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Performance Specified Surface Dressing for Trunk Roads

Sub-task 1
Specification for Highway Works Clause 955 and NG 955

Report

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955 Ageing Profile Test Using a Modified Rolling Thin Film Oven Test (MRTFOT)

Scope

1 (xx/13) This Clause specifies the procedure for obtaining samples of 'Aged Binder' after various stages of conditioning. In this way, a plot of binder properties providing an 'Ageing Profile' may be accomplished in a short time using the Modified Rolling Thin Film Oven Test (MRTFOT) to simulate ageing in road materials over many years in service.

The MRTFOT utilises the Rolling Thin Film Oven (ASTM 2872) specified in BS EN 12607-1.

The 'Ageing Profile' is generated by determining the rheological properties of complex shear (stiffness) modulus G* and phase angle δ of samples taken from the oven at intervals throughout the test period, typically one day. Other properties such as durability of cohesion may also be assessed.

Three protocols are used depending on the type of binder and its use:

(i) a method to simulate ageing of polymer modified binder or paving grade bitumen used in the manufacture of asphalt;
(ii) a method to remove the water phase and volatile flux from polymer modified bituminous emulsions and simulate ageing and;
(iii) a method to test the potential for preservatives to reduce the ageing rate of bitumen.

The 'Ageing Profile' in terms of rheological characteristics provides 'Product Identification' and this is essential to ensure consistency and to enable proprietary binders to be identified.

Warning

2 The use of this test method can involve hazardous materials, operations and equipment. This Clause does not purport to address all of the safety problems associated with the use of the test method. It is the responsibility of the user of these test methods to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

Asphalt Binders

3 Binders used to manufacture hot mixed asphalt (asphalt binders) are first subjected to a 'Short-term Ageing Test' to simulate the effects of manufacture, transport and laying. In this respect, the test described in this Clause has been shown to be equivalent to BS EN 12607-1. The binder is continually stirred by rotating stainless steel screws in sample cans to obtain a homogeneous sample; the procedure also benefits from a faster oxidation rate (rapid ageing), because of the increased exposure to the jetted air stream and, therefore, a shorter test duration.

The binder samples are then aged at a lower temperature and samples removed at intervals and tested to obtain an 'Ageing Profile'. This is particularly useful for asphalt, because different air void contents and binder contents in a road pavement result in different ageing rates.

The 'Long-term Aged' binder sample, where the characteristics are equivalent to those obtained after 65 hours using the Pressure Ageing Vessel PAV85 Test, is obtained after 7 hours; therefore, the test may be carried out in one day. PAV85 is detailed in BS EN 14769.

Binders used for manufacturing 'Warm Asphalt' are mixed at much lower temperatures than for hot mixed asphalt and they do not need the same high temperature protocol for 'Short-term Ageing'; this part of the procedure is therefore amended for these binders. All the ageing is done at the lower temperature.

Bituminous Emulsion Binders

4 Bitumen and polymer modified bituminous emulsions are first subjected to a 'Rapid Recovery Test' which removes any water phase and volatile oils to produce a 'Recovered Binder'. The 'Recovered Binder' has similar properties to that produced from other evaporative tests, but is achieved over a shorter

period and the use of nitrogen, replacing air, reduces risks associated with volatile oils in the oven. The 'Recovered Binder' in rheological terms is equivalent to that recovered using BS EN 13074-1.

The 'Ageing Profile' is provided by continuing the ageing of the 'Recovered Binder' in the MRTFO at a higher temperature, replacing the nitrogen gas with air and removing samples after specified periods for testing. A 'Stabilised Binder', equivalent in rheological terms to that produced using BS EN 13074-2, may be obtained from the 'Ageing Profile' Plot for a particular emulsion. Fluxed and vegetal fluxed binders where volatile oils are thought to be present may also be tested using this protocol.

Preservatives

5 40/60 penetration grade bitumen, used as a control, is subjected to 'Short-term Ageing' in the MRTFOT followed by an extended period at a lower temperature to simulate the ageing of the bitumen in a road pavement. This aged bitumen control is then cooled to ambient and treated with the preservative. Most of the volatile oils are then removed by conditioning the samples in the MRTFO using a nitrogen blanket. The aged bitumen, treated by preservative, is then subjected to further ageing with the MRTFOT. The properties of the aged bitumen, after action of the preservative, are assessed by an 'Ageing Profile' using rheology and compared with the 'Ageing Profile' of the control bitumen.

Definitions

6 For the purposes of the procedures specified in this Clause the following definitions apply:

(i) **Laboratory Sample:** sample of bituminous binder intended for laboratory tests. It may be a spot sample, a composite sample or a part thereof (a divided sample) as described in BS EN 58.

(ii) **Test Sample:** sample of bituminous binder produced by treatment or subdivision of a laboratory sample for individual test (BS EN 58).

(iii) **Recovered Binder:** material remaining after removal of water or flux from a polymer modified or unmodified bituminous emulsion or fluxed binder in the presence of nitrogen, equivalent to the recovered binder using BS EN 13074-1.

(iv) **Aged Binder:** material remaining after the Ageing protocol.

(v) **Short-term Aged Binder:** material obtained during the MRTFOT which is equivalent to that provided by BS EN 12607-1(RTFOT)

(vi) **Long-term Aged Binder:** material obtained during the MRTFOT which is equivalent to that provided by BS EN 14769

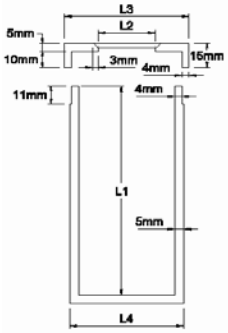
(vii) **Ageing Profile:** graphical plot of performance-related results from samples tested during the ageing protocol of this method. Performance-related testing is carried out using a Dynamic Shear Rheometer (DSR), providing G* and phase angle data at a determined temperature and frequency.

Test Apparatus (common to all protocols)

7 The following test apparatus shall be used:

(i) RTFOT apparatus to BS EN 12607-1.

(ii) Eight identifiable sample cans with threaded screw top lids (or other fixing system) manufactured from aluminium and coated on all internal surfaces with polytetrafluoroethylene (PTFE) with internal dimensions and aperture diameter (L2) as shown below:



Bottle dimensions	
L1	129 ± 1.2 mm
L2	31.75 ± 1.2 mm
L3	70 ± 1.2 mm
L4	64.0 ± 1.2 mm
Note: All other dimensions are approximate.	

