

NZTA T21: 2024

Test Methods for Epoxy Modified Open-Graded Porous Asphalt

1 Scope

This specification describes the procedures used to determine:

- (a) The concentration of epoxy modified bitumen (as weight percentage) and its components in the binder used to manufacture epoxy modified open graded porous asphalt. The methods were developed for analysis of Type V epoxy bitumen supplied by ChemCo Systems Ltd (California, USA) but may be applicable to other acid curing epoxy systems based on bisphenol-A diglycidyl ether epoxy resin or similar materials containing an aryl-alkyl ether group. Either of the three alternative methods using infrared spectroscopy (Methods A and B) or Fast Neutron Activation Analysis (FNAA) may be used.
- (b) The durability of oxidised, compacted specimens of epoxy modified open graded porous asphalt. Durability is measured using AGPT T236 Asphalt Particle Loss (the “Cantabro” test) after oxidation of specimens at 85°C for 40 days. The procedure is used to assess epoxy bitumen formulations for approval purposes according to the NZTA P11 specification.

DISCLAIMER

THESE TEST METHODS INVOLVE USE OF POTENTIALLY HAZARDOUS MATERIALS AND EQUIPMENT AND SHOULD ONLY BE UNDERTAKEN BY PROPERLY TRAINED LABORATORY PERSONNEL FOLLOWING APPROPRIATE HEALTH AND SAFETY PRACTICES.

2 Definitions

- (a) Epoxy modified open graded porous asphalt (EMOGPA): EMOGPA is an open graded mixture of coarse and fine aggregates, mineral filler, and an epoxy modified bitumen binder produced hot in a mixing plant and meeting NZTA P11 specification.
- (b) Epoxy modified bitumen: Epoxy modified bitumen is an acid curing, two-part thermosetting material consisting of bitumen and reactive components that cure through epoxide ring opening and cross-linking.
- (c) Diluted epoxy modified bitumen: Diluted epoxy modified bitumen is epoxy modified bitumen to which bitumen, compliant with NZTA M01 or NZTA M01-A specification and free from solvents, polymers, acid modifiers or other additives, has been added in some proportion.

3 Related Documents

- (a) NZTA M01 Specification for Roading Bitumens
- (b) NZTA M01-A Specification for Performance-Graded Asphalt Binder
- (c) NZTA P11 Specification for Open-Graded Porous Asphalt
- (d) AGPT T212 Gyrotory Compactor Test Method
- (e) Austroads AGPT T236 Asphalt Particle Loss
- (f) AASHTO T 312 Standard Method of Test for Preparing and Determining the Density of Asphalt Mixture Specimens by Means of the Superpave Gyrotory Compactor
- (g) ASTM D3203 Percent Air Voids in Compacted Dense and Open Bituminous Paving Mixtures
- (h) ASTM D3549 Standard Test Method for Thickness or Height of Compacted Bituminous Paving Mixtures
- (i) ASTM D6925 Preparation and Determination of the Relative Density of Hot Mix Asphalt (HMA) Specimens by Means of the Superpave Gyrotory Compactor

4 Determination of the Epoxy Bitumen Concentration by Infrared Spectroscopy

4.1 General

The concentration of epoxy resin (part A) and hardener (part B) in epoxy bitumen is determined by infrared spectroscopy. Either of the two alternative methods A and B may be used.

4.2 Method A

4.2.1 General

Changes in the infra-red (IR) spectra due to curing of the epoxy components between sampling and measurement are accounted for using a curing-correction curve.

4.2.2 Equipment

- (a) Infra-red spectrometer with an Attenuated Total Reflectance (ATR) accessory capable of measuring samples over a range of at least 800cm^{-1} to 4500cm^{-1} at a resolution of 4cm^{-1} . The instrument shall have a method of removing spectral interferences from carbon dioxide and water in the atmosphere, for example by purging with dry, carbon dioxide-free air or through software subtraction.
- (b) A balance, readable and accurate to 0.01g .
- (c) Fan assisted oven capable of maintaining a temperature of $125^{\circ}\text{C} \pm 5^{\circ}\text{C}$.

