



**Transport**  
Roads & Maritime  
Services

# Test method T737

## Rubber content of a scrap rubber mix

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## Revision Summary

Ed/Rev Number	Clause Number	Description of Revision	Authorisation	Date
		Reformatted and Revision Summary added	D. Dash	Jan 2000
Ed 1/ Rev 0	All	New Issue. Title Revised	J. Friedrich	Mar 2009
Ed 2/ Rev 0	All	Reformatted RMS template	J. Friedrich	November 2012

Note that Roads and Maritime Services is hereafter referred to as 'RMS'.

The most recent revision to Test method T737 (other than minor editorial changes) are indicated by a vertical line in the margin as shown here.

# Test method T737

## Rubber content of a scrap rubber mix

### 1. Scope

This test method sets out the procedure for determining the particulate rubber content of digested scrap rubber in a bitumen mixture using reflux extraction.

*NOTE: The method is adapted from the ARRB method AIR 286–5, 1982 and has been compared with Austroads' method AG:PT/T142.*

### 2. General

- (a) The method is applicable to bituminous mixes containing rubber particles, which were produced by comminuting vehicle tyres.
- (b) The test includes the following steps:
  - (i) The rubber bitumen binder sample is dissolved in solvent to extract the bitumen. The process involves continuous washing with clean solvent provided by the Soxhlet apparatus. The recovered rubber is dried and mass determined.
  - (ii) A Rubber Recovery factor is used to allow for loss of soluble rubber components.
  - (iii) The Rubber Content is determined for the mass of recovered rubber.

- (c) The following materials are required for the test:

- (i) Solvent – commercial grade toluene conforming to AS 3529.

*NOTE: Other solvents such as 1,1,1-trichloroethane or 1-bromopropane may be used but must be approved by the client and clearly stated on the report.*

- (ii) 4 extraction thimbles cellulose, single thickness, external dimensions 62 x 180 mm (Whatman Cat No. 2800 608) or 28 x 80 mm (Cat No. 2800 288).

*NOTE: This procedure uses 4 size 28 x 80 mm Soxhlet thimbles in a size 75 mm Soxhlet chamber. However, other combinations of thimbles may be appropriate for this method. An additional 4 thimbles may be required to determine the rubber recovery factor.*

- (iii) Cotton balls and lint-free tissue paper.

- (d) Referenced documents include:

- (i) Austroads Test Method AG:PT/T102 Protocol for Handling Polymer Modified Binders in the Laboratory (abbreviated as AG:PT/T102) available from:  
[http://www.austrroads.com.au/pdf/TestMethod/T102\\_Handling.pdf](http://www.austrroads.com.au/pdf/TestMethod/T102_Handling.pdf)

### 3. Apparatus

- (a) Soxhlet Apparatus with extraction chamber about 75 mm diameter and 190 mm high and condenser, complete with all fittings and a suitable round-bottomed flask.
- (b) Heating mantle with thermostat control.
- (c) A thermostatically controlled oven with good air circulation, which can be maintained within the range 105° to 110°C.
- (d) A balance of at least 500 g capacity and a limit of performance of 0.01 g.
- (e) Metal spoon slightly squeezed along minor axis of bowl.

*NOTE: Size of a teaspoon is suitable but not to be used for food afterwards.*

- (f) Beaker with 1 L capacity.
- (g) Desiccator.
- (h) Boiling chips.

- (i) Option: 75  $\mu\text{m}$  AS sieve (refer to Step 5.2(h)).

#### 4. Preparation

Prepare the sample of binder with additives according to AG:PT/T102.

#### 5. Procedure

##### 5.1 Specimen

- (a) Wrap a cotton wool ball in paper tissue so that it is a tight fit into a Soxhlet thimble. Mark the thimble in a manner which will not be erased by hot solvent. Repeat for all Soxhlet thimbles.

*NOTE: Small nicks or crimp marks may be used to distinguish samples.*

- (b) Heat thimbles in an oven at 105° to 110°C for at least 3 hours then cool in a desiccator.
- (c) When cool and without delay:
- (i) Remove one thimble from the desiccator.
  - (ii) Determine the mass of the thimble and cotton plug ( $M_1$ ) to 0.01 g.
  - (iii) Return the thimble to the desiccator.
- (d) Stir softened sample (refer to Step **Error! Reference source not found.**) with the spoon until the spoon is heated.
- (e) Taking one Soxhlet thimble at a time:

*NOTE: Replicate testing is not required by this method.*

- (i) Remove cotton plug and add sample by spoon to just under fill half the thimble to form the specimen.

*NOTE Aim for a mass between 10 and 30 g.*

- (ii) Replace the cotton plug so that it is tight all round and immediately return the specimen upright in the desiccator.

