBs. The difference in test frequency between the wheel tracking test (1 Hz) and the binder test (7,8 Hz) is given as a possible cause for this poor correlation.

Besides temperature and frequency, stress amplitude is also an important test condition. DSR tests are generally performed in the linear domain. However, the question has been raised [4.07] about whether the linear domain is really a good option when the aim is to predict the in-service rutting resistance under conditions of high shear stress. In a study with 16 binders, correlations with wheel tracking test results were shown to be better for viscosity and $G^*/\sin\delta$ measured at higher shear rates in the non-linear domain. However, it would be extremely difficult to standardise “simple” procedures for carrying out non-linear tests and to guarantee the repeatability and reproducibility of the results.

At sufficiently low frequencies, it has been concluded [4.25] only G^* needs to be considered because taking account of the loss angle does not really improve the correlation with rut depth. However, it has also been found [4.35] that it is absolutely necessary to use $G^*/\sin\delta$

instead of G*. The latter study involved a low modulus SBS modified binder, of which the rutting resistance was strongly underestimated by not considering the loss angle.

It has been suggested [4.04, 4.21, 4.22] that the difference in binder morphology between the binder samples and the binder in the asphalt mixture is an explanation for the poor correlation in the case of PMBs. This problem is also related to the sample preparation conditions because preparation conditions (especially heating temperature) are known to affect the binder morphology, which in turn is known to have an impact on binder rheology [4.39].

RTFOT simulation of short term ageing has a negative impact on the correlation between $G^*/\sin\delta$ and wheel tracking in the case of PMBs [4.07, 4.22], possibly because of the impact of the ageing procedure on the binder morphology. A study [4.38] of the correlation between $G^*/\sin\delta$, both before and after RTFOT, with wheel tracking rate for two different mixtures found that, for one mixture, the correlation was better with the fresh binder properties while, for the other mixture, the correlation was better with the binder properties measured after RTFOT. However, the differences were not significant enough to draw firm conclusions.

To summarise, the correlation with asphalt tests is generally good for unmodified binders, while the rut resistance of modified binders is mostly underestimated. The closer the test temperature and frequency of the binder test are to the conditions adopted in the asphalt test, the better the correlation becomes.

That $G^*/\sin\delta$ at ω = 10 rad/s underestimates the rut resistance of certain PMBs is also recognised in the United States because the SHRP indicator is being questioned for PMBs and alternatives are being considered [4.40].

4.6.3 Relationship with Site Experience

One mixture prepared with five different binders belonging to the same Performance Grade were tested in an accelerated loading facility [4.36] and led to the same ranking of the binders with the ranking based on $G^*/\sin\delta$.

Data on direct correlations between binder properties and rut depth in the field were not found, but good resistance in the wheel tracking test is reported to agree with good rut resistance in larger accelerated load facilities [4.41] and in the field [4.11].

4.6.4 Durability

It is commonly known that the resistance to permanent deformation improves as a result of ageing (excluding rutting induced by excessive ravelling).

4.6.5 Overview of DSR Test as Descriptor for Permanent Deformation

Correlations have been investigated between the SHRP parameter (or variants with respect to test frequency) and the results from wheel tracking tests, triaxial tests, uniaxial tests and other tests. The binder property cannot be a surrogate for asphalt testing because permanent deformation of asphalt is influenced by other factors as well as binder rheology, including aggregate type, mixture composition and component compatibility.

It has been shown [4.22] that the sensitivity of permanent deformation to the DSR result depends on the mixture formulation. In high modulus mixtures such as EME, it is reported [4.31] that the wheel tracking test is not capable of distinguishing between the various binders. This inability is due to the aggregate skeleton being very stable, combined with all the binders considered belonging to the same class of hard binder.

As an average estimation, an increase by a factor of 10 in $G^*/\sin\delta$ leads to a decrease by a factor of 10 in the rut depth, with variations on these numbers depending on the mixture type. This finding confirms an earlier conclusion [4.37].

Obviously, the range of values covered by $G^*/\sin\delta$ depends on the test temperature and test frequency. Examples are:

- from 5 kPa to 100 kPa at 60 °C and 10 rad/s [4.22], and
- from 2 kPa to 200 kPa at (45 – 60) °C and 10 rad/s [4.07].

DSR testing has the advantage that both the test equipment and the experience in performing the tests are widely spread, in Europe as well as in the United States. Precision data are also available (see Section 2.8.3).

The SHRP parameter $G^*/\sin\delta$ at ω = 10 rad/s is a good indicator for the rut resistance of mixtures containing unmodified binders, but it cannot properly predict the rut resistance of asphalt mixtures with PMBs.

The correlation between DSR test results and asphalt tests for permanent deformation is best when the test conditions of temperature and frequency are within the same range. Thus, an interesting question is which test temperature and frequency should be adopted to obtain the best possible correlation with in-field rutting. Measuring at a test frequency as low as possible, but not necessarily zero, should be the objective because rutting is most serious at elevated service temperatures and under heavy, slow traffic. It is interesting to note that the DSR test is then basically the same as the oscillation ZSV (Section 2.13).

4.7 Force Ductility Test

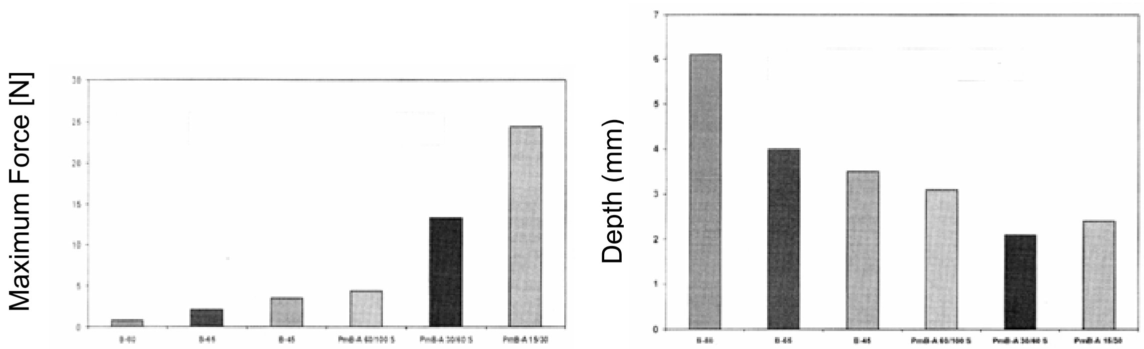
4.7.1 Description

For description, equivalent standardised tests and precision, see Section 2.9.

4.7.2 Relationship with Asphalt Tests

The results of force ductility tests have been compared with the tendency of asphalt mixtures to permanent deformation [4.42]. The force ductility tests were performed at 13 °C and 25 °C and the wheel tracking tests were undertaken at 60 °C. Unmodified, elastomer- and thermoplastomer-modified bitumens were tested with both methods. The maximum force at the beginning of the force ductility test gives information about the contribution of the binder to the stability of asphalt mixtures at high temperatures. Generally, elastomer-modified bitumens with low concentration of polymer show a decreasing force after the first maximum and a tendency to the formation of a plateau. Bitumen with a high concentration of elastomer, show a characteristic second maximum, which can be explained with the formation of a polymer network.

A very close correlation was found between the first maximum force and the rutting depth (Figure 4.3). High forces of the force ductility curve of bitumen are interpreted as a high resistance against rutting of the corresponding asphalt mixtures. This relationship is valid for similar types of bitumens. The contribution of the binder to the rutting resistance of asphalt mixtures depends primarily on the first maximum force of the force ductility curve. It was claimed [4.42] that each sort of bitumen generates a characteristic and well distinguishable curve. The type of the binder and even the sort can be identified, at the first glance, even without the knowledge of the results of other test methods. The specific form of the force-elongation-curve allows conclusions about the original bitumen and about the type and concentration of the polymer.



Force Ductility Test | Rutting Test

Figure 4.3 – Comparison of the maximum force and the rutting depth

4.7.3 Relationship with Site Experience

No references were found that deal explicitly with the correlation between the force ductility test and site experience with respect to rutting.

4.7.4 Durability

No references were found that deal explicitly with the correlation between the force ductility test and the maintenance of the properties measured with time or the general durability of the asphalt.

4.8 Oscillation Zero Shear Viscosity Test

4.8.1 Description

For description, equivalent standardised tests and precision, see Section 2.13.

4.8.2 Relationship with Asphalt Tests

The oscillation ZSV test was used in the ARBIT study (1998-99) on a set of 36 binders including PMBs to investigate the correlation with results of the Hamburg wheel tracking test on SMA [4.15], as shown in Figure 4.4 and Table 4.2. ZSV produced the highest correlation coefficients, compared to R&B softening point and the SHRP indicator $G^*/\sin\delta$.

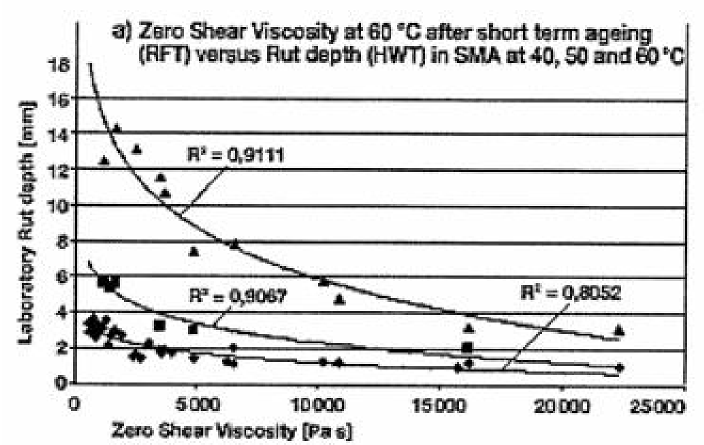


Figure 4.4 – Oscillation ZSV at 60°C versus rut depth at 40 °C, 50 °C and 60 °C [4.15]

Table 4.2 – Correlation data for oscillation zero shear viscosity test

Ref.	Temp. (°C)	No. of binders	Binder types	Range of Oscillation ZSV (Pa.s)	Correlation coefficient
[4.15]	40, 50 & 60	36	Pure and PMB, after RTFOT	$5 \times 10^2 - 25 \times 10^3$	0,81 at 40 °C 0,91 at 50 °C & 60 °C
[4.16]	52 – 76	7	Pure and PMB, fresh	$1 \times 10^3 - 1 \times 10^5$	0,43
[4.43]	50	12	Pure and PMB, fresh	$5 \times 10^2 - 35 \times 10^4$	0,83 without PMBs, 0,82 with PMBs

A correlation was also observed [4.16] between oscillation ZSV and rutting in a laboratory test track, as shown in Table 4.2. The correlation coefficient is not very high, but this is mainly explained by the fact that the asphalt rut data were obtained partly in an accelerated load facility and partly in-field. Thus, the precision of the asphalt data is relatively low. A more sophisticated version of the oscillation ZSV test has been applied [4.16, 4.18] in which ZSV is derived from the master curve of the binder. The method is quite complicated and time-consuming because it requires the performance of frequency sweeps at various temperatures and special software to do the analysis. However, the ZSV obtained from a single frequency sweep at the SHRP high PG temperature was found [4.16] to produce results that correlate just as well to the asphalt tests.

Good correlations have been found [4.43] between the oscillation ZSV (or LSV) and asphalt tests on an asphalt concrete mixture with both the wheel tracking test and the triaxial cyclic compression test. Figure 4.5 shows the correlation obtained between the rut depth in the wheel tracking test and the LSV measured at the corresponding test temperature (50 °C) at a test frequency of 0,01 Hz. The correlation is just as high for the PMBs as for paving grade bitumens.

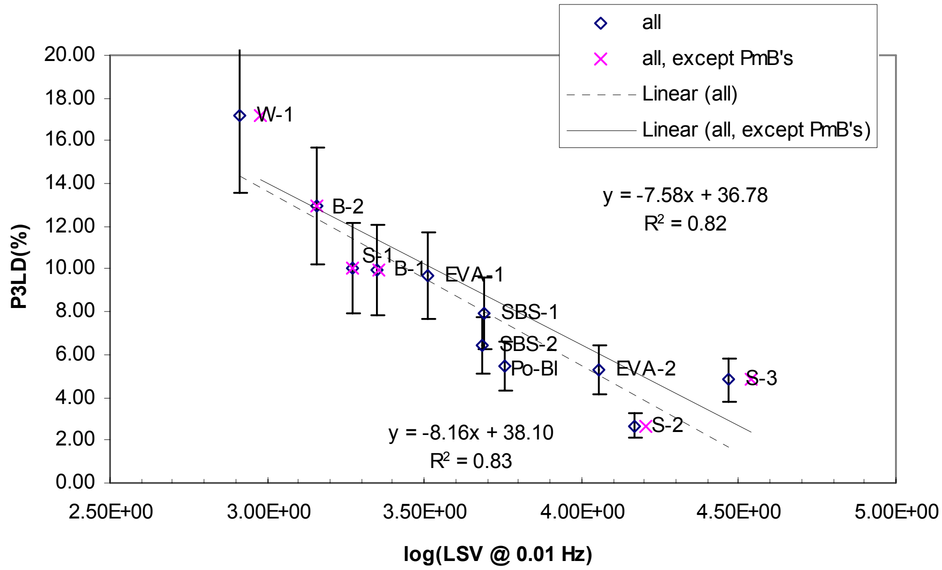


Figure 4.5 – Oscillation LSV at 0,01 Hz versus rut depth at 50 °C [4.43]

4.8.3 Relationship with Site Experience

Of seven binders considered [4.16], the test results of five binders were correlated with rutting data in an accelerated load facility and the test results of two binders were correlated with in-field rutting data.

4.8.4 Durability

It is commonly known that the resistance to permanent deformation improves as a result of ageing.

4.8.5 Overview of Oscillation Zero Shear Viscosity Test as Descriptor for Permanent Deformation

The number of papers that have investigated the correlation between oscillation ZSV and asphalt rutting is still limited. Good correlations have been found [4.43] with both the wheel tracking test and the triaxial compression test.

The test procedure is relatively simple because it only requires a single frequency sweep at a low frequency. Good repeatability and reproducibility of the test results have been reported [4.16, 4.39, 4.44] that were obtained from a low frequency sweep at elevated service temperatures.

The test procedure described in the draft European Standard is more complex because it is designed to determine the equi-viscous temperature for LSV equal to 2000 Pa.s. Precision data have been determined for this test (Section 2.13.3). Correlations between this equi-viscous temperature and asphalt tests have not yet been investigated.

4.9 Repeated Creep Test

4.9.1 Description

For description, equivalent standardised tests and precision, see Section 2.16.

4.9.2 Relationship with Asphalt Tests

A correlation was observed [4.16] between creep stiffness and rutting in a laboratory test track, as shown in Table 4.3. The correlation coefficient is not very high, but this poor precision is mainly explained by the fact that the asphalt rut data were obtained partly in an accelerated load facility and partly in-field. Thus, the precision of the asphalt data is relatively low.

Table 4.3 – Correlation data for repeated creep test

Ref.	Temp. (°C)	No. of binders	Binder types	Range of η_0 (Pa.s)	Correlation coefficient
[4.16]	52 - 76	7	Pure and PMB	$100 – 4x10^4$	0,32
[4.43]	50	12	Pure and PMB	$100 – 5x10^4$	0,73
[4.45]	45 & 60	15	Pure and PMB	$100 – 5x10^4$	0,80

A better correlation has been found between the creep stiffness and the wheel tracking rate in the British wheel tracking test [4.45]. The binder tests were performed at a stress level of 300 Pa. Surprisingly, the correlation was just as good when only the first cycle of the test was considered.

A good correlation was also reported between the creep viscosity and the wheel tracking test to EN 12697-22 [4.43], but the correlation coefficient is smaller than for the oscillation ZSV (Section 4.8.2).

4.9.3 Relationship with Site Experience

Of seven binders considered [4.16], the test results of five binders (three unmodified bitumens and two PMBs) were correlated with rutting data in an accelerated load facility and the test results of two binders (one unmodified bitumen and one PMB) were correlated with in-field rutting data.

4.9.4 Durability

It is commonly known that the resistance to permanent deformation improves as a result of ageing.

4.9.5 Overview of Repeated Creep Test as Descriptor for Permanent Deformation

Three references investigated the correlation between this binder property (η_0) and asphalt rutting. A correlation exists [4.16], but it is not as good as the correlations obtained for the creep ZSV test (using creep tests at various levels and the Carreau model to fit the data) or the oscillation ZSV test (from a single frequency sweep). A good correlation was also found

[4.45], but the conclusions included the cautious suggestion that further research was needed. The third reference also reported a good correlation [4.43], but less good as for the oscillation ZSV.

The range of values of η_0 is of the same order as creep ZSV or oscillation ZSV because of the interrelation between these parameters.

The test is relatively complex because of the large number of test parameters (stress level, number of cycles, loading periods and unloading periods). TRB sponsored an expert task group to evaluate the test method. It was found [4.16] that “the test will require more sophisticated DSR instrumentation and software than currently in use by a majority of laboratories”.

Although precision data were not found, a small inter-laboratory comparison [4.44] between two laboratories on four binders (including two highly modified PMBs) revealed that the reproducibility of this test was not good, compared to the oscillation ZSV test.

4.10 Softening Point (Ring and Ball) Test

4.10.1 Description

For description, equivalent standardised tests and precision, see Section 2.17.

4.10.2 Relationship with Asphalt Tests

There is a general consensus [4.15, 4.21, 4.22, 4.26, 4.38, 4.46] that R&B softening point is a good indicator for permanent deformation of mixtures with pure binders, but not for that of mixtures containing PMBs.

4.10.3 Relationship with Site Experience

No references were found that deal explicitly with the correlation between R&B softening point of the binder and site experience with respect to rutting.

4.10.4 Durability

No references were found that deal explicitly with the correlation between R&B softening point of the binder and the maintenance of that property with time or the general durability of the asphalt.

4.10.5 Overview of Softening Point Test as Descriptor for Permanent Deformation

R&B softening point should not be considered as an indicator for permanent deformation because the aim is to look for performance indicators that work as well for PMBs as for pure bitumen.

4.11 Recommendations for Permanent Deformation

Eight binder tests were identified and reviewed as having a potential relationship with asphalt permanent deformation. Six of the tests are designed for measuring the viscosity of the binder at elevated service temperatures, the exceptions being the R&B softening point test and the DSR test for measuring *G**. However, the R&B softening point test can be excluded because the R&B softening temperature is not capable of correctly ranking PMBs in accordance with their rutting sensitivity. The capillary viscometer test is also not appropriate because the test only applies to unmodified binders.

The number of references found was limited for the coaxial cylinder test and the cone and plate viscosity test. This lack does not mean that the test results would necessarily correlate poorly with permanent deformation. The main reason is probably that these test geometries are less used than the more commonly used parallel plates.

The DSR test is relevant for permanent deformation when the complex modulus is considered at elevated service temperatures and at low frequencies. The DSR test is then equivalent to the oscillation test for ZSV (see below).

The validity of the creep ZSV test can be regarded as an indicator for permanent deformation. However, questions remain concerning:

- The precision of the results for PMBs with a high polymer content. This concern was also the conclusion of the round robin test conducted by CEN TC336 WG1/TG1. It should be further investigated if the reproducibility can be improved by refining the test protocol (e.g. by fixing criteria for stopping the creep test).
- The duration of the creep test. From a practical point of view, a creep period of 8 h or more is not desirable. Shorter creep periods may lead to good correlations with asphalt rutting, but this assumption can only be demonstrated by actually performing asphalt tests and looking at correlations with creep tests after various creep periods.
- The large deformations applied. With creep periods of hours and more, the deformations become very large and outside the range of deformations normally encountered in asphalt mixtures. It is not clear to what extent this difference in range also has an impact on the degree of correlation.

It should be noted that the creep test will always be limited to binders with a ZSV below a maximum value, because of the precision and the duration of the test.

The oscillation ZSV test is currently the most interesting candidate because:

- Good correlations have been reported, coming from three different sources.
- The test is relatively simple and the duration is acceptable (depending on how low the test frequency is).

It has not yet been shown which exact test conditions (temperature and frequency) will give results that correlate best with asphalt rutting. However, the following guidelines generally apply:

- the correlation will be best if the test temperature is as close as possible to the temperature at which rutting occurs; and
- test frequencies from 0,01 Hz to 0,001 Hz lead to a good correlation, while still being practically feasible.

For the equi-viscous temperature based on the oscillation ZSV, as described in the European draft test method, the precision data are satisfying, including for the PMBs. Correlations with asphalt tests have not yet been reported, but the correlations are expected to be good because the oscillation ZSV at a given temperature correlates closely with asphalt rutting tests at the same temperature. However, it is an important recommendation for future work because the validation of the test method depends on the demonstration of this correlation.

The repeated creep test is also an interesting candidate, for which good correlations with asphalt rutting tests have been reported. However, the validation of this test still requires some work, including the estimation of the precision under conditions of reproducibility. Nevertheless, it has already been reported in the USA that the reproducibility of the test is not good because the test requires very high standards for the DSR equipment [4.44]. Therefore, it is questionable to invest more research into following this trail.

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5. Stiffness

5.1 Asphalt tests

The stiffness of asphalt is a structural property that can be used in the design of pavements but which varies with temperature and frequency, as with many asphalt properties. The relevant European asphalt test is EN 12697-26: 2004, Stiffness, containing several options (two-, three and four-point bending, indirect tension, direct tension-compression and direct tension) that give mutually consistent resultants. The scope states that the test method is used to rank asphalt mixtures on the basis of stiffness, as a guide to relative performance in the pavement, to obtain data for estimating the structural behaviour in the road and to judge test data according to specifications for asphalt.

5.2 Bending Beam Rheometer (BBR) Test

5.2.1 Description

For description, equivalent standardised tests, precision and relationship with other bitumen tests, see Section 2.1.

5.2.2 Relationship with Asphalt Tests

The temperature for a BBR binder stiffness of 300 MPa of initial and recovered samples of a control and three polymer-modified binders are reported together with graphs of the stiffness by ITSM of an asphalt concrete with each of the binders at 5 ºC, 10 ºC and 20 ºC for both laboratory and site samples [5.01]. The control binder, with a temperature of -19 ºC, had the highest stiffness moduli, then the PMBs with -24 ºC, -31 ºC and -29 ºC in order. Therefore, the results show no clear relation between BBR temperature and stiffness modulus.

However, based on analysis of previous papers as well as laboratory results, it has been found by others [5.02] that the stiffness modulus of a mixture, as measured in two-point bending on trapezoidal specimens, can be predicted from the BBR binder stiffness using the Bonnaure prediction method [5.03]. Although the method over-predicts the mixture stiffness slightly (unlike another method which significantly underestimated it), it is on the conservative side regarding the low-temperature design of asphalt mixtures. The method derives the stiffness modulus of the mixture, S_m, (N/m²) as:

- Equation (5.1) for 5×10^6 N/m² < S_b < 10^9 N/m².
- Equation (5.2) for 10^9 N/m² < S_b < 3×10^9 N/m².

$$\log(S_m) = \frac{(S89 + S68) \times (\log(S_b) - 8)}{2} + \frac{(S89 - S68) \times \left|\log(S_b) - 8\right|}{2} + S_m 108 \qquad (5.1)$$

$$\log(S_m) = S_m 108 + S89 + \frac{(S_m 3109 - S_m 108 - S89) \times (\log(S_b) - 9)}{\log(3)} \qquad (5.2)$$

where:

S_b is the BBR creep stiffness of the binder (N/m²)

V_b is the binder content by volume in the mixture (%)

V_g is the aggregate content by volume in the mixture (%)

$$S_m 3109 = 10{,}82 - 1{,}342 \times \frac{100 - V_g}{V_g + V_b} \quad (5.3)$$

$$S_m 108 = 8 + 5{,}68 \times 10^{-3} \times V_g + 2{,}135 \times 10^{-4} \times V_g^2 \quad (5.4)$$

$$S68 = 0{,}6 \times \log\left(\frac{1{,}37 \times V_b^2 - 1}{1{,}33 \times V_b - 1}\right) \quad (5.5)$$

$$S89 = \frac{S_m 3109 - S_m 108}{\log(30)} \quad (5.6)$$

5.2.3 Relationship with Site Experience

Results from both laboratory-prepared and site samples [5.01] showed that the stiffness moduli were higher for laboratory-prepared samples than for site samples, although the difference was explained by different air voids contents. Therefore, the same conclusion can be drawn for site samples as for laboratory samples with a lack of correlation between penetration and stiffness modulus of the mixture. However, no data were found that related the BBR results of the binder to the stiffness of the pavement rather than laboratory estimates of the stiffness of the material.

5.2.4 Durability

No references were found for laboratory or field studies that indicated that the test will predict the change in stiffness with time. However, the typical change of BBR temperature after (short) and long term ageing has been found to be about 2 °C [5.04].

5.2.5 Overview of BBR Test as Descriptor for Stiffness

The data from the two references found produce conflicting conclusions about whether the BBR test results can be used as a surrogate for low-temperature stiffness or even are a parameter that affects the stiffness. However, this difference results from one paper investigates the BBR temperature (and, hence, the measurements are not at the same temperature as the measurement of the mixture stiffness) whilst the other investigates the stiffness modulus itself at the same temperature.

5.3 Dynamic Shear Rheometer (DSR) Test

5.3.1 Description

For description, equivalent standardised tests, precision and relationship with other bitumen tests, see Section 2.8.

5.3.2 Relationship with Asphalt Tests

Five papers [5.05, 5.06, 5.07, 5.08, 5.09] demonstrate that the shift factor for producing the master curves of binder and mixture stiffness are strongly correlated with correlation coefficients, R^2, of greater than 0,99 in most cases.

It has been found [5.10] that the stiffness modulus of mixtures, $|E^*|$, containing PMBs as well as paving grade bitumen can be predicted from the complex modulus, G^*, of the binder together with the volumetric composition of that mixture according to equation (5.7).

$$|E^*|(T,f) = E_\infty . R^*(T,f) \qquad (5.7)$$

where:
E_∞ = purely elastic modulus which is a constant for a given composition
R^* = the reduced modulus (0<R*<1) describing the shape of the modulus master curve.

$$E_\infty = 14360\left(\frac{V_a}{V_l}\right)^{.55} \exp(-.0584v)(MPa) \qquad (5.8)$$

$$\log(R^*) = \log(B^*)\left[1 - 1.35F\left(\frac{V_a}{V_l}\right).G(B^*)\right] \qquad (5.9)$$

$$G(B)^* = 1 + 0.11\log(B^*) \qquad (5.10)$$

$$F\left(\frac{V_a}{V_l}\right) = 1 - \exp(-0.13\frac{V_a}{V_l}) \qquad (5.11)$$

In equation (5.7), the impact of the mixture composition is mainly determined by E_∞., but it also depends on $F(V_a/V_l)$.

Figure 5.1 gives the measured versus so-calculated stiffness modulus at different temperatures and for a frequency of 10 Hz for different asphalt mixtures: twelve asphalt concrete mixtures and eight porous asphalt mixtures.

It is also been reported from a theoretical review [5.12] that the

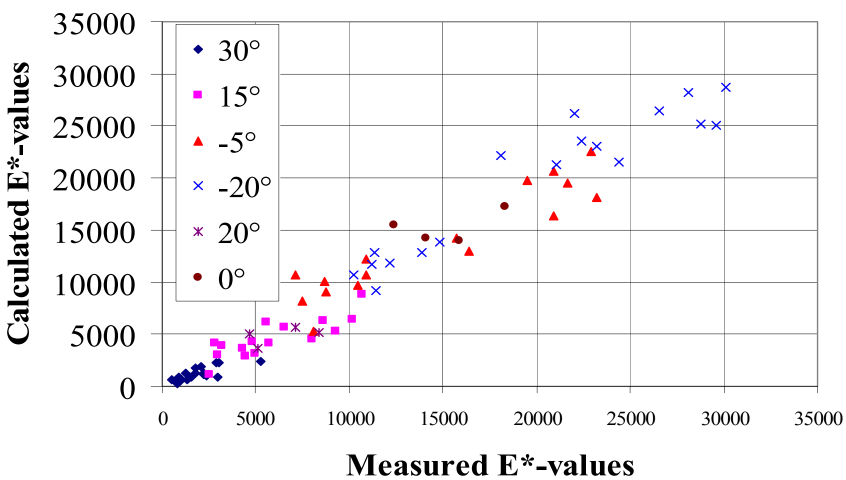


Figure 5.1 – Correlation between measured and calculated mixture stiffness at 10 Hz, making use of equation (5.7) [5.11]

mixture stiffness, S_{mix} (MPa), within a certain range, can be calculated from the binder stiffness, S_{bit} (MPa), according to equation (5.12).

$$S_{mix} = R \times 3{,}56 \times 10^4 \times \frac{V_b + V_a}{V_b} \times e^{-0{,}1 \times V_a} \quad (5.12)$$

where:

$$\log(R) = \log(B) - 1{,}35 \times \left(1 - \exp\left(-0{,}13 \times \frac{V_g}{V_b}\right)\right) \times \log(B) \times \left(1 + 0{,}11 \times \log(B)\right) \quad (5.13)$$

$$B = \frac{S_{bit}}{2000 \times (1 + v)} \quad (5.14)$$

V_a = proportion of air by volume (%)
V_b = proportion of bitumen by volume (%)
V_g = proportion of aggregate by volume (%)
v = Poisson's ratio

From another analysis of models for the behaviour of mixtures [5.09], a relationship has been found for the complex modulus of the mixture, E^*_{mix}, from Equation (5.15).

$$E^*_{mix}(\omega, T) = E_{0,mix} + \left(E^*_{binder}(10^{\alpha}\,\omega, T) - E_{0,binder}\right) \times \frac{E_{\infty,mix} - E_{0,mix}}{E_{\infty,binder} - E_{0,binder}} \quad (5.15)$$

where:
ω = the pulsation = $2 \times \pi \times f_r$
f_r = frequency (Hz)
T = temperature (°C)
E_0 = minimum asymptotic value of the norm of the complex modulus of the mixture or binder
E_∞ = maximum asymptotic value of the norm of the complex modulus of the mixture or binder
α = parameter that can depend on the considered mix design and/or ageing during mixing

Whilst these equations are relatively sophisticated, they do support the concept that the mixture stiffness is correlated with the binder stiffness.

There have been several projects in which laboratory data has been produced for both the binder and mixture stiffnesses. The dynamic stiffness moduli, G^*, of six binders were measured at combinations of two temperatures (10 ºC and 20 ºC) and two frequencies (1 Hz and 0,1 Hz) together with the dynamic stiffness modulus, E^*, of three asphalt mixtures at the same combinations [5.13]. Two of three mixtures were replicated with two aggregate sources, one of which was considered to have poor adhesion characteristics. The precise methods for determining G^* and E^* were not identified. From the results, it was found that there is a relationship between G^* and E^*, but that the aggregate grading and the quality of the aggregate also had very significant influences. To quote from the report [5.13], "*The binder has an important influence on the mix behaviour, but the best binder can be killed by a bad mix formulation and poor quality aggregates*". Nevertheless, it has been found [5.14] that, given a mix design and a binder, there is a direct relationship between the binder and mixture moduli.