4.2.3 Procedure

Use the following method to record the infra-red spectrum:

- (a) Follow the manufacturer's instructions for proper setup, absorbance and wavelength calibration of the infrared spectrometer.
- (b) Run a background infra-red spectrum over the range of at least 800cm^{-1} to 2000cm^{-1} .
- (c) With a spatula, smear a small amount of the diluted epoxy modified bitumen to be tested onto the ATR crystal. The film thickness should be such that the light path is completely attenuated within the sample and the sample completely covers the exposed crystal surface.
- (d) Measure the infra-red spectrum over the range of at least 800cm^{-1} to 2000cm^{-1} using the background spectrum from step (b) above. An example spectrum for a diluted epoxy modified bitumen is shown in Figure 1 in clause 7.
- (e) Repeat the procedure in steps (c) and (d) above twice more so that triplicate spectra are obtained. If the spectra are run more than 30 minutes apart, a fresh background spectrum must be recorded.
- (f) From the triplicate spectra determine the mean value to at least 3 decimal places for each of A_{1249} , A_{1725} , H_{1708} , and H_{1738} , where:
 - (i) A_{1249} = The area of the absorbance peak at 1249cm^{-1} using a baseline drawn from 1203cm^{-1} to 1275cm^{-1} .
 - (ii) A_{1725} = The area of the doublet absorbance peak centred at 1725cm^{-1} using a baseline drawn from 1675cm^{-1} to 1772cm^{-1} .
 - (iii) H_{1708} = The height of the absorbance peak at 1708cm^{-1} using a horizontal baseline drawn from 1810cm^{-1} .
 - (iv) H_{1738} = The height of the absorbance peak at 1738cm^{-1} using a horizontal baseline drawn from 1810cm^{-1} .
- (g) Curing affects the area of the absorbance peaks measured in step (d). Calculate the curing correction value, α , (to 3 decimal places) as:

$$\alpha = \frac{H_{1708}}{H_{1738}}$$

4.2.4 Concentration calibration curves

- Prepare at least six different standard mixtures of diluted epoxy modified bitumen using the epoxy bitumen components in the relative proportions specified by the epoxy bitumen supplier. The epoxy bitumen components and diluting bitumen used must be from the same bulk supply as that used for manufacture of the plant samples being tested. The concentrations of the standard mixtures must span the target epoxy bitumen concentration by at least $\pm 10\%$ (e.g. for a target concentration of 25% epoxy bitumen, standard mixtures should be prepared in the range of 15% to 35%).
- To minimise weighing errors each standard mixture must have a total weight between 100g and 200g. Weigh each component to a precision of 0.01g into a 250ml metal can. Calculate the weight percentage concentrations of Part A and Part B to one decimal place.
- Heat each standard mixture in a fan assisted oven at 125°C for 10 ± 1 minutes, remove, stir well with a spatula for up to one minute and allow to cool to room temperature. Record the spectra of each standard mixture as described in 4.2.3 steps (a) to (f) within one hour of mixing (so α can be assumed to be negligible).
- Plot the percentage Part A (x-axis) against A_{1249} (y-axis) and similarly percentage Part B against A_{1725} and perform a regression to calculate the best fit line or polynomial in each case. As the epoxy is a reactive system, a quadratic function generally gives a better fit than a linear regression. Determine the regression parameters, to four decimal places.

4.2.5 Curing correction curves

- Prepare a standard mixture of diluted epoxy modified bitumen at the target epoxy bitumen concentration using the epoxy bitumen components in the relative proportions specified by the epoxy bitumen supplier.
- The standard mixture must have a total weight between 100g and 200g. Weigh each component to a precision of 0.01g into a metal can. Heat the mixture in a fan assisted oven at 125°C for 10 ± 1 minutes, remove and stir well with a spatula for up to one minute.
- Remove a zero-time sample (1g to 2g), and take additional samples as follows.
Heat the can on a hotplate so that the temperature of the binder is between 120°C and 140°C . The binder must be gently stirred without splashing or entrainment of air. Ensure that material on the sides of the can is occasionally scraped and mixed back with the bulk material. A mechanical overhead stirrer operating at 100rpm to 120rpm is suitable, but if stirred manually the material must not be left unstirred for more than one minute at any time. Remove at least 12 samples from the mixture at about 5 minute intervals. Approximately 1g to 2g each is sufficient.
- Allow samples to cool to room temperature and for each sample record triplicate spectra as described in 4.2.3 steps (a) to (g).
- The samples should span the curing ratios expected from the plant samples to be analysed. Repeat the process with a freshly prepared standard mixture to obtain additional samples if required.
- From each sample's triplicate spectra determine the mean value (to at least 3 decimal places) for each of $A_{1249\text{CURE}}$, $A_{1725\text{CURE}}$, $H_{1708\text{CURE}}$, and $H_{1738\text{CURE}}$, where:
 - $A_{1249\text{CURE}}$ = The area of the absorbance peak at 1249cm^{-1} using a baseline drawn from 1203cm^{-1} to 1275cm^{-1} .
 - $A_{1725\text{CURE}}$ = The area of the doublet absorbance peak centred at 1725cm^{-1} using a baseline drawn from 1675cm^{-1} to 1772cm^{-1} .
 - $H_{1708\text{CURE}}$ = The height of the absorbance peak at 1708cm^{-1} using a horizontal baseline drawn from 1810cm^{-1} .
 - $H_{1738\text{CURE}}$ = The height of the absorbance peak at 1738cm^{-1} using a horizontal baseline drawn from 1810cm^{-1} .