The taper in the throat (from L2 aperture to lid top) is 45 ± 5°

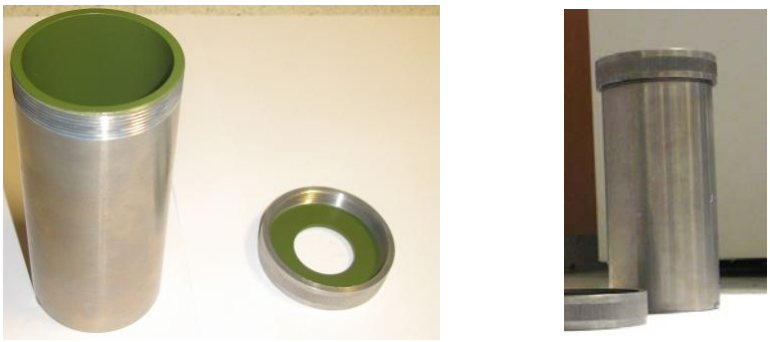
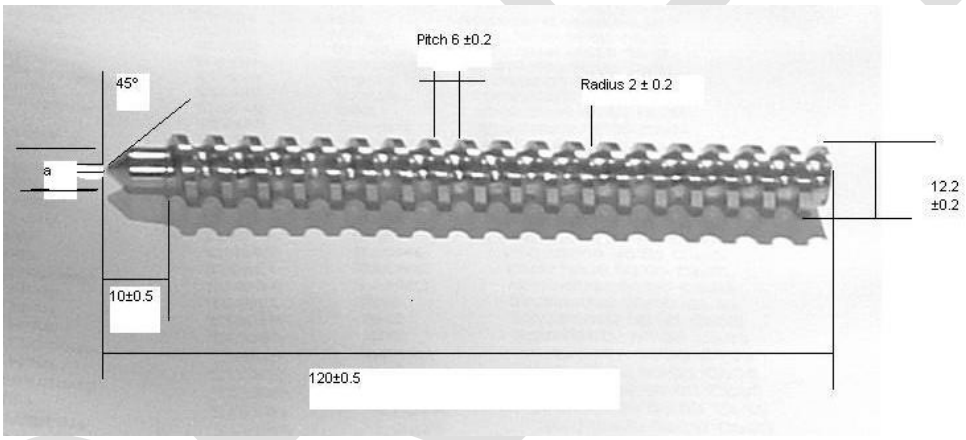


Figure 9 /1: Sample Can and Lid (both with threads)

(iii) Eight screws manufactured from high quality stainless steel (surgical quality) complying with BS EN 10088-3, designation 1.4404 to a 'fine machined finish'. All screws shall have dimensions: diameter 12.2 ± 0.2mm, length 120 ± 0.5mm and have between 18 and 19 turns with a pitch of 6 ± 0.2mm. The depth of cut shall be 2 ± 0.2mm with a semi-circular profile – see **Figure 9/2**. At one end, the screw shall have an unthreaded shank 10 ±0.5mm long and 7.5 ± 0.5mm diameter, ending in a 45° cone, truncated to give a flat end at the apex 1.5 to 2.0mm diameter. The minimum weight of a screw shall be 70g. The direction of screw shall be such that the binder sample is drawn to the closed end of the sample can bottle when the can containing the screw is rotated in the carousel. If the RTFO carousel rotates anticlockwise, viewed from the front, screws with a right-hand thread are required and if the carousel rotates clockwise, screws with a left hand thread are required.



Dimension a = 1.5mm to 2mm

Figure 9/2 Screw with Right-handed Thread (for anti-clockwise rotating carousel)

(iv) A spatula for removing the binder from the sample cans (a flat blade with a square end has been found suitable).
(v) A balance accurate to 0.05g.
(vi) A timer capable of timing 100 minutes, accurate to 1 second in five minutes and, for the MRTFOT, a timer capable of timing 25 hours to the same accuracy.
(vii) Nitrogen gas supply (for Rapid Recovery Test and preservative test).
(viii) Air supply, clean dry air (for MRTFOT).
(viiii) An oven to pre-heat the sample cans and the binder to the required temperature.

8 Principle of the Ageing Profile Test for Polymer Modified and Unmodified Bituminous Binders for Asphalts

A thin film of binder is rotated in aluminium sample cans with screw top lids, both coated internally with PTFE (replacing glass bottles), using the Rolling Thin Film Oven Test (RTFOT) apparatus, as described in BS EN 12607-1. Special screws are used to disturb the binder and maintain a homogenous material during 'Ageing'. Air is jetted over the film of binder for a much longer period than in the conventional test and a lower temperature used in order to simulate the 'ageing' of the binder in the road after application. The screws accelerate the process, and are necessary to maintain a homogeneous sample,

especially when testing polymer modified binders, which tend to form a skin and may phase separate in static ageing tests.

The initial conditioning test is to simulate the changes to the properties of the binder caused by the high temperatures during manufacture, transport and laying, termed 'Short-term Ageing'.

Further ageing is carried out at a reduced temperature and samples removed after set periods and tested, so that an 'Ageing Profile' may be plotted.

Test Procedure for Determining the Ageing Profile of Hot Mixed Polymer Modified and Unmodified Bituminous Binders for Asphalts

9 Obtain a sample in accordance with BS EN 58. Stir the sample thoroughly to ensure it is completely homogenized. If sub-samples are taken these shall have a minimum volume of 250ml. Ensure that the RTFO is level so that the horizontal axes of the sample cans when in position in the carousel are level. Pre-heat the oven to 163 ± 1°C, 1h is generally sufficient. For binders used to manufacture warm mixed asphalts, substitute 135°C for 163°C throughout the test procedure.

Insert a stainless steel screw into each of the sample cans with the tapered end of the screw towards the aperture of the container and record the mass of each assembly to 0.1g. Heat the sample cans and the stainless steel screws to 163 ± 1°C for 90 ± 10 min.

Heat the sample to 170 ± 3°C. Thoroughly stir the sample to ensure homogeneity immediately prior to decanting into the sample cans.

Pour 19.0 ± 0.5g of the sample into a preheated sample can and record the mass with lid. Immediately after weighing, roll the sample cans on the bench to ensure distribution of the binder on the walls of the can.

Mount the sample can in the carousel of the RTFO. Repeat for the remaining sample cans. Close the door of the RTFO between mounting each sample can in the carousel. The period between weighing the first sample can with binder and mounting of all the sample cans and the start of the rotation of the carousel shall not exceed 20 minutes.

When the carousel is fully loaded, rotate the carriage assembly at a speed of 15.0 ± 0.2min^{-1}. Start the flow of air at a set rate of 4000 ± 200ml/min. Start the timer. Maintain the samples in the oven and the air flowing for 45 ± 1min from the time the test temperature reaches 163 ± 1°C.

If the test temperature of 163°C is not reached within the first 10 minutes, discontinue the test.

After 45 ± 1min, sample cans (minimum 2) may be removed for testing and to determine mass loss, if required. This is the Short-term Aged binder, equivalent to RTFOT, BS EN 12607-1.

To continue the Ageing Profile test, maintain the temperature at 163°C for a further 15 minutes, with the carousel rotating in the air flow, then reduce the temperature of the oven to 135 ± 1°C. Note the time the RTFO takes to reach 135°C. Once the temperature has been achieved, continue to rotate the samples in the air supply and remove sample cans to obtain samples of aged binder as required for testing after three periods:
2h ± 10min; termed Aged$_2$ Binder
4h ± 10min; termed Aged$_4$ Binder
6h ± 10min; termed Aged$_6$ Binder: this is the Long-term Aged Binder, equivalent to PAV85.
Other periods may be used either for convenience or to allow other degrees of ageing to be investigated.

