# Residual Binder Extraction from Emulsions for Quality Assurance Testing

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# **Executive summary**

The trend to the use of emulsified binders for chip seal construction instead of cutback bitumen has created issues relating to the recovery and testing of the emulsified binders. The absence of an agreed protocol for recovery of the binder from the emulsion has allowed various techniques to be used by testing laboratories, and these techniques can affect the rheological properties of the recovered binder. There is a consequent risk that these effects could result in acceptable binders being rejected, or non-compliant materials being accepted.

A survey of methods used internationally was carried out and a draft binder recovery protocol developed based on the techniques best suited to materials and conditions in New Zealand. The main issue that has to be resolved in the development of the New Zealand binder recovery method is minimisation of effects on the properties of the binder. Testing a sample of penetration-grade bitumen through the draft process under a nitrogen blanket showed that the method did not affect the penetration result. Without the nitrogen blanket there was a consistent reduction in penetration between samples. This was not found to be the case for other properties or binders containing volatile diluents or modifiers.

The experiments carried out in this research show that the binder was irretrievably altered by emulsification and subsequent recovery. The properties of the binder after extraction from the emulsion were always different from those of the binder before it was emulsified. Once this was understood the focus became the development of a method that was simple and repeatable for extracting the binder, accepting that the binder properties will be different from the binder prior to emulsification. The draft method is contained in section 10 of this document.

Selection of suitable tests for the extracted binder created some difficulties. It was found that there were no appropriate tests that did not involve heating and remixing of the recovered binder so it was decided to use simple standard tests commonly used within the industry: Penetration, Softening Point and Torsional Recovery. These methods all include a standard method of sample preparation that requires heating to high temperatures to remove water and air bubbles and also to reduce the binder viscosity to allow the binder to flow into test moulds.

The test methods require the recovered binder to be heated to up to 160° C, which is up to 100° C higher than pavement service temperatures. In addition, as the binder deposited from the emulsion during road construction does not undergo a homogenisation process at 160° C, the properties measured in the laboratory are unlikely to be identical to the properties of the binder on the road. Once this was accepted then the method of sample preparation was used to ensure property test results would be comparable from laboratory to laboratory. A consistent thermal and mixing history of the residue is crucial to produce consistent test results.

#### **Abstract**

Safety and environmental drivers are pushing the New Zealand road construction industry away from hot, cutback binders and towards the use of emulsified bitumen as the delivery medium for chipseal binders. However, there is no agreed standard method for recovery and testing of the emulsified binder. The absence of such a method could potentially lead to dispute over the quality and contractual compliance of emulsified binders due to the effects of differing laboratory techniques for binder extraction and sample preparation.

This research project reviews techniques used worldwide and recommends a method suitable for New Zealand materials and conditions.

Results from the trials of the new test method show that, in spite of completing the extraction of the binder under conditions similar to those encountered in the field, the binder must be reheated to remove water and air from the test sample. This treatment changes the binder rheological properties compared with the same binder prior to emulsification and extraction.

Thus the objective of this research was to develop a simple repeatable method acceptable to suppliers and purchasers to extract the emulsified binder to allow confirmation of the binder properties for quality assurance purposes.

This report outlines the literature review, the method development, the laboratory trials of the method and results from contractor trials of the method.

#### 1. Introduction

Safety and environmental factors are driving the New Zealand road construction industry away from hot, cutback binders towards the use of emulsions as the delivery medium for chip seal binders.

Safety statistics show that in the New Zealand road surfacing industry there are on average 10-12 lost time injury days each year resulting from burns caused by hot bitumen. In contrast there are no recorded lost time injury days resulting from emulsion burns over the past 20 years of emulsion use.

An environmental comparison (Slaughter 2004) between emulsified and cutback bitumen showed that emulsion produces one third of the CO<sub>2</sub> emissions and one sixth of the volatile organic compound emissions when compared with cutback bitumen on a per-tonne of binder basis.

It is not sustainable to continue to use products that emit greenhouse gases and smog forming pollutants when there are safer and less polluting options available.

Development of the extraction method proposed here will facilitate greater use of bitumen emulsions for the construction of chip seals.

At present in New Zealand there is no industry-accepted method of extracting the residual binder from emulsion. Thus there is no recognised way for producers and clients to demonstrate that the correct binder was applied to the road surface.

So currently end users are not able to check that they are getting the binder that they specified and suppliers are not able to easily demonstrate that compliant materials have been supplied. The absence of a universally accepted procedure to recover and test the emulsified binders has created a barrier to the wider use of emulsified binders for chipsealing.

This project aimed to develop and validate a method (suitable for quality assurance testing and auditing) of extracting the binder from bitumen emulsions. The following methodology was adopted:

- a) Review current methods in use around the world.
- b) Develop an industry-accepted method of extracting the residual binder from emulsions based on current international best practice.
- c) Validate the method by comparing binder properties from before emulsification with those of the same binder after recovery.
- d) Test samples from all emulsion suppliers in the New Zealand Roading Industry to ensure the method is widely applicable and will gain acceptance in the industry.

Once this methodology has been universally accepted, it will mean the removal of one impediment to the greater use of the safer, more environmentally benign bitumen emulsion technology.

# 2. Recovery Method Philosophy

#### 2.1 The need

The driver for developing a recovery method has come from the increasing use of emulsions for chipsealing and the requirement from clients that they can confirm that they have received the product that they wanted.

In many cases clients want the product that they have previously had that has worked or a product with similar properties. They are interested in the properties of the residual binder as the emulsion is considered to be a delivery system only. Generally in performance-based resurfacing contracts, which are the predominant form of chip seal contract in New Zealand, the client specifies:

- 1. The grade (penetration) of the bitumen complying to M/1.
- 2. The quantity of flux oil for unmodified binders.

These properties are specified because they contribute to the long term properties of the seal and Transit New Zealand and other clients are responsible for the binder and chipseal long after the contract maintenance period is finished.

The client also specifies various properties for polymer modified binders such as:

- a) The minimum softening point based on the maximum road temperature in the region; and
- b) Torsional recovery depending on the amount of modification required.

The object of the research was to develop a simple, rapid, quality assurance test procedure that would, employ low temperatures and make use of inexpensive readily available laboratory equipment. It was therefore apparent that with these constraints the recovered residues may not have properties identical to those of the unemulsified binder as the emulsification process itself introduces emulsifier salts (and possibly other proprietary additives) which cannot be easily removed. Similarly preventing the loss of volatile diluents during recovery is impractical for a quality assurance test and cannot be avoided, although low temperatures will minimise losses. The procedure must also easily provide sufficient residue for testing purposes (penetration, softening point torsional recovery etc).