The values of G*/sin(δ) and stiffness (using the two-point method on trapezoidal specimens) were reported [5.15] for two binders in two mixtures. The mixture results are reproduced in Table 5.1 with the associated binder properties. The results indicate that the binder with the higher value of G*/sin(δ) had lower values of stiffness in the two mixtures at 15 ºC and 10 Hz. However, the number of observations is too limited to be able to make any assumptions about the universality of such a relationship.

Table 5.1 – Mixture stiffness with associated binder properties [5.15]

	Modified		50/70	
	Surface course	Binder course	Surface course	Binder course
Stiffness (2-point) (GPa)	5,00	6,60	6,22	8,84
G*/sinδ (kPa)	7,52		4,01	
Penetration (0,1 mm)	63		60	
R&B softening point (ºC)	70		48	
Dynamic viscosity (kPa.s)	5,5		0,15	
Fraass point (ºC)	-20		-13	

A test programme of stiffness testing was carried with two mixtures (AC and PA) using eight different binders in order to establish the universality of the master curve approach [5.16, 5.17]. The eight binders were an 80/100 pen paving grade bitumen, a recycled elastomer, three SBS-modified bitumens, one EVA modified bitumen, a pitch-bitumen and a PCP-EVA modified bitumen.

These data were re-analysed [5.11] and the impact of the binder stiffness on the stiffness of the mixture for the eight asphalt concrete mixtures, of the same composition, but prepared with different binders, was shown (Figure 5.2).

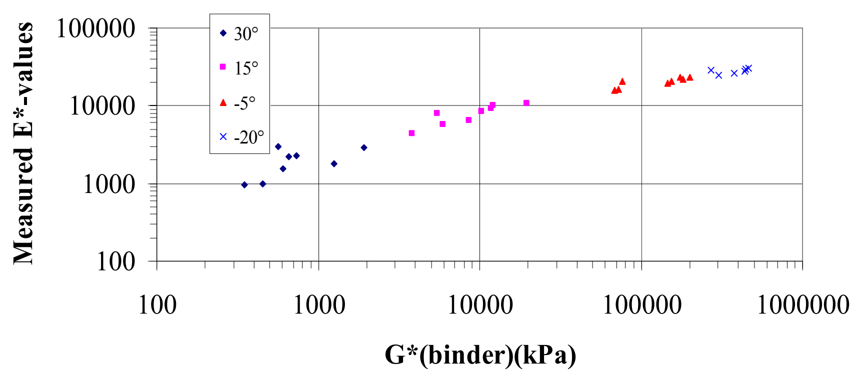


Figure 5.2 – Impact of binder stiffness on mixture stiffness for eight different binders applied in a same AC mixture composition [5.11]

There was a clear relation between binder and mixture stiffness with a change in binder stiffness by a factor of 100 implying a change in the mixture stiffness by a factor of 10. Therefore, the mixture stiffness varies approximately with the square of the binder stiffness. However, the authors noted that this should be checked further with the literature.

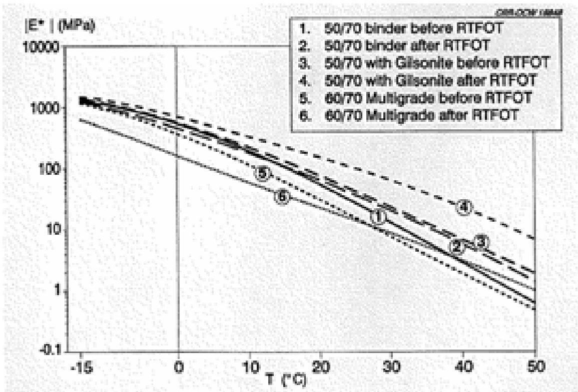
The data in two papers [5.16, 5.17] can be reanalysed to find if there are any relationships between the binder and asphalt properties. The correlation coefficients found from these analyses are given in Table 5.2.

Table 5.2 – Correlation coefficients, R^2, for binder and asphalt stiffness properties [5.16, 5.17]

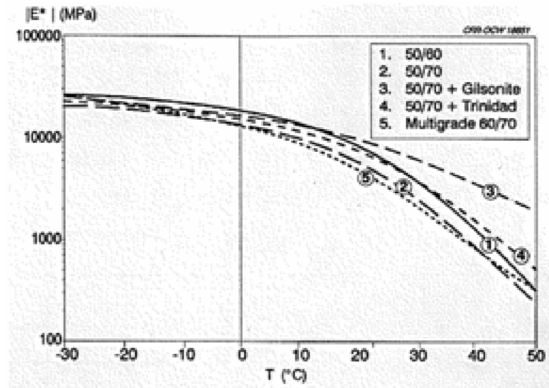
	Asphalt Stiffness @ 30 ºC & 10 Hz Stiffness Mod, E^*	Asphalt Stiffness @ 30 ºC & 10 Hz Phase Angle, δ	Asphalt Stiffness @ 25 ºC & 1,6 Hz Stiffness Mod, E^*	Asphalt Stiffness @ 25 ºC & 1,6 Hz Phase Angle, δ	Elastic Component, E_∞
Binder Properties	Porous Asphalt Mixture				
Penetration (0,1 mm)	0,79	0,35	0,78	0,26	0,16
R&B softening point (ºC)	0,32	0,44	0,24	0,50	0,37
Penetration Index	0,46	0,50	0,36	0,53	0,38
Stiffness @ 30 ºC & 10 Hz	0,67	0,22	0,70	0,25	0,51
Phase angle @ 30 ºC & 10 Hz	0,00	0,13	0,00	0,25	0,22
Stiffness @ 25 ºC & 1,6 Hz	0,65	0,21	0,67	0,24	0,53
Phase angle @ 25 ºC & 1,6 Hz	0,00	0,15	0,00	0,26	0,23
Elastic component, E_∞	0,00	0,12	0,00	0,10	0,01
Binder Properties	Dense Asphalt Mixture				
Penetration (0,1 mm)	0,54	0,11	0,52	0,07	0,07
R&B softening point (ºC)	0,14	0,31	0,20	0,37	0,36
Penetration Index	0,23	0,31	0,29	0,36	0,36
Stiffness @ 30 ºC & 10 Hz	0,57	0,16	0,74	0,17	0,01
Phase angle @ 30 ºC & 10 Hz	0,01	0,15	0,00	0,23	0,02
Stiffness @ 25 ºC & 1,6 Hz	0,54	0,15	0,71	0,17	0,01
Phase angle @ 25 ºC & 1,6 Hz	0,01	0,17	0,00	0,25	0,02
Elastic component, E_∞	0,02	0,08	0,03	0,11	0,58

None of the relationships have particularly good correlations, but some are definitely better than others. In particular, the stiffness modulus of a particular asphalt grading can be predicted reasonably well by the stiffness modulus of the binder, but also by the simpler property of its penetration. The binder stiffness under the same or very similar conditions gave generally the best correlation with the mixture stiffness. No other relationships had correlations over 0,50 for both mixtures.

A comparison of mixture properties manufactured with five different binders (50/60 bitumen, 50/70 bitumen, 50/70 bitumen with TLA, 50/70 bitumen with Gilsonite and 60/70 Multigrade) [5.18] include the complex stiffness modulus of both the binders (before and after RTFOT) by DSR and the mixtures by the two-point trapezoidal bending. The graphs of these characteristics are reproduced in Figure 5.3. The isochrones for 50/60 bitumen and 50/70 bitumen with TLA are not included because the former is almost identical with 50/70 bitumen and the latter would be invalidated by the presence of filler.



Three binders (before and after RTFOT)



Five mixtures

Figure 5.3 – Isochrones of the stiffness moduli at 27 Hz

The results show a consistency between the stiffness modulus of the binder and that of the mixtures for a specific aggregate skeleton. However, it can be seen that any relationship is imperfect which, according to the authors, resulted from the DSR-testing procedure not having been completely established at that time.

Comparisons with two mixtures using seven binders and a further mixture using four other binders [5.06] measured both the binder and mixture stiffness at four temperature and frequency combinations. Comparing the properties at the same conditions gave correlation coefficients, R^2, of 0,991, 0,984 and 0,996 for the three mixtures. The correlation coefficients, R^2, averaged over all the mixtures for each binder property and each mixture stiffness are given in Table 5.3.

Table 5.3 – Average correlation coefficients, R^2, for binder and asphalt stiffness properties [5.06]

Asphalt / Binder	E* @ 10 ºC & 0,1 Hz	E* @ 10 ºC & 1,0 Hz	E* @ 20 ºC & 0,1 Hz	E* @ 20 ºC & 1,0 Hz
Penetration	0,73	0,57	0,89	0,79
Pen after RTFOT	0,70	0,55	0,83	0,72
R&B softening point	0,17	0,05	0,44	0,20
SP after RTFOT	0,15	0,05	0,41	0,19
G* @ 10 ºC & 0,1 Hz	0,97	0,92	0,86	0,93
G* @ 10 ºC & 1,0 Hz	0,95	0,97	0,78	0,91
G* @ 20 ºC & 0,1 Hz	0,91	0,76	0,96	0,91
G* @ 20 ºC & 1,0 Hz	0,97	0,89	0,91	0,95

The results confirm that the binder stiffness under the same conditions gave the best correlation with the mixture stiffness (highlighted cells), although the binder stiffness at other condition also generally correlated well.

The study was extended with a further mixture using 16 different binders [5.07], although the basic binder properties were not reported. The correlation coefficients, R^2, for each binder and mixture stiffness combination are given in Table 5.4.

Table 5.4 – Correlation coefficients, R^2, for binder and asphalt stiffness properties [5.07]

Asphalt / Binder	E* @ 10 ºC & 0,1 Hz	E* @ 10 ºC & 1,0 Hz	E* @ 20 ºC & 0,1 Hz	E* @ 20 ºC & 1,0 Hz
G* @ 10 ºC & 0,1 Hz	0,54	0,66	0,35	0,54
G* @ 10 ºC & 1,0 Hz	0,48	0,53	0,28	0,47
G* @ 20 ºC & 0,1 Hz	0,61	0,57	0,51	0,59
G* @ 20 ºC & 1,0 Hz	0,60	0,62	0,46	0,56

With the PMB binders, the correlations are not as good and the binder stiffness under the same conditions did not necessarily give the best correlation with the mixture stiffness (highlighted cells).

Results from a mixture with 16 different binders [5.19] measuring both the binder and mixture stiffness at 50 ºC and 10 Hz gave correlation coefficients, R^2, between the binder and mixture rheological properties as given in Table 5.5. Unfortunately, the values of G^* are not reported separately for the binder.

Table 5.5 – Correlation coefficients, R^2, for binder and asphalt stiffness properties [5.19]

Binder	Asphalt G* @ 50 ºC, 10 Hz	Asphalt G*/sin(δ) @ 50 ºC, 10 Hz
High temperature PG	0,52	0,59
G*/sin(δ) @ 50 ºC, 10 rad/s	0,66	0,72

An investigation into the SHRP deformation resistance criteria [5.20] included a relationship between the binder stiffness after RTFOT and the mixture stiffness with a correlation coefficient, R^2, of 0,92 for a single aggregate skeleton and a selection of modified and unmodified binders.

A review of the relationship between binder and mixture rheology [5.21] found that there is direct relationship between binder and mixture stiffnesses when the moduli are plotted at the same temperature and frequency. The correlation coefficient, R^2, was 0,98 for paving grade bitumen and 0,59 for polymer-modified binders.

Results from another single mixture with five binders, of which two were PMBs, [5.08] produced Equation (5.16), a polynomial between the mixture stiffness, S_{mix} (MPa), and the binder stiffness, S_{bit} (MPa), with a correlation coefficient, R^2, of 0,99.

$$S_{mix} = -2\times10^{-6}\times S_{bit}^4 + 0{,}0021\times S_{bit}^3 - 0{,}937\times S_{bit}^2 + 215{,}18\times S_{bit} \qquad (5.16)$$

Obviously, the equation is specific to the aggregate skeleton used.

5.3.3 Relationship with Site Experience

No references were found that relate the test results with results from site measurements of stiffness (or surrogates for stiffness) other than that reported in 5.3.4.

5.3.4 Durability

Six of eight dense mixtures that were monitored [5.17] were also measured for stiffness modulus at 10 Hz and three temperatures (-10 ºC, 15 ºC and 30 ºC) together with the elastic component of the stiffness modulus of the mixtures recovered after 12 years in service. The stiffness modulus for all mixtures had decreased at low temperatures with a decrease in the cohesion of the samples. However, there was limited correlation between the original and 12 year values with coefficients, R^2, of 0,33, 0,15, 0,05 and 0,01. Furthermore, the slope was negative at 15 ºC whereas it was positive for all other data sets. Therefore, the initial values are not a good indicator as how quickly the stiffness will change.

The dynamic stiffness modulus, G^*, of one paving grade and ten PMBs after RTFOT was measured at combinations of two temperatures (10 ºC and 20 ºC) and three frequencies (1,6 Hz, 1,0 Hz and 0,1 Hz) together with the dynamic stiffness modulus, E^*, of an asphalt mixture at the same combinations [5.14] . The precise methods for determining G^* and E^* were not identified. From the results, it was found that the correlation was quite poor for the PMBs compared to the paving grade binder until the PMBs were separated into different polymer types. Therefore, the ageing during RTFOT is different for different polymers although the possibility that the ageing on site is also different was not explicitly considered.

5.3.5 Overview of DSR Test as Descriptor for Stiffness

Both the theoretical and laboratory studies reviewed indicate that there is generally a good correlation between the DSR stiffness of the binder and the stiffness of the mixture. The fact that the actual stiffness is dependant on the aggregate mixture in which it is used does not affect the relevance because the goal is to identify the binder contribution. However, there is a dearth of data to directly validate that the binder DSR stiffness relates to field performance. Furthermore, there is limited and confused information on whether the durability of stiffness can be evaluated by conditioning the binder. The RTFOT method appears to work, but only if separate criteria are set for each type of binder modifier and for unmodified binder.

The precision of DSR measurements is approximately equivalent to that of the R&B softening point test, which is generally accepted as adequate for contractual purposes.

Given the above, it is suggested that DSR measurements can be used to indicate the contribution of the binder towards the performance of asphalt in respect to stiffness.

5.4 Fraass Breaking Point Test

5.4.1 Description

For description, equivalent standardised tests, precision and relationship with other bitumen tests, see Section 2.10.

5.4.2 Relationship with Asphalt Tests

The values of Fraass breaking point and stiffness (using the two-point method on trapezoidal specimens) were reported [5.15] for two binders in two mixtures. Mixture results are reproduced in Table 5.1, above. The results show a reduction in stiffness for a lowering of the Fraass breaking point, but the single value is insufficient to develop a general hypothesis.

5.4.3 Relationship with Site Experience

No references were found that relate the test results with results from site measurements of stiffness (or surrogates for stiffness).

5.4.4 Durability

No references were found for laboratory or field studies that indicated that the test will predict the change in stiffness with time.

5.4.5 Overview of Fraass Test as Descriptor for Stiffness

The references found contained insufficient relevant data to identify whether the Fraass breaking point can be used as a surrogate for stiffness or even are a parameter that affects the stiffness.

The precision of the Fraass breaking point is poor, which is one of the main reasons why an alternative measure of the low temperature behaviour of binders is being sought.

It is not recommended that Fraass breaking point is used to indicate the contribution of the binder towards the performance of asphalt in respect to their stiffness.

5.5 Penetration Test

5.5.1 Description

For description, equivalent standardised tests, precision and relationship with other bitumen tests, see Section 2.14.

5.5.2 Relationship with Asphalt Tests

From the results of comparisons with two mixtures and eight binders [5.16, 5.17] given in Table 5.2, the stiffness modulus of asphalt can be predicted for a specific grading as well by the penetration of the binder as by its stiffness modulus. Comparisons with two mixtures using seven binders and a further mixture using four other binders [5.06] showed that the penetration gave reasonable correlations with the mixture stiffness (Table 5.3), but the correlations were better still with the binder stiffness. The correlation was marginally better for the basic penetration value than on binder that has been conditioned by RTFOT.

Research into fatigue [5.22, 5.23] provided data on stiffness modulus at five combinations of temperature and frequency together with the basic binder properties both before and after RTFOT. The correlation coefficients, R^2, between the binder and mixture properties are given in Table 5.6.

Table 5.6 – Correlation coefficients, R^2, for binder and asphalt stiffness properties [5.22, 5.23]

Binder Properties	Asphalt Stiffness Modulus @ 10 ºC & 10 Hz	@ 10 ºC & 25 Hz	@ 20 ºC & 10 Hz	@ 20 ºC & 25 Hz	@ 20 ºC & 40 Hz
Penetration	0,35	0,27	0,67	0,54	0,36
Penetration after RTFOT	0,32	0,22	0,63	0,45	0,33
R&B softening point	0,16	0,10	0,41	0,48	0,07
R&B after RTFOT	0,12	0,05	0,35	0,35	0,04
Penetration Index, PI	0,06	0,04	0,27	0,21	0,02
PI after RTFOT	0,01	0,00	0,15	0,10	0,00

In separate research on durability [5.24], four sections on two road sites and seven sections on an airfield site were monitored, including penetration and R&B softening point of the binder and ITSM at one or two temperatures of the asphalts. The correlation coefficients, R^2, between the binder and mixture properties are given in Table 5.7.

Table 5.7 – Correlation coefficients, R^2, for binder and asphalt stiffness properties [5.24]

Binder Properties	Asphalt Indirect Stiffness Modulus				
	Motorway A9	Highway N241		Schiphol Airfield	
	ITSM @ 20 ºC	ITSM @ 5 ºC	ITSM @ 15 ºC	ITSM @ 0 ºC	ITSM @ 20 ºC
Penetration	0,94	0,31	0,17	0,57	0,24
R&B softening point	0,63	0,21	0,77	0,00	0,14
Penetration Index	0,71	0,18	0,55	0,09	0,00

The correlations coefficients between penetration and mixture stiffness from both studies are very variable and generally weak, indicating that any relationship is casual rather than fundamental. The use of binder after conditioning by RTFOT marginally reduces that correlation. However, there is a very good correlation (and even very universal) between penetration and binder stiffness (determined in DSR at the same temperature and loading time (frequency) as the penetration test), as shown in Figure 5.4 [5.25]. Hence, there should also be a good correlation between penetration and mixture stiffness providing the temperature and loading time are the same.

The initial and recovered penetration values are reported for two binders [5.26] together with stiffness by ITSM at 2,5 Hz in a high modulus base mixture with each binder at various air void contents. The binders were 15 pen bitumen (initial 15 x 0,1 mm, recovered 6 x 0,1 mm) and a polymer-modified binder (initial 12 x 0,1 mm, recovered 8 x 0,1 mm). The stiffness moduli were plotted on a graph with field results having higher air void contents and lower stiffness moduli than the laboratory results and the trend line for the PMB mixture being above that for the HMB15 mixture (although there was no distinction on the individual points as to which mixture they related). Given that there are results from only two binders, the data can, at best, only be an indicator that a lower penetration could indicate a higher stiffness modulus, as would be expected intuitively.

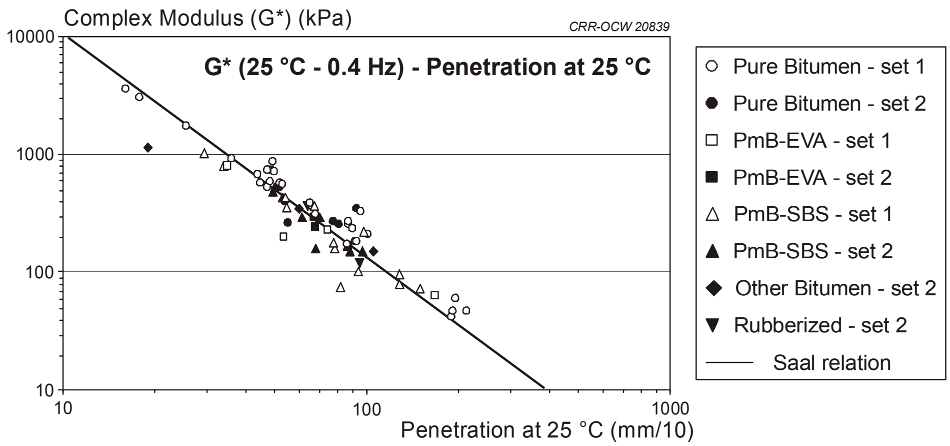


Figure 5.4 – Binder modulus at 25 °C and 0.4 Hz versus penetration for about 100 binders of different origin [5.25]

The penetration values of five binders was reported together with the results from stiffness sweeps to an in-house procedure for one mixture (DBM) with each of the binders and for a further two HRA mixtures with two of the binders [5.27]. The DBM with a linear SBS, the binder with the highest penetration at 81 x 0,1 mm, had the lowest complex modulus values of the DBMs while the DBMs with three other binders having penetrations of between 49 x 0,1 mm and 61 x 0,1 mm had very similar moduli. The DBM with the remaining binder, penetration 39 x 0,1 mm, had the highest modulus. Therefore, the findings also indicate that a lower penetration binder produces an asphalt with a higher stiffness modulus.

The initial and recovered penetration for a control and three polymer-modified binders are reported together with graphs of the stiffness by ITSM of an asphalt concrete with each of the binders at 5 ºC, 10 ºC and 20 ºC for both laboratory and site samples [5.01]. The control binder, with a penetration of 91 x 0,1 mm, had the highest stiffness moduli, then the PMBs with 98 x 0,1 mm, 126 x 0,1 mm and 134 x 0,1 mm in order. Therefore, the results support the findings that a lower penetration binder produces an asphalt with a higher stiffness modulus.

The values of penetration and stiffness (using the two-point method on trapezoidal specimens) were reported [5.15] for two binders in two mixtures. The mixtures results are reproduced in Table 5.1, above. The results support the basic hypothesis found from other papers, although the difference in penetration was marginal.

A series of hot rolled asphalt mixtures with 50 pen, TLA blend, Shell *Multiphalte* and Shell *Cariphalte* [5.28] were tested for ITSM using both 100 mm and 150 mm diameter specimens and the results compared with the penetration of the binders. The Pearson's Correlation values with the two sized specimens were -0.54 and -0.73, respectively, showing an imperfect relationship.

Research into the relationship between elastomeric modified binders and mixture performance [5.29] concluded that the penetration, as well as the R&B softening point, cannot be related to the mechanical performance of the mixture, including static stiffness modulus.

5.5.3 Relationship with Site Experience

Two of the papers reviewed [5.01, 5.26] contained results from both laboratory-prepared and site samples. The stiffness moduli were higher for laboratory-prepared samples than for the site samples, although the difference was explained by different air voids contents [5.01]. Therefore, the same conclusion can be drawn for site samples as for laboratory samples of a partial correlation between penetration and stiffness modulus. However, no data were found that related the penetration of the binder to the stiffness of the pavement rather than laboratory estimates of the stiffness of the material.

5.5.4 Durability

The research on durability at four sections on two road sites [5.24] monitored the indirect stiffness modulus over three years. However, the correlation coefficients, R^2, between the initial penetration and ITSM after ageing were as variable to the initial ITSM given in Table 5.7. This would be expected because there are consistently high linear correlations of ITSM with time for each of sections on the A9 motorway, with R^2 values between 0,53 and 0,77, and similar shaped polynomial relationships on the N241 Highway.

The recovered penetration was reported in two papers [5.01, 5.26] but no stiffness moduli were measured on the recovered samples. Therefore, the penetration results reported cannot be used to establish a correlation with long-term stiffness.

5.5.5 Overview of Penetration Test as Descriptor for Stiffness

The results from the papers reviewed indicate that there is a general trend for a decrease in penetration to be associated with an increase in stiffness modulus. This relationship, as observed, appears to be true for both polymer-modified and unmodified binders. Good correlation between penetration and mixture stiffness can be expected at 25 °C and 0.4 Hz (when the temperature and loading time of both tests are the same), but not necessarily for stiffness determined at any other temperature-frequency combination. Therefore, the test can be regarded as a parameter that affects stiffness (i.e. will provide a ranking, all other parameters remaining constant). Nevertheless, trials involving the change in penetration and stiffness modulus from recovered cores of a selection of binders and, preferably, mixtures would be useful to fully validate the relationship. Observation of the differences in pavement performance from different binders would be useful but is not essential.

The penetration test has been used for many years in contractual specifications without problems and, therefore, the precision is regarded as acceptable. Also, the penetration test is practical and not over-complex to performing. Therefore, the penetration should be considered when assessing the binder contribution to mixture stiffness, if only at a low level or at temperatures close to 25 °C.

5.6 Penetration Index

5.6.1 Description

For description, equivalent standardised tests, precision and relationship with other bitumen tests, see Section 2.15.

5.6.2 Relationship with Asphalt Tests

Penetration Index combined with other empirical binder characteristics can be used in the Van der Poel relation to predict binder stiffness, although the relationships are only valid for pure binders. However, from the correlation coefficients given in Table 5.2 [5.16, 5.17], penetration index is not a good predictor of the stiffness modulus of asphalt. From the results given in Table 5.6 [5.22] and Table 5.7 [5.24], the correlation between penetration index and asphalt stiffness is variable and generally weak, indicating supporting the lack of any fundamental relationship.

5.6.3 Relationship with Site Experience

No references were found that included both site data and penetration index results.

5.6.4 Durability

The research on durability at sections with the same four different binders on two road sites [5.24] monitored the indirect stiffness modulus over three years. However, the correlation coefficients, R^2, between the initial penetration index and ITSM after ageing were as variable to the initial ITSM given in Table 5.7. This would be expected because there are consistently high linear correlations of ITSM with time for each of sections on the A9 motorway, with R^2 values between 0,53 and 0,77, and similar shaped polynomial relationships on the N241 Highway.

5.6.5 Overview of Penetration Index as Descriptor for Stiffness

The penetration index is not as good a predictor as penetration, and so should not be considered when assessing the binder contribution to mixture stiffness

5.7 Softening Point (Ring and Ball) Test

5.7.1 Description

For description, equivalent standardised tests, precision and relationship with other bitumen tests, see Section 2.17.

5.7.2 Relationship with Asphalt Tests

From the results of comparisons with two mixtures and eight binders [5.16, 5.17] given in Table 5.2, R&B softening point is not a good predictor of the stiffness modulus of asphalt. Comparisons with two mixtures using seven binders and a further mixture using four other binders [5.06] showed that the R&B softening point gave poor correlation with the mixture stiffness (Table 5.3). The correlation was marginally better for the basic R&B softening point value than on binder that has been conditioned by RTFOT. From the results given in Table 5.2 [5.16, 5.17], R&B softening point is not a good predictor of the stiffness modulus of asphalt. From the results given in Table 5.6 [5.22] and Table 5.7 [5.24], the correlation between R&B softening point and asphalt stiffness is variable and generally weak, indicating supporting the lack of any fundamental relationship. A review of four binders [5.30] reported the resilient modulus of the mixture together with R&B softening point, torsional recovery and viscosity at 180 ºC of the binder, but the correlation coefficient, R^2, was only 0,17 for the R&B softening point and less for the other binder properties measured.

The initial and recovered R&B softening points are reported for two binders [5.26] together with stiffness by ITSM at 2,5 Hz in a high modulus base mixture with each binder at various air void contents. The binders were 15 pen bitumen (initial 70,0 ºC, recovered 92,5 ºC) and a polymer-modified binder (initial 75,0 ºC, recovered 92,0 ºC). The stiffness moduli were plotted on a crude graph with field results having higher air void contents and lower stiffness moduli than the laboratory results and the trend line for the PMB mixture being marginally above that for the HMB15 mixture (although there was no distinction on the individual points as to which mixture they related). Given that there are results from only two binders, the data can, at best, only be an indicator that a higher R&B softening point could indicate a higher stiffness modulus, as would be expected intuitively.

The R&B softening point of five binders was reported together with the results from stiffness sweeps to an in-house procedure for one mixture (DBM) with each of the binders and for a further two HRA mixtures with two of the binders [5.27]. The DBM with a linear SBS, the binder with the highest R&B softening point at 95 ºC, had the lowest complex modulus values of the DBMs while the DBMs with three other binders having R&B softening points between 52 ºC and 73 ºC had very similar moduli. The DBM with the remaining binder, R&B softening point 60 ºC, had the highest modulus. Therefore, the findings are inconclusive but, if anything, contrary to that of the previous report [5.26].

The initial and recovered R&B softening point for a control and three polymer-modified binders are reported together with graphs of the stiffness by ITSM of an asphalt concrete with each of the binders at 5 ºC, 10 ºC and 20 ºC for both laboratory and site samples [5.01]. The control binder, with a R&B softening point of 46 ºC, had the highest stiffness moduli, then the PMBs with 63,5 ºC, 51 ºC and 62,5 ºC in order. Therefore, the findings support the concept of an increase in R&B softening point being associated with a reduction in stiffness modulus except for the results for the PMB with a R&B softening point of 63,5 ºC which, according to that hypothesis, would have been expected to have the lowest stiffness modulus rather than the second highest.

The values of R&B softening point and stiffness (using the two-point method on trapezoidal specimens) were reported [5.15] for two binders in two mixtures. The mixtures results are reproduced in Table 5.1, above. The results support the hypothesis of an increase in R&B softening point being associated with an increase in stiffness modulus.

A series of hot rolled asphalt mixtures with 50 pen, TLA blend, Shell *Multiphalte* and Shell *Cariphalte* binders [5.28] were tested for ITSM using both 100 mm and 150 mm diameter specimens and the results compared with the R&B softening point of the binders. The Pearson's Correlation values with the two sized specimens were -0.25 and -0.14, respectively, showing a weak relationship.

Research into the relationship between elastomeric modified binders and mixture performance [5.29] concluded that the R&B softening point, as well as the penetration, cannot be related to the mechanical performance of the mixture, including static stiffness modulus.

The results from the papers reviewed indicate that there is no consistent trend between the R&B softening point of the binders and the stiffness modulus of the asphalts.

5.7.3 Relationship with Site Experience

Two of the papers reviewed [5.01, 5.26] contained results from both laboratory-prepared and site samples. The stiffness moduli were higher for laboratory-prepared samples than for the site samples, although the difference was explained by different air voids contents [5.01]. Therefore, the same conclusion can be drawn for site samples as for laboratory samples of a lack of correlation between R&B softening point and stiffness modulus. However, no data were found that related the R&B softening point of the binder to the stiffness of the pavement rather than laboratory estimates of the stiffness of the material.

5.7.4 Durability

The research on durability at four sections on two road sites [5.24] monitored the indirect stiffness modulus over three years. However, the correlation coefficients, R^2, between the initial R&B softening point and ITSM after ageing were as variable to the initial ITSM given in Table 5.7. This would be expected because there are consistently high linear correlations of ITSM with time for each of sections on the A9 motorway, with R^2 values between 0,53 and 0,77, and similar shaper polynomial relationships on the N241 Highway.

The recovered R&B softening point was reported in two papers [5.01, 5.26] but no stiffness moduli were measured on recovered samples. Therefore, the R&B softening point results cannot be used to establish a correlation with long-term stiffness.