Table 9/3 Summary of sampling times					
Stage	Gas	RTFO Temperature	Time	Sampling	Cumulative time
Ageing	Air	163°C	45min	RTFOT Equivalent	45mins
	Air	163°C	15min	None	1h 00min
	Air	135°C	2h	$Aged_2$ test samples	3h 00min
	Air	135°C	4h	$Aged_4$ test samples	5h 00min
	Air	135°C	6h	$Aged_6$ test samples (equivalent to PAV85)	7h 00min

For binders used to manufacture 'Warm Asphalt', 'Short Term Ageing' at 163°C is not required, therefore, replace 163ºC by 135ºC in Table 9/3 and report in addition $Aged_1$ result (ageing at 135°C after a cumulative time of 1h ± 5min).

Handling of the Aged Binder

10 If the binder is to be tested immediately, remove a sample can from the carousel, extract the screw using a small spatula and set the screw aside for cleaning. Place the sample can(s) in an oven preheated to the softening point of the binder plus 100°C (if known), or 170°C if the properties of the binder are unknown, for 20 ± 5min.

Place the containers on their sides in an oven. This allows the binder to drain to one side of the container, which is more easily removed with the spatula.

At the end of 20 ± 5min, remove the sample can from the oven, unscrew the lid and, using the spatula, scrape the aged binder from the sample can and prepare the test samples in accordance with the relevant test method. Warming the spatula to the same temperature as the binder facilitates removal of the binder.

If the aged binder from more than one sample can is needed to produce a test sample, combine the aged binder from the sample cans and re-heat. Re-heating for testing shall be in accordance with BS EN 12594, ensuring that aged binder is heated for no longer than is absolutely necessary. The binder shall be thoroughly homogenized by stirring after heating and before being used for testing, while taking care not to entrain any air.

If the binder is not to be tested immediately, remove a sample can from the RTFO, extract the screw using a small spatula and set the screw aside for cleaning. Unscrew the lid and, using the spatula, scrape the aged binder from the sample can and place on a silicon sheet. The binder shall be sealed and stored at between 0°C to 5°C. The sample shall be re-heated in accordance with BS EN 12594 and homogenised immediately before testing.

If the aged samples are to be transferred to another laboratory for testing, they shall not be subjected to temperatures greater than ambient after ageing and before testing, and the delay before testing shall not exceed 120 hours.

The thermal history of the sample shall be recorded.

Principle of the Ageing Profile Test for Polymer Modified or Unmodified Bituminous Emulsions or Fluxed Binders

11 A thin film of binder is rotated in aluminium sample cans with screw top lids, both coated internally with PTFE (replacing glass bottles), using the RTFOT apparatus, as described in BS EN 12607-1, to evaporate water from bituminous emulsions and/or the light solvent or highly volatile fraction from fluxed or other binders. Special screws are used to disturb the binder and maintain a homogenous material during breaking and/or curing. Nitrogen gas instead of air is jetted over the film of bituminous binder at a lower temperature in order to minimize ageing effects and simulate the condition of the binder soon after application. The first part of the process is carried out with the RTFO set at 85°C to prevent frothing of the binder. The temperature is then increased to 135°C to drive off any remaining water and/or solvent to provide the 'Recovered Binder', which is equivalent to the recovered binder obtained from BS EN 13074-1. The 'Ageing Profile' is provided by continuing the ageing of the binder in the RTFO at 135°C, replacing the nitrogen gas with air and removing samples after specified periods for testing.

12 Obtain a sample in accordance with BS EN 58. Stir the sample thoroughly to ensure it is completely homogenized. The temperature of the binder sample shall not be less than 70% of the normal application temperature in degrees Celsius and shall be recorded (for example, high binder content surface dressing emulsion may require a minimum of 60°C, or a polymer modified fluxed binder, 130°C). The sample history if known shall be recorded; this is important, as some ageing may have occurred. If sub-samples are taken these shall have a minimum volume of 250ml. Ensure that the oven is level so that the horizontal axes of the sample cans, when in position in the carriage, are level. Pre-heat the oven to 85 ± 1°C, 1h is generally sufficient.

Insert a stainless steel screw into each of the sample cans with the tapered end of the screw towards the aperture of the container and record the mass of each assembly to 0.1g. Heat the sample cans, lids and the stainless steel screws to 85 ± 1°C for 90 ± 10min.

Heat the sample to the application temperature of the binder, or, if this is not known, to 60 ± 3°C. Thoroughly stir the sample to ensure homogeneity immediately prior to decanting into the sample cans, taking care to minimise loss of water content and/or any volatile oil. Some cut-back binders and other binders such as primers having low flash points are not suitable for this test. If there is any doubt concerning the volatile nature of the sample for test, a suitable flash point test must be carried out before proceeding with the 'Ageing Profile test' or heating to 60°C.

Pour 19.0 ± 0.5g of the sample into a preheated sample can and record the mass. Immediately after weighing, roll the containers on the bench to ensure distribution of the binder round the bottle.

Mount the sample can in the carousel of the RTFO. Repeat for the remaining sample cans. Close the door of the RTFO between mounting each sample can in the carousel. The period between weighing the first sample can with binder and mounting of all the sample cans and the start of the rotation of the carousel shall not exceed 20 minutes.

When the carousel is fully loaded, rotate the carriage assembly at a speed of 15.0 ± 0.2min^{-1}. Start the flow of nitrogen at a set rate of 4000 ± 200ml/min. Start the timer. Maintain the samples in the oven and the nitrogen flowing for 75 ± 1min from the time the test temperature reaches 85°C. At this stage there may still be residual water in emulsion binders, especially those with high polymer contents.

If the test temperature of 85°C is not reached within the first 10 minutes, discontinue the test.

Increase the temperature of the oven to 135 ± 1°C. Note the time the RTFO takes to reach 135°C. Once the temperature has been achieved, continue to rotate the carousel for a further 30 ± 1min at 135°C. At the end of the 30minutes, turn off the nitrogen. If required, sample cans may be removed and the samples may be used for testing or determination of mass loss. This is the 'Recovered Binder' and is equivalent to that obtained using BS EN 13074-1.

The Ageing Profile can be determined by maintaining the RTFO at 135°C and switching to an air supply set at 4000 ± 200ml/min.