#### 2.2 Consistent approach

The aim of the project was to develop a simple and repeatable test method that all producers and clients could use, so that the results of the extraction were consistent between testing agencies.

Previous experience had shown that, where different test methods were used by suppliers and customers to extract the binder from emulsions, one laboratory reported compliant test results and the other non-compliant results.

The consistency of the results is the most important factor in the method development so that clients have confidence that they are getting what they asked for.

Simple property tests were selected to ensure all laboratories and parties could obtain the equipment and perform the testing, however there are opportunities for variation in the treatment of the residue to form the test samples.

# 2.3 Comparison with unemulsified binder

Bitumen emulsion has long been marketed as an environmentally friendly delivery system for bitumen to replace cutback bitumen. The argument has been that, once the water has evaporated from the emulsion, the properties of the residual binder are identical to a similar binder applied hot.

Although emulsification is described as a delivery system for the binder, once the binder has been emulsified it cannot be retrieved as the same binder again. The residuum is chemically modified during the emulsification process while being divided into small particles by the emulsion mill, and then, after being sprayed, it coalesces into a continuous film. The removal of the water and formation of the film does not remove the chemicals and they remain part of the binder residue.

It is hence reasonable to expect that the properties of the recovered binder could be different from the unemulsified binder, even if the recovery method does not have an effect on the binder properties. However the comparison with the unemulsified binder is still a useful measure of the effect of the recovery method and allows fine-tuning of the method to keep the difference between the two at a minimum.

This difference can be simulated by adding emulsifying chemicals to the unemulsified binder. Thus the 'perfect' recovery method should produce a recovered binder with these properties.

Other factors that could cause change to the binder properties other than the emulsification process include:

- a) Oxidation of the binder during recovery and during heating for sample preparation;
- Loss of volatile diluents from the binder during recovery and during heating for sample preparation; and
- c) Increased homogeneity from heating and stirring for sample preparation.

It is important that the binder recovery test method should mimic what happens in the field as closely as possible so that the residual binder properties represent reality. However for consistent repeatable test results the binder has to be heated to temperatures high enough to enable homogenising by stirring or mixing of the sample and to prepare test specimens without entrained air.

#### Recovery Method Philosophy

These temperatures are much higher (>150° C) than those encountered in the field. In addition, the binder deposited from sprayed emulsions does not undergo homogenisation. It is necessary to use high temperatures for sample preparation in the laboratory to obtain consistent repeatable test results. Otherwise test results would be significantly affected by entrained air or residual water.

The reheating and remixing of the modified binders may cause significant changes in the binder properties but these changes can not be quantified. These issues are further compounded when investigating the differences in properties between modified binders and latex-modified emulsions and their respective extracted residuum properties.

In consideration of the issues discussed above, this project was focussed on developing a method that produces repeatable and reproducible test results for the recovered binder. It is clearly necessary to accept that there may be an unquantifiable offset between the laboratory test results and the 'real' binder properties as applied in the field.

Thus the actual properties of the binder in the field may not be accurately determined, but the test properties will consistently characterise the binder for quality assurance purposes.

When a binder performs well in the field then the field performance should be recorded and the quality assurance test properties used to benchmark the properties of the binder.

#### 3. International Method Review

International technical and specification literature was reviewed in detail using the TELIS library service of Opus International Consultants. Below is a brief description of the methods identified in this review, listing the pertinent parts of the method test conditions such as temperatures and timing. As this is a very active field of research internationally with papers and research constantly being released, the review was only current until June 2007.

#### 3.1 Description of existing test methods

A large number of test methods have been published that describe methods to determine binder content or to provide binder residue for subsequent testing. These methods are often very similar and with one exception remove water from the emulsion by evaporation or distillation.

#### 3.1.1 Distillation and high temperature evaporation methods

- a) BS 2000-458:2000 (BS EN 1431:2000, Identical with IP 458-2000). Methods of Test for Petroleum and its products bitumen and Bituminous binders Determination of Recovered Binder and Oil Distillation from Bitumen Emulsions by Distillation.
   200 g of emulsion is heated in an aluminium still, in air, to 260° C over 30 60 minutes, and then held at 260° C for a further 15 minutes.
- b) ASTM D 6997 04. Standard Test Method for Distillation of Emulsified Asphalt<sup>1</sup>. 200 g of emulsion is heated in an aluminium still, in air, to 260 °C over 30 60 minutes, and then held at 260° C for a further 15 minutes.
- c) International Slurry Seal Association Distillation method. Same as ASTM 6997, except a maximum temperature of 138° C is used.
- d) Texas DOT method Tex 521-C. Same as ASTM D6997 except that for polymer modified emulsions a maximum temperature of 177° C is used.
- e) ASTM D 6934 -04. Standard Test Method for Residue by Evaporation of Emulsified Asphalt. 50 g of emulsion is heated in air in 1000 ml glass beakers at 163° C for three hours (the sample is stirred after two hours).
- f) AS/NZS 2341.30:1996. Methods of Testing Bitumen and Related Roadmaking Products. Method 30: Recovery of Residue from Bituminous Emulsion. 60 g of emulsion is heated under carbon dioxide or nitrogen in metal containers of up to one litre capacity. Containers of 90 mm diameter by 105 mm deep are recommended. The sample is heated to 130° C repeatedly, until constant weight to within 0.2 g (0.33%) is achieved (a time limit for the process is not specified).
- g) California Test method 331 (California Department of Transportation). Method of Test for Residue by Evaporation of Latex Modified Emulsified Asphalt. 40 g of emulsion is heated in air in metal containers. Containers of 87 mm diameter by

<sup>&</sup>lt;sup>1</sup> Note on ASTM D244 and AASHTO T 59-01. ASTM D244 is a compilation of methods for testing emulsions and specifies ASTM D6934 or ASTM D6997 to obtain residues. AASHTO T 59-01 is essentially the same as ASTM D244.