5.7.5 Overview of Softening Point Test as Descriptor for Stiffness

There was no consistent relationship between the R&B softening point of binders and the stiffness modulus of the asphalt. Therefore, the test cannot be regarded as a parameter that affects stiffness.

Nevertheless, the R&B softening point test has been used for many years in contractual specifications without problems and, therefore, the precision is regarded as acceptable. Also, the R&B softening point test is practical and not over-complex to performing.

5.8 Recommendations for Stiffness

Six binder tests were identified and reviewed as having a potential relationship with asphalt mixture stiffness. Of these tests:

1. The BBR test can be discounted at this time because there were only two papers with diametrically opposing conclusions with regard to the ability of that test to reflect potential asphalt stiffness, if only because the testing was carried out under different conditions. However, because this divergence neither proved nor disproved any relationship, further work could make it relevant.
2. The DSR is covered by many papers which, in general, supported there being a relationship between the binder stiffness and asphalt stiffness. The relationship is particularly strong when using the same temperature and frequency conditions for both the binder and the mixture. However, the relationship is also dependent on the aggregate skeleton of the mixture.
3. The data on the Fraass test was limited to data from a single paper and, therefore, insufficient to draw conclusions at this time. However, the Fraass brittle temperature would not be expected theoretically to be related to mixture stiffness.
4. Penetration was found to correlate well with mixture stiffness, especially at the same temperature and loading time, although generally not as well as the DSR binder stiffness which is able to evaluate binder stiffness over a large range of temperatures and frequencies. The relationship was less good for PMBs than for paving grade bitumen. Nevertheless, it has potential for initial assessments because the test is simpler to perform than the DSR.

5. The penetration index generally had a marginally worse correlation with the mixture stiffness than the penetration whilst being a more complicated measure, so there appears no justification to use it as the binder measure for asphalt stiffness.
6. The R&B softening point generally has a significantly worse correlation with the mixture stiffness than the penetration, so there appears no justification to use it as the binder measure for asphalt stiffness.

Therefore, the best options for identifying the potential binder contribution to asphalt stiffness are DSR binder stiffness and/or penetration, with the former being able to evaluate stiffness for many temperature/frequency combinations that are encountered in practise but is also more complex.

There is sufficient data from the papers identified to validate the relationship between DSR binder stiffness and the mixture stiffness, particularly when using the same temperature and frequency conditions. However, the durability implications and any relationship to pavement performance are effectively missing. Furthermore, several of the papers indicated that DSR binder stiffness after RTFOT was less well correlated with mixture stiffness than the results from unaged binder. Therefore, there is justification to undertake further research in order to understand the measures required to identify long-term changes in mixture stiffness from binder properties, although such research would take some time to reach any conclusion.

There are sufficient data from the papers to support the use of the penetration test as a simple surrogate for the potential for mixture stiffness, at least for paving grade bitumens. Because it is a pragmatic test, the limitations in the relationship for durability and field performance would have to be accepted.

Penetration could also be used as a simple quality control test.

5.9 References

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6. Low Temperature Cracking

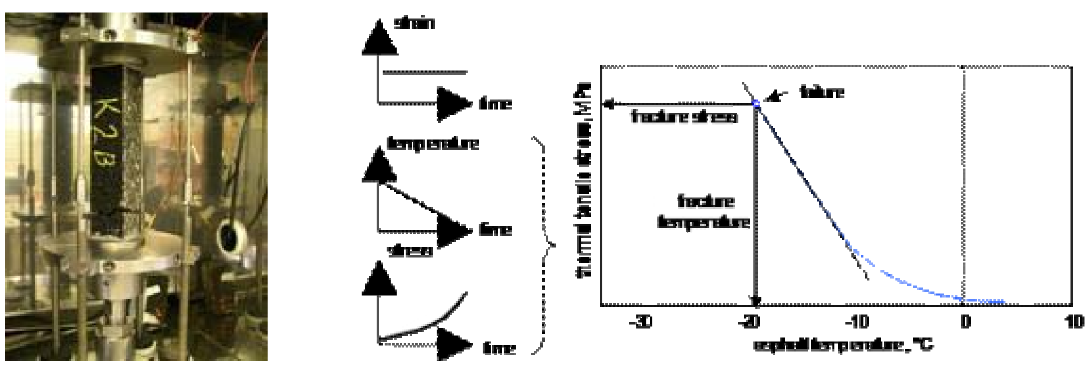
6.1 Asphalt tests

Low temperature cracking is particularly important for evaluating the low temperature behaviour of asphalt. Currently, four tests for determining low temperature properties of asphalt are known. These are:

- unrestrained thermal dilation test (TST),
- relaxation test (RT),
- tensile stress restrained specimen test (TSRST), and
- uniaxial tensile strength test (UTST).

In order to avoid errors in the test results arising from the use of different test equipment, all tests listed are performed employing the same testing equipment. The test methods described below are not yet standardised in a European standard, but there are technical test procedures described for the tests [6.01].

For the tensile stress restrained specimen test (TSRST), a beam specimen is mounted in a load frame which is enclosed in a cooling chamber. During the experiment, the length of the specimen is kept constant and the temperature is decreased with a constant cooling rate. Any movement of the specimen as a consequence of thermal shrinkage is monitored by LVDTs, activating a screw jack that stretches the specimen back to its original length. This process continues until the tensile stress exceeds the tensile strength and, hence, the specimen fractures. While different cooling rates are possible, the standard procedure starts at 20 °C and has a cooling rate of 10 °C/h. An illustration of the test procedure of the TSRST is given in Figure 6.1.



(a) Experimental setup (b) Illustration of result from TSRST

Figure 6.1 – Tensile stress restrained specimen test

In contrast to the TSRST, the asphalt specimen in the TST is not restrained. This test derives the thermal expansion (α_T). Test parameters such as the start temperature and the cooling rate are the same as for the TSRST.

The relaxation test (RT) is conducted at constant temperature (isothermal condition). Relaxation time (τ) and stiffness (E) are monitored at the considered testing temperatures. The test is performed in three steps. First, the specimen is cooled to the testing temperature (stress-free). A strain increment is then applied so that the instantaneous (elastic) material response can be determined. In the third step, the strain is kept constant and the relaxation of stress is monitored. In order to avoid damage to the specimen, the stresses induced by the strain increment in step three may not exceed 30 % of the tensile strength of the asphalt at the temperature considered. Commonly, the relaxation test is performed at +20, +5, -10 and -25 °C.

Similar to the RT, the UTST is conducted at the specified temperatures of +20, +5, -10 and -25 °C. After stress-free cooling of the asphalt to the testing temperature, the tensile strength test is performed by applying a constant strain rate (1 mm/min) until the specimen fractures [6.01].

6.2 Bending Beam Rheometer (BBR) Test

6.2.1 Description

For description, equivalent standardised tests, precision and relationship with other bitumen tests, see Section 2.1.

6.2.2 Relationship with Asphalt Tests

Five papers [6.02, 6.03, 6.04, 6.05, 6.06] have been reviewed dealing with correlations between results from BBR measurements and TSRST measurements. Although the BBR limiting temperature of the binders is reported [6.02, 6.03, 6.04] to be correlated to the mixtures failure temperature from TSRST measurements, it has also been reported [6.05] that only the stiffness *S* and the *m*-value of binders determined at -24 °C are correlated to the failure temperature of the TSRST results. The BBR limiting temperatures are the temperatures at which the creep stiffness *S* reaches 300 MPa and the *m*-value reaches 0,3. The mixture failure temperature from TSRST measurements corresponds to the maximum stress occurring during TSRST.

The failure temperature of asphalt was found [6.02] to correlate with the BBR limiting temperature of binders with a correlation coefficient of $R^2 > 0{,}80$. For this study, three base bitumens were blended with four different polymers, giving a total of 15 binders of which 12 are PMBs. The results of binder and asphalt testing are presented in Figures 6.2 and 6.3, respectively. The addressed low temperature parameters of bituminous binders and mixtures can be seen to be dependent on the base bitumen.

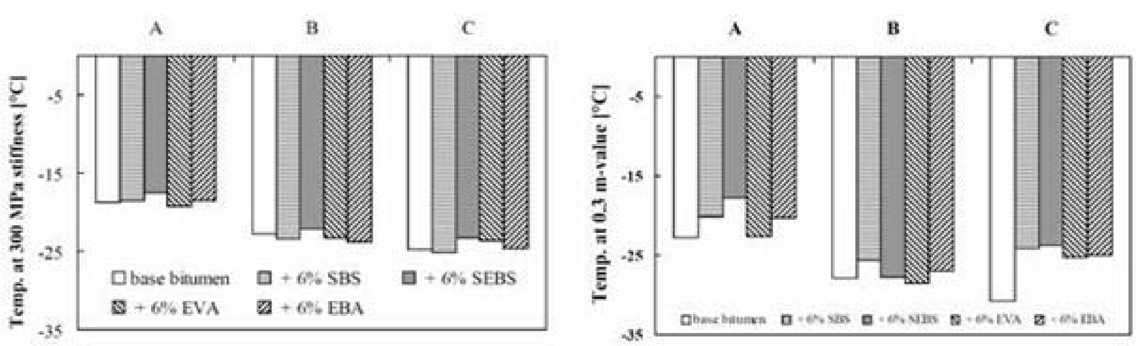


Figure 6.2 – BBR limiting temperatures from pure and modified binders [6.02]

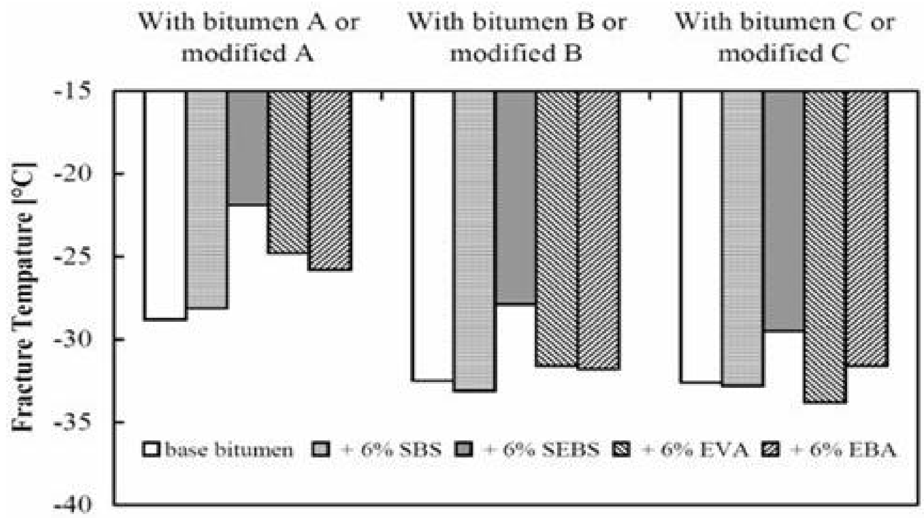


Figure 6.3 – TSRST failure temperatures of asphalt mixtures [6.02]

A much better correlation between TSRST failure temperature and BBR limiting temperature was determined [6.03] in which R^2 = 0,99 (Figure 6.4). This study included three paving grade bitumens and two modified binders. BBR testing was performed on RTFOT aged binders. Particularly in the case of modified binders, this study refers to the BBR limiting temperature compared to Fraass breaking point in order to relate to the low temperature behaviour of an asphalt mixture. It has to be remembered that this study includes only five binders.

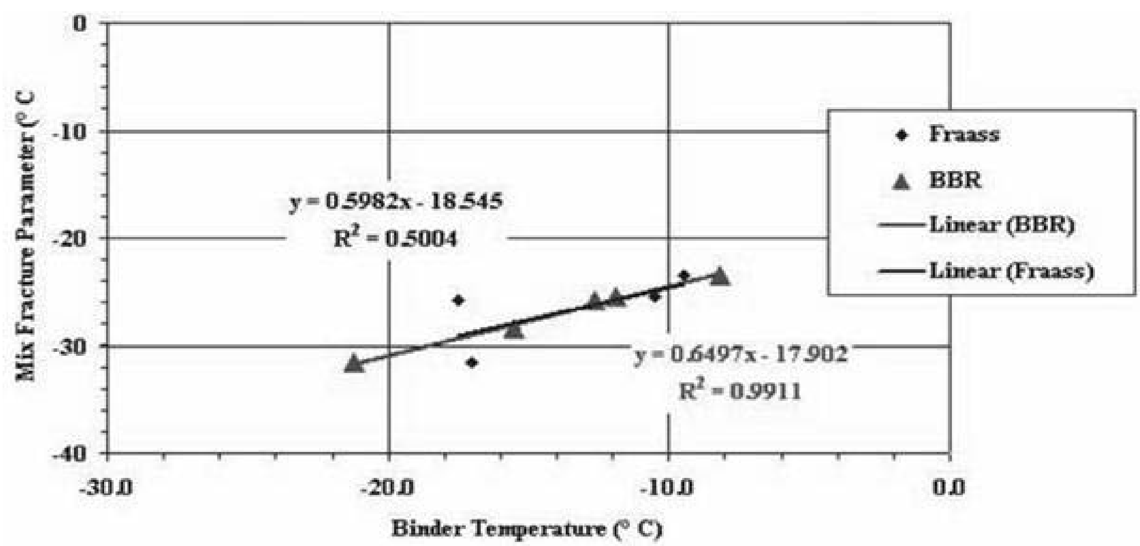


Figure 6.4 – Relationship between binder and mixture low temperature parameters [6.03]

The correlation coefficient between the BBR limiting temperature of the binder and the TSRST failure temperature has been found [6.04] to be R^2 = 0,76 (Figure 6.5). For this study, 36 binders were evaluated of which 13 were PMBs. All binder and asphalt tests were performed on unaged samples.

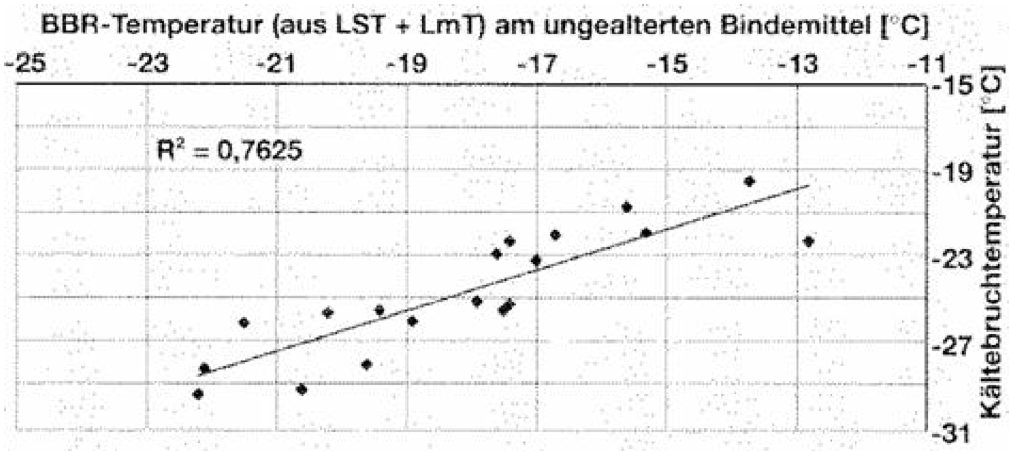


Figure 6.5 – Relationship between BBR limiting temperature and mixture failure temperature from TSRST measurements [6.04]

A comparison [6.06] has been made of the BBR limiting temperatures and the mixture failure temperatures from TSRST measurements (failure temperature) conducted with both regular and notched asphalt test samples. Eleven RTFOT-aged binders were evaluated of which seven were PMBs and four were paving grade bitumens. A summary of the results on binder and asphalt testing is given in Table 6.1.

Table 6.1 – Low temperature parameters of binder and asphalt [6.06]

Binder	Parameters for bitumens			Parameters for mixtures	
	T (S = 300 MPa)	T (m = 0,3)	T (ε_f = 1,3 %)	T_{TSRST}, un-notched	T_{TSRST}, notched
A60	-13,0	-15,0	-11,0	-25,0	-20,9
A60 + 5% SBS	-13,5	-13,5	-15,0	-23,0	-21,3
A100	-15,0	-18,5	-14,0	-28,1	-25,5
A100 + 3% SBS	-15,5	-18,0	-16,0	-27,5	-24,8
A100 + 5% SBS	-16,0	-17,0	-17,5	-27,7	-28,1
A100 + 7% SBS	-18,0	-17,0	-20,0	-27,8	-27,6
A200	-19,0	-23,0	-18,0	-31,9	-27,8
A200 + 5% SBS	-21,0	-22,5	-22,0	-31,0	-29,8
B85	-17,0	-21,0	-18,0	-28,0	-27,9
B85 + 5% SBS	-17,5	-18,5	-18,5	-26,3	-26,9
B85 + 5% SBS + 0,2% S	-20,5	-23,0	-21,0	-31,0	-31,5

In Table 6.2, the respective correlation coefficients between the binder and asphalt low temperature parameters are presented, ranging from 0,73 to 0,87 for the BBR. The results show that, for correlation purposes, the notched TSRST is better than the regular TSRST without a notch.

Table 6.2 – Correlation coefficients, R², for binder and asphalt low temperature properties [6.06]

Asphalt	T_{TSRST}	
Binder	un-notched	notched
T (S = 300 MPa)	0,73	0,87
T (m = 0,3)	0,84	0,76
T (ε_f = 1,3%)	0,53	0,83

The stiffness *S* and the *m*-value from BBR testing of twelve binders, of which seven were paving grade bitumens and five were PMBs, were correlated with TSRST results (failure temperature) [6.05]. The binders were subjected to RTFOT ageing before being tested in the BBR. The stiffness *S* and the *m*-value of the binders were determined at -24 °C. The results of the BBR and TSRST experiments are displayed in Table 6.3.

Table 6.3 – Results of BBR, DTT and TSRST experiments [6.05]

Sample	BBR @ -24 ºC		DTT @ -30 ºC			TSRST	
	S (MPa)	m –	δ_f (MPa)	ε_f (%)	J_f (1/GPa)	T_{max} (ºC)	δ_{max} (MPa)
U1	231	0,359	4,67	2,38	5,10	-40,2	4,62
U2	291	0,289	3,98	1,61	4,05	-34,8	4,35
U3	439	0,260	3,22	0,94	2,92	-30,7	4,56
U4	670	0,249	2,87	0,59	2,06	-26,0	3,87
U5	865	0,204	4,11	0,83	2,02	-29,6	4,25
U6	155	0,376	3,13	1,30	4,15	-33,7	4,36
U7	231	0,373	5,85	2,49	4,26	-34,1	4,25
Mean U1–U7	348	0,301	3,87	1,28	3,32	-32,7	4,32
P1	169	0,349	5,04	2,57	5,10	-35,8	4,61
P2	186	0,340	6,23	2,77	4,45	-32,7	3,95
P3	224	0,357	5,00	1,94	3,88	-33,5	4,43
P4	205	0,347	4,33	2,32	5,36	-34,8	4,14
P5	–	–	3,77	0,86	2,28	-2,86	4,89
Mean P1–P5	195	0,348	4,81	1,94	4,04	-33,1	4,39

Plots of the BBR stiffness *S* and *m*-value against the TSRST failure temperature (T_{max}) are displayed in Figure 6.6. The BBR results, both stiffness *S* and *m*-value, and the TSRST data (failure temperature) do not appear to correlate well (R^2 = 0,53 and R^2 = 0,50, respectively).

6.2.3 Relationship with Site Experience

No references were found that relate the test results with results from site measurements of low temperature cracking.

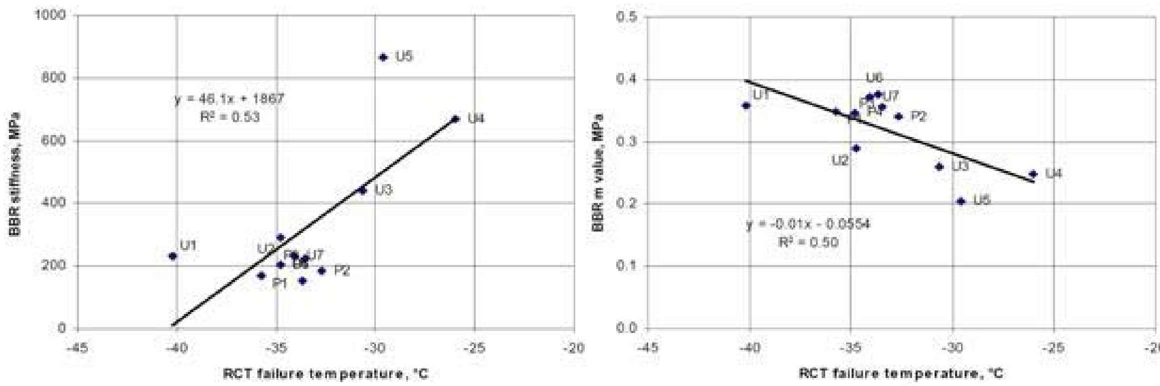


Figure 6.6 – BBR stiffness *S* (left) and *m*-value (right) against TSRST failure temperature [6.05]

6.2.4 Durability

No references were found for laboratory or field studies that indicated that the test will predict the change in low temperature properties with time.

6.2.5 Overview of Bending Beam Rheometer (BBR) Test as Descriptor for Low Temperature Cracking

Five reviewed papers, all from different sources, deal with relationships between BBR results and TSRST results. Four of them address the BBR limiting temperature and one paper deals with BBR stiffness *S* and *m*-value as low temperature parameters.

The studies reviewed indicate that there is generally a good correlation between the BBR limiting temperature of the binder and the TSRST failure temperature of the mixture. Table 6.4 provides the corresponding correlation coefficients for the papers reviewed dealing with BBR limiting temperature. The relationship appears to be true for both polymer modified and unmodified binders. The BBR limiting temperature provides a ranking for the corresponding asphalt's low temperature behaviour. Nevertheless, there is a lack of data concerning field performance and durability.

Table 6.4 – Sources for BBR limiting temperature against TSRST failure temperature with correlation coefficient and number of binders tested

Source	Correlation coefficient R^2	Total number of binders tested	Number of modified binders tested
[6.02]	0,80	15	12
[6.03]	0,99	5	2
[6.04]	0,76	36	13
[6.06]	0,73; 0,84 → un-notched 0,87; 0,76 → notched	11	7

In contrast to the BBR limiting temperature, BBR stiffness *S* and *m*-value determined at -24 °C are not well correlated with the TSRST failure temperature. The BBR stiffness *S* and *m*-value do not appear to be as good predictors for the low temperature behaviour of an asphalt as the BBR limiting temperature. However, it should be kept in mind that only one source reviewed [6.05] dealt with the correlation between *S* and *m*-value and TSRST failure temperature.

6.3 Direct Tensile Test (DTT)

6.3.1 Description

For description, equivalent standardised tests, precision and relationship with other bitumen tests, see Section 2.7.

6.3.2 Relationship with Asphalt Tests

Results from DTT measurements were correlated to the low temperature performance of mixtures [6.05, 6.06, 6.07]. The tests conducted with asphalt mixtures were TSRST and UTST.

DTT results have been correlated [6.05] with TSRST results of mixtures containing the same binders. Twelve binders, including seven conventional and five polymer-modified binders, were tested in the DTT. The binders were subjected to RTFOT ageing before being tested in the DTT. The DTT experiments were conducted at a temperature of -30 °C and a cross-head speed of 0,1 mm/min. Failure stress (σ_f), failure strain (ε_f) and failure compliance (J_f) from DTT as well as the failure temperature from TSRST are given in Table 6.3. The failure compliance J_f is defined as the failure strain divided by the failure stress. Correlation plots including the DTT and the TSRST results are shown in Figure 6.7. The DTT failure strain values (ε_f) appear to correlate reasonably well with the TSRST failure temperatures (R^2 = 0,69). An even better correlation is observed between DTT failure compliance (J_f) and the TSRST failure temperature (R^2 = 0,79). This study indicates that there is a good correlation of low temperature asphalt cracking and binder properties determined in the DTT.

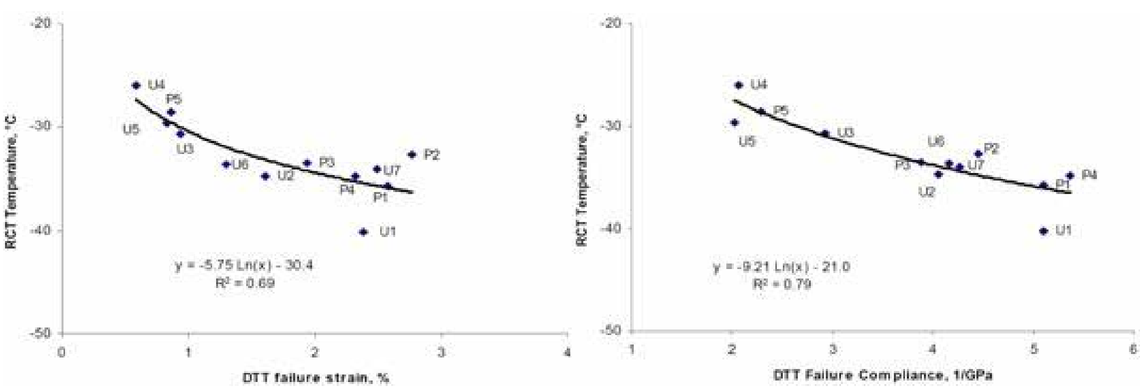


Figure 6.7 – TSRST failure temperature against DTT failure strain (left) and DTT failure compliance (right) [6.05]

A research programme [6.07] aiming at correlating the low temperature behaviour of different binders to asphalt mixtures was conducted. Two paving grade bitumens and three modified binders and the according mixtures were tested. The asphalt mixtures were produced with one type of aggregate and grading. DTT was performed at 1 mm/min and at constant temperatures with six repeats at each temperature.

The determined binder parameters were the temperature leading to failure at 1% strain ($T_{\varepsilon=1\%}$) and a brittle/ductile transition temperature of binders (T_{bdb}), which is the temperature at which the tensile strength peaks in the axes tensile strength-temperature (Figure 6.8). The low-temperature parameters for asphalt mixtures were T_{bdm} (at two strain rates) from UTST and the failure temperature T_{TSRST} from TSRST testing. In addition to T_{bdb}, a brittle/ductile transition temperature of asphalt mixtures (T_{bdm}), determined from direct tensile tests on asphalt (UTST) and depending on the applied strain rate (Figure 6.9), was introduced. The according parameters relating to a different strain rate are designated with T_{bdm} (300 µm/m/h) and T_{bdm} (45 000 µm/m/h). Figure 6.10 compares the tensile strength of binders found with the DTT to the tensile strength of mixtures, which are quiet close to each other.

The values obtained of T_{bdb} and $T_{\varepsilon=1\%}$ as well as of T_{TSRST} as T_{bdm} are given in Table 6.5. For the set of asphalts considered, the tensile tests on binders and mixtures and the T_{TSRST} rank the materials in the same manner. Table 6.6 presents the correlation coefficients (linear regression) between low-temperature parameters already discussed. The parameters T_{bdb} and $T_{\varepsilon=1\%}$ are well correlated with T_{bdm} (300 µm/m/h), T_{bdm} (45 000 µm/m/h) and T_{TSRST}, with correlation coefficients ranging from 0,90 to 0,99. That means these parameters can be good surrogates for each other.

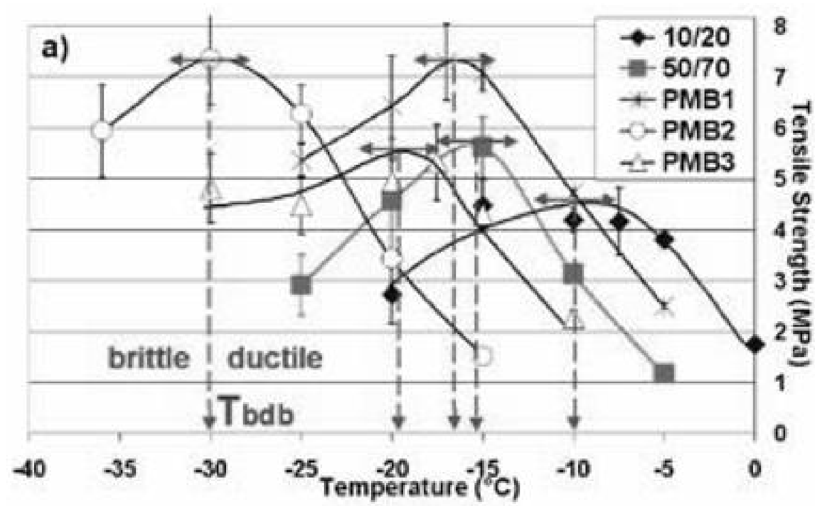


Figure 6.8 – DTT results for tensile strength of 5 binders according to temperature [6.07]

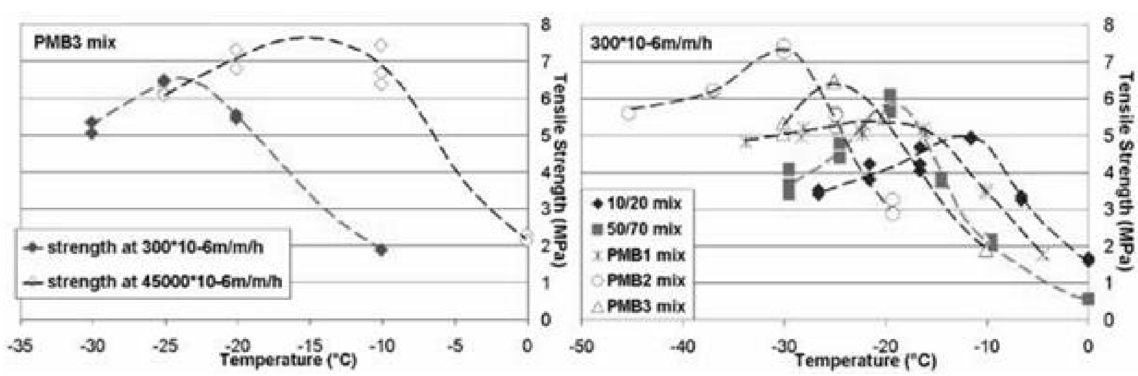


Figure 6.9 – Tensile strengths at constant temperatures of PMB3 mix (left) (different strain rates) and of five mixes (right) [6.07]

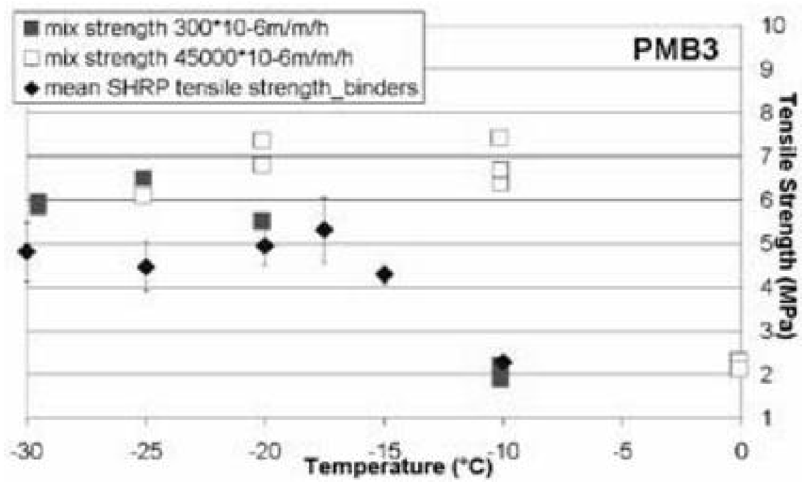


Figure 6.10 – Tensile strength of binders and tensile strength of mixes at two different strain rates [6.07]

Table 6.5 – Low temperature parameters of bitumens and asphalts [6.07]

	Binder	10/20	50/70	PMB1	PMB2	PMB3
Binder Parameters	T_{bdb} (°C)	-10	-15,5	-16,5	-30	-20
	$T\varepsilon$ = 1 % (°C)	-11	-18,5	-20,5	-32,5	-20,5
	K_{Ic} (kPa*m$^{1/2}$)	45,0 ± 5,8	43,0 ± 5,1	59,7 ± 12,4	129,8 ± 36,0	50,8 ± 2,7
	G_{Ic} (J/m^2)	3,0 ± 0,6	4,9 ± 1,1	17,3 ± 8,7	78,9 ± 32,5	8,0 ± 0,7
Asphalt Parameters	T_{bdm} (300 µm/m/h) (°C)	-11	-19,5	-20	-30,1	-24
	T_{bdm} (45 000 µm/m/h) (°C)	-3	-13	-11	-22,5	-16
	T_{TSRST} (°C)	-17,8	-25,5	-26,1	-40,5	-28,5

Table 6.6 – Correlation coefficients (linear regression) between low-temperature parameters [6.07]

	T_{bdm} (300)	T_{bdm} (45000)	T_{TSRST}
T_{bdb} (°C)	0,94	0,92	0,99
$T\varepsilon$ = 1 % (°C)	0,92	0,90	0,99
K_{Ic} (kPa.m$^{1/2}$)	0,60	0,57	0,80
G_{Ic} (J/m^2)	0,61	0,58	0,81

The temperature at which the DTT failure strain reaches 1,3 %, $T_{\varepsilon=1,3\%}$, was correlated [6.06] with the mixture failure temperature of TSRST measurements, T_{TSRST}, on both regular and notched samples. Eleven RTFOT-aged binders were evaluated, of which four binders were paving grade bitumens and seven PMBs. A summary of the results on binder and asphalt testing is given in Table 6.1. In Table 6.2, the resulting correlation coefficients, R^2, between the binder parameters and the asphalt parameters are given. As already mentioned in

Section 6.2 (BBR), these results show that the notched TSRST is better than the regular TSRST without a notch. This difference seems to be especially true for the DTT, where the correlation coefficient for the un-notched samples accounts for 0,53 in contrast to 0,83 for the notched samples.