(v) $A_{1249ZERO}$ and $A_{1725ZERO}$ values are respectively $A_{1249CURE}$ and $A_{1725CURE}$ values determined for the zero-time sample (clause 4.2.4 step (d)).

(g) For each sample calculate the curing correction value, α_{CURE} (to 3 decimal places) as:

$$\alpha_{CURE} = \frac{H_{1708CURE}}{H_{1738CURE}}$$

(h) For each sample calculate the curing correction factor $A_{1249CURECF}$ values as:

$$A_{1249CURECF} = \frac{A_{1249ZERO}}{A_{1249CURE}}$$

(i) Similarly calculate the curing correction factor $A_{1725CURECF}$ values as:

$$A_{1725CURECF} = \frac{A_{1725ZERO}}{A_{1725CURE}}$$

(j) Plot α_{CURE} (x-axis) against $A_{1249CURECF}$ (y-axis) and perform a piece-wise linear regression to calculate the best fit lines to the data. Repeat the process for the $A_{1725CURECF}$ values (see clause 7 Figure 2 for an example).

(k) For each test sample use the value α determined in 4.2.3 step (g) to calculate the appropriate value of $A_{1249CURECF}$ and $A_{1725CURECF}$ from the respective regression equations determined in 4.2.4 step (d).

(l) Calculate the corrected value of A_{1249} , $A_{1249CORR}$, to two decimal places as:

$$A_{1249CORR} = A_{1249}(A_{1249CURECF})$$

(m) Calculate the corrected value of A_{1725} , $A_{1725CORR}$, to two decimal places as:

$$A_{1725CORR} = A_{1725}(A_{1725CURECF})$$

4.2.6 Calculations

(a) Using the concentration calibration curve for Part A (4.2.4 step (d)) and the value of $A_{1249CORR}$ (4.2.5 step (l)) calculate the corrected percentage Part A, % Part A (corr).

(b) Using the concentration calibration curve for Part B (4.2.4 step (d)) and the value of $A_{1725CORR}$ (4.2.5 step (m)) calculate the corrected percentage Part B, % Part B (corr).

(c) Calculate the component ratio β , as:

$$\beta = \frac{\% \text{ Part A (corr)}}{\% \text{ Part B (corr)}}$$

(d) Using the component proportions specified by the epoxy bitumen supplier, where % Part A (STD) and % Part B (STD) are the percentages of Part A and B respectively specified by the supplier for a 25% diluted epoxy bitumen blend, calculate:

$$\beta_{STD} = \frac{\% \text{ Part A (STD)}}{\% \text{ Part B (STD)}}$$

(e) Calculate the component ratio difference, β_{DIFF} , as:

$$\beta_{DIFF} = \beta_{STD} - \beta$$

(f) Calculate the percentage epoxy bitumen content as:

$$\% \text{ epoxy bitumen} = \% \text{ Part A (corr)} + \% \text{ Part B (corr)}$$

4.2.7 Reporting

Report the following:

- The corrected percentage Part A calculated in 4.2.6 step (a) to the nearest percent.
- The corrected percentage Part B calculated in 4.2.6 step (b) to the nearest percent.
- The component ratio difference (β_{DIFF}) calculated in 4.2.6 step (c) to two decimal places.