Rotate the samples in the air supply and remove sample cans to obtain samples of aged binder as required for testing after three periods:

3h ± 10min; termed $Aged_3$ Binder
5h ± 10min; termed $Aged_5$ Binder, and
22h ± 10min, termed $Aged_{22}$ Binder

Other periods may be used either for convenience or to allow different degrees of ageing to be investigated. Long-term Ageing equivalent to PAV85 has been found to occur at 22 hours. It takes longer to achieve than with asphalt binder, because there is no short-term ageing at 163°C. The PAV85 ageing period is not thought to be relevant for emulsion binders used in most road applications. If samples at 22 hours are not required to simulate PAV85, a combined sample from three sample cans may be useful at 3 and 5 hours, or another aged sample from two cans, say at 4 hours, may be obtained for test to enhance the 'Ageing Profile'

Table 9/4 Summary of sampling times					
Stage	Gas	RTFO Temperature	Time	Sampling	Cumulative time
Recovery	Nitrogen	85°C	75±1min	none	1h 15min
	Nitrogen	Ramp to 135°C	20min approx	none	1h 35min
	Nitrogen	135°C	30±1min	'Recovered binder' test samples	2h 05min
Ageing	Air	135°C	3h	$Aged_3$ test samples	5h 05min
	Air	135°C	5h	$Aged_5$ test samples	7h 05min
	Air	135°C	22h (optional)	$Aged_{22}$ test samples	24h 05min

Handling of the Recovered Binder

13 If the binder is to be tested immediately, remove a sample can from the carousel, extract the screw using a small spatula and set the screw aside for cleaning. Immediately transfer the sample can (s) to an oven and heat until at the required test temperature. Place the containers on their sides in an oven. This allows the binder to drain to one side of the container, which is more easily removed with the spatula.

Unscrew the lid of the sample can and, using the spatula, scrape the recovered binder from the sample can and place in the test apparatus. Warming the spatula to 135°C facilitates removal of the binder.

If the recovered binder from more than one sample can is needed to produce a test sample, combine the recovered binder from the sample cans and re-heat. Re-heating for testing shall be in accordance with BS EN 12594, ensuring that recovered binder is heated for no longer than is absolutely necessary. The recovered binder shall be thoroughly homogenized by stirring after heating and before being used for testing, while taking care not to entrain any air.

If the recovered binder is not to be tested immediately, remove a sample can from the RTFO, extract the screw using a small spatula and set the screw aside for cleaning. Unscrew the lid and, using the spatula, scrape the recovered binder from the sample can and place on a silicon sheet. The binder shall be sealed and stored at between 0°C to 5°C. The sample shall be re-heated in accordance with BS EN 12594 and homogenised immediately before testing.

If the recovered samples are to be transferred to another laboratory for testing, they shall not be subjected to temperatures greater than ambient after recovery and before testing, and the delay before testing shall not exceed 120 hours.

The thermal history of the sample shall be recorded.

Handling of the Aged Binder

14 If the binder is to be tested immediately, remove a sample can from the carousel, extract the screw using a small spatula and set the screw aside for cleaning. Place the sample can (s) in an oven preheated to the softening point of the binder plus 100°C (if known), or 170°C if the properties of the binder are unknown, for 20 ± 5min. Place the sample cans on their sides in an oven. This allows the binder to drain to one side of the sample can, which is more easily removed with the spatula.

At the end of 20 ± 5min, remove the sample can from the oven, unscrew the lid and, using the spatula, scrape the aged binder from the sample can and prepare the test samples in accordance with the relevant test method. Warming the spatula to the same temperature as the binder facilitates removal of the binder.

If the aged binder from more than one sample can is needed to produce a test sample, combine the aged binder from the containers and re-heat. Re-heating for testing shall be in accordance with BS EN 12594, ensuring that aged binder is heated for no longer than is absolutely necessary. The binder shall be thoroughly homogenized by stirring after heating and before being used for testing, while taking care not to entrain any air.

If the binder is not to be tested immediately, remove a sample can from the RTFO, extract the screw using a small spatula, and set the screw aside for cleaning. Unscrew the lid and, using the spatula, scrape the

aged binder from the sample can and place on a silicon sheet. The binder shall be sealed and stored at between 0°C to 5°C. The sample shall be re-heated in accordance with BS EN 12594 and homogenised immediately before testing.

If the aged samples are to be transferred to another laboratory for testing, they shall not be subjected to temperatures greater than ambient after ageing and before testing, and the delay before testing shall not exceed 120 hours.

The thermal history of the sample shall be recorded.

Principle of Assessing the Action of Preservatives using the MRTFOT

15 A thin film of 40/60 penetration grade binder is rotated in aluminium sample cans, coated internally with PTFE (replacing glass bottles), using the Rolling Thin Film Oven Test (RTFOT) apparatus, as described in BS EN 12607-1. Special screws are used to disturb the binder and maintain a homogenous material during conditioning. The bitumen is subjected to 'Short-term Ageing' followed by an extended period in the RTFO to simulate the ageing of the bitumen in a road pavement. The properties of the 'Long-term Aged' bitumen may be determined at this stage. The aged bitumen control in the remaining samples is then cooled to ambient, treated with the preservative and left for 24 hours. Most of the remaining highly volatile oils are subsequently removed by conditioning the samples in the RTFO using a nitrogen blanket. The aged bitumen, treated by preservative, is then subjected to further ageing with the Modified RTFO. The properties of the aged bitumen, after action of the preservative, are assessed by an 'Ageing Profile' test, using rheology values of complex shear (stiffness) modulus G* and phase angle δ, in accordance with BS EN 14770. Cohesion durability may also be determined.

Test Procedure for Determining the 'Ageing Profile' of Aged Paving Grade Bitumen as a Control Treated with a Preservative

16 Obtain a sample of 40/60 paving grade binder in accordance with BS EN 58. Stir the sample thoroughly to ensure it is completely homogenized. If sub-samples are taken these shall have a minimum volume of 250ml. Ensure that the RTFO is level so that the horizontal axes of the sample cans when in position in the carousel are level. Pre-heat the oven to 163 ± 1°C, 1h is generally sufficient.

Insert a weighed stainless steel screw into each of the sample cans with the tapered end of the screw towards the aperture of the can lid; record the mass of each assembly to 0.1g. Heat the sample cans, lids and the stainless steel screws to 163 ± 1°C for 90 ± 10min.

Heat the sample to the maximum storage temperature of the binder. Thoroughly stir the sample to ensure homogeneity immediately prior to decanting into the sample cans.

Pour 19.0 ± 0.5g of the sample into a preheated sample can and record the mass. Immediately after weighing, roll the sample can on the bench to ensure distribution of the binder round the walls of the can.

Mount the sample can in the carousel of the RTFO. Repeat for the remaining sample cans. Close the door of the RTFO between mounting each sample can in the carousel. The period between weighing the first sample can with binder and mounting of all the sample cans and the start of the rotation of the carousel shall not exceed 20 minutes.

When the carousel is fully loaded, rotate the carriage assembly at a speed of 15.0 ± 0.2min^{-1}. Start the flow of air at a set rate of 4000 ± 200ml/min. Start the timer. Maintain the samples in the oven and the air flowing for 45 ± 1min from the time the test temperature reaches 163°C.