6.3.3 Relationship with Site Experience

No references were found that relate the test results with results from site measurements of low temperature cracking.

6.3.4 Durability

No references were found for laboratory or field studies that indicated that the test will predict the change in low temperature properties with time.

6.3.5 Overview of Direct Tensile Test (DTT) as Descriptor for Low Temperature Cracking

Three papers reviewed are dealing with correlations between DTT and asphalt mixture properties. All papers come from different sources.

In general, DTT parameters appear to correlate reasonably well with low temperature mixture properties. Promising parameters for addressing the contribution of a binder to the low-temperature behaviour of an asphalt are the failure compliance, the temperature leading to failure at a specific strain rate and a temperature designated T_{bdb}, which addresses the brittle/ductile transition temperature of binders. Even though there were only five materials tested, the two latter parameters correlated to the TSRST failure temperature very well with a correlation coefficient of $R^2 = 0,99$. Despite the promising results, the repeatability of the DTT still remains to be improved.

Again, there is a lack of data concerning field performance and durability.

6.4 Fraass Breaking Point Test

6.4.1 Description

For description, equivalent standardised tests, precision and relationship with other bitumen tests, see Section 2.10.

6.4.2 Relationship with Asphalt Tests

Fraass breaking point is a commonly used parameter for the determination of a binder's low temperature behaviour. The values of Fraass breaking point and correlations to TSRST measurements are reported in four sources [6.02, 6.07, 6.03, 6.04]. In general, the correlation between Fraass breaking point and the mixture failure temperature from TSRST measurements was found to be very weak. The correlation coefficients, R^2, range from 0,42 to 0,72 as summarised in Table 6.7. The relationship between TSRST failure temperature and Fraass breaking point is visualised in Figures 6.4 and 6.11 for [6.03] and [6.04] respectively.

Table 6.7 – Sources of Fraass test results with correlation coefficient and number of binders tested

Source	Correlation coefficient R^2	Total number of binders tested	Number of modified binders tested
[6.02]	0,50	15	12
[6.03]	0,50	5	2
[6.04]	0,42	36	13
[6.07]	0,72	5	3

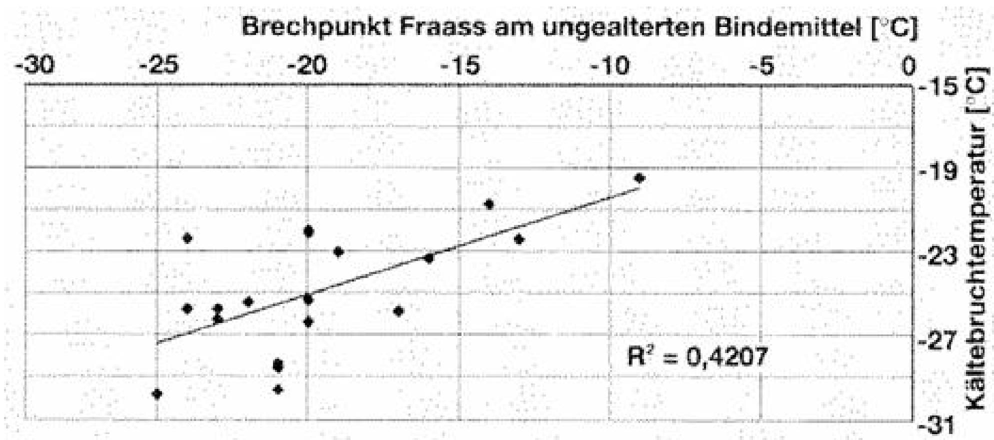


Figure 6.11 – Relationship between Fraass breaking point and TSRST failure temperature [6.04]

6.4.3 Relationship with Site Experience

No references were found that relate the test results with results from site measurements of low temperature cracking.

6.4.4 Durability

No references were found for laboratory or field studies that indicated that the test will predict the change in low temperature properties with time.

6.4.5 Overview of Fraass Breaking Point Test as Descriptor for Low Temperature Cracking

The correlation between Fraass breaking point and low temperature properties of asphalt mixtures was found to be only poor. Therefore, it is not recommended that Fraass breaking point is used to indicate the contribution of the binder towards the performance of asphalt mixtures in respect to their low temperature properties.

The precision of the Fraass breaking point is poor.

6.5 Fracture Toughness Test (FTT)

6.5.1 Description

For description, equivalent standardised tests, precision and relationship with other bitumen tests, see Section 2.11.

6.5.2 Relationship with Asphalt Tests

As already referred to in Section 6.3, a research programme [6.07] aiming at correlations between binder properties and mixture properties was conducted. One part of this study was to determine the low temperature fracture properties of binders. Five different binders, two paving grade bitumens and three PMBs, and the five respective mixtures were evaluated. The asphalt mixtures were produced with one type of aggregate and grading.

The low temperature fracture properties of bitumen were determined with a three point bending test on pre-notched bitumen samples. Fracture testing was performed based on ASTM E399-90 [6.08] and ISO 13586 [6.10] (Figure 6.12) at -19,5 °C with a rate of loading of 1 mm/s. The specimens were monotonically loaded until the fracture crack propagated.

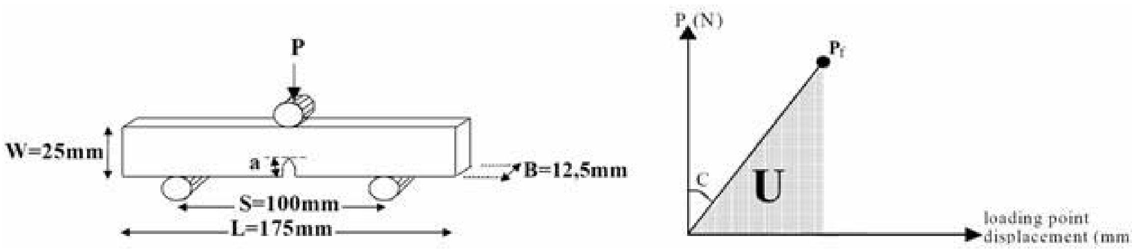


Figure 6.12 – Mode-I fracture test (left) and schematic of the load-loading point displacement diagram [6.07]

Fracture toughness, K_{Ic}, was calculated from tests conducted on six samples. Fracture energy, G_{Ic}, was determined from the energy *U* (Figure 6.12) derived from integrating the load versus loading point displacement diagram.

The binder parameters, K_{Ic} and G_{Ic}, are given in Table 6.5. Although the results seem to distinguish between pure bitumens and modified bitumens, the repeatability of the results is poor. The low-temperature parameters for mixtures, i.e. T_{bdm} (for two considered strain rates) from UTST testing and the mixture failure temperature, T_{TSRST}, from TSRST testing are also reported in Table 6.5.

In Table 6.6 the correlation coefficients (linear regression) between low temperature parameters are displayed. The binders fracture properties, K_{Ic} and G_{Ic}, are not well correlated with the low temperature asphalt properties, T_{bdm} and T_{TSRST}. This lack of correlation is explained by the way that the K_{Ic} and G_{Ic} parameters are independent of those of other tests and, thus, may be complementary to the parameters derived from DTT (Section 6.3).

In Table 6.8 and Figure 6.13, the correlation coefficients for exponential regression between K_{Ic} and G_{Ic} and the low temperature asphalt properties T_{bdm} and T_{TSRST} are given. The correlation coefficients appear to be higher relative to the linear regression results presented in Table 6.6.

Table 6.8 – Correlation coefficients (exponential regression) between K_{Ic} and G_{Ic} [6.07]

	T_{bdm} (300)	T_{bdm} (45000)	T_{TSRST}
K_{Ic} (kPa.m$^{1/2}$)	0,62	0,57	0,81
G_{Ic} (J/m^2)	0,74	0,67	0,86

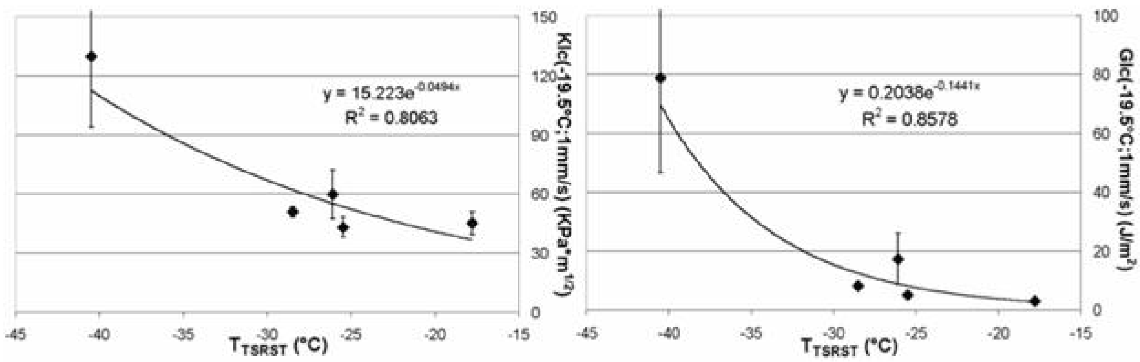


Figure 6.13 – Relationship between T_{TSRST} and K_{Ic} & G_{Ic} for the five studied binders [6.07]

6.5.3 Relationship with Site Experience

No references were found that relate the test results with results from site measurements of low temperature cracking.

6.5.4 Durability

No references were found for laboratory or field studies that indicated that the test will predict the change in low temperature properties with time.

6.5.5 Overview of Fracture Toughness Test (FTT) as Descriptor for Low Temperature Cracking

The binder fracture toughness K_{Ic} and fracture energy G_{Ic} require further research as performance indicators for low temperature cracking. Only one paper reviewed dealt with the fracture toughness and fracture energy of binders in which only five asphalts were tested. Although the results are promising, they still have to be checked with other kinds of bitumens before any definitive conclusion can be drawn.

The K_{Ic} and G_{Ic} parameters are independent and, thus, may be complementary to other reasonable low temperature binder properties, for example the parameters from the DTT. How both types of parameters need to be combined to adequately reflect and predict low temperature behaviour of asphalt mixtures will, of course, need further investigation.

With regard to the precision of the binder parameters K_{Ic} and G_{Ic}, the repeatability still remains to be improved.

6.6 Critical Cracking Temperature

6.6.1 Description

The concept of critical cracking temperature covers the determination of low-temperature properties of asphalt binders using data from both the bending beam rheometer (Section 2.1) and the direct tensile test (Section 2.7). It can be applied to both unaged material and material aged using RTFOT (Section 3.1) or PAV (Section 3.5).

The DTT data required is the failure stress at a minimum of two test temperatures. Two BBR data sets at two different temperatures are required with deflection measurements at 8 s, 15 s, 30 s, 60 s, 120 s, and 240 s. The stiffness master curve is calculated from the stiffness versus time data measured in the BBR. The stiffness master curve is then converted to the creep compliance curve, which is again converted into the relaxation modulus using different fitting procedures. Subsequently, the thermally induced stress is calculated. The calculated thermal stress is then compared to the failure stress from the DTT. The intercept of the failure stress from the DTT with the thermally induced stress rounded to the nearest 0,1 degree is the critical cracking temperature T_{cr} (see Figure 6.14).

Detailed information and the exact procedure for calculating the critical cracking temperature T_{cr} is described in [6.11]. A special software for calculating T_{cr} called TSAR (Thermal Stress Analysis Routine) has been developed.

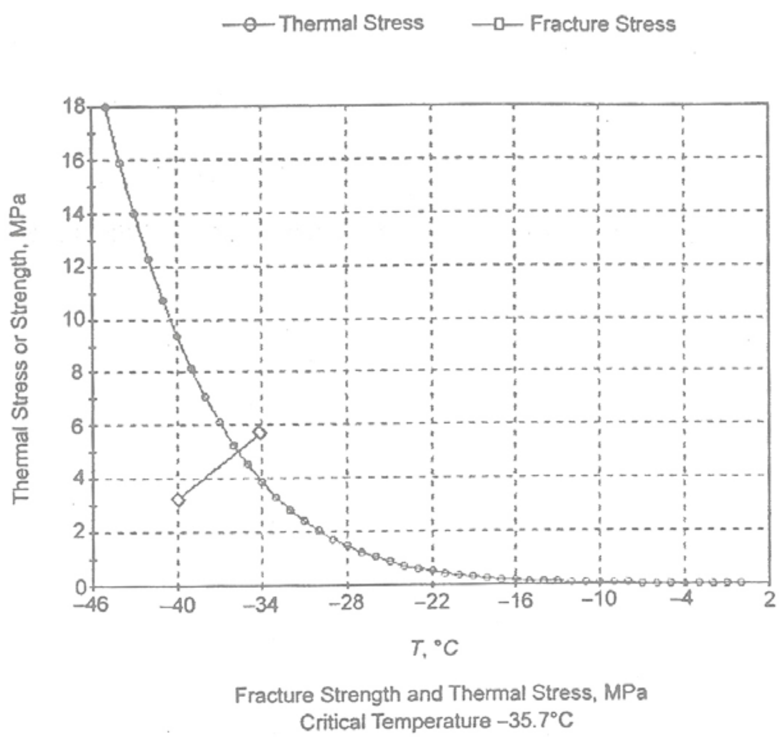


Figure 6.14 – Example of determination of the critical cracking temperature T_{cr} [6.11]

6.6.2 Relationship with Asphalt Tests

The critical cracking temperature, T_{cr}, of non-modified and modified binder types have been compared with the temperatures of the limit values for the low PG [6.12]. The measurements showed a very good agreement of the limiting temperatures according to the stiffness *S*, the *m*-value and the tensile strength was observed for the non-modified binders. With the modified binders, the critical cracking temperature generally ranged between the limit temperatures (PG precise) of the stiffness and the *m*-value.

6.6.3 Relationship with Site Experience

No data found.

6.6.4 Durability

No data found.

6.6.5 Overview of Critical Cracking Temperature as Descriptor for Low Temperature Cracking

The critical cracking temperature, T_{cr}, could be used as an alternative to using either the BBR or DTT on their own. However, more information is necessary to ensure that the extra work involved in using both measures does give greater reliability and relevance to the result.

6.7 Recommendations for Low Temperature Cracking

Four binder tests were identified and reviewed as having a potential relationship with asphalt mixture low temperature cracking. The outcomes of these tests are:

1. The BBR test is covered by many papers which indicate that there is generally a good correlation between the BBR limiting temperature and the TSRST failure temperature. The BBR limiting temperature provides a ranking for the corresponding asphalt's low temperature behaviour. In contrast to the BBR limiting temperature, BBR stiffness *S* and *m*-value determined at -24 °C, are not well correlated with the TSRST failure temperature, although the latter data was limited to a single paper. With regard to SBS modified bitumen, it is further reported that the BBR test underestimates the low temperature performance.

2. The results of the DTT were found to correlate reasonably well with mixture low temperature parameters, i.e. derived from TSRST and UTST. Promising candidates for addressing the binder's contribution to the low temperature behaviour of an asphalt mixture are the failure compliance of the binder, the temperature leading to failure at a specific strain rate and a brittle/ductile transition temperature of binders, designated T_{bdb}. It has to be noted that the DTT data were from three papers, with each paper addressing different characteristic values of DTT testing correlated with asphalt parameters. Despite the promising results, the repeatability of the DTT still remains to be improved.

3. The Fraass Breaking Point Test generally has a poor correlation with the asphalt mixture's low temperature behaviour besides a bad precision of the test. Therefore, it is not recommended that Fraass breaking point is used to indicate the contribution of the binder towards the performance of asphalt mixtures in respect to their low temperature properties.

4. The data of the Fracture Toughness Test is limited to data from a single paper and, therefore, the binder fracture toughness K_{Ic} and fracture energy G_{Ic} as performance indicators for low temperature cracking still require further research. The fracture parameters appear to be independent and, thus, may be complementary to other reasonable low temperature binder properties, for example to parameters from the DTT. How both types of parameters need to be combined to adequately reflect and predict low temperature behaviour of asphalt mixtures will, of course, need further research. The repeatability of the binder parameters K_{Ic} and G_{Ic} still remains to be improved.

From this point of view, the best options for identifying the potential binder contribution to the low temperature behaviour of asphalt are either BBR limiting temperature or a DTT parameter. An alternative might be the use of the concept of critical cracking temperature, which is a combination of BBR and DTT results to determine a low-temperature parameter called the critical cracking temperature, T_{cr}. In addition, a "real" fracture property, such as the fracture toughness or the fracture energy, could be used to complement the BBR and DTT results. The critical cracking temperature and fracture toughness both show great promise, but will need time to confirm its suitability and it is unlikely to be available in the immediate future.

There are sufficient data from the papers identified to validate the relationship between BBR limiting temperature of the binder and the mixture's failure temperature from TSRST measurements. With regard to both the DTT and the FTT parameters, there is insufficient data from the papers at this time and further research will be necessary before any definitive conclusion can be drawn. Such research should concentrate on the performance characterisation of modified bitumen. Nevertheless, the durability and any relationship to field performance are effectively missing for all tests. Therefore, there is justification to undertake further research to identify field performance, although such research would take some time to come to a robust conclusion.

6.8 References

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7. Fatigue Cracking

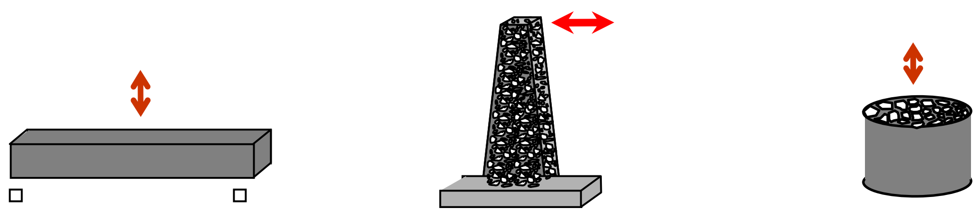
7.1 Asphalt tests

7.1.1 General

The cracks due to mechanical fatigue depend on the road pavement structures (i.e. thickness of layers, stiffness modulus and the rheological behaviour of the materials), on the traffic (particularly heavy lorries), on the climatic conditions and on the evolution (ageing) of the characteristics of the bitumen.

Fatigue tests on asphalt are undertaken under cyclic loading on specimen cut from pavements or manufactured in the laboratory. Two main types of sinusoidal loading are applied on the specimens:

- Flexural loading on prismatic specimens (applied at three or four points, including restraints, Figure 7.1a) or trapezoidal specimens (applied at two points, Figure 7.1b).
- Push-pull (or tension/compression) loading on cylindrical specimens (Figure 7.1c).
- Diametral loading on cylindrical samples.



(a) (b) (c)

Figure 7.1 – Shapes of asphalt specimens for fatigue tests

Cyclic stress or strain displacements are imposed depending on the level of the layer in the pavement to be designed or evaluated. The European standard for fatigue tests takes into account the two main types of loading, as shown in Figure 7.1.

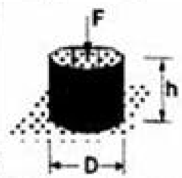
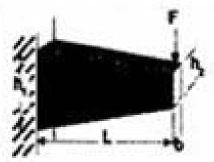
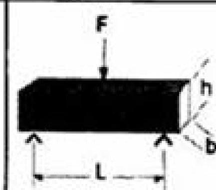
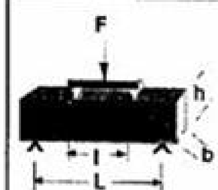
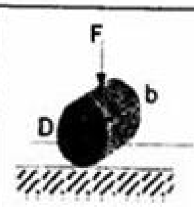
An inter-laboratory investigation on fatigue [7.01] with eleven different test methods (Table 7.1) included uniaxial push-pull, 2-, 3- and 4-point bending (2PB, 3PB and 4PB) and indirect tension tests was organised by RILEM Committees TC 101 BAT and was completed in 1996. The testing conditions specified were sinusoidal excitation at 10 Hz and 10 °C using controlled strain and stress modes. In total, more than 150 fatigue tests were carried out during the investigation. The conclusions of the investigation were:

- The fatigue lives determined are significantly affected by the test method employed.
- No correlation was found between the fatigue lives obtained from stress- (load-) and strain- (displacement-) controlled fatigue tests.
- The results of the beam tests (2PB, 3PB and 4PB) appeared to be dependent on the kind of test type used as well as on the size of the sample.
- For a given strain (or stress) amplitude, the beam tests (2PB, 3PB, and 4PB) generally resulted in longer life durations compared to homogeneous tension/compression (T/C) tests. This increase was due to the influence of specimen shape (the stress and strain values are always smaller in the sample). It did not seem possible to transpose results

from one test to another or to use the same formula to predict degradation or cracking of roads. Therefore, if a correlation is found between fatigue life and a characteristic of bitumen, it must be relative to a particular asphalt fatigue test.

- The complex modulus obtained at the beginning of the fatigue tests is independent of the type of test.
- A fatigue damage law can be defined and a unique set of parameters derived that accurately describes push-pull (or tension-compression) tests for both strain and stress controlled conditions and describes the controlled strain 2PB tests. However, further work is still needed.
- Biasing effects, which are not fatigue, exist during a fatigue test which affect the result. One effect is the heat caused by the accumulation of dissipated viscous energy and another, less well known, one appears to be due to the thixotropy of the binder.

Table 7.1 – Types of fatigue test

Type	Test Geometry	Type of loading/ Country of the team	Amplitude (10^{-6} m/m or MPa)
T/C		Tension-Compression "Homogeneous" F_1, S_1	Strain: (80), 100,140, 180 Stress: 0.9
2PB		Two-Point Bending "Non Homogeneous" F_2, B_1, B_2	Displacement; max strain: 140, 180, 220 Load; max stress: 1.4
3PB		Three-Point Bending "Non Homogeneous" N_1	Displacement; max strain: 140, 180, 220 Load; max stress: 1.4
4PB		Four-Point Bending "Non Homogeneous" N_2, P, PL, UK	Displacement; max strain: 140, 180, 220 Load; max stress: 1.4
ITT		Indirect Tensile Test "Non Homogeneous" S_2	Load; max strain: at first cycle: ~25, ~40, ~65

7.1.2 Typical behaviours of some asphalts

With constant stress, the fatigue life of asphalt increases with the modulus of rigidity (stiffness). With constant strain, the fatigue life of asphalt is greater at higher temperatures and less with higher stiffnesses. Thus, the resistance to fatigue decreases when the modulus increases from lower levels of compaction (as is the case with dense graded mixtures), whereas it increases with asphalts made from sand and gravel.

Experimental designs were carried out [7.02] to evaluate the influence of parameters, including the nature of the bitumen, the bitumen content, the granularity, the temperature and the level of compaction, on the fatigue strength.

Figure 7.2 represents the plane of variations in the resistance to fatigue that result from the binder content and the level of compaction (expressed as the degree of tightening, ΔC, equal to the usual level of compaction achieved on site minus the level of compaction of laboratory prepared specimens) for a particular asphalt mixture.

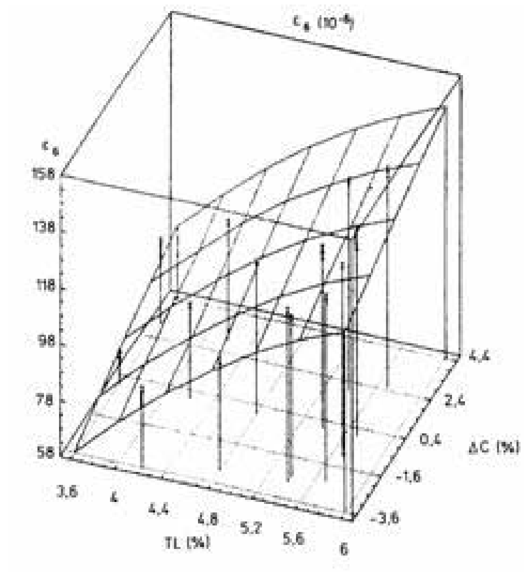


Figure 7.2 – Variations in resistance to fatigue depending on binder content and level of compaction

7.1.3 Effect of the temperature on the fatigue test

For one grade of bitumen (50/70 pen) and one bitumen content (5,4 %), the response of admissible strain at one million cycles (ε_6) can be deduced for temperatures between -10 °C and +30 °C by Equation (7.1), which reaches a minimum at a temperature of θ = 3 °C.

$$\varepsilon_6 = 10^{-4}(1.21 - 0.0088 * \theta + 0.00148 * \theta^2) \tag{7.1}$$

It has been found [7.03] that the test temperature influences the fatigue life according to Equation (7.2).

$$N_f = 10^{(K_1 + K_2\theta)} * \varepsilon^{(K_3 + K_4\theta)} \tag{7.2}$$

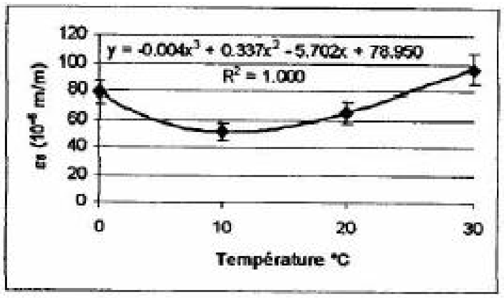
where:
N_f = the fatigue duration expressed in a number of cycles
K_i = the characteristic parameters of asphalt mixtures
ε = the initial strain
θ = is the temperature

For a given asphalt mixture, the second order model has been used [7.04] to obtain the results presented in Figure 7.3a. Figure 7.3b shows the correlations existing between the results of the fatigue resistance when estimated and when measured at four selected temperatures.

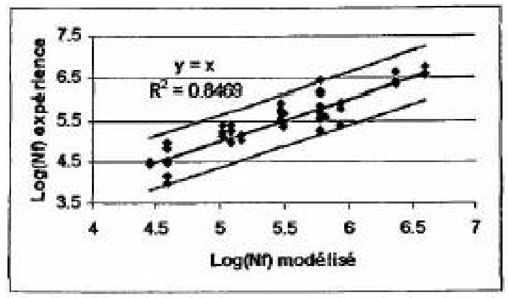
7.1.4 Effect of the granularity for Béton Bitumineux Semi Grenu (BBSG)

When the upper size *D* of the aggregate is changed but all other parameters (natural aggregate, nature and bitumen content, etc) are held constant, then:

- The level of compaction varies (it is all the more significant because *D* is significant).
- The fatigue strength remains the same with the tightening of the granular structure compensating for the embrittlement generated by the concentration of the large size granular.



(a) Variation of fatigue lives ε_6 with temperature



(b) Prediction of N_f with initial strain for four temperatures

Figure 7.3 – Effect of temperature on fatigue

- The stiffness modulus of the material varies appreciably in line with the level of compaction (500 MPa by point).

The level of compaction is closely related to the bitumen content and, for this type of asphalt mixture, the relationship for the strain is expressed by Equation (7.3).

$$\varepsilon_6 = -290 + 27 * TL - 1.5TL^2 + 3.3 * C \qquad (7.3)$$

where:
TL = the bitumen content (%)
C = the level of compaction (%)

7.1.5 Effect of the immersion in water

Water weakens the material, which can then lose a significant proportion of its fatigue resistance after three weeks of immersion. This loss is partly recovered after one week in dry storage following the three weeks of immersion.

7.1.6 Classification of the influence of the parameters for the same bitumen

The temperature of the test is the most significant parameter for fatigue. It is followed by the bitumen content and, to a lesser extent, by the aggregate gradation and the level of compaction. For the modulus at 15 °C and 10 Hz, the binder content and the level of compaction are of the same order of effect. The filler content has a rigidifying effect.

7.1.7 Effect of bitumen type using the same paving grade

The fatigue behaviour of an asphalt mixture, when compacted in a standard way, with various binders (bitumen, modified and special) can vary by a factor of three. Thus, the choice of binder can affect the resistance to fatigue of the asphalt [7.02, 7.05, 7.06].

It has also been found [7.07] that styrene-butadiene-styrene (SBS) modified bitumens can increase the breaking strength.

7.1.8 Stiffness modulus

The stresses and strains developed in the asphalt layer under the effect of the traffic loads are related to the stiffness modulus of the asphalt. Therefore, it is necessary to relate the pavement behaviour to the complex modulus under specific conditions, such as the value of the modulus at a given frequency and temperature or the variation of this modulus with changes of frequency or temperature. It has been shown [7.08] that there are relationships between the fatigue behaviour and of the values of asphalt mixture modulus.