- (d) The percentage epoxy bitumen calculated in 4.2.6 step (f) to the nearest percent.

4.3 Method B

Changes in the infra-red spectra due to curing of the epoxy components between sampling and measurement are removed by heating the samples for an extended period. This method is intended to determine epoxy bitumen concentrations of approximately 25% and may not be suitable for determining concentrations above 35%.

4.3.1 Equipment

- (a) Infrared spectrometer with an Attenuated Total Reflectance (ATR) accessory capable of measuring samples over a range of at least 800cm^{-1} to 4500cm^{-1} at a resolution of 4cm^{-1} . The instrument shall have a method of removing spectral interferences from carbon dioxide and water in the atmosphere, for example by purging with dry, carbon dioxide-free air or through software subtraction.
- (b) A balance, readable and accurate to 0.01g.
- (c) Fan assisted oven capable of maintaining a temperature of $125^{\circ}\text{C} \pm 5^{\circ}\text{C}$.

4.3.2 Pre-treatment

- (a) Heat the test sample in the oven at 125°C for 2 hours \pm 5 minutes, remove, stir well with a clean spatula for approximately 30 seconds and replace in the oven for another 8 hours \pm 5 minutes. Remove the samples and stir well with a clean spatula for approximately 30 seconds. Record the spectra of each standard mixture as described in clause 4.3.3 steps (a) to (f).

Note: The total pre-measurement heating time of 10 hours may be reduced if evidence satisfactory to the principal is provided to show that the peak areas defined in 4.3.3 (f) have reached a constant value. The same premeasurement heating time must be used for the calibration standards (4.3.4) and the test samples.

4.3.3 Record the infra-red spectrum

- (a) Follow the manufacturer's instructions for proper setup, absorbance and wavelength calibration of the infrared spectrometer.
- (b) Run a background infrared spectrum over the range of at least 800cm^{-1} to 2000cm^{-1} .
- (c) With a spatula, smear a small amount of the diluted epoxy modified bitumen to be tested onto the ATR crystal. The film thickness should be such that the light path is completely attenuated within the sample and the sample completely covers the exposed crystal surface.
- (d) Measure the infrared spectrum over the range of at least 800cm^{-1} to 2000cm^{-1} using the background spectrum from 4.3.3 (b). An example spectrum for a diluted epoxy modified bitumen is shown in clause 7 Figure 1.
- (e) Clean the ATR crystal and repeat the procedure from steps (c) and (d) above twice more so that triplicate spectra are obtained. If the spectra are run more than 30 minutes apart a fresh background spectrum must be recorded.
- (f) From the triplicate spectra determine the mean value (to at least 3 decimal places) for each of A_{1249} , A_{1725} where:

A_{1249} = The area of the absorbance peak at 1249cm^{-1} using a baseline drawn from 1203cm^{-1} to 1275cm^{-1} .

A_{1725} = The area of the doublet absorbance peak centred at 1725cm^{-1} using a baseline drawn from 1675cm^{-1} to 1772cm^{-1} .

4.3.4 Concentration calibration curves

- (a) Prepare at least five different standard mixtures of diluted epoxy modified bitumen, with epoxy bitumen components in the relative proportions specified by the supplier. The epoxy bitumen components and diluting bitumen used must be from the same bulk supply as that used for manufacture of the plant samples being tested. The concentrations of the standard mixtures must span the target epoxy bitumen concentration by at least $\pm 10\%$ (for a target concentration of 25% epoxy bitumen, standard mixtures of 10%, 15%, 20%, 25%, 30% and 35% should be prepared).
- (b) To minimise weighing errors each standard mixture must have a total weight between 100g and 200g. Weigh each component to a precision of 0.01g into a 250ml metal can. Calculate the weight percentage concentrations of Part A and Part B to one decimal place.
- (c) Heat each standard mixture in the oven at 125°C for 15 ± 1 minutes, remove, stir well with a clean spatula for approximately 30 seconds and replace in the oven for another 9 hours and 45 minutes ± 5 minutes. Remove the samples and stir well with a clean spatula for approximately 30 seconds. Record the spectra of each standard mixture as described in 4.3.3 steps (a) to (f).