If the test temperature of 163°C is not reached within the first 10minutes, discontinue the test.

After 45± 1min at 163 ± 1°C, sample cans (minimum 2) shall be removed for testing and to determine mass loss. This is the 'Short-term Aged Binder'.

Reduce the temperature of the oven to 135 ± 1°C. Note the time the RTFO takes to reach 135°C. Once this temperature has been achieved, continue to rotate the samples in the air supply for 240 ± 10min. At the end of this period, remove the sample cans from the RTFO and determine their mass loss.

Remove the screws and weigh to determine the mass of binder retained on each identified screw.

A quantity of binder shall be removed from each sample can, which shall be:

8 ± 1g, less the mass of binder retained on the particular screw removed.

It may be necessary to warm the sample cans to facilitate the removal of the screws and binder. After removing the binder, lay the sample cans on their sides and allow them to cool to ambient temperature.

Add 10 ± 0.5g of preservative to each sample container and swill the preservative around the sample container for approximately 15 seconds. Determine the mass of each sample can.

The sample cans with the binder and preservative are stored on their sides, with the pooled binder at the bottom, in a ventilated fume cupboard at ambient temperature for 24 hours. This process allows any very low flashpoint volatile oil to safely evaporate and consequently reduce the amount of hydrocarbon solvent within the sample can.

Pre-heat the oven to 50 ± 1°C, and transfer the sample cans from the fume cupboard to the RTFO carousel. Start the carousel and jet nitrogen at 4000 ± 200ml/min over the sample cans for 120 ± 10min.

Identify and weigh eight clean screws and heat the screws to 135°C.

After 2 hours, the RTFO temperature is raised from 50°C to 135°C and air jetted at 4000 ± 200ml/min substituted for the nitrogen supply. Note the time when the oven achieves 135°C.

When the RTFO has reached 135°C, stop the carousel and add a preheated screw to each sample can, with the tapered end of the screw towards the aperture/lid of the sample can (and noting which screw is added to which sample can). Restart the carousel and continue the conditioning of the samples at 135°C in air jetted at 4000 ± 200ml/min for 120 ± 10 min.

Remove the sample cans from the RTFO to obtain samples of aged binder as required for testing and determine the mass of each sample can.

The control bitumen may be substituted with a binder recovered from a road or from a laboratory aged sample, in which case the ‘Short-term Ageing’ and subsequent ageing protocol are not carried out, because the binder will already have been aged and heated. In this case, the recovered binder sample is heated in accordance with BS EN 12594 and 11 ± 0.5g added to heated cans (135°C) which are then rolled to achieve a film around the walls. After this, they are cooled, 10 ± 0.5g preservative added and the preservative treated samples subjected to the same Ageing Profile protocol as described above.

Handling of the Preservative Treated Aged Bitumen

17 If the binder is to be tested immediately, remove a container from the carousel, extract the screw using a small spatula and set the screw aside for cleaning. Place the sample can (s) in an oven preheated to 140°C for 20 ± 5min. Place the containers on their sides in the oven. This allows the binder to drain to one side of the container, which is more easily removed with the spatula.

At the end of 20 ± 5min, remove the container from the oven, unscrew the lid and, using the spatula, scrape the aged binder from the container and prepare the test samples in accordance with the relevant test method. Warming the spatula to the same temperature as the binder facilitates removal of the binder.

If the aged binder from more than one container is needed to produce a test sample, combine the aged binder from the containers and re-heat. Re-heating for testing shall be in accordance with BS EN 12594, ensuring that aged binder is heated for no longer than is absolutely necessary. The binder shall be thoroughly homogenized by stirring after heating and before being used for testing, while taking care not to entrain any air.

If the binder is not to be tested immediately, remove a container from the RTFO, extract the screw using a small spatula and set the screw aside for cleaning. Unscrew the lid and, using the spatula, scrape the aged binder from the container and place on a silicon sheet. The binder shall be sealed and stored at between

0°C to 5°C. The sample shall be re-heated in accordance with BS EN 12594 and homogenised immediately before testing.

If the aged samples are to be transferred to another laboratory for testing, they shall not be subjected to temperatures greater than ambient after ageing and before testing, and the delay before testing shall not exceed 120 hours.

The thermal history of the sample shall be recorded.

Report

18 Sample description (e.g. type, age/condition and source).

The Ageing Profile test procedure adopted for the work.

The change in mass of the sample from each stage of the recovery and /or ageing protocol shall be plotted to develop an Ageing Profile for mass change.

Similarly, results of tests (for example G* using BS EN 14770), carried out after each stage of recovery and /or ageing protocol, shall be plotted to develop an Ageing Profile for that property. These results shall include:

- A plot of G* (0.4Hz) over a range of temperatures between 0°C and 60°C
- A plot of phase angle (0.4Hz) over a range of temperatures between 0°C and 60°C
- G* and phase angle at 0.4Hz and 25°C
- Temperature (0.4Hz) where the phase angle is 45 degrees (Viscous Elastic Transition (VET) temperature)
- G* (0.4Hz) at the VET temperature where phase angle is 45 degrees

For Clause 930 hard grade binder samples and other samples where they are considered to have a softening point above 60°C the temperature range shall be increased to 80°C.

The thermal history of the samples, if they are not tested immediately, shall be reported.

NG 955 Ageing Profile Test Using a Modified Rolling Thin Film Oven Test (MRTFOT)

1 The MRTFOT uses screws to continually stir and mix polymer modified bituminous binders in the sample cans so they may be aged without the problems of skinning and polymer phase separation. The air jetted into the glass bottles normally used in the RTFOT has sometimes caused polymer binders to creep out of the bottles, because of the return air flow. The MRTFOT uses the screws to pull binder to the back of the cans as they rotate in the carousel and this counteracts the air flow to the front. If the RTFO carousel rotates anticlockwise, viewed from the front, screws with a right-hand thread are required and if the carousel rotates clockwise, screws with a left hand thread are required. The screws also help in the mixing and binder film exposure accelerating oxidation. Glass bottles do not have screw tops so removal of the whole sample is difficult. To further aid the removal of binder, the MRTFOT uses PTFE coated cans which also assists with the cleaning. Glass bottles do not have precision made walls and in order for the screws to uniformly scrape the internal walls, aluminium PTFE coated cans were designed. A heavy stainless steel screw with a flat edge was developed to enhance scraping and pressure at the interface and a semi-circular thread incorporated to maximise binder movement. The design (unthreaded shank) also minimises the opportunity for the screw to worm itself out of the orifice. The stainless steel screws are manufactured from high quality surgical steel so that they are resistant to corrosion, which is particularly a problem with emulsions. The screws, produced to a 'fine machined finish', and the sample cans manufactured to the dimensions stated, with screw top lids, are all commercially available. A certificate of conformity should be obtained. It is convenient to have the PTFE coating a contrasting colour to the binder (e.g. green) so that the binder sample can be clearly identified.