- 60 mm deep are recommended. The sample is heated at 118° C for 30 minutes, the temperature raised to 138° C for a further 1.5 hours at which time the contents are stirred and then heated for another 1 hour (a total heating time of three hours).
- h) Virginia DOT Test Method 78. This method is the same as California Test 331.
- i) AS/NZS 2341.23:2002. Methods of Testing Bitumen and Related Roadmaking Products. Method 23: Determination of Residue from Evaporation. Water in the emulsion is evaporated on a hot plate. The temperature and time of the procedure is not specified. The method is not intended for recovery of residue for further testing.
- j) California Test 330 (California Department of Transportation) Method of Test for Residue by Evaporation of Emulsified Asphalt. A 25 g sample of emulsion is heated at 149° C for up to 30 minutes. The method does not appear intended for the recovery of residue for further testing.
- k) Distillation of Polymer-Modified Asphalt Emulsions. Button, J. 21st Annual meeting of the Asphalt Emulsion Manufacturers Association, March 23-26, 1994. Hollywood, Florida, pp. 87-94. Six different recovery methods were compared: distillation at 260° C, 178° C and 138° C, thin film evaporation at 138° C and a rotary evaporator method at 138° C using 600 mm Hg vacuum and a 1000 ml/min nitrogen flow. The penetration and viscosity at 60° C and 135° C of the residue were measured for comparison with the base bitumen. Bitumens with natural rubber latex, styrene butadiene rubber and without additives were studied. Distillation at 260° C resulted in softening of the recovered binder relative to the original indicating polymer degradation. Distillation at 178° C and 138° C resulted in hardening as did simple thin film evaporation at 138° C (California method 331, see (g) above). Recovery results using the rotary evaporator method were more scattered but also showed hardening.
- I) A Performance-Graded Binder Specification for Surface Treatments. Barcena, R., Epps Martin, A., Hazlett, D. Texas Transportation Institute, Texas A&M University, Report 1710-1, 2001. 74 pp. Various recovery methods were compared. The method finally adopted was as follows (the 'stirred can method'): 1200 g of emulsion was heated with constant stirring at 163° C for 170 minutes with a nitrogen blanket in a four litre tin. The temperature was maintained using heating tape around the tin. The method produced about 800 g of residue for testing.

#### Other methods investigated in (I) included:

- I. A rotary evaporator method based on ASTM 5404-97; 16 g of emulsion was heated for 30 minutes at 100° C then at 70 minutes at 163° C using a nitrogen purge.
- II. A hotplate method in which ointment tins with 20 g of emulsion were heated at 180° C with stirring for one hour.
- III. An evaporative method based on ASTM 6934-04; 50 g of emulsion was heated in air in 8.4 cm diameter, 12.3 cm high beakers at 163° C for three hours (samples were stirred after two hours).
- IV. ASTM D 6997 04 Standard Test Method for Distillation of Emulsified Asphalt (see b above).

The methods were compared in terms of carbonyl group absorption in the infra-red spectrum and viscosity at 60°C, although values for the original binders are not given nor are the types of binders used, discussed. The 'stirred can' method adopted resulted in the second highest mass loss (presumably all water), behind that of the rotary evaporator method (the theoretical, expected mass loss is not stated).

#### 3.1.2 Low temperature evaporation methods

- a) BS 2000-493:2002 (BS EN 13074:2002, identical with IP 493-2003) Methods of Test for Petroleum and its products bitumen and bituminous binders Recovery of Binder from Bitumen Emulsions by Evaporation. A layer of emulsion is spread at 1.5 to 2.0 kg/m² (≈ 375 500 g), onto 500 mm square, non-stick paper sheets. Water is evaporated in air at ambient temperature for 24 hours, then at 50° C for a further 24 hours in an oven. The method suggests that the recovered residue is 'kneaded' into the forms required for testing at a temperature not exceeding 50° C.
- b) Comparison of Ageing Test Methods for Polymer Modified Road Binders and a Review of the Rapid Recovery Test for Emulsion and Cut-Back Binders. Walsh, I.D., Shrubsolel, P., Heslop, M.F.W. Proceedings 3rd Eurasphalt & Eurobitume Congress, Vienna, 2004, Book 2, pp 1212-1223. The BS 2000-493:2002 (BS EN 13074:2002) method (see above), was compared to the UK Highways Agency 'rapid recovery' method (clause 923 of the UK National Specification for Highway Works). The latter method involves heating 19 g of emulsion at 85° C for 75 minutes in a PolyTetraFluoroEthylene (PTFE) Rolling Thin Film Oven (RTFO) Test bottle. The bottle is modified with a stainless steel screw insert to ensure mixing of polymer modified emulsions. A stream of nitrogen is directed into the bottles. Six different polymer modified and non-polymer modified emulsions were compared. The RTFO method resulted in hardening (as measured by the dynamic shear modulus (G\*) at 25° C) of 1.5 to two times that of the BS method, but moduli were not compared to that of the unemulsified materials.
- c) Polymer Network Formation in the Emulsion Residue recovered by Forced Air Drying. Takamura, K., Heckmann, W. Asphalt Emulsion Manufacturers Association International Symposium on Asphalt Emulsion Technology, Nov 11-14 1999, Washington, pp.185-194. 60 g of emulsion is added to a 5 mm deep, 175 mm square tray and placed in a cabinet (46 cm high, 33 cm wide and 28 cm deep) at room temperature. Fans, positioned just above the trays, drew air through the cabinet at about 60 m/s linear velocity. The emulsion samples were left for 5 hours (90% of the water had evaporated after three hours), resulting in a bitumen film 2 mm thick. The shear moduli of the recovered binders were measured but not compared with the unemulsified materials.
- d) Comparison of Emulsion Residues Recovered by Forced Airflow and RTFO Drying.
   Takamura, K. International Slurry Surfacing Association/ Asphalt Emulsion
   Manufacturers Association Proceedings, march 12-15, 2000, Florida. See e) below.
- e) Evaluation of Asphalt Binders Used for Emulsions. Clyne, T.R., Marasteanu, M.O., Basu, A. Minnesota Department of Transportation, August 2003. 62 pp. This is essentially the same paper as above (d) on the forced air flow method except for a discussion of a round robin study comparing four recovery methods; 'vacuum distillation, 177° C distillation, 260° C distillation and ASTM evaporation'. Details of

the methods are not given (or any literature reference) but presumably the 'vacuum distillation' method is some form of rotary evaporator method; '177° C and 260° C distillation' are ASTM D6997 and 'ASTM evaporation' refers to ASTM 6934. Residues were compared in terms of rutting temperature, softening point and morphology by microscopy. Considerable scatter amongst laboratories was observed. In general distillation at 260°C appeared to adversely affect residues from polymer modified emulsions whereas the 'evaporation' method had the least effect, especially with respect to the appearance of the polymer dispersion under the microscope. Recovered binders were not compared to the initial unemulsified binders. The paper also compared the PTFE RTFO bottle method discussed in b) to the forced air flow method. The rutting resistance temperature of the residue from SBR latex bitumen emulsions was found to be the same for both methods but the RTFO method was favoured because it was faster.

f) Rheological Characterisation of Asphalt Emulsions Residues. Marasteanu, M.O., Clyne, T.R. Journal of Materials in Civil Engineering, 2006, pp 398-407. The authors compared two methods for emulsion residue recovery; the forced air flow method (see c)-e) above) with the addition of two hours in an oven at 60° C, and a method using RTFO bottles. The RTFO method is similar to that in b); 35 g of emulsion is heated at 85° C in an RTFO (in a glass bottle presumably) for 75 minutes with 4 l/minute airflow. The bottles were then placed in a 135° C oven for 15 minutes. No screw device was used as in b). Various polymer and unmodified bitumen emulsions were tested. Comparisons were made between recovery methods but not with unemulsified binders. The RTFO method residues were found to have higher shear moduli than the forced air flow method residues.