Note: The total premeasurement heating time of 10 hours may be reduced if evidence satisfactory to the principal is provided to show that the peak areas defined in 4.3.3 (f) have reached a constant value. The same premeasurement heating time must be used for the standards and test samples.

- (d) Plot the weight percentage Part A (x-axis) against A_{1249} (y-axis) and similarly weight percentage Part B against A_{1725} and perform a regression to calculate the best fit line or polynomial in each case. As the epoxy is a reactive system, a quadratic function generally gives a better fit than a linear regression. Determine the regression parameters, to four decimal places.

4.3.5 Calculations

- (a) Calculate the percentage Part A (% Part A) for the test sample using the concentration calibration curve for Part A (4.3.4 (d)) and the value of A_{1249} (4.3.4 step (f)).
- (b) Calculate the percentage Part B (% Part B) for the test sample using the concentration calibration curve for Part B (4.3.4 (d)) and the value of A_{1725} (4.3.4 step (f)).
- (c) Calculate the component ratio β , as:

$$\beta = \frac{\% \text{ Part A (corr)}}{\% \text{ Part B (corr)}}$$

- (d) Using the component proportions specified by the epoxy bitumen supplier, where % Part A (STD) and % Part B (STD) are the percentages of Part A and B respectively specified by the supplier for a 25% diluted epoxy bitumen blend, calculate:

$$\beta_{STD} = \frac{\% \text{ Part A (STD)}}{\% \text{ Part B (STD)}}$$

- (e) Calculate the component ratio difference, β_{DIFF} , as:

$$\beta_{DIFF} = \beta_{STD} - \beta$$

- (f) Calculate the percentage epoxy bitumen content as:

$$\% \text{ epoxy bitumen} = \% \text{ Part A} + \% \text{ Part B}$$

4.3.6 Reporting

Report the following:

- (a) The percentage Part A calculated in 4.3.5 (a) to the nearest percent.
- (b) The percentage Part B calculated in 4.3.5 (b) to the nearest percent.
- (c) The component ratio difference (β_{DIFF}) calculated in 4.3.5 (e) to two decimal places.
- (d) The percentage epoxy bitumen calculated in 4.3.5 (f) to the nearest percent.

5 Determination of the Epoxy Bitumen Concentration by Fast Neutron Activation Analysis

5.1 General

The total concentration of epoxy resin (Part A) and hardener (Part B) in epoxy bitumen is determined by measurement of the elemental oxygen content using fast neutron activation analysis (FNAA).

5.2 Equipment

- (a) A suitable neutron source, gamma ray detector and associated sample handling and data processing instrumentation are required. These are currently not commercially available in New Zealand. Suitable testing facilities are available through the Texas A&M University, Center for Chemical Characterisation & Analysis (<https://research.tamu.edu/centers/center-for-chemical-characterization-and-analysis/>)
- (b) A balance, readable and accurate to 0.01g.
- (c) Fan assisted oven capable of maintaining a temperature of $125^{\circ}\text{C} \pm 5^{\circ}\text{C}$.

5.3 Procedure

5.3.1 Concentration calibration curves

- (a) Prepare at least ten different standard mixtures of diluted epoxy modified bitumen (with the epoxy bitumen components in the relative proportions specified by the epoxy bitumen supplier). The epoxy bitumen components and diluting bitumen used must be from the same bulk supply as that used for manufacture of the plant samples being tested. The concentrations of the standard mixtures must span the target epoxy bitumen concentration by at least $\pm 10\%$ (e.g. for a target concentration of 25% epoxy bitumen, standard mixtures should be prepared in the range of at least 15% to 35%) and must also include a 0% standard (i.e. the diluting bitumen).
- (b) To minimise weighing errors each standard mixture must have a total weight between 100g and 200g. Weigh each component to a precision of 0.01g into a 250ml metal can. Calculate the weight percentage concentrations of epoxy bitumen to one decimal place.
- (c) Heat each standard mixture in the oven at 125°C for 10 ± 1 minutes, remove and stir well with a spatula for up to one minute and transfer 15g - 20g of material to a clean glass jar for subsequent FNAA analysis.
- (d) Have the elemental oxygen content of the standards measured by FNAA. Plot the percentage oxygen content (y-axis) against the epoxy bitumen concentration (x-axis) and perform a regression to calculate the best fit line.