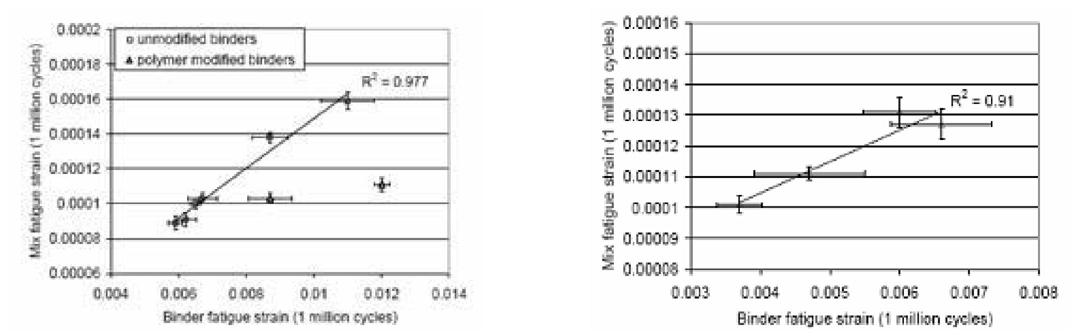
7.2 Bitumen Fatigue Testing

7.2.1 Description

For description, equivalent standardised tests, precision and relationship with other bitumen tests, see Section 2.2.

7.2.2 Relationship with Asphalt Tests

Various tests are under development to compare the results of fatigue resistance of the bitumens with those of the asphalt. A comparison [7.09] of the mixture fatigue ($\varepsilon_{6\ mix}$) with the binder fatigue ($\varepsilon_{6\ bit}$) at equi-stiffness conditions (G* = 30 MPa) and differing temperatures (Figure 7.4a) found that, for paving grade bitumen, the agreement was good but, for the PMBs tested, there was an increase in the binder fatigue life which was not observed in the behaviour of the mixture. For tests at constant temperature, the relationship between binder and mixture fatigue (Figure 7.4b) indicates that the binder versus mixture strain level was independent of the gap defined in rheometry measurement. A good relation between (unmodified) binder and mixture behaviour is obtained.



(a) equi-stiffness and differing temperatures (b) constant temperature

Figure 7.4 – Comparison of binder and mixture strain levels with fatigue lives of one million cycles

However, the relation between binder and mixture fatigue behaviour will be dependent on the mixture type.

Figure 7.5 [7.10] shows the relationship between the average mixture performance and the binder fatigue life as determined by the number of cycles to 50 % of the initial *G** value. There is a high correlation (R^2 = 84 %) for the nine binders that showed binder fatigue failure. This result is very encouraging and indicates that the newly developed binder fatigue test is promising and could be a better indicator of the fatigue damage of mixtures.

The dissipated energy ratio approach is proposed [7.10] as the method to determine the fatigue life of binders because of the independent nature of this approach on loading modes. Although the geometry has certain effects on the results, this approach can give reliable results that are found to correlate well with mixture performance by selecting the proper test conditions.

7.2.3 Relationship with Site Experience

No papers have been found that relate to bitumen fatigue testing with site experience.

7.2.4 Durability

No papers have been found that relate to bitumen fatigue testing with durability.

7.2.5 Overview of Bitumen Fatigue Test as Descriptor for Fatigue Cracking

The bitumen fatigue test is in progress but no significant results are known.

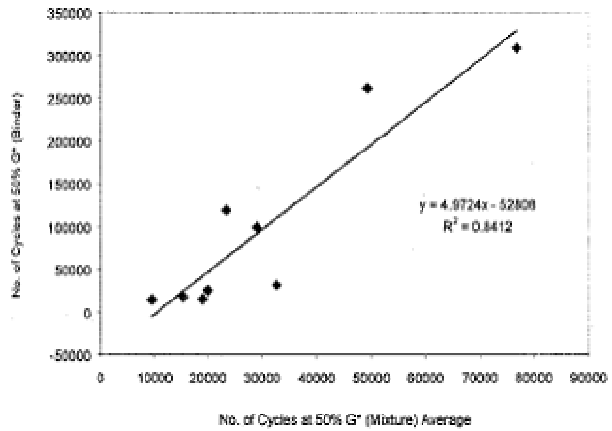


Figure 7.5 – Correlation between binder fatigue life and average mixtures fatigue life measured at the same temperature and frequency

7.3 Binder Complex Modulus Test

7.3.1 Description

For description, equivalent standardised tests, precision and relationship with other bitumen tests, see Section 2.8.

7.3.2 Relationship with Asphalt Tests

The complex moduli of binders are quite closely correlated to the complex moduli of asphalts [7.11]. The exact relationship depends on the mixture formulation [7.10]. For one type of asphalt mixture and different kind of binders, the admissible strain (at 10^6 cycles) results in fatigue tests seem to be correlated to the norm of the mixture complex modulus [7.06] (Figure 7.6). However, the correlation became poor (Figure 7.7) when using the rheological property of the binder G*sinδ [7.10].

It has been concluded [7.12] that binder rheology alone is insufficient to predict mixture fatigue performance. This is not surprising because fatigue is generally recognised to be a relatively complex failure mechanism, including crack initiation and propagation, crack stopping and healing effects. Meanwhile, a relationship between the slope of fatigue law and the tangent of the phase angle obtained to 10°C or 20°C and 10 rad/s has been proposed [7.13, 7.14].

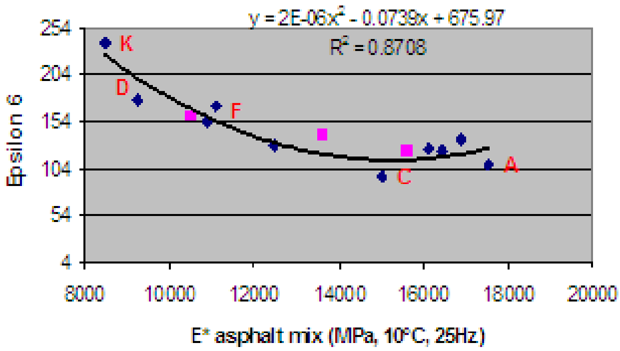


Figure 7.6 – Complex modulus of the asphalt mixture and mixture fatigue lives

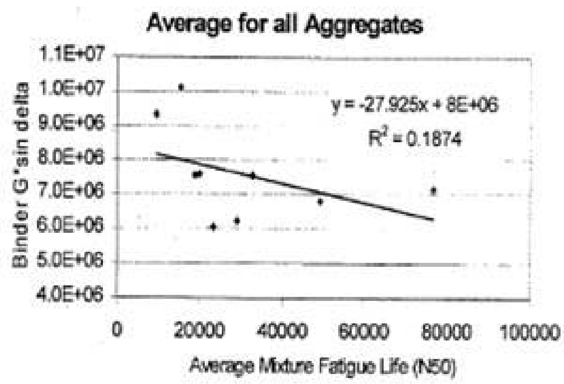


Figure 7.7 – Complex modulus of the binder and mixture fatigue lives

7.3.3 Relationship with Site Experience

Wheel track tests conducted on asphalt mixtures containing six bitumens and one aggregate from the SHRP Materials Reference Library (MRL) have been reported [7.03]. The experimental arrangement of the thin asphalt-concrete slab in a slab testing facility was such that the resulting structure represented a relatively thick, full-scale, in-service pavement structure and controlled-stress mode of loading. The conclusions were that:

- For fatigue life, the rankings of cores of MRL bitumens based on fatigue life (N1) from wheel track testing were similar to those based on fatigue life obtained from the flexural beam fatigue tests.
- For modified mixtures, validation results were generally inconclusive, except that both wheel track and flexural beam test results suggest that modification with one modifier was detrimental to the fatigue life of mixtures containing one bitumen.

Allowances have been made [7.06] to take into account controlled stress, controlled strain and rest periods in order to explain the shift that still exist between prediction of life duration obtained from the tests with rest periods and the actual behaviours of pavements. The effect of healing needs further study.

Periods of rest, which strongly increase the fatigue life of the asphalt tested, have been introduced [7.03, 7.04, 7.15, 7.16, 7.17] in order to take into account the heating during a fatigue test which reduces the stiffness modulus of the asphalt.

7.3.4 Durability

The durability of the fatigue properties is intuitively related to the ageing of materials. Only one article [7.18] discusses the subject in term of correlations between the threshold of cracking observed on site and about the physico-mechanical characteristics of the bitumens. Critical points about the appearance of mixture cracking have been proposed for the rheological parameters of the bitumens that are considered as relevant. However, even if these parameters are known for an unaged binder and after RTFOT plus PAV aging, it is not possible to predict sensitivity to cracking on site after the coating process and ageing.

7.3.5 Overview of binder complex modulus Test as Descriptor for Fatigue Cracking

It appears that mixture fatigue (as measured by tests conducted at controlled rate of strain with no resting time) is, for a given mixture formulation, closely related to changes in the modulus of the constituent bitumen. More research is needed to determine the precise bitumen rheological parameters in relation to the fatigue test. Meanwhile, all measurements of the evolution of the mechanical properties under conditions, such as those proposed for the fatigue of the bitumen or after ageing at the temperature for which the phase angle reaches a given threshold (e.g. delta = 27° @ 10 rd/s), must be retained.

7.4 Force Ductility Test

7.4.1 Description

For description, equivalent standardised tests and precision, see Section 2.9.

7.4.2 Relationship with Asphalt Tests

A correlation was undertaken between force ductility and fatigue behaviour of asphalt concrete with both paving grade bitumen and EVA, SBS and tyre-rubber modified bitumens [7.19]. The test parameters of temperature and deformation speed were the most significant parameters in the test. Nevertheless, the results indicated that there are relationship between tenacity of the binders and both the number of load applications to failure and the cumulative dissipated energy of the asphalt. Unfortunately, the relationships were different for crumb tyre-rubber modified bitumen and for EVA- and SBS-modified bitumens. Therefore, the tenacity of the binder alone cannot be used to predict the asphalt performance in fatigue with a given aggregate skeleton.

7.4.3 Relationship with Site Experience

No papers have been found that relate to force ductility testing with site experience.

7.4.4 Durability

No papers have been found that relate to bitumen force ductility testing with durability.

7.5 Bitumen Technological Tests (Penetration, Ring and Ball, Viscosity, Cohesion, BBR and DTT) Before and After Ageing

7.5.1 Description

For description, equivalent standardised tests, precision and relationship with other bitumen tests, see the relevant sections of Chapter 2.

7.5.2 Relationship with Asphalt Tests

For the last 10 years, there have been no articles devoted to the existing relations between the usual properties and the resistance to fatigue of the asphalt. Further data will have to wait for the results from ongoing projects.

7.5.3 Relationship with Site Experience

Each time cracking was observed on the roadway [7.18], the values of the R&B softening point, the asphaltenes content, the temperature for a phase angle at a given frequency or the temperature for which the slope "*m*" in BBR test reaches the value of 0,3 (all after laboratory ageing) exceed a critical point. All the other parameters did not present any singularity.

7.5.4 Durability

No relevant papers found.

7.5.5 Overview of Binder Technological Tests as Descriptor for Fatigue Cracking

The field correlations between fatigue cracking and the standard technological characteristics of the bitumens remain very significant, allowing quick evaluation of the quality of the unmodified bitumen.

7.6 Recommendations for Fatigue Cracking

Further research is needed for a more explicit correlation between the fatigue behaviour of asphalt mixtures and some characteristics of binders. The fatigue of bitumens and typical parameters of complex modulus seem to be the best properties in relation to the behaviour with the fatigue of the asphalt mixture. While awaiting the results from such research, the basic characteristics (such as penetration, softening point or viscosity) and some rheological characteristics, both before and after ageing (such as by RCAT or RTFOT and PAV), remain the best criteria to assess the fatigue behaviour of asphalt.

In particular, the relationship between bitumen fatigue and mixture fatigue at the number of cycles to achieve a 50 % reduction in G^* looks particularly promising and it is recommended that this work is continued to include a range of PMBs and other mixture types. The research should involve two aspects:

- Validation with field performance is critical and there is a lack of information relating laboratory fatigue behaviour with performance in practice. More effort is required in this area, particularly in relating the in-situ bitumen properties with fatigue cracking.
- PMBs cover a wide range of polymer types and polymer contents which give them widely differing properties. If further studies are intended to be carried out on asphalt mixtures containing PMBs, it is recommended that a range of the different families of PMBs are examined to ensure that specific conclusions can be attributed to the different families of materials.

The force ductility test shows some promise, but the relationship appears to be dependent on the type of modifier which makes it of limited use. Furthermore, the research is limited, currently being restricted to a single paper.

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8. Adhesion

8.1 Asphalt Tests

It is generally accepted that adhesion is the property which characterises the bond between two materials. In the case of asphalt mixtures, it reflects the interaction of the binder or bitumen and the aggregate material. Adhesion is an interface phenomenon because it occurs at the surface of materials (e.g. binder, aggregates and fillers). The term adhesivity is the ability or capacity of a material such as bitumen to form a bond that results in adhesion.

While the term adhesion (also referred to, less precisely, as affinity or compatibility) is wide spread, its direct quantitative measure is less evident. Often, it is far easier to define the lack of adhesion because that phenomenon is directly correlated with failure or *in situ* damage (e.g. ravelling). Moreover, water has a detrimental effect on adhesion. Therefore, the susceptibility to moisture or the resistance towards debonding is considered to be a good indirect indicator of the power of a binder to adhere to aggregates. In this context, it is frequently referred to as a stripping phenomenon. This idea has already been taken up in the standards EN 12697-11 [8.01] and EN 13697-12 [8.02] dealing with the assessment of the adhesion of asphalt mixtures. Frequently in such procedures, a ratio (expressed in per cent) of a mechanical property (e.g. strength or modulus) of an asphalt mixture is calculated from a series of measurements conducted before (dry specimens) and after water conditioning.

Although, adhesion is defined as the bond between only two materials, the situation is more complex in the case of composite materials such as asphalt mixtures. There are often difficulties in the interpretation of test results or failure mechanisms because the separation of adhesion characteristics within the asphalt mixture is hampered by other binder or mixture properties such as cohesion and the mix design. As a consequence, researchers have developed test procedures that focus both on a single binder/aggregate pair as well as compacted asphalt specimens. However, because adhesion is an interface event, generally all test procedures include both the binder and the aggregate.

Stripping is known to be a demonstration of interfacial tension between bitumen and aggregate in the presence of water and could, therefore, be explained and estimated by thermodynamic theories. Several research studies report the measurement and use of such thermodynamic characteristics (e.g. surface tension) to develop quantitative models describing interface phenomena such as adhesion or stripping (the lack of adhesion) [8.03, 8.04]. Such studies require a more fundamental insight into material characteristics governing adhesion.

The development of tests to determine adhesion began in the 1930s. Since then, numerous tests have been developed in an attempt to identify and quantify adhesion properties. The moisture susceptibility tests can be divided in two major categories:

- Test conducted on loose coated aggregate (e.g. rolling bottle, boiling test and immersion test).
- Test conducted on compacted mixtures (e.g. Cantabro, Indirect Tensile Strength (ITS) and abrasion tests).

The first family of tests makes use of simpler equipment and is less time consuming to perform. However, the results are often qualitative and more difficult to interpret in relationship with the adhesion phenomenon. The second family of tests, when conducted with conditioning, is considered as an indirect measure for adhesion. They take into consideration the traffic, environment and mixture properties but need more elaborate testing equipment, longer testing time and/or laborious test procedures. Specific test methods to evaluate the role of the mastic (mixture of filler and bitumen) on the adhesion properties of a mixture are currently not available.

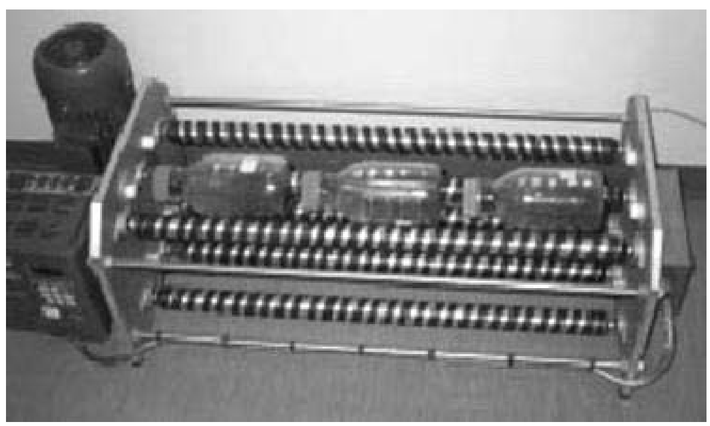
The following overview is based on the test methods available in the BiTVal database which are described or utilised to access adhesion in particular or, more generally, to investigate durability aspects of asphalt mixtures. This summary is not comprehensive because of the limited number of papers (about 30) which correlated with adhesion (see also the conclusions in Section 8.13.3).

8.2 Rolling Bottle Test

8.2.1 Description

The rolling bottle (flask) method (EN 12697-11 revision, part A [8.01]) is used to determine the affinity between single size aggregate and bitumen. In the test, 510 g of aggregate particles (8/11 mm fraction sieved in accordance with EN 12697-2) are coated with 16 g of bitumen (film thickness ± 0.1 mm). The coated aggregate is then placed in a 500 ml flask containing deionised water and a glass rod and rotated at 60 cycles/min for 6 h at ambient temperature (15 - 25°C) (Figure 8.1). The residual degree of bitumen coverage of the particles is estimated visually. For each rolling time, the average degree of bitumen coverage (mean of the two operators' results) is calculated and rounded to the nearest 5 %.

Test bottle



Bottle rolling machine

Figure 8.1 – Rolling bottle test equipment

Originally, the volume of the test bottles were 250 ml, rather than 500 ml, and most of the experience has been obtained with the smaller bottles.

8.2.2 Equivalent Standardised Tests

None found.

8.2.3 Precision

The precision of this test has not yet been established officially. However, the following data are estimated from normal practice:

Repeatability: r = 20 %

Reproducibility: R = 30 %

8.2.4 Relationship with Other Tests

None found.

8.2.5 Experience with Test

Two papers in the BiTVal database report the use of the rolling bottle method for evaluating the influence of adhesion promoters on the adhesion properties of asphalt mixtures. The benefit of a modifier called 'road bitumen stabiliser' (rbs) was evaluated [8.05] in comparison with other additives such as hydrated lime or amines. A series of adhesion promoters were tested [8.06] with a wide range of bitumens (including PMB) and aggregates, referring to the existing methodology as stated in specification DIN 1996, part 10 [8.07].

8.2.6 Relationship with Site Experience

No relationship with site experiences reported.

8.2.7 Durability

No data available.

8.2.8 Overview of Rolling Bottle Test as Descriptor for Adhesion

Generally, the test is simple and easy to perform. Due to the visual evaluation, the test result is rather subjective and, therefore, the precision is limited and it is generally used as a screening technique.

8.3 Boiling Water Stripping Method

8.3.1 Description

The boiling water stripping method (EN 12697-11 revision, part C [8.01]) is used to determine the affinity between any mineral aggregate/bitumen combinations in which the mineral is calcareous, silico-calcareous or siliceous by nature by a more objective means (assessment by acid/base titration) than the rolling bottle method (Section 8.2). The procedure involves the coating of 1.5 kg of aggregate (10 to 14 mm fraction) with 1.8 % by mass of binder. Mixture of uncoated (= bare) aggregate and coated aggregate are then produced and subjected to chemical attack to produce a calibration curve of acid consumption against the proportion of uncoated aggregate. In a next step, 200 g of the coated aggregate is boiled in 600 ml of demineralised water for 10 min, allowed to dry and subjected to an acid attack in order to determine, while using the calibration curve (Figure 8.2), the extent of stripping.

8.3.2 Equivalent Standardised Tests

An equivalent test method exists in the USA and is described in ASTM D 3625 [8.08].

8.3.3 Precision

A repeatability coefficient of variation of 15 % of the determined value has been found, with an absolute threshold of the stripping percentage of 2 %.

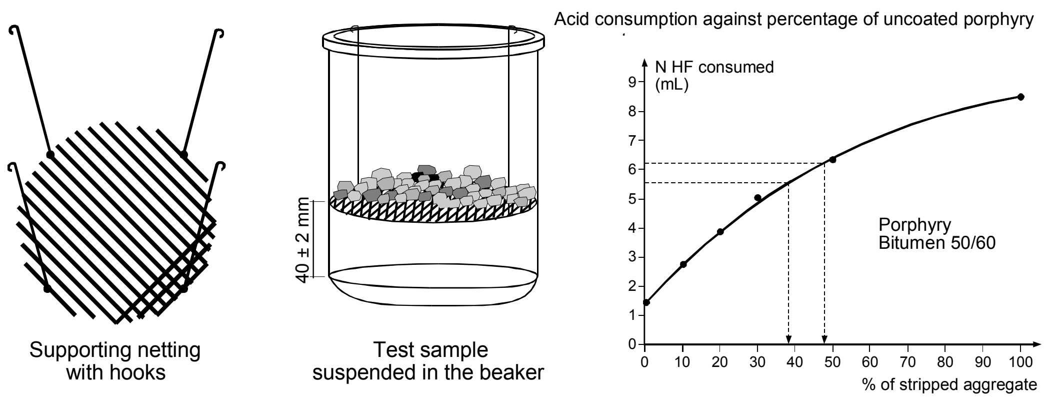


Figure 8.2 – Experimental set up and Example of a calibration curve

8.3.4 Relationship with Other Tests

None found.

8.3.5 Experience with Test

Based on extensive experience [8.09], the boiling water stripping method (EN 12697-11 revision, part C [8.01]) permits the definition of three performance ranges of bitumen/aggregate combinations with respect to resistance to stripping. The ranges are < 15 % (good to excellent), 15 – 30 % (fair) and > 30 % (critical).

Currently, no papers available in the BiTVal database include the above European test method. However, the idea of an accelerated stripping test is taken up by two research groups [8.10, 8.11]. Some data (including only 2 aggregates and 2 binders) was obtained from several stripping test differing in the experimental test conditions (temperature, time, etc) [8.10]. However, additional data was found to be required in order to establish the validity of the proposed test (which is already very similar to the proposed revision to EN 12697-11) as a descriptor for adhesion.

A detailed discussion of a similar method [8.11], which includes both the bitumen absorption and the stripping after several time intervals, gives measurements from which a coating value or index is calculated. The reported coating values allow a ranking as a function of the bitumen evaluated and permits an identification of aggregate/bitumen combinations that are diagnosed as critical for adhesion. Three performance ranges are proposed, these being < 25 % (critical), 25 % - 45 % (suspected) and > 45 % (performing well).

8.3.6 Relationship with Site Experience

No data available.

8.3.7 Durability

No data available.

8.3.8 Overview of Boiling Water Stripping Method as Descriptor for Adhesion

The test is the most objective of the binder/aggregate affinity tests, but the use of hydrochloric acid with calcareous aggregates and hydrofluoric acid with silico-calcareous or siliceous aggregates as the reagent presents health and safety concerns.

8.4 Ultrasonic Method

8.4.1 Description

An ultrasonic method has been developed [8.12] to measure the resistance to stripping of coated aggregates. In the test, polished stone specimens (thickness about 10 mm, diameter 45 mm) are coated with 0,2 g bitumen to give a binder thickness of 0,12 mm. Subsequently, the test piece is subjected to the ultrasonic action of water which strips the bitumen mechanically from the polished stone. The degree of stripping is evaluated by either weighing or by visual assessment (Figure 8.3).

Figure 8.3 – Test pieces after ultrasonic handling subjected to visual evaluation

8.4.2 Equivalent Standardised Tests

None found.

8.4.3 Precision

The precision of the test has not yet been established. Nevertheless, standard errors varying from 0,7 to 32,1 % was reported. Authors also indicate that the repeatability of the test needs to be improved.

8.4.4 Relationship with Other Tests

None found.

8.4.5 Experience with Test

Preliminary test results have been reported [8.11] that show the importance of the experimental set up in terms of the soaking and stripping times, temperature and fabrication of the test pieces. Although the test procedure is rather simple other than requiring polished pieces of the aggregate to be tested, the test procedure appears to be fairly sensitive to even small changes in the experimental set up. As a consequence, both the repeatability and the reproducibility of the test are unsatisfactory. The range of values reported is large, from almost no stripping (< 2 %) up to 80 %.

Only one paper from the database deals with this method with no follow up article by the same research group having been identified to date. Nevertheless, the idea has recently (TRB 2005) been picked up [8.13]. They report on the use of an Ultrasonic Accelerated Moisture Conditioning procedure (UAMC) as a one day test for the evaluation of moisture sensitivity of HMA mixtures. A good repeatability for the test (including the continuously measurement of the weight loss over time) was demonstrated. Moreover, a significant relationship exists between the slope of the regression function as determined in the UAMC test and the tensile strength ratio after one cycle of freeze-thaw conditioning.

8.4.6 Relationship with Site Experience

The reported study is rather preliminary and deals mainly with the experimental set up. No validation with in situ experience has been made yet.

8.4.7 Durability

No data available on this topic.

8.4.8 Overview of Ultrasonic Method as Descriptor for Adhesion

The test has good precision but is sensitive to the experimental set up. The limited number of papers reviewed makes it difficult to assess its potential.

8.5 Net Adsorption Test

8.5.1 Description

The Net Adsorption Test (SHRP Designation M-001, AASHTO designation PP5) was developed as a screening procedure for selecting bitumen and aggregates [8.14] as well as determining the effectiveness of anti-stripping agents [8.15]. The test is based on measurements of adsorption isotherms (Langmuir equations) of the amount of solute (bitumen) adsorbed from a toluene solution[1] onto aggregates. The test determines the affinity between bitumen and aggregate by measuring the initial adsorption (by UV-spectro-photometry at 410 nm) and the moisture sensitivity of the bond following the addition of water into the system. Values of the initial amount of binder being adsorbed are expressed in per cent. In a modified test procedure, it has been suggested [8.16] that the data should be re-evaluated by expressing the initial and net adsorption as a proportion of the total bitumen in the solution.

The test consists of adding 140 ml of a bitumen solution in toluene to 50 g of 0/4,75 mm aggregate. The mixture is shaken for 6 h, at which the bitumen concentration is measured in order to determine the amount of binder adsorbed. Subsequently, 2 ml of water is added and the system is shaken for another 8 h. The bitumen desorbed is assessed by the appropriate UV-absorption. The bitumen which remains on the aggregate is termed the net absorption.

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[1] Toluene is a solvent which should only be used with extreme care.

8.5.2 Equivalent Standardised Tests

None found.

8.5.3 Precision

The standard deviation of the modified procedure (which also included a more specified aggregate grading) is 1,7 % and 1,8 % for the initial and net adsorption, respectively [8.17].

8.5.4 Relationship with Other Tests

See Section 8.7.4.

8.5.5 Experience with Test

The method should give an early indication in terms of likelihood of in-service failure but it has been suggested that further work is needed in order to provide information about predicting the likely in-service performance [8.16]. Other work focusing on the interaction of fillers and binders using the Net Adsorption Test [8.18] has found that more research before using the test while selecting a 'durable' combination resistant to stripping.

The results of the Net Adsorption Test have been used by SHRP to define the following recommendations for aggregate/bitumen performance (Table 8.1).

8.5.6 Relationship with Site Experience

No validation with in field experience has been noted.

Table 8.1 – SHRP recommendations for aggregate/bitumen adhesion

% Net adsorption (SHRP value)	Aggregate/binder adhesive performance
> 70	Good
55 – 70	Marginal
< 55	Poor

8.5.7 Durability

No data available.

8.5.8 Overview of the Net Adsorption Test as Descriptor for Adhesion

The method should give an early indication in terms of likelihood of in-service failure but further research is needed in order to validate this assumption. The limited number of papers reviewed also makes it difficult to assess its potential.

8.6 Vialit Plate Test

8.6.1 Description

The Vialit plate test is a resistance test to wrenching by shocks (Figure 8.4) [8.19] in order to estimate a measure of the adhesion of an aggregate at a bitumen film. This test is specific to surface dressing. In the test, aggregate particles are pressed onto a tray of bitumen. The tray is turned upside down and a steel ball is dropped onto the reverse side. The impact of the ball may cause detachment of the aggregate particles depending on the test conditions (e.g. temperature, following ageing, wetting). The number of detached aggregate particles together with the increasing number of impacts may be used as an indicator of performance. Visual inspection of the detached aggregate can usually determine the type of failure in terms of whether it is cohesive or adhesive.

8.6.2 Equivalent Standardised Tests

None found.

8.6.3 Precision

The repeatability of the test has been estimated [8.19] at 15 % for traditional mixtures. Since 1963, there has been no other data published on the repeatability. However, the experience of experts generally confirms these results today.

8.6.4 Relationship with Other Tests

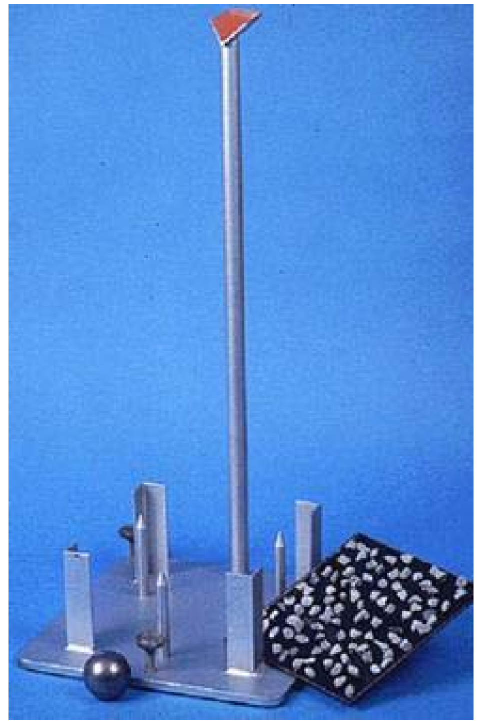

Figure 8.4 – Vialit plate apparatus

The Vialit plate shock test has been used for the study of the adhesive properties of binders in a series of asphalt mixtures designed for heavy duty pavements [8.20]. No relationship with other test results is reported and only a ranking of the evaluated modified binders is provided. This ranking is expressed as a percentage of improvement as compared with a reference unmodified bitumen (70/100 pen).

8.6.5 Experience with Test

The plate shock test has been usual practice in France since the 1970s for surface dressing [8.20]. A study [8.21] with 8 anhydrous binders (paving grade bitumen and PMBs) and three types of aggregate (flint, quartzite, and microdiorite) highlights the influence of the temperature of the support at the time of gritting (Figure 8.5) and the influence of the purity of the aggregates.

8.6.6 Relationship with Site Experience

To test the presence of dust, the Vialit test is practised with dry and not washed materials. A result higher than 80 % indicates the presence of dust is not a problem whereas a value lower than 80 % implies that there may be a need to treat the aggregates [8.22].

Practised in a wet atmosphere on un-washed and wet aggregates, this test gives information about the active adhesiveness. If the result is higher than 90 %, the active adhesiveness is satisfactory whilst the binder-aggregate combination is rejected in the contrary case. In this case, the addition of a compound that enhances adhesiveness is necessary in order to reach the value threshold of 90 %.