#### 3.1.3 Ethanol precipitation

Characterisation of the Different Phases in the Life Cycle of the Binder in a Bitumen Emulsion: Recovery Methods. Gueit, C., Robert, M., Durand, G. In Transportation Research Circular E-C122, October 2007, pp 1 -10. Transportation Research Board, Washington DC. See also: Caracterisation des Differentes Phases de la vie du Liant d'une Emulsion de Bitume: Evaluation de Diverses Methods de Recuperation (1st partie). (Characterisation of Different Life Phases of a Binder of a Bitumen Emulsion: Evaluation of Different recovery Methods.)<sup>2</sup> Gueit, C., Robert, M., Durand, G. RGRA No, 848, April-May 2006. Method based on the addition of ethanol was developed and compared with various other methods. Ethanol is added to break the emulsion and precipitate the bitumen. The bitumen is removed, pressed, washed with water then dried at 140° C. The volume ratio of ethanol to emulsion, the rate of addition and drying time are not specified.

Other methods evaluated in this paper were:

- I. EN 13074: Ambient and low oven temperature evaporation (see 3.1.2 a) above).
- II. EN 1431/ ASTM D244: High temperature distillation (see 3.1.1 a) above).
- III. Belgian Standard Method 08-34: an emulsion sample is heated in a 2 l high-form beaker with a Bunsen burner and under constant agitation to 163° C. The beaker is placed in oven at 163° C for a further 0.5 hours.

<sup>&</sup>lt;sup>2</sup> Text is in French. Summary is based on a partial translation.

IV. Spanish Standard Method NLT 147: 50 g of emulsion is heated in a 60 ml beaker at 163° C, a method similar to ASTM 6934-04.

Other methods (Pr EN 14895, XPT 66-063 and T66-031) that include simulation of 6 months to one year field 'ageing' were also studied.

Emulsions of unmodified 160/220 penetration and 35/50 penetration, 160/220 with SBS and 160/220 with latex were used. Residual binders were compared on the basis of softening point, penetration, and morphology by microscopy, cohesion (EN 13588 pendulum test), elastic recovery (EN 13398) and infra-red spectroscopy.

The distillation 3.1.3 II, Belgian 3.1.3 III and ethanol precipitation methods were most successful in terms of having the least effect on recovered bitumen properties. Surprisingly the elastic recovery results were similar for all methods including the distillation. Softening point and penetration results were the most sensitive to recovery method.

#### 3.2 Existing methods

The existing recovery methods used for extracting the binder residue from bitumen emulsions can be classified broadly as:

- a) High temperature distillations where the emulsion is heated at high temperatures over 150° C, sometimes using vacuum or nitrogen to prevent oxidation.
- b) Thin film (1-2 mm) evaporation at temperatures up to 163° C, static or with occasional or continual stirring (as for the RTFO methods) and sometimes with a nitrogen blanket.
- c) Precipitation methods using solvents (e.g. ethanol) to accelerate the binder deposition before the removal of the water phase.

Most of these recovery methods employ relatively high temperatures (>100° C) to remove the water from the emulsion.

In general these high temperatures are more likely to promote oxidation of the residue, loss of volatiles from the binder, and in the case of polymer modified binders, chain scission. All of these processes may affect the properties of the residue, although investigation of the significance of such effects during recovery has been limited and the results contradictory.

In principal high temperature treatment should be limited if possible to that necessary for testing of the residue. (As is the case for standard bitumen samples.) Of the methods identified in the literature review, method 6.1.2 (a) (BS 2000-493:2002) best meets this criterion as it employs a maximum temperature of 50° C, well within temperatures likely to be experienced in the field. The procedure has the additional practical advantage of not requiring expensive specialised test equipment. The test however takes 48 hour to complete and a shorter test time is preferable. Also the test suggests that the recovered residue is 'kneaded' into formed needed for testing at a temperature not exceeding 50° C.

This procedure does not adequately homogenise the residue and is likely, particularly in the case of polymer modified binders, to entrain air bubbles which could affect the property test

results (e.g. penetration) and the repeatability of the method. the residue from whatever extraction method is employed must be heated at temperatures that enable proper mixing and specimen preparation for subsequent testing.

The ethanol precipitation method is novel and appears very simple although the drying time required at 140° C is not specified. This high temperature may not be a disadvantage as the residue from whatever method is employed must be heated at similar temperatures to enable proper mixing and specimen preparation for measurement of penetration, softening point etc.

Following the literature review it was decided to focus on a simple low temperature oven treatment method, but some preliminary trials using ethanol precipitation and a new electrolytic deposition were also carried out.

# 4. Preliminary Investigations

#### 4.1 Ethanol precipitation

Absolute ethanol (150 ml) was added in 50 ml increments to 38.95 g of cationic 75% solids styrene-butadiene-styrene (SBS) modified bitumen emulsion in a beaker. A substantial degree of breaking and bitumen precipitation was noted after the first addition but subsequent additions of ethanol produced no noticeable change. After standing for 30 minutes at room temperature the liquid phase was still a dark brown colour indicating the presence of unbroken emulsion or very fine broken particulates. The liquid phase was decanted off and the precipitated bitumen washed with additional clean ethanol and then with water until the washings were clear. The residue was dried at 105° C in an oven for 18 hours, cooled and weighed. The ethanol had precipitated only 79% of the binder (i.e. 21% of the binder remained in an emulsified state).

The method has two drawbacks:

- Complete phase separation is not achieved and full recovery would require an additional filtration or centrifugation of the liquid phase; and
- The bulk precipitated material contained a considerable amount of entrained water that required physical manipulation (kneading) and oven drying to remove. Another solvent or breaking agent may provide a better separation but the reagents used must be volatile for ease of removal.

#### 4.2 Electrolytic deposition

The particle charge apparatus described in AS 2341.22 was used to form a circuit in which a shallow aluminium tray (0.0512 m²) forms the cathode. Cationic styrene-butadiene-styrene (SBS) modified bitumen emulsion (412 g, 75% binder content) was placed in a tray and the circuit connected. After ten minutes current had ceased to flow and the experiment was stopped. A large amount of unbroken emulsion remained and was poured off leaving, after washing with water, an approximately 1 mm layer of bitumen adhering to the tray. The deposited bitumen after careful removal of surface water with paper towels accounted for 7.4% (22.8 g) of the binder originally present, decreasing to 7.3% after oven drying at 105° C for 18 hours.

