

COMMENTARY TO AG:PT/T142 - RUBBER CONTENT OF DIGESTED CRUMB RUBBER BINDERS – TRICHLOR BATH METHOD

PREFACE

This modified binder test method was developed by ARRB Group on behalf of Austroads. Representatives of Austroads, ARRB Group and the Australian Asphalt Pavement Association have been involved in the review of this test method.

FOREWORD

Crumb rubber modified binders are prepared from mixtures of bitumen, crumb rubber and additives selected to optimise the properties of the digested binder system. The concentration of crumb rubber is considered to be an important performance controlling characteristic of these binders. This test method provides a measure of the crumb rubber concentration through the use of a trichlor-ethylene degreasing bath.

SCOPE

This method describes a procedure for determining the crumb rubber content of a digested crumb rubber modified binder. The concentration of rubber can be lower than the original composition would suggest due to the potential loss in the digestion process. This test determines the particulate rubber content of digested crumb rubber/bitumen binder mixtures used for sprayed sealing work.

Future Developments

None.

RUBBER CONTENT OF DIGESTED CRUMB RUBBER BINDERS – TRICHLOR BATH METHOD

1. REFERENCED DOCUMENTS

The following documents are referred to in this test method:

AUSTROADS

AG:PT/T101 Method of sampling polymer modified binders, polymers and crumb rubber.

AS/NZS

1141.3.1 – Methods for sampling and testing aggregates: Sampling - Aggregates

1152 - Test Sieves

ARRB GROUP

Method 25 A bulk density test to characterise the morphology of rubber particles.

2. APPARATUS and MATERIALS

The following apparatus and materials are required in the performance of this test:

- a. Solvent - commercial grade 1.1.1 – trichloroethane or trichlor-ethylene,
- b. Filter papers - Whatman No. 4 or equivalent, 320 mm in diameter.
- c. White, 3 ply Valentine table napkin 430 x 420 mm.
- d. Wire Mesh Basket - constructed of woven wire cloth of aperture 150 μm , which conforms to the requirements of AS 1152. The basket shall be 120 mm high by 90 mm square and reinforced along the edges by metal rod.
- e. Balance - accurate and readable to 0.01 g
- f. Vacuum oven - capable of being maintained at $100^{\circ}\text{C} \pm 5^{\circ}\text{C}$ and a pressure of 3 kPa or less.
- g. Oven - capable of being maintained at $160^{\circ}\text{C} \pm 10^{\circ}\text{C}$
- h. Wooden spatula
- i. Metal clips - to secure edges of filter to wire basket.
- j. Container with lid - to hold solvent, of sufficient size to accommodate one or more wire mesh baskets.
- k. Vessels - to dispense and receive solvent poured through the basket.

3. PROCEDURES

3.1 Sample preparation

Samples for testing shall be provided in accordance with AG:PT/T101.

For bulk samples of less than 1 kg mass a sub-sample shall be obtained by pouring the bulk sample evenly over the top of the riffle box and thus dividing it into two representative parts, which are collected in boxes placed beneath the chutes. The contents of one box is then divided into two by further riffling and the process repeated until a sub-sample of suitable size (20 to 25 g) for sieving is obtained.

For bulk samples greater than 1 kg mass, a sample less than 1 kg shall be obtained by a systematic sample reduction procedure such as that used for fine mineral aggregates and as described in AS 1141.3.1.

3.2 Method

The procedure shall be as follows:

- a. Dry filter papers in the vacuum oven at $100^{\circ}\text{C} \pm 5^{\circ}\text{C}$ and a pressure of 3 kPa or less,
- b. Allow the papers to cool for 5 minutes and weigh. Designate this mass as M_1 .
- c. Fold No.4 filter paper and line the wire mesh basket then line the filter paper with coarsely folded napkin and secure the napkin to the edge of the basket with wire clips.
- d. Heat sample in oven at $160^{\circ}\text{C} \pm 10^{\circ}\text{C}$ until contents are sufficiently fluid to pour.
- e. Tare prepared basket on balance and, using wooden spatula, transfer as much of the sample as possible to the basket. Record mass of sample as M_2 .
- f. Allow sample to cool and place in container of solvent so that the solvent level is 25 mm below the top of the basket,
- g. Place lid on container and allow to stand overnight.
- h. Remove basket and allow solvent to drain.
- i. Pour fresh solvent through the basket assembly until issuing solution is pale yellow in colour (see Note 1). The basket should be filled to no higher than 25 mm from the top.
- j. Remove filter paper assembly and rubber and Allow to air dry overnight.
- k. Dry filter papers and rubber in the vacuum oven for 1 hour at $100 \pm 5^{\circ}\text{C}$ and a pressure of 3 kPa or less.
- l. Cool and weigh filter papers and rubber. Designate this mass as M_3 .