A new monitoring programme on the performance of the pavement was due to be followed up to at least 2004 [8.20]. However, no subsequent information from this trial has been found.

Quartzite 10/14

@ 20 ºC @ 5 ºC

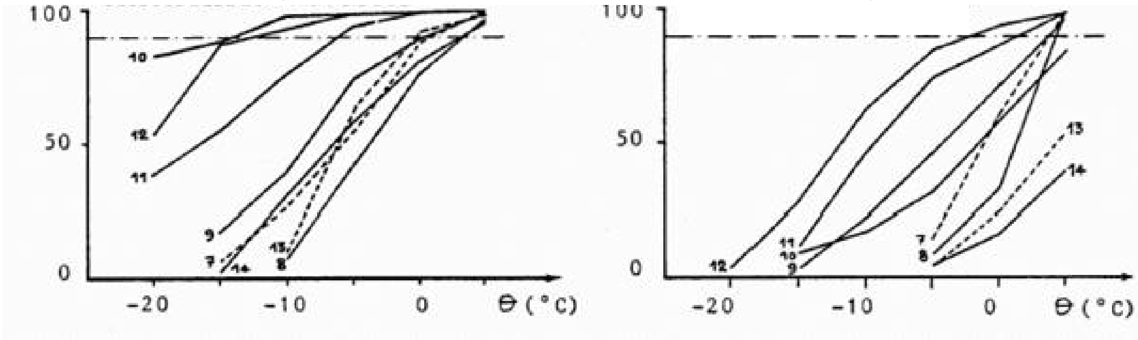

Figure 8.5 – Proportion of aggregate particles remaining on the plate depending on the temperature of manufacture and the temperature of the test
(Note: Binders 7 and 13 are paving grade, the remainder are polymer modified)

8.6.7 Durability

No data available.

8.6.8 Overview of Vialit Plate Test as Descriptor for Adhesion

This test with the Vialit plate tries to assess the influence of the purity of the aggregates and the action of the presence of water and does not claim to address all the causes of lack of adhesion of the binder to the aggregate. Therefore, the usefulness of the test for assessing the adhesive properties of binders can be questioned. Moreover, it is far from straightforward to explain the test results in terms of adhesion. Other failure mechanisms, such as cohesion and/or ductility, do play a role.

8.7 Cantabro Test

8.7.1 Description

In the Cantabro test [8.23], a compacted specimen is put in a Los Angeles drum without any steel balls and given 300 rotations with a speed of 30 rpm. The specimens are weighed before and after the rotations and the weight loss is calculated (Cantabro loss value). The test result strongly depends on the temperature.

8.7.2 Equivalent Standardised Tests

None found.

8.7.3 Precision

The following precision data have been established for tests carried out at 25°C:

Weight loss < 40 %: repeatability r = 2 % and reproducibility R = 4 %

Weight loss ≥ 40 %: repeatability r = 5 % and reproducibility R = 8 %

8.7.4 Relationship with Other Tests

An attempt was made to correlate the results of the Cantabro test with the Net adsorption test [8.16]. However, the ranking varied as a function of the number of rotations, with the best correlation being at 150 rotations.

Other test results were only performed to assess cohesive properties of mixtures (varying in bitumen source) at low temperature (10 °C) [8.20]. However, only a ranking of the proportional improvement relative to a reference mixture is given.

8.7.5 Experience with Test

The Cantabrian Test was initially developed at the University of Cantabria, Spain to determine the degree of cohesion in open cold mixtures. In order to access adhesion rather than cohesion, the fine material has been excluded from the test mixture (UCL-method) [8.24, 8.25]. Samples can also be tested after exposure to water (the retained Cantabro test).

The Cantabro method is a test, specific to porous asphalt and discontinuous open-grade hot mixtures for thin surface layers, for evaluating the resistance to particle loss by abrasion and the effect of impact. Therefore, the Cantabro loss is used as an indicator for bonding properties between bitumen and aggregate. In Belgium, specifications for porous asphalt allow a maximum weight loss of 15 % and 20 % for PMB and unmodified bitumen, respectively, with the Cantabro test being carried out at 18 °C.

Spanish specifications state (tests carried out at 25 ± 1°C), as part of the mix design criteria, the following limits:

- Porous mixtures:
  - Maximum 20 % (dry specimens) and 35 % wet/dry loss ratio with heavy traffic.
  - Maximum 25 % (dry specimens) and 40 % wet/dry loss ratio with lighter traffic.
- Discontinuous open-grade hot mixes for thin surface layers:
  - Maximum 15 % (dry specimens) and 35 % wet/dry loss ratio with heavy traffic.
  - Maximum 15 % (dry specimens) and 40 % wet/dry loss ratio with lighter traffic.

The method is also known to be used commonly in Japan to evaluate the particle loss resistance of porous asphalt under winter conditions.

The repeatability has been assessed when the test was used for the determination of the cohesive strength of mixtures [8.26]. For loss values in the range < 35 % to 40 %, a variation of < 2 % was found at a confidence level of 95 % whilst, for an elevated loss value in the range of 60 % to 90 %, a variation > 5 % was found. No corresponding data is given for the retained Cantabro test when assessing adhesive properties.

8.7.6 Relationship with Site Experience

A monitoring programme of the performance of a pavement was to be followed to at least 2004 [8.20]. However, the corresponding data could not be identified up to now.

8.7.7 Durability

Test results have been obtained following the ageing of specimens at 163 °C for different times [8.26]. The drop in penetration of the binder (0,1 mm) was found to correlate with the loss in value for the Cantabro test at 25 °C.

8.7.8 Overview of Cantabro Test as Descriptor for Adhesion

Only three papers referring to this test method have been identified in the BiTVal database. Therefore, only a limited amount of data is available. Nevertheless, there is a belief that the test results could predict the in situ performance.

The fact that a single test method (with some modifications in the test procedure) such as the Cantabro abrasion test has been used for both assessing cohesive and adhesive properties of a compacted asphalt mixture indicates the difficulty in isolating one property (such as adhesion) when interpreting the test results. Therefore, such an exercise is often tentative. However, both phenomena (i.e. adhesive and cohesive failures) can occur simultaneously in practice.

8.8 Wheel fretting Test (WFT)

8.8.1 Description

A treated tyre, inflated at 6 bar and with a load of 3000 N, is run on a circular band consisting of asphalt test specimens under an inclination angle of 2 to 5° with respect to the horizontal plane of rotation, thus inducing fretting (Figure 8.6). The axis of the load lies in the horizontal plane. Up to 3 million revolutions are made (about 1 month period). During the test, the temperature on the surface of ± 20 °C is maintained. The specimens are dried and weighed before and after testing. The fretting performance is expressed as the mass loss after a given number of wheel passages.

Figure 8.6 – The wheel fretting test

8.8.2 Equivalent Standardised Tests

None found.

8.8.3 Precision

The use of the WFT has been evaluated while investigating the resistance to ravelling (short term) of porous asphalt (including both modified and unmodified binders) [8.27]. The reported standard deviations varied (N = 2) between 12 % and 43 %, increasing with mass loss.

8.8.4 Relationship with Other Tests

The mass loss in the WFT at 20 °C seems to be related to the penetration at the test temperature. For low temperatures (4 °C), the mass loss increases with decreasing penetration. For high temperatures (20 °C), the mass loss increases with increasing penetration.

The mass loss in the WFT at 20 °C and the Californian Abrasion Test (CAT) (Figure 8.7) at 4 °C behave qualitatively the same as a function of the computed penetration for the test temperature, passing through a minimum.

8.8.5 Experience with Test

A single paper referring to this abrasion test was found [8.27]. The test was only utilised to study the performance in terms of resistance to ravelling of porous asphalt produced with PMB in comparison those produced with unmodified binders. The direct relationship between the test results and the adhesive properties of the binder is unclear, making it difficult to elaborate on this issue based on data originating form such abrasion test methods.

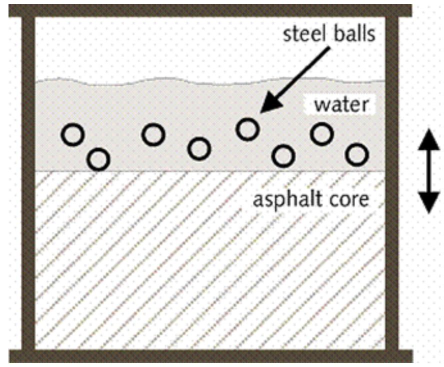

Figure 8.7 – Schematic representation of the CAT (mass loss is the performance indicator)

8.8.6 Relationship with Site Experience

No data available.

8.8.7 Durability

No data stated.

8.8.8 Overview of the Wheel Fretting Test as Descriptor for Adhesion

The single paper that refers to this test does not allow any conclusions to be drawn with regard to its suitability. Furthermore, the test requires specialist equipment not generally and is both time consuming and costly.

8.9 Indirect Tensile Strength Test

8.9.1 Description

Figure 8.8 – ITS apparatus

The most commonly used method to determine the water sensitivity of a compacted asphalt mixture includes the measurement of the Indirect Tensile Strength (ITS) before and after conditioning in water (Figure 8.8 and 8.9). It is probably the most typical example of a test where the samples are conditioned in water to simulate the in-service conditions and assessment of the moisture damage is made by the ratio of conditioned to unconditioned strength (ITSR) or stiffness (modulus can be assessed simultaneously by measuring the displacement in the plane perpendicular to the applied stress, as shown in Figure 8.10). The principle of the test is described in EN 12697-12 [8.28].

The procedure for the ITS test is given in EN 12697-23 [8.29]. It includes the measurement of the maximum tensile stress applied to a cylindrical specimen loaded diametrically until break at the specified test temperature (25 °C is recommended in combination with EN 12697-12 [8.02]) and speed of displacement of the compression testing machine. The cylindrical specimen is either a laboratory-made (e.g. gyratory or impact compacted) moulded specimen or core taken from a bituminous layer or slab.

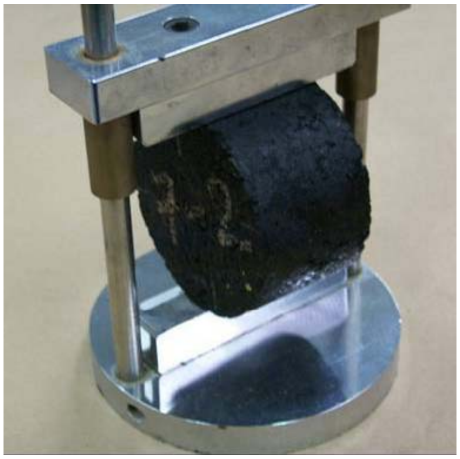

Figure 8.9 – Detail of the appropriate test accessories

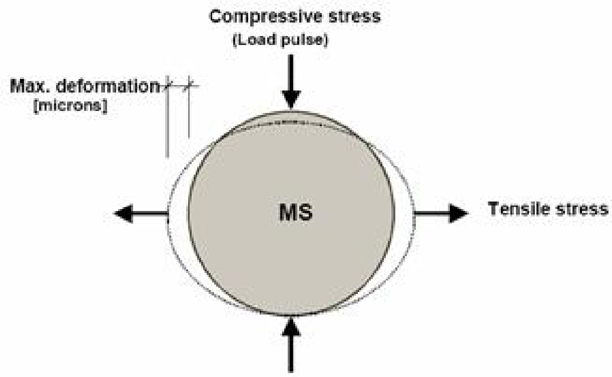

Figure 8.10 – Schematic representation of a dynamic indirect tensile test

8.9.2 Equivalent Standardised Tests

A series of similar test procedures which differ only in the experimental part (saturation levels, temperature, time of water conditioning, etc.) of the methodology is wide spread. Typical examples include AASHTO T283 (Modified Lottman ITS test procedure) [8.30], the Swedish FAS method 449-98 [8.31] and ASTM D 4867 [8.32]. For a more complete overview of water sensitivity tests on compacted asphalt mixtures, see Section 8.11.

8.9.3 Precision

In EN 12697-12 [8.02], the following precision data are estimated based on an experiment carried out in the USA:

Repeatability, *r*, is approximately 15 %
Reproducibility, *R*, is approximately 23 %

It is expected that the precision of the test method will improve when a test temperature of 25 °C is generally used and experience in the laboratory is obtained.

8.9.4 Relationship with Other Tests

None found.

8.9.5 Experience with Test

The influence of the experimental conditions in relation to the significance or discriminating power of the test results has been examined [8.33, 8.35]. The test temperature (25 °C) has been criticised as being too high [8.33] because of the great impact of the temperature on the type of fracture induced during the ITS-test. It has also been suggested [8.35] that a conditioning temperature of 60 °C leads in an additional step to more significant differences in ITS test results. However, the conditioning of foam bitumen samples at this temperature is believed to have over-cured the binder [8.34].

A critical value of 30 % for the loss of resilient modulus is proposed [8.10] when evaluating the adhesive strength of asphalt mixtures (including both normal and modified binders), although no field validation was mentioned.

It has been proposed [8.13] that the measurement of the resilient modulus is the best test procedure to evaluate the moisture sensitivity of a mixture when the proportional difference in strength between the unconditioned and conditioned specimens is small. However, if this difference exceeds 20 %, the evaluation using the tensile strength is preferred.

A more fundamental assessment on the testing of moisture sensitivity of asphalt mixtures [8.36] draws attention to the water damage in mixtures can be complicated by aggregate structure and type. Hence, each mixture property for each mixture is affected differently and to different degrees by water damage. The evaluation of water damage needs to take account of these different effects in a consistent manner. Therefore, the use of a single parameter to describe moisture damage must be questioned.

8.9.6 Relationship with Site Experience

An evaluation of ITSR results have been validated [8.37] utilising the Westrack set up (accelerated pavement test facility) to monitor the performance of a whole series of asphalt mixtures (which included none with modified bitumen but all mixtures were treated with hydrated lime). The moisture sensitivity test results for the Westrack mixtures were frequently below the recommended ITSR of 0,80 (17 out of 34 mixtures). However, after nearly 4,9 million ESALs, moisture damage was not evident. While it might be argued that the test was relatively short term, Nevada DOT experience with lime treatment on similar materials suggested otherwise. Therefore, the criteria for the ITSR or the test itself was considered suspect although the Westrack set up is located near Reno, Nevada, where the climatic conditions are not representative with regard to the possible impact of rainfall.

The adhesive properties of AC mixtures with modified bitumens and the addition of an adhesion promoter have been evaluated using the ITS test [8.38]. No significant influence after water conditioning was measured although samples were characterised by a 7 % void content. The suitability of the method was questioned but this study did deal with surface courses for airfields already containing either an adhesion promoter or polymer modified binders. The ITS tests were undertaken at 10°C.

The objective of an extensive moisture damage study [8.39] was to evaluate the relationship between pavement performance in the field and ITSR values measured in the laboratory on the original asphalt mixtures. This study, concluded in 1999, raised specific questions about the meaning of the ITSR results because of repeatability problems and the lack of clear evidence of a relationship between the ITSR values and moisture damage in the field. The pavement condition information was determined a performance distress index. The conclusion was that the current ITSR protocol could not be used as a quantifiable measure of moisture damage effects on pavement performance.

The effectiveness of several tests methods yielding ITSR values was quantified on the basis of test data from various researchers [8.40]. The success/failure ratings presented in Table 8.2 are base on comparing the laboratory predictions with the field performance ratings. A higher proportion of success implies a larger number of ‘correct’ predictions.

The field experiences from four US states (California, Nevada, Texas and Virginia) regarding their history with moisture sensitivity of asphalt mixtures in relationship with performance prediction and the tools to identify any problems correlated to moisture damage have been compiled [8.41]. The experience in all the states indicated the need of an improved laboratory test or criterion to identify moisture sensitive HMA mixes (until 2003, the AASHTO T283 procedure [8.30] had been applied). All cited exceptions to, or lack of, a correlation between the wet-dry TSR requirement and field performance. Moreover, larger states are not satisfied with the repeatability and reproducibility of the test method. As a consequence, Texas abandoned the wet-dry TSR as the criterion for identifying moisture sensitivity of HMA mixtures in 2003.

Table 8.2 – Success rates of test methods [8.40]

Test method	Minimum Test Criterion	Success
Modified Lotmann (AASHTO T283)	TSR = 70 % TSR = 80 %	67 % 76 %
Tunnicliff-Root (ASTM D4867)	TSR = 70 % TSR = 80 %	60 % 67 %
10 min boil test	Retained coating 85 % – 90 %	58 %
Immersion-compression (AASHTO T165)	Retained strength 75 %	47 %

8.9.7 Durability

No data available.

8.9.8 Overview of ITS Test as Descriptor for Adhesion

A total of ten papers report test results obtained by ITST. The temperature of the test is questioned but it has been proposed as the best test for evaluating the adhesive strength and the moisture sensitivity of asphalt mixtures, although the latter only when the difference between the results from unconditioned and conditioned specimens is small. There are considerable data on the relationship with site performance but they show limited correlation. Furthermore, the precision is not particularly good. Therefore, the indirect tensile stiffness test cannot be seen as a good descriptor for the adhesion properties of the binder.

8.10 Pneumatic Adhesion Tensile Testing Instrument (PATTI)

8.10.1 Description

The pneumatic adhesion tensile testing instrument (PATTI) was initially developed [8.42] for evaluating the pull-off strength of a coating on rigid substrates such as metal, concrete or wood. The procedure has been used to evaluate the adhesive loss of binder-aggregate systems exposed to water [8.43, 8.44]. The PATTI device and a drawing of its cross-section are shown in Figures 8.11 and 8.12.

To perform a test, air pressure is transmitted to the piston which is placed over the pull stub and screwed on the reaction plate. The air pressure induces an airtight seal formed between the piston gasket and the aggregate surface. When the pressure in the piston exceeds the cohesive strength of the binder or the adhesive strength of the binder-aggregate interface, a failure occurs. The pressure at failure is recorded and converted into the pull-off tensile strength (kPa) at 25 °C.

Figure 8.11 – PATTI 110 device

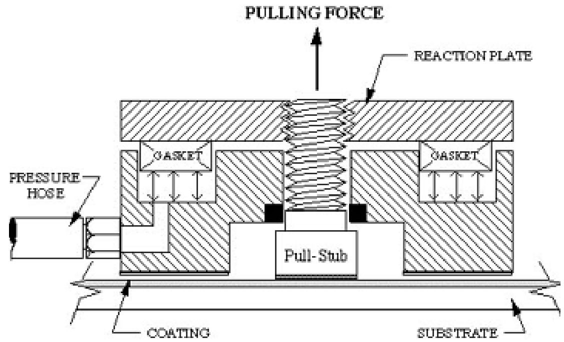

Figure 8.12 – Schematic cross-section of the PATTI device

Although a glass plate was initially used as the substrate, the test method was extended to aggregate surfaces. Moreover, specimens can be conditioned in water (up to 48 h) and the failure surface can be examined in order to define adhesive versus cohesive failure (Figure 8.13).

Figure 8.13 – Type of failure from the PATTI-test
(left: cohesive failure, right: adhesive failure)

8.10.2 Equivalent Standardised Tests

None found.

8.10.3 Precision

Based on statistical analysis [8.43], a coefficient of variation (Cv) of 15 % or less has been reported in most cases (average value of 10,5 % for unaged binders). Approximately 55 % of all tests conducted indicate a Cv value of ≤ 10 % while 10 % of the test revealed a Cv value of ≥ 20 %. The tests were undertaken using glass substrates. It has been found [8.45] that repeatability could be significantly improved by including a temperature controlling system and a standard methodology for detecting the type of failure of the specimens.

8.10.4 Relationship with Other Tests

An attempt has been made to correlate some of the results obtained by the PATTI test method with data acquired by performing the indirect tensile strength test (ASTM D4867 [8.32]) following conditioning in water [8.46]. However, the results of the correlation are based on a simplistic multi-linear model of tensile strength values which comprises both binder cohesive properties as well as the pull-off strength under water-exposed condition (PATTI plus conditioning).

8.10.5 Experience with Test

The PATTI test, including the conditioning of specimens, has been found to enable a ranking of binders with respect to their moisture susceptibility [8.43, 8.44]. However, attention needs to be paid to examining the entire soak-time/pull-off strength curve while evaluating binder properties.

8.10.6 Relationship with Site Experience

No relationship with site experiences reported.

8.10.7 Durability

No data available.

8.10.8 Overview of PATTI Test as Descriptor for Adhesion

All data published so far indicate that the mode of failure changes with the water conditioning. Unconditioned specimens are all characterised by a cohesive failure whereas an adhesive failure mechanism is observed after soaking in water. Consequently, the interpretation of the test results in terms of adhesion only is not that straightforward.

Little attention is given to the preparation and characterization of substrates other that glass. Questions concerning the surface chemistry or roughness of substrates remain unanswered.

8.11 Saturation Ageing Tensile Stiffness (SATS) Test

8.11.1 Description

A set of five cylindrical samples are partially saturated in a vacuum desiccator with a residual pressure of (68 ± 3) kPa for 30 min. The surface dried samples are then stored at a temperature of (85 ± 1) °C in a pressure vessel (Figure 8.14) with water to a level between the second and third trays from the bottom (Figure 8.15), as shown in Figure 8.16. At the time of writing, a variant of the test is about to be tried in which the water is raised to between the third and fourth tray (i.e. two samples under water and three above rather than one sample underwater and four above).

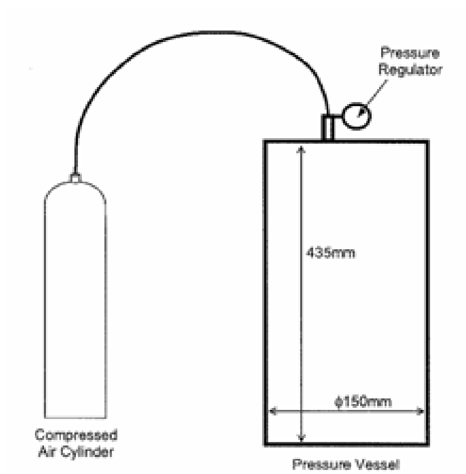

Figure 8.14 – Schematic & dimensions of typical pressure vessel

The pressure is gradually increased from atmospheric pressure to 2,1 MPa over a period of 20 min. The conditioning at a pressure of (2,1 ± 0,1) MPa and temperature of (85 ± 1) °C is maintained for (65 ± 1) h. The temperature is then allowed to cool to 30 °C over 24 h without releasing the pressure. (Much of the early research was done without this step, with damage resulting to some otherwise sound samples from the release of pressure at elevated temperature.) Each sample is surface

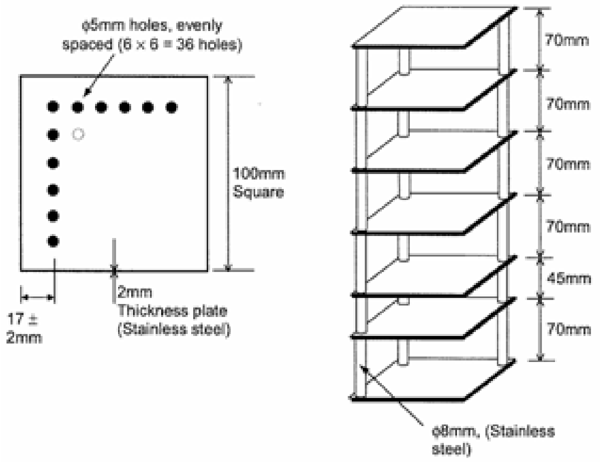

Figure 8.15 – Schematic and dimensions of typical specimen tray

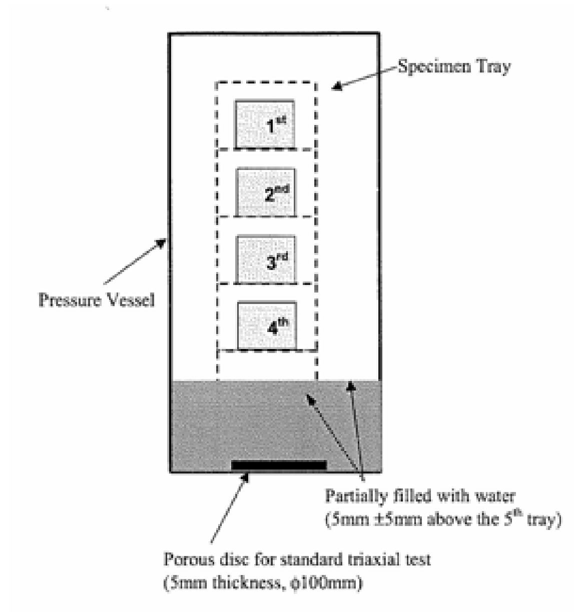

Figure 8.16 – Schematic diagram of the core configuration in the pressure vessel

dried and its wet mass measured within three minutes of removing the cores from the pressure vessel.

The percentage saturation and the indirect tensile stiffness modulus (ITSM) of the samples are determined both before and after conditioning, with the latter being used to determine the stiffness ratio. The stiffness ratio is considered alongside the degree of saturation for assessment purposes, although there are no set levels as yet, the results having to be reported for information at present.

8.11.2 Equivalent Standardised Tests

The test is given as clause 953 of the UK Specification for Highway Works [8.47].

8.11.3 Precision

No precision has been established as yet.

8.11.4 Relationship with Other Tests

The SATS conditioning regime was developed by comparing six durability regimes [8.48 to 8.51]. These regimes were:

- Specimens stored at 10 ºC with no accelerated ageing.
- Dry oven ageing at 85 ºC for 120 h.
- Partially saturated oven ageing at 85 ºC for 120 h (water bath in oven).
- Low pressure oxidation at 3 l/min and 85 ºC for 120 h.
- Pressure aged at 2,1 MPa and 85 ºC for 65 h.
- Partially saturated pressure aged at 2,1 MPa and 85 ºC for 65 h (SATS).

The regimes behaved similarly with an increase in ITSM after initial ageing followed by a reduction, presumably with loss of adhesion, but the last regime produced the greatest reduction of 60 %.

The recovered binder from the SATS conditioning have been found to have similar rheological properties as binder that has been aged by the PAV85 (HiPAT) regime (Section 3.5) for binders [8.48 to 8.51].

The SATS regime and the modified AASHTO T283 procedure [8.30] were compared [8.51]. For standard mixtures, the two regimes produced similar results but, for a mixture known to have poor performance, the retained stiffness showed a high degree of dependence on the specimen saturation and the test method, with the SATS procedure being better at identifying the poor performance.

8.11.5 Experience with Test

See Section 8.11.4.

8.11.6 Relationship with Site Experience

See Section 8.11.7.

8.11.7 Durability

The ITSM of material had unexpectedly reduced by 60 % after 8 years in service. Investigation showed that the loss of strength was due to access of water through voids and, hence, loss of adhesion with time. The stiffness reduction was replicated in the SATS conditioning on samples of the same mixture [8.48 to 8.51] (Section 8.11.4).

8.11.8 Overview of SATS Test as Descriptor for Adhesion

The SATS tests shows promise as an indicator of the durability of adhesion, but currently the published research is from only one source. Work is now underway by others, and should help to validate this method for assessing the durability of adhesion between the binder and aggregate in asphalt mixtures. However, the results to date show that this durability is more dependent on the aggregate than the binder, which will make any assessment of the binder influence difficult.

8.12 Surface Free Energy Properties of a Bitumen-Aggregate System

8.12.1 Description

Asphalt pavement performance is related to cohesive and adhesive bonding within the bitumen/aggregate system and the cohesive and adhesive bonding are related to the surface free energy characteristics of the system.

Surface free energy is presented in many textbooks on surface physical chemistry [8.52]. By definition, surface free energy of a solid or liquid is the energy needed to create a new element area of surface under a vacuum. The relationship between the Gibbs free energy, work of adhesion and surface free energy can be summarised in Equation (8.1).

$$W^a = -\Delta G^a \tag{8.1}$$

where W^a is the work of adhesion and ΔG^a is the Gibbs free energy of adhesion.

When considering a brittle material, the total work expended per unit of surface area in forming two new surfaces (cohesive failure) is equal to twice the surface energy per unit of area, γ, as described in Equation (8.2).

$$W^c = 2\gamma \quad \& \quad \Delta G^c = -2\gamma \tag{8.2}$$

where W^c is the work of cohesion and ΔG^c is the Gibbs free energy of cohesion.

When two dissimilar materials form an interface by being in contact, a tensile force can split the materials into dissimilar components. In the latter case, Equation (8.3) can be postulated.

$$W^a = \gamma_1 + \gamma_2 + \gamma_{12} \tag{8.3}$$

where:

γ_1 = surface free energy of material 1 (e.g. bitumen)

γ_2 = surface free energy of material 2 (e.g. aggregate)

γ_{12} = interfacial energy of material 1 and 2

The properties of interfaces can normally be described as triple junctions or a three-phase boundary. Young proposed Equation (8.4) for a system in equilibrium to obtain surface tension from the contact angle formed when a drop of a liquid is placed on a perfectly smooth, rigid solid, as shown in Figure 8.17.

Young's equation $$\gamma_{SV} = \gamma_{SL} + \gamma_{LV}\cos\theta \tag{8.4}$$

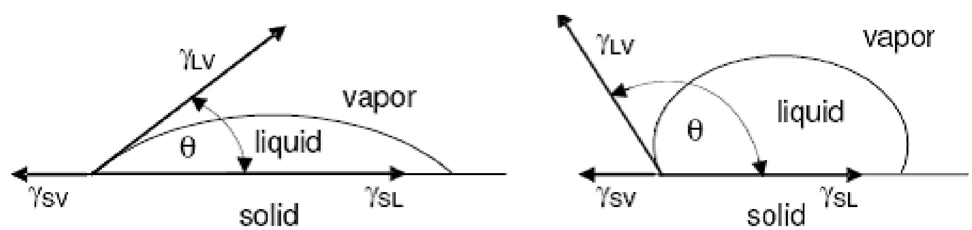

Figure 8.17 – The three-phase boundary of a liquid drop on a solid surface in vapour

Two different situations are depicted in Figure 8.17:

- A contact angle, θ, less than 90°, indicating a favourable interaction between the liquid and the solid resulting in a good spreading.
- A contact angle, θ, larger than 90°, indicating an unfavourable situation where moistening will be incomplete.

The surface energies of asphalt and aggregate are mainly comprised of an apolar and an acid-base component and, therefore, linking the energies to the type interaction and subsequently to the chemical nature of molecules present at the interface, as given in Equation (8.5).

$$\gamma = \gamma^{LW} + \gamma^{AB} \tag{8.5}$$

where:

γ = surface free energy of the bitumen and aggregates
γ^{LW} = Lifshitz-van der Waals (apolar) component of the free surface energy
γ^{AB} = acid-base (polar) component of the free surface energy

The measurement of the contact angle is a rather sophisticated method, although widely used in chemical engineering for the measurement of surface characteristics.

The determination of the surface energy of bitumen has been measured by either the Wilhelmy plate method [8.03, 8.04] or the pending drop methodology [8.53].

The Wilhelmy plate method is a universal method especially suited to check surface tension over long time intervals. A vertical plate of known perimeter, previously coated with bitumen, is attached to a balance and the force due to wetting is measured (Figure 8.18).