Compared to the ethanol precipitation, electrolytic deposition appears to entrain little water but would require a specially designed apparatus with a large cathode surface area to obtain sufficient quantities of bitumen for subsequent testing. Questions also arise as to whether the deposited film is representative of the bulk binder phase and the applicability of the method to all emulsifier types.

#### 4.3 Test procedure

The procedure finally developed was based loosely on method 3.1.2 a) (BS 2000-493:2002). That method specifies an ambient temperature step followed by oven treatment at 50° C. The procedure was modified to use galvanised steel trays instead of non-stick paper, the 24 hour ambient temperature step was omitted, and the residue obtained was heated to allow proper mixing before testing.

In summary, sufficient emulsion to form a 2 mm film of residue is poured into a galvanised steel tray (200 mm X 200 mm) and heated in a 50° C oven for 26.5 hours. The residue is scraped from the trays, combined then heated at 130° C (or 155° C for polymer modified bitumens) for up to 1.5 hours then specimens are poured for testing. A detailed description of the method is given in appendix 2. The test development is discussed below (section 5).

The method was evaluated using a small number of test emulsions from Fulton Hogan plants. These emulsions were manufactured using typical binders used in practice (80/100, 180/200 and polymer modified bitumens). The penetration, shear modulus and phase angle (over a range of frequencies) properties of the recovered residues were compared to that of the original binders before emulsification.

The recovered binders were also examined using infrared spectroscopy for signs of oxidation and polymer degradation.

Once the prototype method was developed, samples of emulsions from manufacturers were tested using penetration and torsional recovery measurements to ensure the procedure was widely applicable.

# 5. Test Development

#### **5.1** Effect of heating time

Weight loss at 50° C was measured to determine the rate of water evaporation from various different cationic emulsions (none of which contained volatile diluents). Sufficient emulsion was used (based on water content determined by Dean and Stark Distillation (AS 2341.9)) to give a 2 mm film thickness (assuming a density at 50° C of 1.0) of residual binder.

A typical weight loss curve is shown in Figure 5.1 and results are summarized in Table 5.1. For comparison, a 2 mm film of distilled water had completely evaporated in under one hour under the same conditions. The data in Table 5.1 indicate that constant weight is achieved after about 26-27 hours. Emulsion 6/06/272 was tested in two different ovens (440 L and 140 L capacity) without any noticeable difference in evaporation efficiency.

By comparison a number of experiments with emulsion 6/07/272 were conducted to measure evaporation at room temperature with the plates place next to a large fan. At an average temperature of  $22.3^{\circ}$  C (range  $17.5^{\circ}$  –  $25.8^{\circ}$  C), approximately 40 hours were needed to reach the expected binder content whilst at  $18.2^{\circ}$  C (range  $11.4^{\circ}$ - $24.0^{\circ}$  C) 60 hours were needed.

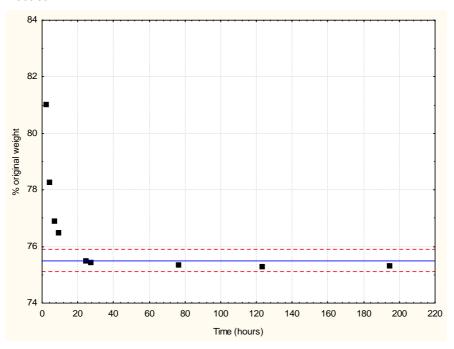


Figure 5.1 Typical evaporative weight loss at  $50^{\circ} \pm 4^{\circ}$  C for emulsion 6/07/272 (2% SBS polymer modified bitumen). Solid and dashed lines represent Dean & Stark binder content and upper and lower repeatability limits respectively.

Table 5.1 Weight loss data at 50.0° ± 1° C.

	Emulsion SBS polymer modifie nder content* (by Dea	(180/2 Binder con	ion 6/07/89 200 bitumen) itent* (by Dean & = 59.9 ± 0.4%		
	Oven 1		Oven 2		Oven 1
Time (hrs)	% Original Wt# (mean of 8 replicates)	Time (hrs)	% Original Wt# me (hrs) (mean of 6 Time replicates)		% Original Wt# (mean of 4 replicates)
0	100.0	0	100.0	0	100.0
1.5	81.8 ± 0.3*	2.5	81.0 ± 0.9	1.25	71.34 ± 1.3
4.75	78.2 ± 0.3	4.25	78.3 ± 0.5	17.75	59.47 ± 0.04
7.25	76.8 ± 0.3	7	76.9 ± 0.4	20.5	59.45 ± 0.04
23	75.8 ± 0.3	9.2	76.5 ± 0.3	24	59.45 ± 0.06
24.5	75.7 ± 0.3	24.75	75.5 ± 0.3	26.5	59.45 ± 0.05
26.5	75.7 ± 0.3	27.25	75.4 ± 0.3	40.3	59.40 ± 0.05
		76.5	75.4 ± 0.2	89.0	59.39 ± 0.04
		123	75.3 ± 0.3		
		194.5	75.3 ± 0.3		

<sup>\*</sup> Repeatability as given in ASTM D244-04

Measured binder contents after 26.5 hours heating for various emulsions are compared with the Dean and Stark derived binder contents in Table 5.2, and show good agreement. In absolute terms, for the quantity of emulsion used, a difference in binder content of 0.5% is equivalent to about 0.13 g of water per plate. It must be noted that all the recovered binders foamed to a small degree when subsequently heated for testing even that obtained after 194 hours (eight days).

Table 5.2 Measured binder contents after 26.5 hours for various emulsions.

	Emulsion (binder type)	Calculated Binder Content (%)	Expected Binder Content (%)
6/07/272	(2% SBS polymer modified binder)	75.7 ± 0.3	75.5 ± 0.4
6/07/272	(2% SBS polymer modified binder)	75.5* ± 0.3	75.5 ± 0.4
6/07/89	(180/200 bitumen)	59.45 ± 0.05	59.9 ± 0.4
6/07/127	(5% SBS polymer modified binder)	75.5 ± 0.1	75.9 ± 0.4
6/07/127	(5% SBS polymer modified binder)	75.5 ± 0.3	75.9 ± 0.4
6/07/87	(180/200 bitumen)	70.0 ± 0.4	69.3 ± 0.4

<sup>\*</sup>Interpolated from the weight loss curve.

### **5.2** Residue properties

Test on emulsion residues were carried out after the 26.5 hour 50° C treatment as described in 5.1 above. Penetration (ASTM D5), softening point (ASTM D36) and, for polymer modified emulsions, torsional recovery (Austroads AG:PT/T122 were selected as they are basic tests widely used within the industry. Other methods of analysis were also employed and are discussed below.