*NOTE: Ensure that the entire cotton plug is replaced otherwise  $M_1$  will be changed.*

- (f) Record the specimen number against the record of thimble marking.
- (g) When cool and without delay (allow about 2 hours):
- (i) Remove one specimen from the desiccator.
  - (ii) Determine the mass of the thimble, specimen and cotton plug ( $M_2$ ) to 0.01 g.
  - (iii) Return the specimen to the desiccator until required for testing.

##### 5.2 Testing

- (a) Remove the specimens from the desiccator.
- (b) Load the appropriate number of specimens (i.e. Soxhlet thimbles) into the Soxhlet apparatus. Position so that the lip of the thimble is above the overflow level of the Soxhlet.
- (c) Add to the boiling flask sufficient solvent for refluxing. Add several boiling chips.
- (d) Assemble the Soxhlet with the reflux condenser on top.
- (e) Switch on the Soxhlet and run the apparatus:

*NOTE: Splashing of reflux solvent should not be excessive.*

*This step typically requires 10 hours refluxing spread over 3 days. The process may be turned off at night.*

- (i) While running, monitor the process to ensure that condenser water is still flowing and apparatus is functioning correctly.

*NOTE: Monitoring should be at least every hour.*

- (ii) If the process is turned off, ensure that the sample chamber is at least half full of solvent.

- (iii) Continue the process until the siphoning liquid is clear or slightly “straw” in colour and then turn off the apparatus.
- (f) Remove each thimble and specimen from the Soxhlet apparatus and leave in fume cupboard until dry.
- (g) Place each thimble in an oven and increase temperature from ambient to 105° to 110°C and dry to Constant Mass.

NOTE: Initially allow at least 3 hours or overnight.

- (i) Remove the thimble and specimen from the oven, place in desiccator and allow to cool to touch (about 1 hour).
- (ii) Determine the mass.
- (iii) Compare successive mass results. Constant Mass ( $M_3$ ) has been achieved if the difference in mass from successive determinations is within 0.1%.
- (iv) Otherwise return the thimble and specimen to the oven for a further period of at least 2 hours and repeat Step (g).
- (h) Determine whether rubber particles were lost from around the seal of the cotton plug:

NOTE: For example if the thimbles in the Soxhlet became fully submerged.

- (i) Pour the toluene remaining in the flask at the end of the test through a 75 µm sieve. Remove the boiling chips.
- (ii) Determine the mass of rubber retained on the sieve ( $M'$ ) to 0.01 g.
- (iii) Reject the test if  $\frac{M'}{M_3 - M_1} \times 100\% > 1\%$  and retest the sample.

### 5.3 Rubber Recovery Factor

- (a) The default value for the Rubber Recovery Factor is  $P = 0.8$ .

NOTE: The Rubber Recovery Factor allows for oils compounded with the rubber as well as rubber sufficiently degraded by the digestion process to make it soluble in the extraction process.

- (b) Where requested, determine the Rubber Recovery Factor ( $P$ ) of a sample of rubber of the same type or source as present in the binder as follows:
  - (i) Carry out Steps 5.1 and 5.2 using a sample of the rubber instead of the binder.

NOTE: The subscript “R” is used to denote the mass for the rubber.

- (ii) Calculate the Rubber Recovery Factor ( $P$ ) according to Calculation 6(a).

## 6. Calculations

- (a) Where required in Step 5.3(b), calculate the Rubber Recovery Factor ( $P$ ) as follows:

$$P = \frac{(M_{R3} - M_{R1})}{(M_{R2} - M_{R1})}$$

Where:

$$\begin{aligned}
 P &= \text{Rubber Recovery Factor to 0.1} \\
 M_{R3} &= \text{Mass of rubber sample dried to constant mass (g)} \\
 M_{R2} &= \text{Mass of rubber sample in thimble when prepared (g)} \\
 M_{R1} &= \text{Mass of dry thimble (g)}
 \end{aligned}$$

- (b) Calculate the Rubber Content ( $R$ ) of the sample as a percentage as follows:

$$R = \frac{(M_3 - M_1)}{P \times (M_2 - M_1)} \times 100\%$$

Where:

R = Rubber Content to 0.1%

P = Rubber Recovery Factor to 0.1

$M_3$  = Mass of specimen dried to constant mass (g)

$M_2$  = Mass of specimen in thimble when prepared (g)

$M_1$  = Mass of dry thimble (g)

*NOTE: Repeatability. Duplicate test results obtained by the same operator should not be considered suspect unless R differs by more than 0.8%.*

## 7. Reporting

Include the following information, data and results in the report:

- (a) Sample details.
- (b) The Rubber Recovery Factor ( $P$ ) assumed or determined to 0.1.
- (c) The Rubber Content ( $R$ ) rounded to 0.1 of a percent.
- (d) Reference to this test method.