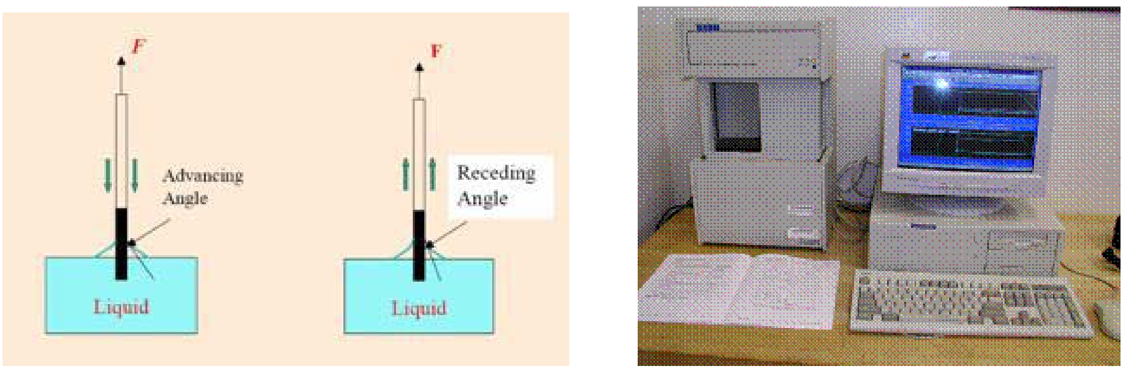

Figure 8.18 – Wilhemly Plate Method

Figure 8.19 – Device for contact angle measurement using the pending drop method

The surface and interfacial tension can also be measured by the pending drop method by analysing the geometry of a drop of liquid optically. This technique also permits running experiments at high temperatures and pressures (Figure 8.19).

The latter technique, while using test fluids of well-known surface energy (e.g. water or organic solvents), can also be used to determine the surface energy of a solid such as polished aggregates.

The measurement of the surface energy of aggregates was also performed by using the universal sorption device (USD) [8.03]. The USD is comprised of a magnetic suspension balance system, PC, temperature control, high quality vacuum and regulator, pressure transducer, solvent container and a vacuum dissector (Figure 8.20).

The methodology by which the USD is used to measure surface free energies of aggregates involves the following steps:

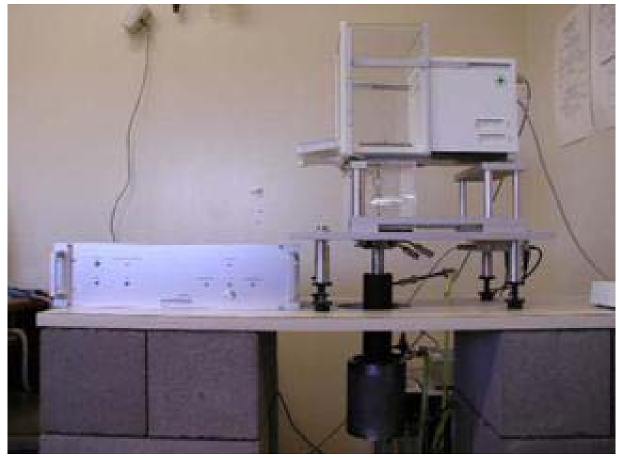

Figure 8.20 – Universal Sorption Device (USD)

- The selection of three solvents of well-known surface energies (and components).
- Measurement of the amount of solvent adsorbed to the surface of the aggregates.
- Correction for buoyancy effects.
- Calculation of the specific surface area by using the BET equations.
- Calculation of the spreading pressures at the saturation vapour pressure.
- Calculation of the surface free energies of the aggregate.

Although this methodology is quite long, it can accommodate the peculiarity of the irregular shape, size, mineralogy and rough surface texture of the aggregate used in asphalt mixtures.

8.12.2 Precision

No values for *r* and *R* have been reported to date. Nevertheless, a relative standard deviation of less than 10 % has been found routinely while accessing surface free energies of bitumen by using the Wilhemly plate method. Experience gained at BASt indicates a good precision of the pending drop method, but the sample preparation is crucial.

8.12.3 Relationship with other Tests

None found.

8.12.4 Experience with Test

The use of surface free energy properties of a bitumen-aggregate system to predict damage potential (especially stripping phenomena) has been described in detail in the literature [8.03, 8.04]. Nevertheless, almost all test results reported originate from research conducted by only one group (i.e. within the framework of the NCHRP 9-37 project). More recently, the evaluation of surface characteristics of both bitumen and aggregates in relationship to cohesive and adhesive properties has been picked up by research at BASt. However, the concept of surface energy has not been generally used up to now within the field of asphalt mix design because the research is relatively academic, requiring knowledge of thermodynamics and physico-chemistry.

8.12.5 Relationship with Site Experience

Not studied yet.

8.12.6 Durability

None data reported.

8.12.7 Overview of Contact Angle as Descriptor of Adhesion

The main advantage of the use of contact angles and, therefore, surface energies of materials such as bitumen and aggregate is its thermodynamic basis. Consequently, the test results enable a direct correlation to be made between the cohesive and adhesive properties of materials occurring in an asphalt mixture. However, studies also include stripping phenomena because the presence of a third component, such as water, can be taken into account.

The state of the art currently provides enough data to build up a database ranking materials with respect to their surface energy. Therefore, a good idea of compatible binder-aggregate combinations is possible. However, no validation in situ has been conducted yet nor is any planned.

One major disadvantage of the method is the need for sophisticated instrumentation and, therefore, qualified personnel to conduct the measurements. Moreover, it is generally accepted that sample preparation (often long and tedious) is critical to obtain reproducible test results. This disadvantage is not surprising because surface characteristics may differ from bulk properties of a material. Moreover, the concept of contact angles is only valid for systems in equilibrium. Consequently, systems which have the capacity to evolve with time in order to minimise the surface energy may be difficult to access in a reasonable time. This is particularly the case when studying composite systems produced at high temperatures or which contain surface active additives (e.g. polymers or adhesion promoters).

8.13 Water Immersion Test, Aggregate Method

8.13.1 Description

EN 13614 [8.54] specifies a method for determining the adhesion of a cationic bitumen emulsion coated onto a 6,3/10 mm or 8/11 mm light-coloured reference aggregate when immersed in water. The emulsion is mixed thoroughly with the aggregate and, after the emulsion has completely broken under specified conditions, the mixture is immersed in water in a glass container. After a given time and under specified conditions, the proportion of the aggregate surface covered with binder is visually assessed in per cent. The conditions specified depend on whether the emulsion has limited storage stability (breaking index lower than 120) or can be stored (breaking index higher than 120).

8.13.2 Precision

No values for *r* and *R* have been reported to date. However, EN 13614 claims that tests carried out by the same operator have shown that the same result is generally achieved for any given bitumen emulsion.

8.13.3 Relationship with other Tests

No data reported.

8.13.4 Experience with Test

No data reported.

8.13.5 Relationship with Site Experience

No data reported.

8.13.6 Durability

None data reported.

8.13.7 Overview of Water Immersion Test, Aggregate Method as Descriptor of Adhesion

Because the test is for cationic bitumen emulsions rather than bitumen itself, references to have not been found in the sources searched. However, the test may not be suitable for the generality of bitumens for the same reason.

8.14 Shaking Abrasion Test

8.14.1 Description

EN 12274-7 [8.55] specifies a test method for determining the suitability of 0/2 mm aggregates, cationic bitumen emulsions and, where appropriate, additives for slurry surfacings by determining the water sensitivity of the slurry surfacing mixtures. The mixtures are prepared to a standard grading and binder content at room temperature. Four 30 mm diameter by 25 mm high cylindrical specimens are statically compacted in a mould and then conditioned by storage under water in 60 mm diameter by 400 mm high cylinders with a vacuum applied prior to testing. The cylinders are rotated end over end in a suitable device and the material loss from the samples measured. The abrasion is mean result from the four samples.

8.14.2 Precision

No values for *r* and *R* have been reported to date.

8.14.3 Relationship with other Tests

No data reported.

8.14.4 Experience with Test

No data reported.

8.14.5 Relationship with Site Experience

No data reported.

8.14.6 Durability

None data reported.

8.14.7 Overview of Shaking Abrasion Test as Descriptor of Adhesion

Because the test is for slurry surfacings manufactured with cationic bitumen emulsions rather than hot mix asphalt manufactured with bitumen itself, references to this test have not been found in the sources searched. However, the test may not be suitable for the generality of bitumens for the same reason.

8.15 Recommendations for Adhesion

It is generally recognised that the primary function of bitumen is to act as an adhesive. As a consequence, the study of this property has been the subject of numerous investigations in recent decades. The range of descriptions of the various test methods in Sections 8.2 to 8.12 devised to assess the adhesive properties of binders illustrates the research efforts in this area. Furthermore, it is appreciated that the list of test methods taken up in this chapter is not comprehensive. However, other methods reported often only vary in the choice of test parameters or experimental set up and can be found elsewhere [8.41, 8.42, 8.56].

Other tests, such as the freeze-thaw pedestal test, the immersion wheel tracking test, the Hamburg wheel tracking device with water conditioning and the environmental conditioning system (ECS), have been omitted from the overview. The latter test methods can be related only indirectly to adhesion phenomena (immersion in water while carrying out the tests) but are otherwise not developed as adhesion test for binders or mixtures. Therefore, such tests were considered as not really relevant to the topic of adhesion and, consequently, not discussed further.

Unlike other chapters in this report, the conclusions for adhesion are not straightforward. Although, research on this topic has been going on for a long time, both the understanding as well as the assessment of the adhesive properties of bitumen is still an area of focus. An attempt is made to clarify some of the remaining issues in Table 8.3 by reviewing both the advantages as well as the limitations of several test methods described in this chapter. However, Table 8.3 is not comprehensive (as noted above) but it does illustrate some of the unanswered questions relating to adhesion.

The review given in Table 8.3 serves as a guide to formulate the following general conclusions with respect to adhesion:

- Adhesion is an interface phenomenon and, therefore, applies to material combinations (e.g. binder/aggregate or mastic/aggregate). Water has to be included into the relationship because of the durability issues with asphalt mixtures. As a result, the direct assessment of adhesion is complicated (a variety of test specimens ranging from a bitumen/aggregate pair to compacted asphalt mixtures subjected to water conditioning and/or traffic).
- The surface properties of a sample (e.g. of bitumen) could differ significantly from the bulk material. All materials will tend to minimise their surface energy if possible (depending on their capacity to rearrange at a given temperature).
- The approach of using surface energies of materials enables some fundamental insights about adhesion to be gained. However, equilibrium in an asphalt mixture is probably never realised.

Table 8.3 – Summary of advantages/limitations of test methods assessing adhesion

Test method	Advantages	Limitations
Rolling bottle test	Simple and easy to perform	Visual and, therefore, subjective evaluation of test result, making it a screening technique
Boiling water stripping test	Objective test	Need for chemicals
Ultrasonic method	High sensitivity	Highly dependant of experimental set up
Net adsorption test	Thermodynamic basis (Langmuir isotherms)	Further research needed in order to predict the in service performance
Vialit plate test	Considerable experience	Interpretation in terms of adhesion hampered by other parameters such as cohesion and ductility
Indirect tensile strength	Takes into account the effect of water conditioning Test carried out on a asphalt mixture	Validation with in situ performance not straightforward Interpretation of test results by the use of a single parameter (moisture) is questionable
PATTI	Well established test used in coating industry	Mode of failure changes with water conditioning (cohesive to adhesive)
SATS	Replicates observed loss of adhesion	Limited experience Results highly dependent on aggregate
Surface energies of materials	Based on solid thermodynamic principles	Sophisticated instrumentation needed Theoretical model only valid for systems in equilibrium
Water immersion test, aggregate method	–	Designed specifically for bitumen emulsions only
Shaking abrasion test	–	Designed specifically for bitumen emulsions in slurry surfacings only

- The interpretation of test results with respect to adhesion is often hampered by the interference of other phenomena. The occurrence of a cohesive failure mode often accompanies the observations made during an experiment. Moreover, water conditioning could introduce a change in the failure mechanism and, hence, lead to inconclusive test results (e.g. PATTI).
- The effect of the water conditioning can vary with the accessibility of the asphalt mixture (e.g. voids content) and, therefore, the mix design itself could interfere with the test result (e.g. ITS).
- An asphalt mixture is a dynamic system (e.g. ageing and/or traffic introduced damage) and, therefore, its susceptibility to moisture damage or loss in adhesive properties could vary over time (e.g. introducing cracks enables water to penetrate the system).

- The majority of the tests lack in situ experience. Validation with in-service performance needs to be evaluated.

In contrast to other tests, interfacial properties and, therefore, adhesion is possibly the most difficult one to conceive. It has to describe the suitability of binders to adhere to various pavement components such as aggregates, sand and fillers. Although a lot of interesting ideas are included in the tests already described in this chapter, the subject of adhesion still needs future research in order to establish well validated and performance-based specification.

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9. Recommendations

9.1 Data Reviewed

A large number of papers that identified both asphalt and bitumen properties have been reviewed, with the combinations of properties covered in the sections listed in Table 9.1.

Table 9.1 – Location of Reviews

Bitumen Tests	Asphalt Properties Permanent Deformation	Stiffness	Low Temp. Cracking	Fatigue Cracking	Adhesion
Number of references No. of papers No. of standards	46 39 7	30 30 –	12 8 4	19 19 –	54 42 14
BBR	–	5.2	6.2	7.5	Adhesion is not a property of just the binder, so the tests to measure it are either on compacted or uncompacted mixtures. As yet, no proposal has been developed to measure the adhesive properties of binders directly other than the use of uncompacted samples with a control aggregate.
Binder Fatigue	–	–	–	7.2	
Capillary Viscometer	4.2	–	–	–	
Coaxial Cylinder Visc.	4.3	–	–	–	
Cone & Plate Visc.	4.4	–	–	–	
Creep Zero Shear Visc.	4.5	–	–	–	
Direct Tensile	–	–	6.3	–	
DSR	4.6	5.3	–	7.3	
Force Ductility	4.7	–	–	7.4	
Fraass	–	5.4	6.4	–	
Fracture Toughness	–	–	6.5	–	
Oscillation Zero SV	4.8	–	–	–	
Penetration	–	5.5	–	7.5	
Penetration Index	–	5.6	–	–	
Repeated Creep	4.9	–	–	–	
Softening Point	4.10	5.7	–	7.5	
Tensile	–	–	–	–	
Vialit Pendulum	–	–	–	–	

It is on these reviews that the recommendations for the selection of tests (Section 9.2) and further research (Section 9.3) are based.

9.2 Recommendations for Test Selection

9.2.1 Permanent Deformation

The papers covering permanent deformation are reviewed in Chapter 4 with recommendation given in Section 4.11. The overall conclusion is that the DSR test is relevant for permanent deformation when the complex modulus is considered at elevated service temperatures and at low frequencies. The DSR test is then equivalent to the oscillation test for ZSV, which is the preferred method. The oscillation ZSV test is relatively simple and provides good correlations but still needs to be correlated when using the equiviscous temperature for a given zero shear viscosity. However, further research is required, as described in Section 9.3.1.

9.2.2 Stiffness

The papers covering stiffness are reviewed in Chapter 5 with recommendation given in Section 5.8. The overall conclusion is that the best options for identifying the potential binder contribution to asphalt stiffness are DSR binder stiffness and/or penetration, with the former being able to evaluate stiffness for many temperature/frequency combinations that are encountered in practise but is also more complex. Given that PMBs, for which penetration is not suitable, are widely used, the preferred test is DSR. However, further research is required, as described in Section 9.3.2.

9.2.3 Low Temperature Cracking

The papers covering low temperature cracking are reviewed in Chapter 6 with recommendation given in Section 6.7. The overall conclusion is that the best options for identifying the potential binder contribution to the low temperature behaviour of asphalt are either BBR limiting temperature or a DTT parameter. An alternative might be the use of the concept of critical cracking temperature which is combining both the BBR and the DTT results for determining a low-temperature parameter called critical cracking temperature T_{cr}, although time will be needed to confirm its suitability and it is unlikely to be available in the immediate future. In addition, a “real” fracture property, such as fracture toughness or fracture energy, may be used to supplement the information gathered from BBR and DTT. Nevertheless, until further information becomes available, the BBR is probably the best option for use in a performance standard for bitumen. However, further research is required, as described in Section 9.3.3.

9.2.4 Fatigue Cracking

The papers covering fatigue cracking are reviewed in Chapter 7 with recommendation given in Section 7.6. The overall conclusion is that the basic characteristics (such as penetration, softening point and viscosity) and some rheological characteristics, both before and after ageing (such as RCAT or RTFOT and PAV), remain the best criteria to assess the fatigue behaviour of asphalt although the relationship between bitumen fatigue and mixture fatigue at the number of cycles to achieve a 50 % reduction in G^* looks promising. However, further research is required, as described in Section 0.

9.2.5 Adhesion

The papers covering adhesion are reviewed in Chapter 8 with recommendation given in Section 8.13.3. The overall conclusion is that the approach of using surface energies of materials enables some fundamental insights about adhesion to be gained, although equilibrium in an asphalt mixture is probably never realised. However, further research into the definition of standard aggregates is required, as described in Section 9.3.5.

9.3 Recommendations for Further Research

9.3.1 Permanent Deformation

9.3.1.1 Oscillation ZSV Test

Although the oscillation test has shown good correlation, the conditions have not been fixed, particularly for equiviscous temperatures. Therefore, the exact test conditions (temperature and frequency) need to be established that would give results that correlate best with asphalt rutting. The following guidelines would be used as the starting point:

- The tests should find the equiviscous temperature for a zero shear viscosity of 2000 Pa.s.
- Alternatively, the test temperature would be as close as possible to the temperature at which rutting occurs.
- The test frequencies would be between 0,01 Hz and 0,001 Hz, which have led to good correlations whilst still being practical.

The investigation would involve identifying a selection of sites with known level of traffic and histories of deformation achieved for which the precise aggregate skeleton and binder are known. Ideally, the aggregate skeletons would be identical whilst the deformations and the binders would differ significantly. A series of oscillation ZSV tests would be carried out under differing test conditions and the results analysed to maximise the correlation between the test results and the observed deformations after allowing for the traffic. If, as expected, there are a range of aggregate skeletons, some standard asphalt tests for deformation resistance (wheel tracking and/or cyclic compression) would be needed to identify the contribution of the skeleton and, hence, allow for it when maximising the correlation.

If insufficient field data can be found, the site data would have to be developed using accelerated load testing (ALT) of sites or trial lengths with known mixtures. However, ALT will not identify any effect of changes of the binder properties with time. There is a strong possibility that ALT will be necessary.

9.3.1.2 Creep ZSV Test and/or Repeated Creep Test

If the oscillation ZSV test is not taken up as the measure for permanent deformation for any reason, the best alternative is the creep ZSV test with the repeated creep test as an interesting candidate. However, these tests require that the procedures are refined and a precision exercise undertaken on the finalised procedure for them to be viable tests.

The creep ZSV test would need to be repeated on a variety of binders under differing conditions, in particular duration and deformation. The comparative performance of those binders in asphalt mixtures, based on standard asphalt tests for deformation resistance (wheel tracking and/or cyclic compression), would be used to optimise the conditions for relevance, practicality and precision. An inter-laboratory procedure would be undertaken using at least six binders (three PMBs with a high polymer content) in order to assess the resulting repeatability and reproducibility.

The repeated creep test would need an inter-laboratory precision exercise with at least six binders (three PMBs with a high polymer content) included in order to assess the resulting repeatability and reproducibility.

9.3.2 Stiffness

There is sufficient data to validate the relationship between DSR binder stiffness and the mixture stiffness, particularly when using the same temperature and frequency conditions. However, the durability implications and any relationship to pavement performance are effectively missing. Therefore, there is justification to undertake further research in order to understand the measures required to identify long-term changes in mixture stiffness from binder properties.

Ideally, a series of aggregate skeletons with a number of different binders would be laid on a road in a series of trial section over identical formations. The structural stiffness would be monitored regularly on core or other samples and any change or differences related back to DSR measurements made on the binders made unaged, after RTFOT and after RTFOT and PAV and/or RCAT. However, this procedure would take many years to produce the results. Therefore, existing sites would need to be found with the results and with known binders that could be replicated for the DSR tests. It is uncertain how much data could be obtained from such sites.

9.3.3 Low Temperature Cracking

9.3.3.1 Direct Tensile Test

Despite promising results, the precision of the DTT still remains to be improved. Therefore, the test would need to be repeated on a variety of binders under differing conditions. The comparative performance of those binders in asphalt mixtures, based on standard asphalt tests for low temperature cracking (TST, RT, TSRST and/or UTST), would be used to optimise the conditions for relevance, practicality and precision. An inter-laboratory precision exercise would be undertaken using at least six binders (including three PMBs with a high polymer content) in order to assess the resulting repeatability and reproducibility.

9.3.3.2 Critical Cracking Temperature

The concept of critical cracking temperature, which is a combination of BBR and DTT results to determine a low-temperature parameter called the critical cracking temperature, T_{cr}, has shown great promise but has still to be validated.

Sites with known asphalt mixtures would have to be found that have developed low-temperature cracking, ideally together with similar sites using different mixtures and/or binders where low-temperature cracking has not developed. The data that can be collected on the in service performance would be compared with the binder properties to ascertain the extent to which the tests can be used to predict low-temperature cracking.

If insufficient field data can be found, the site data would have to be developed using ALT of sites or trial lengths with known mixtures. However, ALT will not identify any effect of changes of the binder properties with time. There is a strong possibility that ALT will be necessary.

An inter-laboratory precision exercise will not be required because the precision should be calculable from the precision of the BBR and DTT results used to determine the value.

9.3.3.3 Fracture Toughness Test

Data from the fracture toughness test is limited but the fracture parameters do appear to be independent and, thus, may be complementary to other reasonable low temperature binder properties, such as the parameters from the DTT. Research is needed to establish how both types of parameters can be combined to predict the low temperature behaviour of asphalt mixtures.

At least three different aggregate skeletons with six different binders (including three PMBs with a high polymer content), giving a total of 18 mixtures, would be tested for the standard asphalt tests for low temperature cracking (TST, RT, TSRST and/or UTST). The binders would also be tested for fracture toughness and DTT. The relevant parameters from Fracture Toughness and DTT would be correlated to the asphalt properties to identify if a combined parameter could be used to give a better predictor and, if so, whether the extra test time/cost is worth the improvement gained.

If the research is positive, an inter-laboratory precision exercise would be undertaken using at least six binders (three PMBs with a high polymer content) in order to assess the resulting repeatability and reproducibility for the fracture toughness test.

9.3.3.4 Field Performance

With regard to both the critical cracking temperature (BBR and DTT) and the FTT parameters, there are insufficient data on their correlation with the field performance and further research would be necessary before any definitive conclusion can be drawn. In particular, data are needed on the performance characterisation of modified bitumen.

Sites with known asphalt mixtures would have to be found that have developed low-temperature cracking, ideally together with similar sites using different mixtures and/or binders where low-temperature cracking has not developed. The data that can be collected on the in service performance would be compared with the binder properties to ascertain the extent to which the tests can be used to predict low-temperature cracking.

If insufficient field data can be found, the site data would have to be developed using ALT of sites or trial lengths with known mixtures. However, ALT will not identify any effect of changes of the binder properties with time. There is a strong possibility that ALT will be necessary.

9.3.4 Fatigue Cracking

Further research is needed for a more explicit correlation between the bitumen fatigue and mixture fatigue at the number of cycles to achieve a 50 % reduction in G^*. Validation with field performance is critical because there is a lack of information relating laboratory fatigue behaviour with performance in practice.

Asphalt mixtures with a wide range of polymer types and polymer contents, as well as paving grade bitumen, would be manufactured and tested for asphalt fatigue using the two point trapezoidal and four point bending regimes. The results for the number of cycles to achieve a 50 % reduction in G^* would be correlated with the similar value for the binder tested for bitumen fatigue. Where possible, experience from sites with the mixtures tested should be compared with the test results, but there are unlikely for many such sites.

9.3.5 Adhesion

9.3.5.1 Control Aggregates

Adhesion is a property of both the binder and the aggregate to which the binder has to adhere. Therefore, any test needs to involve an aggregate, either the actual aggregate to be used or standard aggregates that can be used to develop a ranking for binders. For a European, or other international, standard, any standard aggregate could not be limited to a single quarry or location. Therefore, they need to be either a mineral (e.g. silica), a manufactured product (e.g. aluminium oxide) or a widely available aggregate type (e.g. limestone), any of which could be restricted by demanding specific properties.

Different options for one or more standard aggregates would be investigated by selecting several sources of the option and undertaking the candidate adhesion tests with the same binder to identify whether the range of sources added excessive uncertainty in the result. If the majority of the results produce consistent results but there are outliers, the reason for those outliers would need to be ascertained and limits applied to the aggregate that would exclude them.

9.3.5.2 Field Performance

The majority of adhesion tests lack data on their correlation with the field performance and further research would be necessary before any definitive conclusion can be drawn. In particular, data are needed on the performance characterisation of modified bitumen.

Sites with known asphalt mixtures have been found to develop adhesion problems would need to be found, ideally together with similar sites using different mixtures and/or binders where adhesion problems have not developed. The data that can be collected on the in service performance would be compared with the adhesion test results to ascertain the extent to which the tests can be used to predict adhesion and its retention.

If insufficient field data can be found, the site data would have to be developed using ALT of sites or trial lengths with known mixtures. However, ALT will not identify any effect of changes of the binder properties with time. There is a strong possibility that ALT will be necessary.

9.3.6 Proposal for Further Work

The items of further work described above can be categorised into three levels of desirability, together with an idea of the time required for the work as short-term (1-2 years), medium-term (2 to 5 years) or long-term (over 5 years), as follows:

<u>Essential</u>:

Deformation resistance	Oscillation ZSV test	Section 9.3.1.1	Short-term
Low temperature cracking	Critical cracking temperature	Section 9.3.3.2	Medium-term
Adhesion	Control aggregates	Section 9.3.5.1	Medium-term

<u>Important</u>:

Low temperature cracking	Direct tensile test	Section 9.3.3.1	Medium-term
Low temperature cracking	Fracture toughness test	Section 9.3.3.3	Medium-term
Fatigue	Bitumen fatigue test	Section 0	Medium-term
Adhesion	Relationship with site data	Section 9.3.5.2	Long-term

Desirable:

Deformation resistance	Creep ZSV/repeated creep tests	Section 9.3.1.2	Medium-term
Stiffness	DSR relationship with site data	Section 9.3.2	Long-term
Low temperature cracking	Relationship with site data	Section 9.3.3.4	Long-term

9.4 Database and Library

The database and library developed during the course of this work is a valuable resource that could be used for future work by the institutes involved in the project, all FEHRL institutes or even more widely. However, the value of them will only be maintained if the information contained is kept up to date. Maintenance can be achieved by either regular updates as conferences are held or by any institute or consortium of institutes using them on a project having to update them from the last time they were updated. The cost of any future work could thereby be minimised whilst, at the same time, adding data to a valuable resource.

The library and database are currently held on a password-protected area of the FEHRL website. The members of the current project have passwords. It would be ideal to allow free access to the area, but there could be copyright issues with some or all of the papers in the library.

Therefore, it is proposed that:

- Access becomes unrestricted to the BiTVal database on the FEHRL website.
- The BiTVal library remains on a password-protected area of the FEHRL website.
- Participants in the BiTVal project plus other FEHRL members are allowed access to the password-protected area.
- The use of the BiTVal database and library for any future project is conditional on the institutes involved on that project updating them.

10. Acknowledgements

The work on this project in the United Kingdom was undertaken by the Network Management Group of the Infrastructure Division of TRL Limited with funding from the Pavement Engineering Team of the Highways Agency, whose support is gratefully acknowledged.

The work on this project in Germany was undertaken by the Federal Highway Research Institute (BASt) in association with the Federal Ministry of Transport, Building and Urban Development (BMVBS) and the Road and Transport Research Association (FGSV), whose support is gratefully acknowledged.

The work on this project in Belgium was undertaken by the Belgian Road Research Centre (BRRC), which wishes to thank the Federal Ministry for Economic Affairs for the financial support in this project under contract CC-CCN 109.

The work on this project in Austria was undertaken by the Institute for Road Construction and Maintenance of the Vienna University of Technology and funded by OMV Aktiengesellschaft as partner of the Institute's Christian Doppler laboratory, whose support is gratefully acknowledged.

The institutes and universities involved in the work would like to thank the stakeholders (CEN, Eurobitume, EAPA and FEHRL) for their support and guidance throughout the project.

Annex A: Acronyms used

AC	Asphalt Concrete
AASHTO	American Association of State Highway & Transportation Officials
ALT	Accelerate Load Testing
ASTM	American Society for Testing & Materials
BBSG	Béton Bitumineux Semi Grenu
BBR	Bending Beam Rheometer
BiTVal	Bitumen Test Validation
CEDR	Conferences of European Directors of Roads
CEN	Comité Européen de Normalisation (European Committee for Standardisation)
CTOD	Crack Tip Opening Displacement
DBM	Dense Bitumen Macadam (a form of asphalt concrete used in the UK)
DSR	Dynamic Shear Rheometer
DTT	Direct Tensile Test
EAPA	European Asphalt Pavement Association
ECS	Environmental Conditioning System
ETFOT	Extended Rolling Thin Film Oven Test
ESAL	Equivalent Single Axle Load
EVA	Ethylene Vinyl Acetate
FEHRL	Forum of national European Highway Research Laboratories
FSCH	Frequency Sweep at Constant Height
FTT	Fracture Toughness Test
HiPAT	High Pressure Ageing Test
HMA	Hot Mix Asphalt
HMB	High Modulus Base (a form of asphalt concrete used in the UK, often with midpoint of bitumen grade given afterwards)
HRA	Hot Rolled Asphalt
HVS	Heavy Vehicle Simulator
IISRP	International Institute of Synthetic Rubber Producers
IR	Infrared
ITS	Indirect Tensile Stiffness
ITSM	Indirect Tensile Stiffness Modulus
ITSR	Indirect Tensile Stiffness Ratio
LTA	Long-Term Ageing
MRL	Materials Reference Library (for SHRP)
MTFOT	Modified Thin Film Oven Test
MRTFOT	Modified Rolling Thin Film Oven Test
NRTFOT	Nitrogen Rolling Thin Film Oven Test
PA	Porous Asphalt
PAV	Pressure Ageing Vessel
PG	Performance Grade (binder grading to SHARP specification)
PMB	Polymer-Modified Binder
R&B	Ring and Ball
rbs	Road Bitumen Stabiliser
RCAT	Rotating Cylinder Ageing Test
RFT	Rotating Flask Test
RMFOT	Rolling Microfilm Oven Test
RSCH	Repeated Shear at Constant Height
RT	Relaxation Test
RTFOT	Rolling Thin Film Oven Test
SATS	Saturation Ageing Tensile Stiffness test

SBS	Styrene-Butadiene-Styrene block copolymer
SENB	Single-Edge Notch Bend
SHRP	Strategic Highways Research Program
SMA	Stone Mastic Asphalt
SST	SUPERPAVE Shear Tester
STA	Short-Term Ageing
TFAAT	Thin Film Accelerated Ageing Test
TFOT	Thin Film Oven Test
TODT	Tilt-Oven Durability Test
TSRST	Tensile Stress Restrained Specimen Test
TST	Unrestrained Thermal Dilation Test
UAMC	Ultrasonic Accelerated Moisture Conditioning procedure
UTST	Uniaxial Tensile Strength Test
WFT	Wheel Fretting Test

Annex B: BiTVal Project Plan

B.1 Introduction

The Comité Européen de Normalisation (European Committee for Standardisation, CEN) technical committee for bituminous binders, TC 336, has already produced a specification for paving grade bitumen that is based on national standards that had existed previously. Working group TC 336/WG1 is now working on a second generation bitumen standard that is intended to be performance-related and so will be able to cover polymer-modified bitumen as well as unmodified paving grade bitumens. Such a performance-related specification should ensure that the optimal binder can be selected for each case that will maximise the performance and/or minimise the cost.