<sup># ± 95%</sup> confidence limits

Penetration, softening point and torsional recovery results are given in Table 5.3, the properties of the residues are compared to the base binder (i.e. the binder before emulsification). In some cases, to estimate the effects of oxidation, emulsions were also dried under an inert nitrogen atmosphere.

Table 5.3 Residue properties after 26.5 hours at 50°C.

Emulsion (binder type)	Treatment	Penetration at 25° C* (dmm)	Torsional Recovery* (30 sec) %	Torsional Recovery* (30 min) %	Softening point* (°C)	Change in Penetration from respective base binder (%)
6/07/272 (2% SBS	Base binder	169 ± 3	21 ± 1	46 ± 2	46.3 ± 0.1	-
polymer modified	Residue	148 ± 7	23 ± 1	52 ± 1	50.2 ± 0.1	-14
binder)	Residue under nitrogen	153 ± 3	-	-	-	-10
6/07/127 (5% SBS	Base binder	142 ± 2	15 ± 1	43 ± 5	52.9 ± 2.5	-
polymer modified binder)	Residue	127 ± 4	9 ± 1	13 ± 2	53.5 ± 4.9	-19
C /07/00	Base binder	174 ± 4	-	-	42.0 ± 3.7	-
6/07/89 (180/200	Residue	167 ± 5	-	-	45.6 ± 3.7	-4
bitumen)	Residue under nitrogen	167 ± 5	-	-	-	-4

<sup>\*</sup>mean of 2-5 replicates ± 95% confidence limits

For all the emulsions, both penetration and softening point results show hardening of the residue compared to the base binder but the effect is most marked with the polymer modified materials. Most of this affect appears unrelated to oxidation as the penetration drops by a similar amount when water is evaporated in the absence of air. The polymer modified emulsions also show (particularly in the 5% case) a drop in torsional recovery value suggesting that the residue is less elastic than the base binder.

To further investigate the effect of oxidation, experiments were carried out using various bitumens poured as 2 mm thick films on the test plates and exposed in air or under nitrogen for 26.5 hours at 50° C. Results are given in Table 5.4.

Table 5.4 Effect of oven treatment used in recovery procedure on binder penetration (26.5 hours at 50°C, unemulsified bitumens).

Bitumen	Treatment	Penetration at 25° C* (± 95% conf limit)	Change in Penetration
2% SBS polymer modified binder	Base binder	169 ± 3	-
(6/06/278)	Heated as 2 mm film	160 ± 3	-7
	Base binder	161 ± 5	-
180/200 bitumen (6/97/372)	Heated as 2 mm film	150 ± 5	-7
100/200 bitumen (0/31/312)	Heated as 2 mm film under nitrogen	161 ± 5	0
80/100 bitumen (6/97/371)	Base binder	71 ± 1	-
00/100 bituilleli (0/97/371)	Heated as 2 mm film	74 ± 1	+4.2

<sup>\*</sup>mean of 2-6 replicates ± 95% confidence limits

The 180/200 bitumen showed a small decrease in penetration similar to that observed for the emulsion residue. The 80/100 showed an increase (softening) but the results are within the single operator precision for the test (ASTM D5). The polymer modified binder also showed a decrease in penetration but the effect is much less than that observed for the emulsion indicating that some other process may be affecting the results. One possibility is

that the polymer morphology in the dried emulsion film is different from that of the base binder.

# 5.3 Infrared spectroscopy and shear moduli

The effect of oven treatment was further investigated by infra-red (IR) spectroscopy. Spectra of recovered binders were measured using 4% solutions in dichloromethane (1 mm KBr cell). Results are given in Table 5.5.

Oxidation of SBS type polymers by atmospheric oxygen leads to a decrease in absorption arising from the C-H bending of trans-disubstituted double bonds in the polymer at about 970 cm<sup>-1</sup> (Ouyang, C., Wang, S., Zhang, Y., Zhang, Y. Polymer Degradation and Stability 91(2006), 795-804). Oxidation of bitumen gives rise to increased absorption in the carbonyl (about 1700 cm<sup>-1</sup>) and sulphoxide regions (1033 cm<sup>-1</sup>) of the spectrum.

For all three emulsions the residue and respective base binder spectra were identical (within error), indicating the absence of any significant oxidation. For the SBS binders tested, the error in the absorbance measurement at 970 cm<sup>-1</sup> (0.02 absorbance units), corresponds to an SBS content of about 0.11% so that any degradation has been minimal for practical purposes.

Table 5.5 Effect of recovery procedure on binder oxidation.

Emulsion	Treatment	Absorbance per gram*		
		1700 cm <sup>-1</sup>	1033 cm <sup>-1</sup>	970 cm <sup>-1</sup>
		$(\pm 0.03)$	(± 0.05)	$(\pm 0.02)$
2% SBS polymer modified binder	Base binder	0.36	0.91	0.84
(6/07/272)	Residue	0.37	0.93	0.86
5% SBS polymer modified binder	Base binder	0.33	0.86	1.10
(6/07/127)	Residue	0.34	0.86	1.12
	Base binder	0.25	0.82	0.56
180/200 bitumen (6/07/87)	Residue	0.28	0.84	0.56
180/200 bitumen (0/07/87)	Residue under nitrogen	0.26	0.82	0.56

<sup>\*</sup>mean of 2- 4 replicates ± 95% confidence limits

Moduli and phase angle measurements (by dynamic shear rheometry) for the residue recovered from a 5% SBS polymer modified bitumen emulsion are given in Table 5.6 and Table 5.7. Large changes in both moduli and phase angle are evident particularly at the low frequency. The moduli and phase angle of the unemulsified base polymer modified bitumen subjected to the recovery procedure, show even larger variations (Table 5.8 and Table 5.9) and the direction of changes are in many cases inconsistent with the data in Table 5.6 and Table 5.7.

Table 5.6 Effect of recovery procedure on modulus (G\*) of 5% SBS polymer modified bitumen emulsion (6/07/127).

Temperature		G* Iz, Pa)	G* increase	(20.0	G* increase	
(°C)	Base binder	Heated as 2 mm film	(0.1 Hz) %	Base binder	Heated as 2 mm film	(20.0 Hz) %
25	22730	30480	34.1	674300	924000	37.0
35	5562	7330	31.8	186500	240200	28.8
45	1452	1572	8.3	56240	70360	25.1
55	512.2	365.9	-28.6	19850	24900	25.4

Table 5.7 Effect of recovery procedure on phase angle ( $\delta$ ) of 5% SBS polymer modified bitumen emulsion (6/07/127).