2 The MRTFOT can be used as an alternative to PAV85 (BS EN 14769) as specified by HAPAS or equivalent Third Party Certification body, to evaluate 'ageing' of the binder on the road. The MRTFOT does not require a Pressure Ageing Vessel (PAV) apparatus, which is considered to be a greater safety hazard when using emulsions or fluxed binders containing highly volatile oil. In static tests such as the PAV, the trays used in BS EN 14769 (3mm thick sample) may result in skinning and heterogeneous ageing. The combination of the screw continually mobilising the binder film and the rotation of the specimen in the air stream in the MRTFOT reduces the test time whilst maintaining a homogeneous sample. This is particularly relevant for heavily polymer-modified binders and is one reason why 135ºC was selected. The protocol minimises the possible phase separation of the polymer from the base binder.

3 Binder samples may lose volatile oil, but may increase in weight due to the products of oxidation, so care should be exercised in evaluating weight changes.

4 Binder samples for test from other apparatus (e.g. recovered binders from roads) may be loaded into the sample cans without carrying out the RTFOT at 163ºC.

5 Binders that have been tested in accordance with BS EN12607-1 in a separate RTFOT apparatus using conventional glass bottles do not necessarily result in identical 'Short-term Aged Binder' after the MRTFOT. This may be the case when the binder is heavily modified, where skinning is likely to occur and the binder does not roll exposing fresh binder faces for oxidation to take place, as originally intended in the ASTM D2872 test, which was designed for unmodified bitumen.

6 The test method produces around 10g of material per sample can. This is sufficient for testing using rheology (see BS EN 14770) or Vialit Pendulum Cohesion (see BS EN 13588). For other tests, sufficient sample cans should be used and the binder combined prior to testing.

7 Polymer modified binders may need to be heated and stirred prior to testing in other apparatus to ensure that internal stresses are not present in the test specimens. This thermal history is to be reported.

Asphalt Binders

8 The Ageing Profile test provides a plot, generally of complex stiffness (shear) modulus G* and phase angle δ, using a Dynamic Shear Rheometer in accordance with BS EN 14770. The change in properties provides an indication of how the binder will age in the road pavement and a plot is much

superior to just two points: ‘Short-term Aged’ and ‘Long-term Aged’. The ‘Long-term Aged’ condition represents the near end-life of asphalt and, since most binders asymptote to similar stiffness after that amount of ageing, it does not reveal a great deal of information; conversely, early-life points can show the rate of loss of beneficial characteristics. The correlation between ageing in road pavements and laboratory ageing methods is not far advanced and this procedure is able to generate a large number of samples at a realistic price to study durability. Longer periods of test providing larger volumes of aged binder, such as generated by the RCAT, may have greater precision, but the cost of this specialist equipment and the time taken to complete the test (a couple of hundred hours), supports the argument for more rapid tests such as the Ageing Profile test to be used.

9 Hard grade bitumens to BS EN 13924 may be assessed using the Ageing Profile test. The viscous elastic transition temperature may be plotted and some correlation with cracking of both stone mastic asphalt and high modulus asphalt has been determined.

Bituminous Emulsions and Fluxed Binders

10 The initial stage of the MRTFOT is able to drive off water from polymer modified bituminous emulsions rapidly at a comparatively low temperature of 85ºC. This is because the stainless steel screws continually disturb the binder and there is a controlled nitrogen gas flow over the binder surface encouraging a homogenous break without skinning. This is important, as higher temperatures would drive off the volatile oils too quickly and/or destroy the microstructure of the binder, which would not, therefore, simulate the residual binder on the road. Some polymer microstructures are more sensitive to temperature than others and this should be considered when heating samples for subsequent testing.

11 Nitrogen gas is used, rather than air, to minimise ageing of the binder and to increase safety, especially with some emulsions (containing some very light oils) and also for fluxed binders.

12 The second stage of the test method uses nitrogen gas at 135ºC for 30 minutes (after ramping the RTFO temperature up from 85ºC to 135ºC) to remove most of the highly volatile oil left within the sample after the first stage. This process results in a ‘Recovered Binder’ equivalent to that obtained by the recovery process described in BS EN 13074-1.These recovery methods simulate the state of a binder film soon after application using conventional spraying or mixing equipment; the method is not intended to drive off all the volatile components, but to enable testing of recovered binder properties as detailed in BS EN 13808 for emulsions. The test method may also be used for conventional unmodified emulsions.

13 The ‘Recovered Binder’ from the sample cans may be conveniently scraped onto a non-adhesive sheet or dish (e.g. PTFE or silicone) in order to collect enough material for a test. Care should be taken to minimise volatile oil loss and opportunity for oxidation by ensuring a quick transfer to a suitable storage pot or test apparatus.

14 The percentage loss of weight should be recorded as an indicator of water and/or volatile oil loss and compared to the binder content of an emulsion or the percentage volatile flux.

15 Recording sample history is important as the method of sampling, whether from spraybar or storage tank or just after manufacture, may affect the properties of the binder. The sample size and its subsequent treatment in terms of, for example, re-heating, exposure to frost and regular stirring, may make a considerable difference to the result for water or volatile oil loss.

Preservatives

16 These may be solvent based materials (typically 50% solvent) with hard base binders, or sometimes resin based systems. Other components are used to improve the aged bitumen in road pavements (e.g. to replace the lost maltene phase). It is the solvent based preservatives that require careful attention to the laboratory protocol, hence the use of nitrogen and fume cupboards. The RTFOT also needs proper venting.

17 The protocol provides binder samples that may be tested using rheology to compare ageing of the control against ageing of the preservative treated aged control binder. The use of the screws ensures mixing so the improvement is the maximum potential that could possibly be expected.

18 After initial addition, preservatives provide improvement of the properties of the binder, because they soften the aged bitumen. However, after subsequent ageing, some preservatives show hardening, which, unless the treatment is repeated on a regular basis, might be detrimental to the performance of the surface of the road pavement. Data are required to correlate test results to field performance.

19 The test provides product identification and an indication of performance, so that any changes in formulation may be detected and accommodated and also consistency of supply may be established.

20 Some preservatives are emulsion systems and may be polymer modified binders. These binders improve the aged control bitumen in the test, but it has not yet been demonstrated that they can penetrate the road surfaces sufficiently to provide the same benefit in situ.