TC 336/WG1 started by investigating tests that are intended to measure different performance properties. The process started by a selection process that included analysis at the Eurobitume Workshop 1999 in Luxembourg and a series of national Bitumen Test Specification (BiTSpec) seminars around Europe during 2002/2003 culminating in the European BiTSpec seminar in Brussels in June 2003. Several of the identified bitumen tests have subsequently been drafted in preparation for the development of the performance-related specification.

However, to be assured that the selected tests are sufficiently related to performance, TC 336/WG1 wanted the Forum of European National Highway Research Laboratories (FEHRL) to validate them. Validation will form a crucial stage in the process for developing the new system of binder specifications in Europe, and there was support for validation at the BiTSpec seminars. The necessity to assess the relevance of test methods to performance was also recognised by the European organisations – representative of the key stakeholders (EAPA, Eurobitume, FEHRL, IISRP and CEDR) – when they were invited by CEN in 2002 to define and express their needs and expectations related to the future binder specification system.

The following three main phases have been proposed for the validation project:

- Phase 1: Assembly and assessment of existing data from all sources that can assist in the validation of the test methods defined by CEN TC 336.
- Phase 2: Laboratory and/or field validation trials needed to complete the validation of the test methods defined by CEN TC 336.
- Phase 3: Research, development and validation of tests for other bitumen properties considered to have a potential relevance for the specification of bitumen.

The FEHRL Board has agreed that Phase 1 should be undertaken by laboratories that could obtain funding from their national highway authority. As part of Phase 1, the additional work needed in Phase 2 and, possibly, Phase 3 will be identified and justified so that the proposal for these phases can be put forward for a project that can be funded centrally by one (or more) relevant European organisations.

B.2 Project Methodology

B.2.1 Data Organisation

B.2.1.1 Standard Record Form

Nynas AB commissioned the Belgian Centre de Recherches Routières (BRRC) to undertake a review of references to bitumen tests in published reports as part of their contribution to the dialogue that resulted in the selection of the tests by CEN TC 336/WG1. Based on the experience it gained on that project, BRRC will develop a standard form for recording the relevance of a paper to the project in terms of the test(s) covered, associated asphalt properties from laboratory and/or site samples, precision, etc. A separate form will be used for the data from each paper or other reference. BRRC will also prepare a spreadsheet on which the data from each form can be stored electronic or the input could be input direct.

B.2.1.2 Questionnaire Preparation

Eurobitume will develop a questionnaire based on the standard record form that will ascertain the required information from organisations outside the project.

B.2.1.3 Preparing a Database

The German Bundesanstalt fuer Straßenwesen (BASt) will set up a database which can input the information from the spreadsheet. The database will have to have fields for all the information on the standard record form together with free fields for the abstract and conclusions.

B.2.2 Data Collection

B.2.2.1 Early International Conferences

The references investigated by BRRC for Nynas AB were compiled from international conferences up to 2000. Therefore, BRRC will be responsible for converting these conferences.

B.2.2.2 Later International Conferences

The Project Manager will produce a list of international conferences, symposia and workshops that cover bitumen and/or asphalt since 2000 with assistance from other members of the Steering Committee. The Project Manager will allocate each seminar to a participating FEHRL laboratory.

B.2.2.3 Data from Conferences

Each participant will obtain copies of the papers for the conferences that are allocated to them, preferably electronic in .pdf format. Each paper will be reviewed for relevance to the project and, if they are relevant, the data will be input onto the spreadsheet, either directly or via the standard record form. A library of the relevant papers will be compiled in .pdf format, scanning in papers for which an electronic version is not available. The library will be for use in the project only.

B.2.2.4 Issuing of Questionnaires

The questionnaires will be sent highway engineering departments, road laboratories (including members of the project), universities with highways departments, road authorities, bitumen producers, asphalt producers and contractors in order to get information on both past and ongoing research projects, test tracks and field sections across the Europe where the results are relevant to the project but have not yet been published or where the information is not readily available. Possible recipients of the questionnaire will be proposed by members of the project and confirmed by the Steering Committee.

B.2.2.5 Questionnaire Returns

There are not participating FEHRL laboratories in every European country, so the Project Manager, in consultation with the Management Committee, will allocate European and major international countries between the participating FEHRL laboratories. The questionnaire returns will be returned to the participating FEHRL laboratory responsible for the country from which the respondee comes. The laboratory will convert the reply into electronic format on the spreadsheet.

B.2.2.6 National and Unpublished Information

If the replies to the questionnaire or local knowledge identify national conferences or published papers on relevant work not reported in the international conferences covered, the laboratory responsible for the country will review the papers similarly to the papers in international conferences in Section B.2.2.3.

B.2.2.7 Compilation of BiTVal Database

Participating FEHRL laboratories will copy their completed spreadsheets and electronic library of references to BASt. BASt will compile the data into the BiTVal database and distribute copies to all members of the Steering Committee.

B.2.2.8 BiTVal Library of References

Participating FEHRL laboratories will copy their electronic library of references to the Project Manager. The Project Manager will compile the complete electronic BiTVal library of references and distribute copies to all members of the Steering Committee.

B.2.3 Data Analysis

B.2.3.1 Compilation of Test Data

The Project Manager, in consultation with the Management Committee, will allocate the CEN TC 336/WG1 tests between the participating FEHRL laboratories. The laboratories will identify the papers relevant to those tests from the BiTVal database and the aspects each cover. The participating laboratory will compile a report on each test method by use of the BiTVal database and the BiTVal library of references that covers the following aspects:

- Relevance of the test result to the intended property of the bitumen.
- Relevance of the test result to the intended property of the asphalt.
- The precision of the test.
- The range of values that can be attained with the test.
- The impact of a change in the test result on the property of the asphalt.

B.2.3.2 Assessment

The Steering Committee will review all the compilations and assess the validity of the test method to a consistent standard. Ideally, the assessment for each test will be one of the following:

- The test produces a result that is a valid and workable measure of the intended property.
- The test does not produce a result that is a valid and workable measure of the intended property.
- The test produces a result that could be a valid and workable measure of the intended property, but there is conflicting indications or there is insufficient information to confirm validity.

B.2.4 Requirements for Phases 2 and 3

B.2.4.1 Identifying Missing Data

For those tests that have been assessed as producing results that could be valid and workable measures of the intended properties but for which there are conflicting indications and/or there are insufficient data to confirm validity, the type and extent of data that is needed to confirm or deny validity will be assessed. The Steering Committee will prepare a detailed proposal for obtaining the missing data, including a realistic timetable and costs.

B.2.4.2 Additional Tests

If CEN TC 336/WG1 identify additional tests after starting Phase 1 or Phase 1 identifies additional tests to replace tests do not produce results that are valid and workable measures of the intended properties or other tests are identified by other research, those additional tests will need to be validated. The Steering Committee will prepare a proposal to validate such additional tests (if any), including a realistic timetable and costs.

B.3 Project Organisation

The project will be managed by Ian Carswell of the British Transport Research Laboratory (TRL). The project manager will be supported by a Management Committee to ensure that the work is undertaken efficiently and a Steering Committee to ensure that the work is targeted correctly. The Management Committee will consist of representatives of the participating FEHRL laboratories and the Steering Committee will consist of the Management Committee plus representative of other key stakeholders (EAPA, Eurobitume and possibly CEDR and/or IISRP).

The management structure for Phase 1 of BiTVal is set out in Figure B.1. The FEHRL laboratories included are the ones known to be interested in taking part, but this list may change due to the laboratories being unable to gain funding and/or other FEHRL laboratories joining.

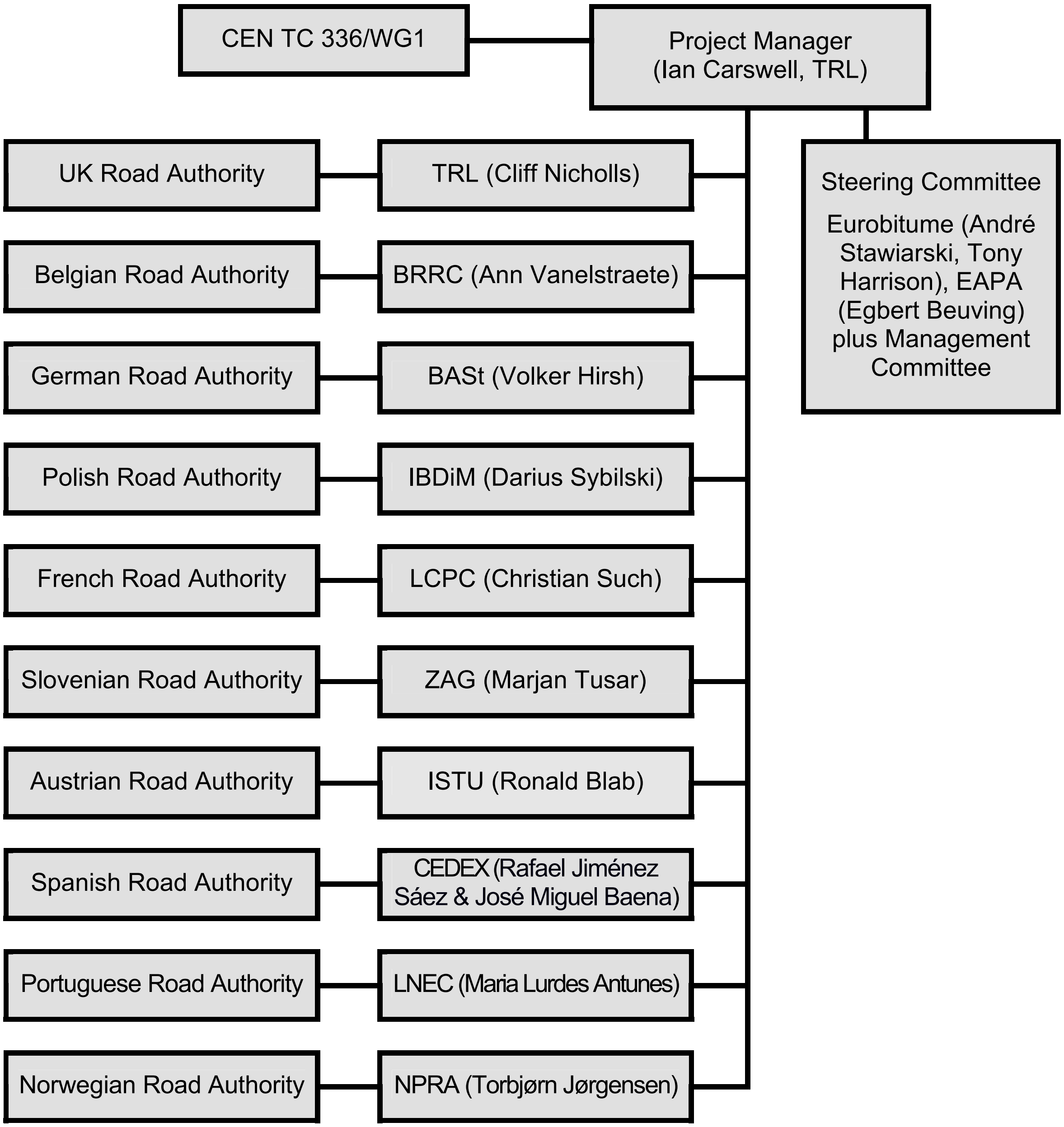


Figure B.1 – Management Structure

B.4 Outputs

B.4.1 Customer

There is no overall customer for the project because Phase 1 of BiTVal is to be undertaken by FEHRL laboratories funded by their national highway authority for the effort that they put into the work. Therefore, CEN TC 336/WG1 will be treated as the nominal customer, but all outputs, including minutes of meetings, will be available for any member of the project team to send to his direct customer. Furthermore, in the absence of an overall customer and with the work shared by several laboratories, the intellectual property rights of the outputs will be vested in the Forum of European Highway Research Laboratories, FEHRL.

B.4.2 Meetings

Formal meetings of the Steering Committee will be held at approximately 6 monthly intervals, including at the start and finish of the project in February 2004 and May 2005, respectively. Other meetings of the Steering Committee and meetings of the Management Committee will be called as appropriate. However, the main medium of communications between members of the committees will be email.

B.4.3 Deliverables

B.4.3.1 BiTVal database and library of references

A database of published, unpublished and ongoing research projects relating bitumen properties and/or test methods to asphalt mixture properties and/or pavement performance, together with electronic copies of the references included in the database in .pdf format. Both the database and library of references will be made available to FEHRL laboratories for future research on behalf of national or European highway authorities.

B.4.3.2 Summary report

A summary report for submission to CEN TC 336/WG1 in which all the available information for each of the CEN TC 336/WG1 selected test methods is reviewed. The conclusion will include the assessment of validity for each test and consequential recommendations.

B.4.3.3 Proposal for Phases 2 and 3

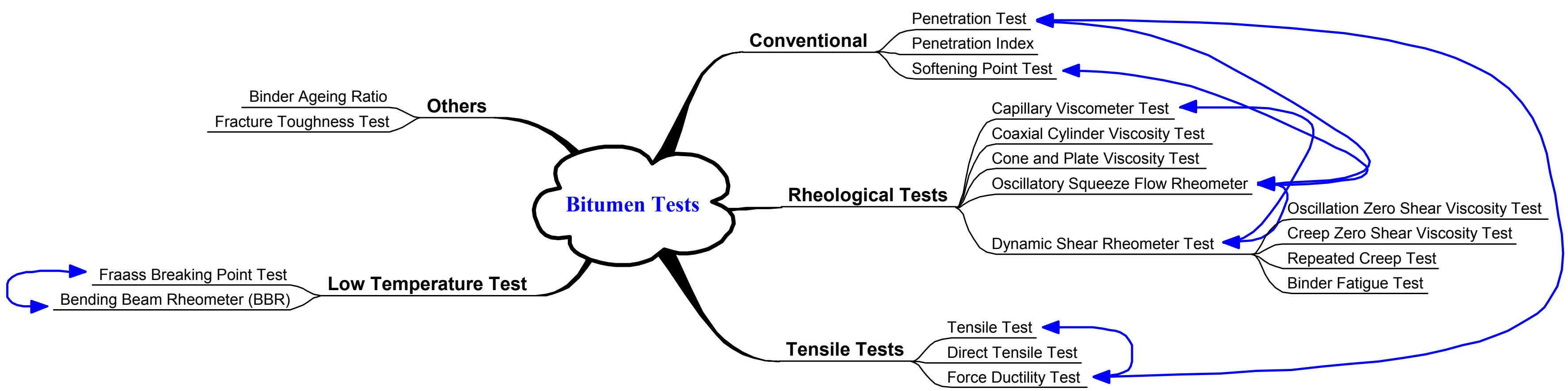
A proposal for the work needed to be undertaken in Phase 2 in order to fill any gaps that have been identified for the validation of any of the selected test methods. The proposal will also include the work needed to be undertaken in Phase 3 if there are additional tests needing validation.

B.4.4 Future Use of BiTVal Outputs

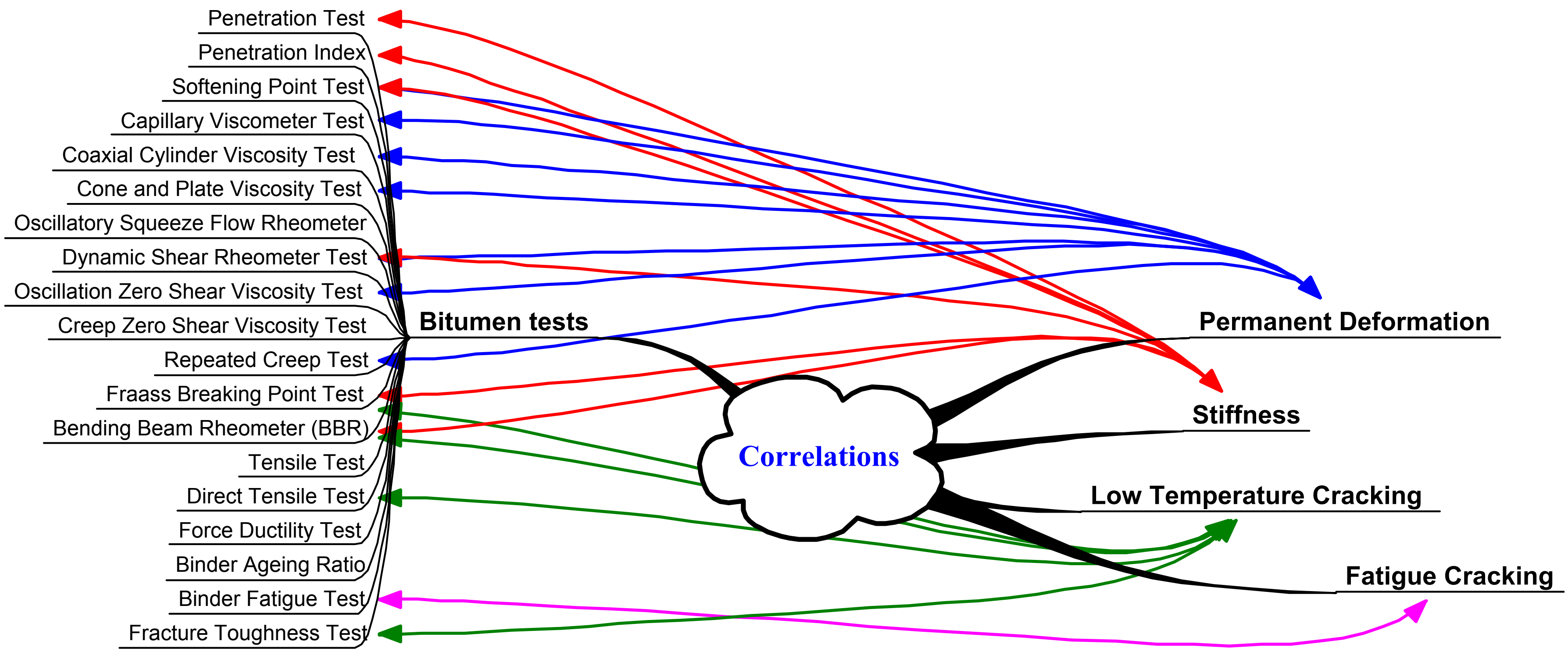
It is hoped that the BiTVal database and library of references will become useful tools for FEHRL laboratories when investigating bitumen and bitumen tests. Researchers would use these facilities as part of the literature search at the beginning of their projects and provide one or more inputs to add to the database and library of references. As such, the BiTVal database and library of references will need to be maintained by a FEHRL laboratory or laboratories.

Annex C: Schematic Relationships

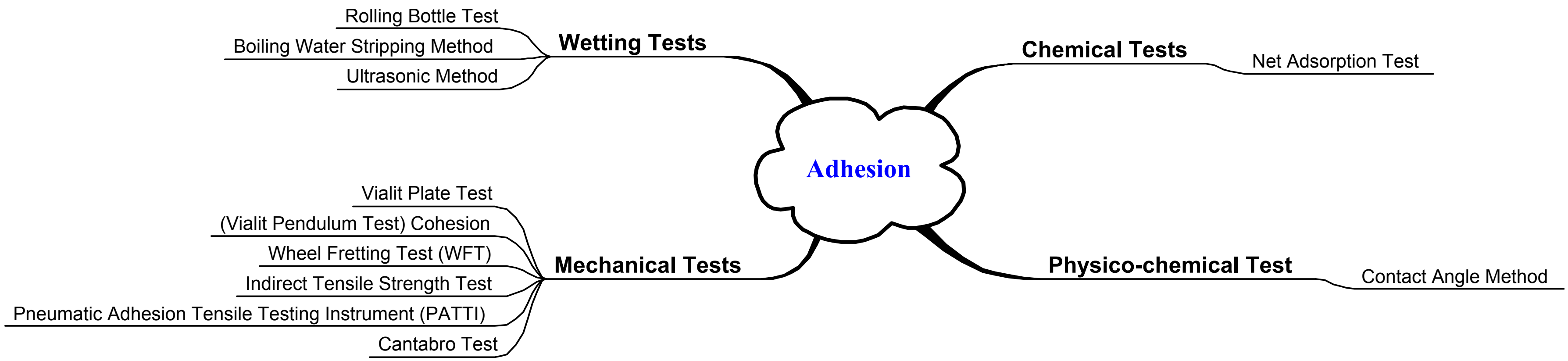
C.1 Bitumen Test Relationships



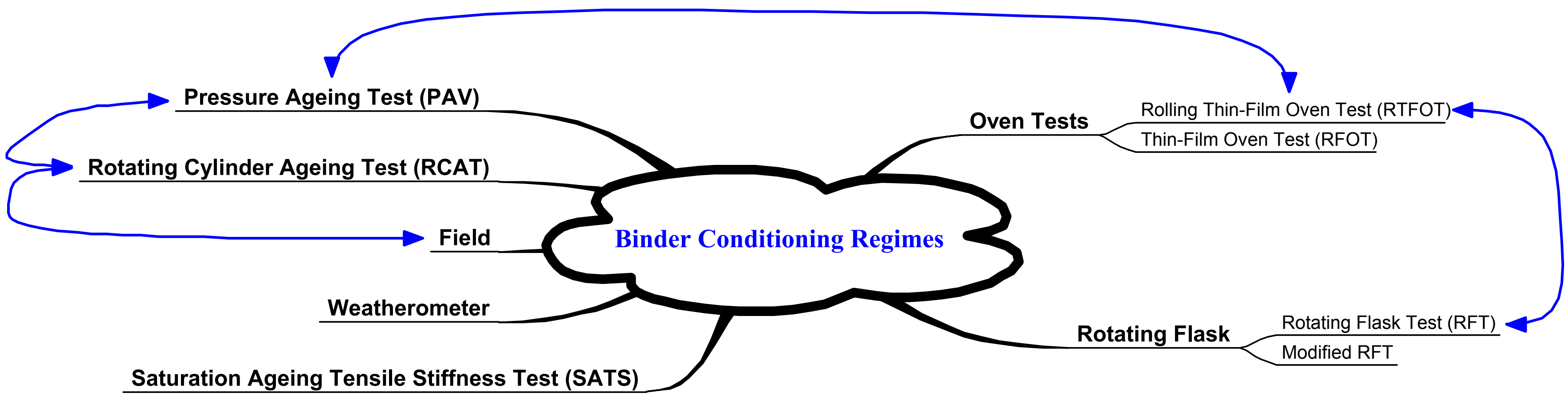
C.2 Asphalt Test Relationships



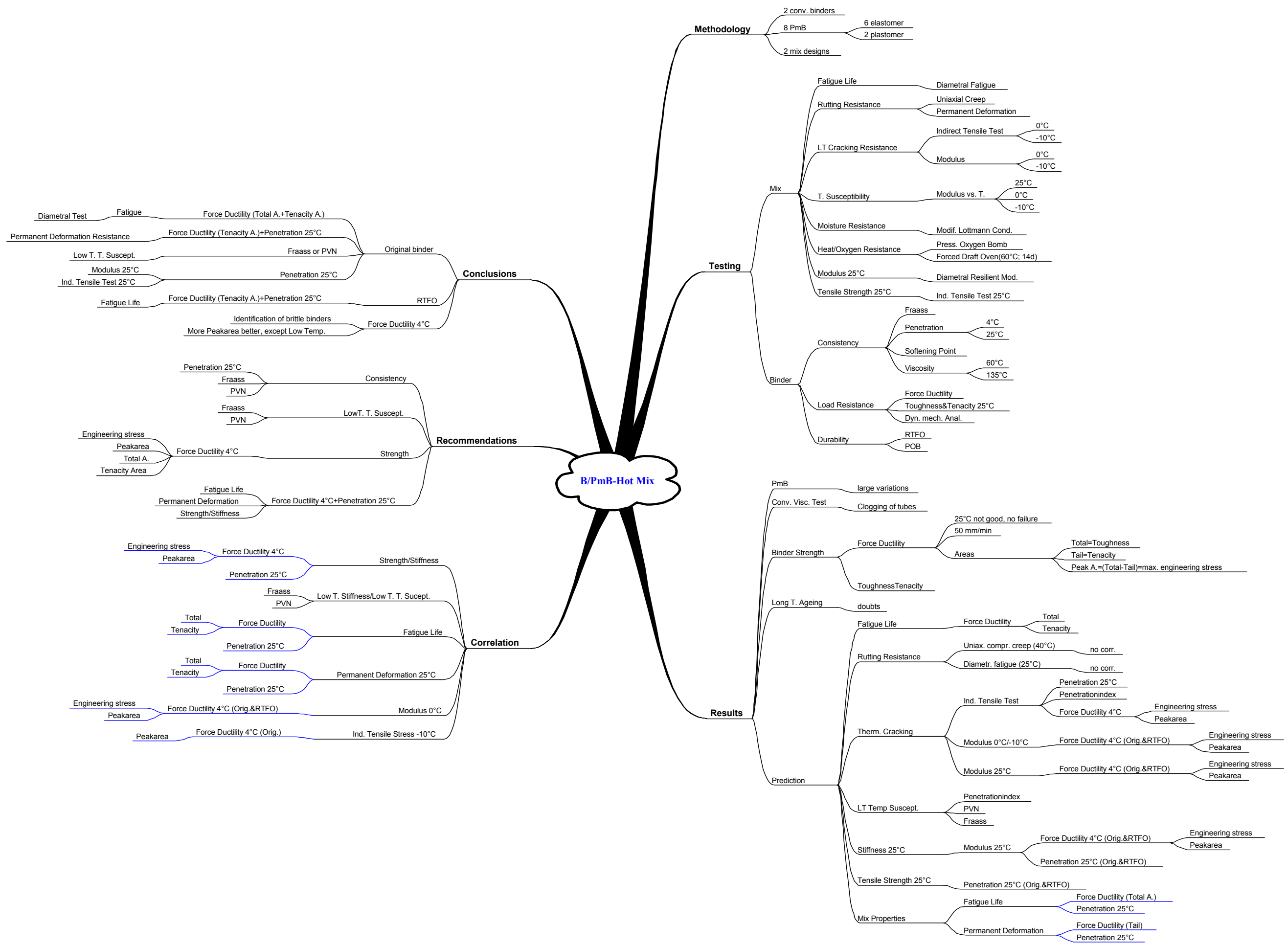
C.3 Adhesion Relationships



C.4 Binder Conditioning Relationships



C.5 Overall Analysis



Annex D: BiTVal Proformas

D.1 Conference Proforma

REVIEWER
Name:..
Affiliation:..

REFERENCE
Title:......................
Authors:.................
Source:..................

Binder properties

Elevated service temperature properties

Complex modulus	DSR	
	other	
Dynamic viscosity	Cone&Plate	
	Coaxial cylinders	
	Capillary viscosimeter	
	other	
Zero Shear Viscosity	Oscillation method	
	Creep method	
	other	
Softening point	R&B	
Creep stiffness	Repeated Creep Test	

Intermediate and/or low service temperature properties

Complex modulus	DSR	
	other	
Penetration	Penetration	
Low temperature stiffness	BBR	
	Direct Tensile Test	
	other	
Cohesion	Force ductility	
	Direct Tensile Test	
	Vialit Pendulum Test	
	Fracture toughness test	
	other	
Fatigue	Binder fatigue test	
	other	

Ageing/Wheathering

short term ageing	RTFOT	
	TFOT	
	RFT	
	Modified German RFT	
	other	
long term ageing	PAV	
	RCAT	
	Modified German RFT	
	other	
wheathering	wheatherometer	

State binder

Pure	
Modified	
Unaged	
Short term aged	
Long term aged	
Recovered	

Mixture properties

Elevated service temperature properties

Stiffness	Stiffness test	
Permanent deformation	Wheel tracking test	
	Cyclic compression test	
	other	

Intermediate and/or low service temperature properties

Stiffness	Stiffness test	
Strength	Indirect tensile test	
	Direct tensile test	
	other	
Low temperature cracking	Thermal stress restrained specimen test	
	other	
Fatigue cracking	Fatigue test	
Adhesion	Aggregate/Binder affinity	
	Particle loss of Porous Asphalt	
	other	

Correlations

Binder/Mix	
Binder/Field	
Mix/Field	

Relevance

High	
Moderate	

Comments:

Abstract:

D.2 Research Proforma

BiTVal Project - Questionnaire on Ongoing Research Projects

Organisations involved	
Project name	

Country (ies)	
Contact points	

PROJECT CONTENT

Binder properties evaluated

Elevated service temperature properties

Complex modulus	DSR	
	other	
Dynamic viscosity	Cone&Plate	
	Coaxial cylinders	
	Capillary viscosimeter	
	other	
Zero Shear Viscosity	Oscillation method	
	Creep method	
	other	
Softening point	R&B	
Creep stiffness	Repeated Creep Test	

Ageing/Wheathering

short term ageing	RTFOT	
	TFOT	
	RFT	
	Modified German RFT	
	other	
long term ageing	PAV	
	RCAT	
	Modified German RFT	
	other	
wheathering	wheatherometer	

State binder

Pure	
Modified	
Unaged	
Short term aged	
Long term aged	
Recovered	

Intermediate and/or low service temperature properties

Complex modulus	DSR	
	other	
Penetration	Penetration	
Low temperature stiffness	BBR	
	Direct Tensile Test	
	other	
Cohesion	Force ductility	
	Direct Tensile Test	
	Vialit Pendulum Test	
	Fracture toughness test	
	other	
Fatigue	Binder fatigue test	
	other	

Mixture properties evaluated

Elevated service temperature properties

Stiffness	Stiffness test	
Permanent deformation	Wheel tracking test	
	Cyclic compression test	
	other	

Intermediate and/or low service temperature properties

Stiffness	Stiffness test	
Strength	Indirect tensile test	
	Direct tensile test	
	other	
Low temperature cracking	Thermal stress restrained specimen test	
	other	
Fatigue cracking	Fatigue test	
Adhesion	Aggregate/Binder affinity	
	Particle loss of Porous Asphalt	
	other	

Study of correlations

Binder/Mix	
Binder/Field	
Mix/Field	

PROJECT DESCRIPTION

Project purpose and short description	
Expected deliverables and publications	
Project timing **Starting date** **Expected ending date**	
Status of progress so far	
Summary of main findings so far	
Other information on the project	

Report prepared by: **Date:**

Annex E: Literature Included in Database

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