Temperature	δ (0.1 Hz)		δ increase	δ (20.0 Hz)		δ increase
(°C)	Base binder	Heated as 2 mm film	(0.1 Hz) %	Base binder	Heated as 2 mm film	(20.0 Hz) %
25	59.23	58.77	-0.8	57.51	54.73	-4.8
35	63.78	66.64	4.5	61.81	60.97	-1.4
45	60.73	77.11	27.0	61.36	61.6	0.4
55	55.31	82.15	48.5	63.11	62.8	-0.5

Table 5.8 Effect of recovery procedure on modulus (G\*) of 5% SBS polymer modified bitumen (6/07/128).

Temperature	(0.1	G* Hz, Pa)	Change in G*	in G* (20.0 Hz, Pa)		Change in G*
(°C)	Base binder	Heated as 2 mm film	(0.1 Hz) %	Base binder	Heated as 2 mm film	(20.0 Hz) %
25	22730	25620	12.7	674300	537100	-20.3
35	5562	9621	73.0	186500	165700	-11.2
45	1452	4339	198.8	56240	58030	3.2
55	512.2	2447	377.7	19850	25000	25.9

Table 5.9 Effect of recovery procedure on phase angle ( $\delta$ ) of 5% SBS polymer modified bitumen (6/07/128).

Temperature	(0.	<b>δ</b> I Hz)	Change in (20.0		<b>5</b> ) Hz)	Change in δ (20 Hz)
(°C)	Base binder	Heated as 2 mm film	% %	Base binder	Heated as 2 mm film	% (20 HZ)
25	59.23	46.52	-21.5	57.51	55.16	-4.1
35	63.78	43.44	-31.9	61.81	56.14	-9.2
45	60.73	36.91	-39.2	61.36	52.33	-14.7
55	55.31	35.36	-36.1	63.11	49.68	-21.3

In contrast results for unemulsified 180/200 bitumen after recovery show much smaller changes (Table 5.10 and Table 5.11). The increase in moduli and decrease in phase angle are, in most cases, close to the precision of the measurement (typically  $\pm$  7%), and are consistent with the limited oxidation caused by the recovery procedure indicated by previous data. To put these values in perspective the moduli of 80/100 grade bitumen over the  $25^{\circ}$  C to  $55^{\circ}$  C temperature range at 20 Hz are 110% to 186% higher than that of the corresponding 180/200 values, and 405% to 472% higher at 0.1 Hz.

The moduli and phase angle data support the earlier contention that changes in the physical properties of the polymer modified bitumen residues on recovery are not artefacts of the recovery method, but are related to changes in polymer morphology occurring due to the emulsification and emulsion breaking process. SBS polymer modified bitumens are inherently unstable two phase systems and variations in sample handling history (heating, stirring) are also likely to be significant factors.

Table 5.10 Effect of recovery procedure on modulus (G\*) of 180/200 bitumen (6/97/372).

Temperature	G* (0.1 Hz, Pa)		Change in G*	(20.0	G* Hz, Pa)	Change in G*
(°C)	Base binder	Heated as 2 mm film	(0.1 Hz) %	Base binder	Heated as 2 mm film	(20 Hz) %
25	14510	17430.0	20.1	956900	1107000	15.7
35	2239	2543.0	13.6	212900	233800	9.8
45	406	454.2	11.9	49850	54370	9.1
55	92.24	101.9	10.5	13470	14710	9.2

Table 5.11 Effect of recovery procedure on phase angle ( $\delta$ ) of 180/200 bitumen (6/97/372).

Temperature	δ (0.1 Hz)		Change in <b>δ</b>	δ (20.0 Hz)		Change in δ	
(°C)	Base binder	Heated as 2 mm film	(0.1 Hz) %	Base binder	Heated as 2 mm film	(20 Hz) %	
25	78.77	77.98	-1.0	63.24	61.81	-2.3	
35	83.92	83.52	-0.5	72.51	71.66	-1.2	
45	87.2	87.03	-0.2	77.52	76.84	-0.9	
55	88.43	88.48	0.1	80.99	80.49	-0.6	

# **6.** Discussion on the Adequacy of the Method

If a simple comparison between the recovered material from a QA sample and the 'expected properties of the residue', stated by the manufacturer, is all that is required then high temperature effects are irrelevant, and any recovery procedure could be used. The expected properties of the residue would be determined beforehand by the manufacturer performing the procedure on their product range thus taking into account any degradation. The difficulty with this practical solution to the difficulties of extracting and testing the residue is that the properties measured from the test results on the extracted residue from production samples used for quality assurance may not bear much resemblance to the actual properties exhibited in the field.

The main issue with the method is that to prepare the test specimens the extracted residuum has to be heated to a significantly higher temperature than would occur in the field. the short period of heating and mixing may not be significant for standard penetration grade binders but for modified binders it may be very significant.

It was decided that it was more important to have a consistent approach so customers could test the product and compare it with what the supplier said they would get.

In performance based contracts the supplier and contractor are responsible for the chip seal system working including the binder and the chip. The client specifies the base binder required for the contract. The extraction method must be able to show that for emulsions made with unmodified binders, the required base binder has been used.

Normally when a client asks for modified emulsions there is a specific purpose in mind for the chipseal, e.g. the binder modification is required for improved performance in extreme climates or improved binder performance on sites with high stress etc. However, in the past hot polymer modified binders have been specified by binder properties and, as can be seen from the trials, the properties of the residuum from a polymer emulsion are not the same as the properties of the polymer modified binder before emulsification. The expectation is that the supplier will provide a technical data sheet for each of their polymer modified emulsions that provide the expected properties of the residua extracted using this method.

If the properties are different in the laboratory this does not mean that the binder will perform worse in fact because of the improved constructability and improved adhesion properties provided by the emulsification delivery system, polymer emulsion seals outperform their hot binder equivalents every time.

Once the concept that the properties of the residuum extracted using this method will differ from the properties of the hot modified binder pre-emulsification is accepted, then the test method can be used to confirm that the agreed material has been supplied.

#### 7. Contractor Trials of Method

#### **7.1** Test results

The trials of the method were limited because the method was not developed until the end of the season and there was very little emulsion being manufactured at this time, especially solvent-free emulsion. However a number of trials were completed with the results shown in Table 7.1.

The Fulton Hogan Dunedin Laboratory tested an emulsion containing solvent because that was all they had access to at the time of testing. The results of this testing showed that the method does not remove all of the kerosene.

The test results from the Fulton Hogan Auckland and Canterbury laboratories that trialled the method on polymer modified binders and polymer modified emulsions made from the binder were quite consistent for all of the three property tests. There were significant differences between the properties of the binder before and after extraction but these were consistent between the tests.

Results from the Higgins laboratory were consistent with those from the other laboratories, the residues were harder than the original binders with the exception of Flexiphalt 350S which showed the opposite behaviour. The change in penetration for the 180/200 binder (9%) is similar to those reported in Tables 5.3 and 5.4.

Table 7.1 Test results from the contractor trials of the test method.