5. CALCULATIONS

Calculate the concentration of rubber in the test portion by the following formula:

$$P = \frac{M_3 - M_1}{M_2 - M_1}$$

M₁ mass of filter papers

M₂ mass of sample, basket and filter papers

M₃ mass of residual sample and filter papers

Percent rubber = P x 100

6. REPORTING

The following should be included in a test report:

- a. Sample identification
- b. The concentration of rubber as the mean of two determinations, made on separate sub-samples, to the nearest 0.1 percent.
- c. Test date
- d. Reference to this method, i.e. AG:PT/T142.

7. PRECISION

The following criteria should be used for judging the acceptability of test results (95 per cent probability).

- a. Repeatability* Duplicate test results obtained by the same operator should not be considered suspect unless they differ by more than 0.8.
- b. Reproducibility. Not determined.

* Based on the results of single determinations of percentage rubber made on 5 specimens separately manufactured to have identical rubber contents (data given in Table I).

Note

1. This can conveniently be done in an industrial degreasing bath which can also be used to distil the contaminated solvent for reuse.

Appendix A Correction for rubber loss on digestion

(Informative)

This test method was developed to measure the rubber content of crumb rubber/bitumen digestions laid in road trials. It has two further applications:

- in the assessment of the homogeneity of crumb rubber/bitumen binder in a sprayer determined by collecting a series of binder samples at intervals during the spraying run
- in routine quality control of commercial crumb rubber blends.

Where an improved estimate of added rubber is required, an estimate of the quantity of rubber lost in the digestion process can be made. To perform this procedure the following steps should be followed:

Repeat the procedure (a) to (l) using a rubber/bitumen reference sample which has been manufactured in the laboratory by digesting a known mass of rubber, M_4 , obtained from the same source as that in the test sample, in bitumen. The digestion time shall not differ from that of the test sample by more than 5 minutes in the case of test samples digested for 30 minutes or less, by more than 10 minutes in the case of test samples digested for 1 hour or less and 15 minutes for test samples digested for more than 1 hour. The digestion temperature of the reference sample shall not differ from that of the test sample by more than 10°C. In this case, designate the mass of dried filter papers as M_5 and the dried filter papers and rubber as M_6 .

The following shall be calculated.

Proportion of rubber recovered by filtration as determined from the reference sample,

$$P = \frac{M_6 - M_5}{M_4}$$

M_4 mass of sample

M_5 mass of filter papers

M_6 mass of recovered sample and filter papers

Mass of rubber in test sample,

$$P = \frac{M_6 - M_5}{M_4}$$

TABLE I
Results of carrying out test procedure on five different samples
manufactured to have the same rubber content
(Rubber Type: Commercial Crumb Tyre with ARRB Improved Morphology)

| | | | | | |
|------------------------------------|-------|-------|-------|-------|-------|
| Digestion Time (h) | 0.5 | 1.0 | 2.0 | 0.5 | 2.0 |
| Proportion of rubber recovered (P) | 0.791 | 0.791 | 0.780 | 0.715 | 0.694 |
| Rubber Content | 14.6 | 14.6 | 14.9 | 14.6 | 14.1 |

- Notes: (1) Test samples were prepared with a rubber content of 14.5 %
(2) Digestion Temperature 190 - 200°C for all samples,

AMENDMENT RECORD

| Amendment No. | Clauses amended | Action | Date |
|---------------|--|--------------|--------------|
| 1 | This method has been developed from ARRB-Transport Research test method (reported in AIR 286-3 April 1981) | New | January 2002 |
| 2 | Commentary Page | New | October 2004 |
| | Footer and header | Format | |
| | Applied revised test method number | Format | |
| | Applied new styles | Format | |
| 3 | Applied new test method numbers | Substitution | March 2006 |
| | Moved notes to the end of the method | Format | |

Key

| | |
|--------------|---|
| Format | Change in format |
| Substitution | Old clause removed and replaced with new clause |
| New | Insertion of new clause |
| Removed | Old clauses removed |