5.3.2 Elemental oxygen content of test samples

Transfer 15g - 20g of the diluted epoxy modified bitumen to be tested to a glass jar and have the elemental oxygen content measured by FNAA.

5.4 Calculation

Using the concentration calibration curve (5.3.1) and the elemental oxygen content of the test sample calculate the percentage of epoxy bitumen present.

5.5 Reporting

Report the epoxy bitumen content calculated in clause 5.4 to the nearest percent.

6 Durability Assessment for Approval of Epoxy Bitumen Formulations

The durability of an epoxy bitumen formulation is assessed by accelerated aging of compacted specimens followed by Asphalt Particle Loss (“Cantabro”) testing. Results for the test formulation are compared to control measurements using unmodified bitumen and the Chemco Type V product.

6.1 Equipment

- (a) Fan assisted oven capable of maintaining a temperature of $125^{\circ}\text{C} \pm 3^{\circ}\text{C}$, for preparing test specimens.
- (b) Fan assisted oven capable of maintaining a temperature of $85^{\circ}\text{C} \pm 3^{\circ}\text{C}$, for curing specimens.
- (c) Fan assisted cabinet capable of maintaining a temperature of $10^{\circ}\text{C} \pm 1^{\circ}\text{C}$, for conditioning specimens prior to testing.
- (d) Enclosure or cabinet capable of maintaining the abrasion test machine at a temperature of $10^{\circ}\text{C} \pm 3^{\circ}\text{C}$ where the temperature is measured in air inside the drum of the machine without the lid in place.

6.2 Procedure

6.2.1 Preparation of Test Specimens

Three sets of six specimens are prepared using the epoxy bitumen formulation under test (“test” formulation), the Chemco Type V epoxy bitumen (“benchmark” formulation) and the diluting bitumen alone (“control” formulation).

Using the epoxy bitumen formulation under test, prepare at least six specimens using the procedure given in AASHTO T 312, or ASTM D6925 or Austroads T212 amended as follows:

- (a) The conditioning time specified shall be 60 ± 10 minutes at 125°C
- (b) The specimens shall be compacted using 50 gyratory cycles.

A mix design meeting the requirements of the EPA 10 mix envelope of NZTA specification P11 must be used. Diluted epoxy bitumen (see clause 2(c)) at the concentration specified by the manufacturer shall be used to prepare the specimens. The total binder content of the mix shall not exceed 5.5% and the air voids shall be in the range of 20% - 25%.

6.2.2 Testing

- (a) Measure the volume of the specimens according to ASTM D3549 and calculate the air voids according to ASTM D3203.
- (b) Tightly wrap the sides only of the test specimens with silicone release paper or similar, to provide support and prevent slumping. Place the specimens in an 85°C oven within 24 to 48 hours of manufacture. To prevent damage to the specimens they must be supported on a flat, solid oven tray (not a wire rack).
- (c) After 480 ± 4 hours (20 days) carefully remove the tray of specimens (without handling the individual specimens) and allow the specimens to cool on the tray to room temperature for 1 to 2 hours. Invert the specimens so their tops are now in contact with the tray and replace immediately into the oven.
- (d) After a total of 960 ± 4 hours (40 days) has elapsed, carefully remove the tray of specimens and allow the specimens to cool on the tray to room temperature for 1 to 2 hours. Place the specimens in a 10°C cabinet and allow to come to temperature for at least 4 hours.
- (e) Determine the Asphalt Particle Loss for each of the test specimens using the method of AGPT T236, with the following amendments to the method:
 - (i) Conduct the abrasion loss testing at a temperature of 10°C .
 - (ii) Omit the moisture conditioning steps of AGPT T236 clause 2.2.

- (iii) Carry our density testing as specified in 6.2.2 above.

All specimens must be tested within 48 hours of removal from the curing oven (6.2.2(d)).

- (f) For “benchmark” formulation, repeat steps 6.2.2 (a) to (e) using the Chemco Type V epoxy bitumen diluted to 25% concentration with the same diluting bitumen used for the test formulation. The binder content, aggregate source and particle size distribution shall be the same as that used for preparation of the “test” formulation specimens.
- (g) For “control” formulation, repeat steps 6.2.2 (a) to (e) using the same diluting bitumen used for the test formulation (without epoxy bitumen). The binder content, aggregate source and particle size distribution shall be the same as that used for preparation of the “test” formulation specimens.