Polymer Modified and Unmodified Bituminous Binders for Asphalts – Ageing Profile Test

MRTFOT

163ºC

45min

Air jetted at 4000ml/min

Stainless Steel Screws

Weight loss reported as described in BS EN 12607-1
Sample:
'Short-Term Aged Binder'

MRTFOT

163ºC

15min

Air jetted at 4000ml/min

Stainless Steel Screws

No sampling

Transition Procedure
Cool from 163ºC to 135ºC

Ageing Profile Test

135ºC

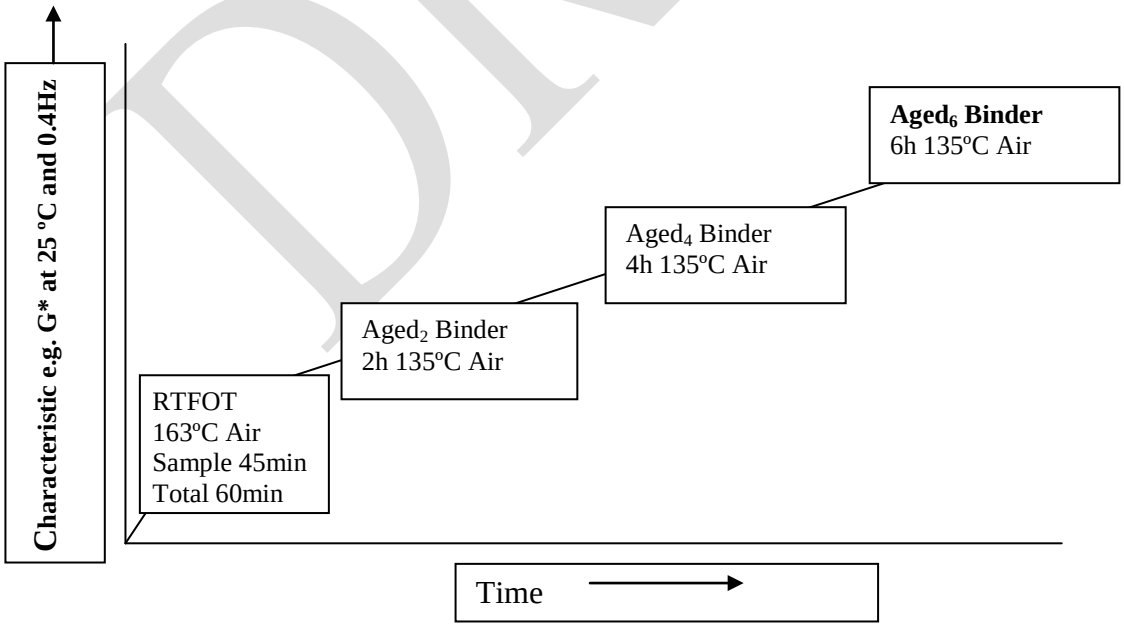
Air jetted at 4000ml/min

Stainless Steel Screws

2h Weight loss reported ($Aged_2$ Binder)

4h Weight loss reported ($Aged_4$ Binder)

6h '$Aged_6$ Binder' Weight loss reported (Sample equivalent to PAV85 'Long-term Aged Binder')



Polymer Modified and Unmodified Bituminous Emulsion and Fluxed Binder - Ageing Profile Test