	Penetration (dmm)	Softening Point (°C)	Torsional Recovery % (30 sec & 25° C)
Higgins Laboratory			
180/200 (Original)	181	40.8	N/A
Residue	165	41.0	N/A
Flexiphalt 330S (Original)	157	43.4	9
Residue	142	43.8	8
Flexiphalt 350S (Original)	135	63.6	52
Residue	153	53.6	13
Fulton Hogan Auckland Laboratory			
SX14 (Original) A	155	53.6	18
EX14 Residue	122	63.4	33
SX14 (Original) B	155	51.8	21
EX14 Residue	132	62.6	31
Fulton Hogan Dunedin Laboratory			
180/200 (Original) CRS1 Residue (containing remnant kerosene)	188 279	41.4 38.7	N/A N/A
Fulton Hogan Canterbury Laboratory			
SX14 (Original)	152	51.8	19
EX14 Residue	135	58.4	28

### 7.2 Test method

The feedback from the Fulton Hogan Auckland Laboratory was that they had had great difficulty removing the bitumen from the tray and they used heat to help. The Fulton Hogan Canterbury Laboratory used Teflon paper to line the tray and had no difficulties with removing the binder.

### 8. Conclusions

A low temperature emulsion binder recovery method has been developed that results in minimal binder oxidation or SBS polymer degradation.

The method provides sufficient residue for standard tests such as penetration, torsional recovery and softening point.

There are significant differences in properties between the residue extracted from polymer modified emulsions and the polymer modified binders before emulsification.

The method produces consistent results for similar products from different production plants and laboratories.

# 9. Recommendations

The proposed method be trialled as the industry standard method of 'residual binder extraction' from bituminous emulsions for quality assurance testing.

That there be a review of the test method within 12 months of the introduction of the method.

That further investigation be carried out to develop and agree binder test methods that do not require homogenisation or heating of the extracted residuum above temperatures likely to be encountered by the binder in the field.

# 10. Draft Emulsion Binder Recovery Procedure

#### **10.1** Scope

This test method describes a standard procedure for the recovery of the non-volatile binder component of bituminous emulsions for testing by standard methods. The method is an output from a Land Transport New Zealand research project under the auspices of an industry working group.

This test method is intended for suppliers, users and clients to recover binders from bituminous emulsions to allow laboratory testing for quality control purposes.

#### 10.2 Equipment required

- a) Fan assisted oven capable of maintaining a temperature of  $50^{\circ} \pm 2^{\circ}$  C.
- b) Four flat bottomed metal trays  $200 \times 200 \pm 2$  mm square with a  $10 \pm 2$  mm raised lip. The trays shall be manufactured from a corrosion resistant material, for example galvanised iron or stainless steel. The metal used to fabricate the trays shall be at least 1 mm thick to minimise distortion of the tray bases.
- c) Hotplate of area at least  $200 \times 200$  mm capable of warming the trays sufficiently to allow easy removal of the bitumen sample from the tray.
- d) Balance of sufficient range to weigh the filled metal trays readable to  $0.1\,\mathrm{g}$  and accurate to better than  $\pm\,0.5\,\mathrm{g}$ .
- e) Wide blade spatula with blade at least 75 mm wide.
- f) 500 ml press top cans
- g) Stirring rod, e.g. brass rod 300 mm long, 2 mm thick suitable for vigorously remixing hot binder in the 500 mL can.

#### 10.3 Procedure

- a) Determine the binder content (% by weight) of the emulsion according to BS 434: Part 1:1984, Appendix F or equivalent method.
- b) Weigh the empty trays to the nearest 0.1 g.

Calculate the weight of emulsion required to obtain an approximately 2 mm film of residual binder as below:

Weight of emulsion 
$$(g) = \frac{788.0}{\text{binder content}}$$

- c) Pour the required weight of emulsion ( $\pm$  0.5g) onto each tray, reweigh and place in the oven at 50°C. Ensure that the trays are reasonably level. Leave the trays in the oven for 26  $\pm$  0.5 hours.
- d) Remove the trays from the oven and allow them to cool to room temperature. Weigh the trays and record the weight. Recalculate the emulsion binder content for each tray using the formula below.

Binder content by evaporation = 
$$\frac{(M3 - M1)}{(M2 - M1)} \times 100$$
 (%)

Where: M1 = mass of tray (g)

M2 = mass of tray and emulsion (g)

M3 = mass of tray and recovered binder (g)

Calculate the average binder content of the emulsion by averaging the results for the trays tested. Note any significant variance from the binder content established in a) above.

e) Warm each tray on a hotplate at  $70^{\circ}$  –  $80^{\circ}$  C for five minutes and scrape the combined binder into a 500 ml can.

Heat the binder in an oven at  $130^{\circ} \pm 5^{\circ}$  C ( $155^{\circ} \pm 5^{\circ}$  C for polymer modified binders) for 1 – 1.5 hours, stirring occasionally until fluid and frothing has stopped before testing for penetration, torsional recovery and softening point.

f) Prepare samples for the required subsequent testing.

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# **Glossary**

Anionic Emulsion A bituminous emulsion that has a negative charge conferred

on each droplet.

Bitumen A viscous liquid or solid consisting essentially of

hydrocarbons and their derivatives. It softens when heated and hardens when cooled. It is black brown in colour and

has waterproofing and adhesive properties.

Binder The adhesive viscous material that binds to both the existing

road surface and the sealing chips as a cohesive mass in a

chipseal.

Bitumen Emulsion A liquid in which a substantial amount of bitumen or

bituminous binder is dispersed as fine droplets (dispersed phase) in water (continuous phase) and stabilised with

emulsifying agents.

Cationic Emulsion A bituminous emulsion that has a positive charge conferred

on each droplet.

Chipseal A wearing course of uniformly sized aggregate (sealing chip)

spread over a film of sprayed bituminous binder.

LTNZ Land Transport New Zealand

TNZ Transit New Zealand

Cutter A volatile distillate added to bitumen to temporarily reduce

its viscosity e.g. Kerosene, or turpentine.

Flux A relatively non-volatile distillate which is blended with

bitumen to permanently or semi-permanently reduce the

viscosity of the bitumen. E.g. AGO (Diesel fuel).

AGO Automotive Gas Oil, Diesel fuel.

Residual Binder The non-volatile fraction of a binder that remains after

evaporation of the volatiles.

named for its penetration grade.

PMB Polymer-Modified Bitumen – a bitumen with polymer added to

reduce the temperature sensitivity of the binder, some

polymers make the binder more elastic.

PME Polymer-Modified Emulsion – an emulsion made with polymer

modified bitumen.

Diluent A substance that has the effect of reducing viscosity when

added to bitumen. See cutter or flux.