6.3 Calculations

- (d) For each set of six specimens (test, benchmark and control formulations), calculate the percentage mass loss, M_L , from each specimen to the nearest percent as:

$$M_L = \frac{(m_i - m_f)}{m_i} \times 100$$

- (e) Examine the data for outliers at the 95% confidence level using Dixon’s Q or Grubbs test. Remove any outliers and calculate the mean (\bar{x}) and standard deviation (s) of the M_L values for the remaining specimens in the set.
- (f) Use the mean mass loss for the test formulation (\bar{x}_{test}) and that of the benchmark formulation ($\bar{x}_{benchmark}$) to calculate the t value:

$$t = \frac{(\bar{x}_{benchmark} - \bar{x}_{test})}{\sqrt{\frac{S_{benchmark}^2}{n_{benchmark}} + \frac{S_{test}^2}{n_{test}}}}$$

Where: s = the sample standard deviation for each set of specimens

n = the number of test specimens in the set

- (g) Calculate the degrees of freedom (rounded to the nearest whole number):

$$\text{Degrees of freedom} = \left\{ \frac{\left(\frac{S_{benchmark}^2}{n_{benchmark}} + \frac{S_{test}^2}{n_{test}} \right)^2}{\left(\frac{S_{benchmark}^2}{n_{benchmark}} \right)^2 + \left(\frac{S_{test}^2}{n_{test}} \right)^2} \right\} - 2$$

6.4 Reporting

For each set of specimens (test, benchmark and control formulations), report the following:

- (a) The M_L value and percentage air voids for each specimen, noting any found to be outliers (see 6.3).
- (b) The mean (\bar{x}) and standard deviation (s) of the M_L values for the sample (excluding outliers).
- (c) The t value and degrees of freedom
- (d) The percentage air voids for each specimen (excluding outliers), measured according to ASTM D3203.

7 Example Infra-Red Spectrum and Curing Correction Curve

Figure 1 is an example infrared spectrum (4.2.3). Figure 2 is an example of a curing correction curve (4.2.5).

Figure 1. Example infrared spectrum of a diluted epoxy bitumen (epoxy bitumen (25% w/w), mixed with 80/100 penetration grade bitumen (75% w/w)). The area of the 1249 cm^{-1} peak and the area of the peak doublet centred at 1725 cm^{-1} , are shaded.

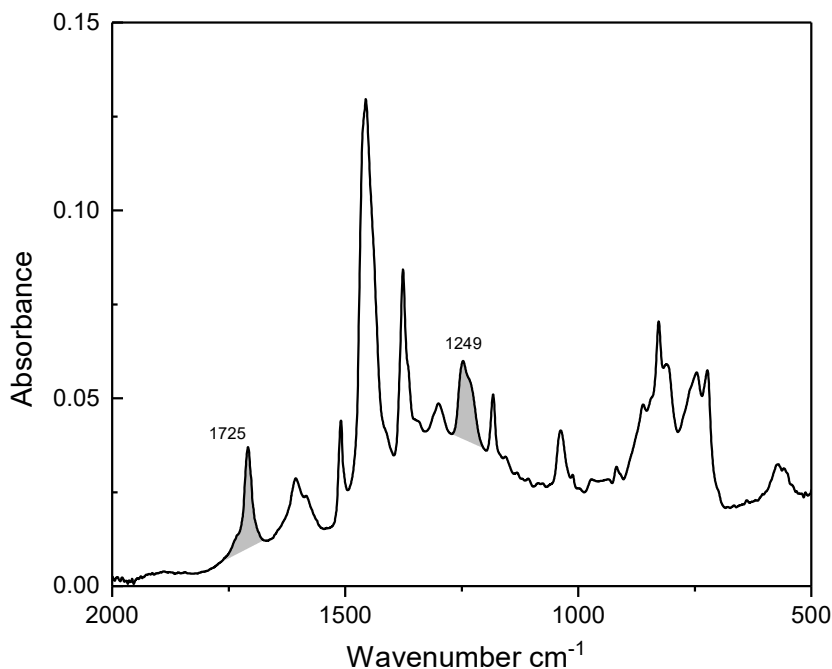


Figure 2 Example curing correction curve for the 1725 cm^{-1} peak area showing a piece-wise linear regression.