MRTFOT

85ºC

75min

Nitrogen gas jetted at 4000ml/min

Stainless Steel Screws

Ramp RTFO to 135ºC approximately 20min

30min at 135ºC

'Recovered Binder' Sample

Ageing Profile Test

135ºC

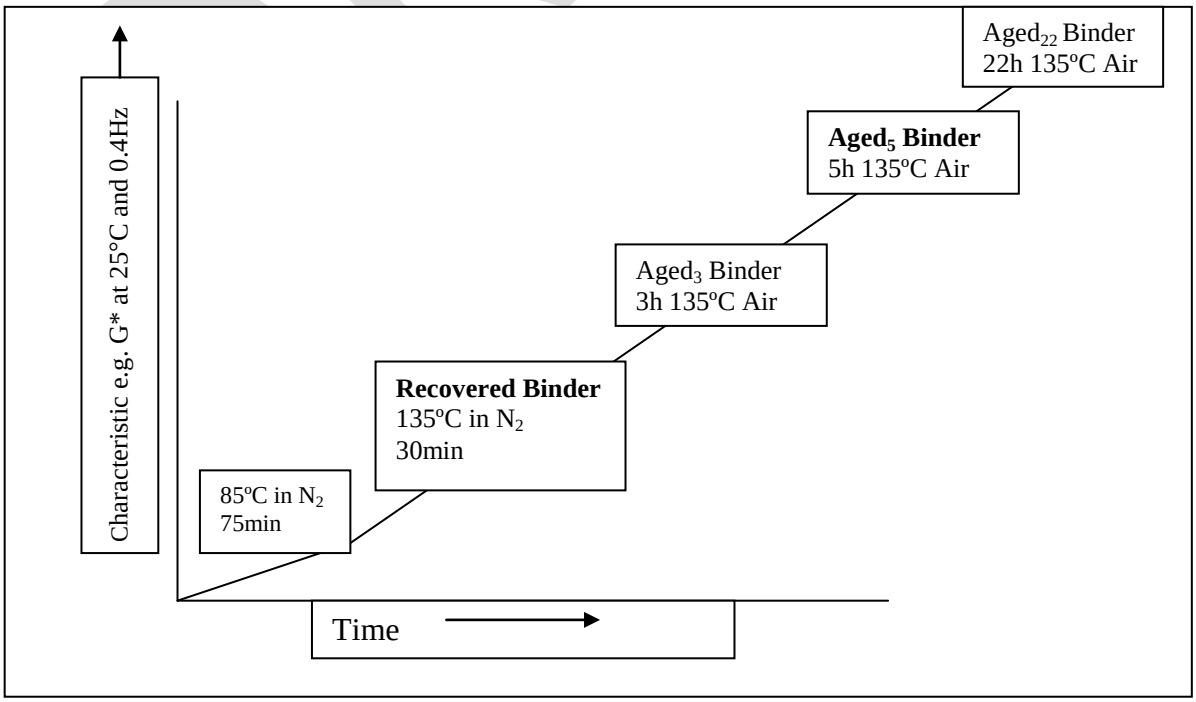
Air jetted at 4000 ml/min

Stainless Steel Screws

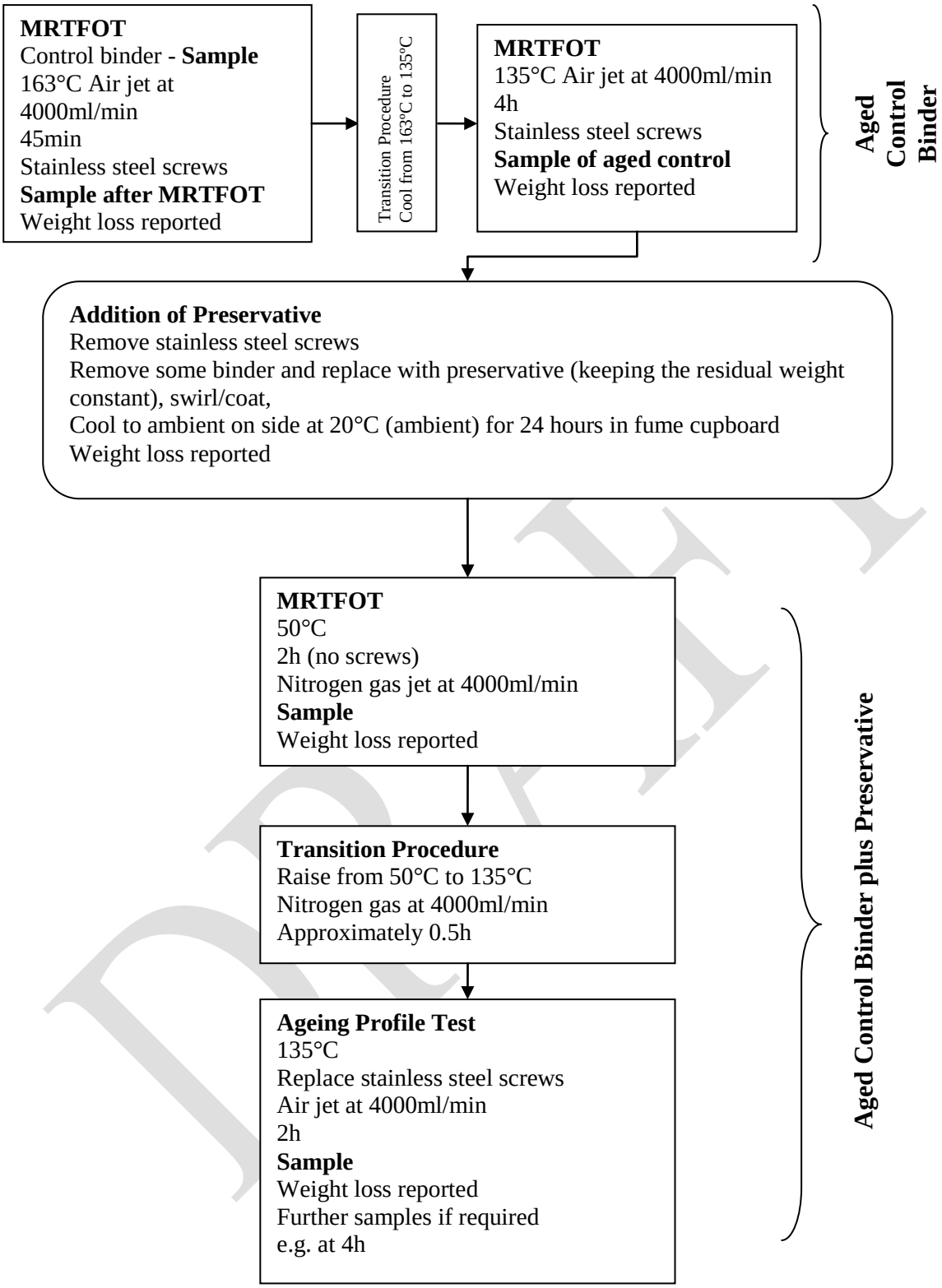
3h $Aged_3$ Binder sample

5h $Aged_5$ Binder Sample

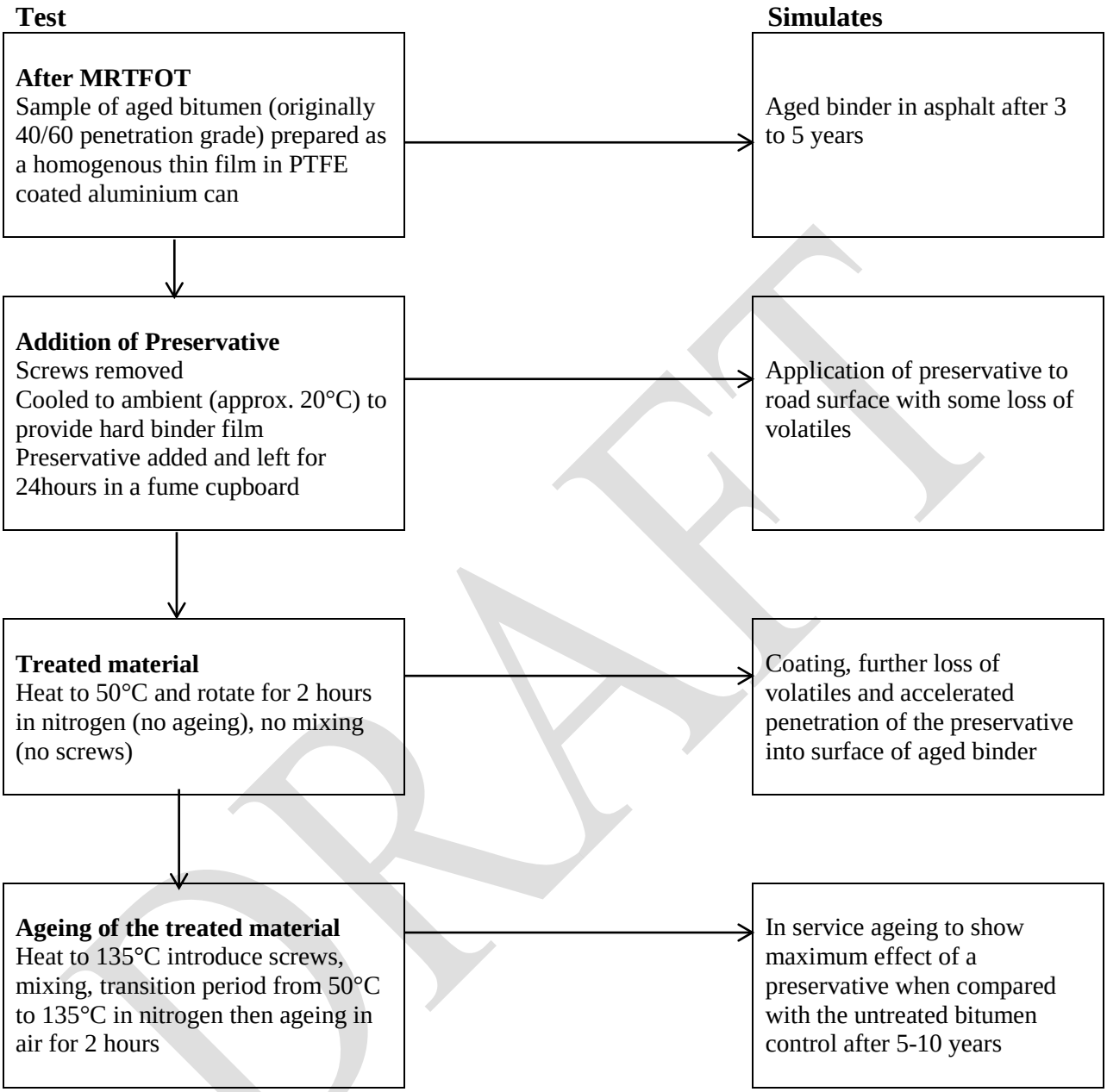
22h $Aged_{22}$ Binder Sample (not normally required, but equivalent to PAV 85)



Preservative Test Protocol



Preservative Protocol - Stages of Simulative Ageing



Preservatives - Improvement in Capacity for Ageing as shown by VET temperature