

NZTA T31: 2025

Method of Measuring the Active Adhesion Between a Bituminous Binder and a Chip Seal Aggregate

1 Scope

The purpose of this test method is to assess the ability of a given combination of bitumen binder and adhesion (anti-stripping) agent to form a bond with a chip sealing aggregate in the presence of water and to determine a minimum dosage rate for use in cutback bitumen chip sealing.

Two methods are specified: Method A which has traditionally been used since the development of this test, and Method B, which some practitioners use for simplicity. The user of this document can select either method, but if dispute arises, Method A should be regarded as the definitive method.

2 Referenced Documents

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| (a) NZ Transport Agency | NZTA M01 Specification for Bitumen |
| (b) NZ Transport Agency: | NZTA M06 Specification for Sealing Chip |
| (c) Standards New Zealand: | NZS 4407 Methods of Sampling and Testing Road Aggregates |
| (d) ASTM International: | ASTM D5 Standard test Method for the Penetration of Bituminous Materials |

3 Apparatus

- (a) Drop-Test Unit (Figure 1). The unit consists of a three-point mount for holding the test plates, attached to a square table possessing screw feet for levelling. A rod is attached to one side of the table; on the top of this rod is a shallow slightly sloped trough mounted at an angle of 3°, sloping downward towards the centre of the apparatus. The slope of the trough is set so that the ball has a sideways component of motion, and thus strikes the test plate once only.

The rod shall be 600mm \pm 0.5mm in length, with the distance from the trough exit to the test plate mount 500mm \pm 0.5mm.

- (b) Steel Ball-Bearing. The ball bearing shall be a standard “two-inch” diameter bearing of weight 534.5g \pm 1.0g.
- (c) Test Plates. These should be made of galvanised sheet steel cut into squares of side 200mm \pm 2mm of thickness nominally 1.90mm. At least four plates are required for each test.

The plates shall be flat. A straight rule laid parallel to any one of the plate edges across either face of the plate shall at no point be more than 0.5mm from the plate surface as measured with a feeler gauge or other suitable equipment.

Note: The test plates may have a nominally 5mm lip on each edge to aid spreading and retention of the bitumen.

- (d) Roller (method A only). A roller with attached handle. The roller cylinder shall be covered with a layer of rubber at least 13mm thick and of hardness such that it satisfies the requirements listed in the annex.

The diameter of the covered roller shall be 250mm \pm 3mm. Its weight, including that of any axle required to secure it to the handle, shall be 25.0kg \pm 0.2kg.

The distance from the roller axis to the unattached end of the handle shall be 1.1m \pm 0.1m and the weight of the handle shall be 5.45kg \pm 0.10kg.

- (e) Aggregate Conditioning Equipment (method A only). This consists of a cylindrical sealable plastic conditioning vessel of 140mm minimum internal diameter and 150mm height. The vessel must contain a flat circular corrosion-proof perforated or wire mesh plate whose diameter is not less than 10mm less than the internal diameter of the vessel. The plate should be supported in the conditioning vessel at least 20mm above the inside base and at least 100mm below the bottom of the vessel lid. When in place in the vessel, the plate must be sufficiently strong to support a kilogram of aggregate spread over its surface.

A piece of absorbent paper, such as blotting paper, is placed against the inside curved surface of the conditioning vessel so as to completely cover the surface from the base of the vessel to the lid.

- (f) Climate chamber or room (method B only). A temperature and humidity-controlled chamber or room, maintained at $23^{\circ}\text{C} \pm 3^{\circ}\text{C}$ and $50\% \pm 5\%$ relative humidity (method B only).
- (g) Drying oven as specified by NZS 4407 Part 1 (method B only).
- (h) Heating oven for sample preparation, set at $140^{\circ} \pm 5^{\circ}\text{C}$.
- (i) Conditioning Bath(s). A water bath capable of maintaining temperature within $\pm 0.3^{\circ}\text{C}$ of the test temperature as specified in Table 1. Total bath volume available shall be sufficient to hold four aggregate-covered test plates and the conditioning vessel immersed to within 25mm of its top. The bath liquid, in the case of the test plates, shall be distilled or deionised water. Sufficient additional bath volume to accommodate a fog-spray bottle (item 3(x)) should also be available if water dispersible agents are to be tested.
- (j) Thermometer to read bath temperature to $\pm 0.3^{\circ}\text{C}$.
- (k) Balance to weigh 1000g, readable to 0.01g and accurate to $\pm 0.03\text{g}$.
- (l) 0.5L and 0.25L cans with press-lid tops as appropriate.
- (m) High shear rate stirrer.
- (n) Sieves, square orifice perforated plate. Appropriate sieves for aggregate sorting are 9.50mm and 13.2mm. For aggregate washing a 4.75mm sieve is suitable. Other sieve sizes should be used if an alternative aggregate fraction to that specified in clause 4.2 is selected for test.
- (o) Electric hotplate, area 200mm x 200mm at least, with variable temperature control up to at least 120°C . The effective area of a hotplate may be increased if necessary by placing on top of it a thick (approximately 13mm) metal plate of at least 200mm x 200mm.
- (p) Device to measure hotplate surface temperature to $\pm 1^{\circ}\text{C}$. A digital thermometer with a surface probe is satisfactory for this purpose.
- (q) Flexible filling knife with blade width approximately 100mm.
- (r) Glass or brass binder stirring rods, length 200mm or greater.
- (s) Open-top metal can with outward-curved lip and loose-fitting top, volume approximately 0.5L.
- (t) Board of low thermal conductivity non-metallic rigid material, e.g. pine wood, with minimum width and length at least 245mm and 590mm respectively, thickness at least 6mm.
- (u) Metal aggregate stirring rod, nominal diameter 6mm, length 200mm or greater (method A only).
- (v) Glass or plastic beaker, nominal volume 1L, for aggregate washing.
- (w) Non-absorbent wide-mesh material to make bags to hold test aggregate during conditioning (method A only). Nylon stocking/pantihose material has been found suitable.
- (x) Fog-spray plastic bottle (for water-dispersible agents only).
- (y) Plastic beaker, 400mL capacity or greater (for water dispersible agents only).
- (z) Square polystyrene pad, thickness 12mm minimum, square side 190mm $\pm 5\text{mm}$.

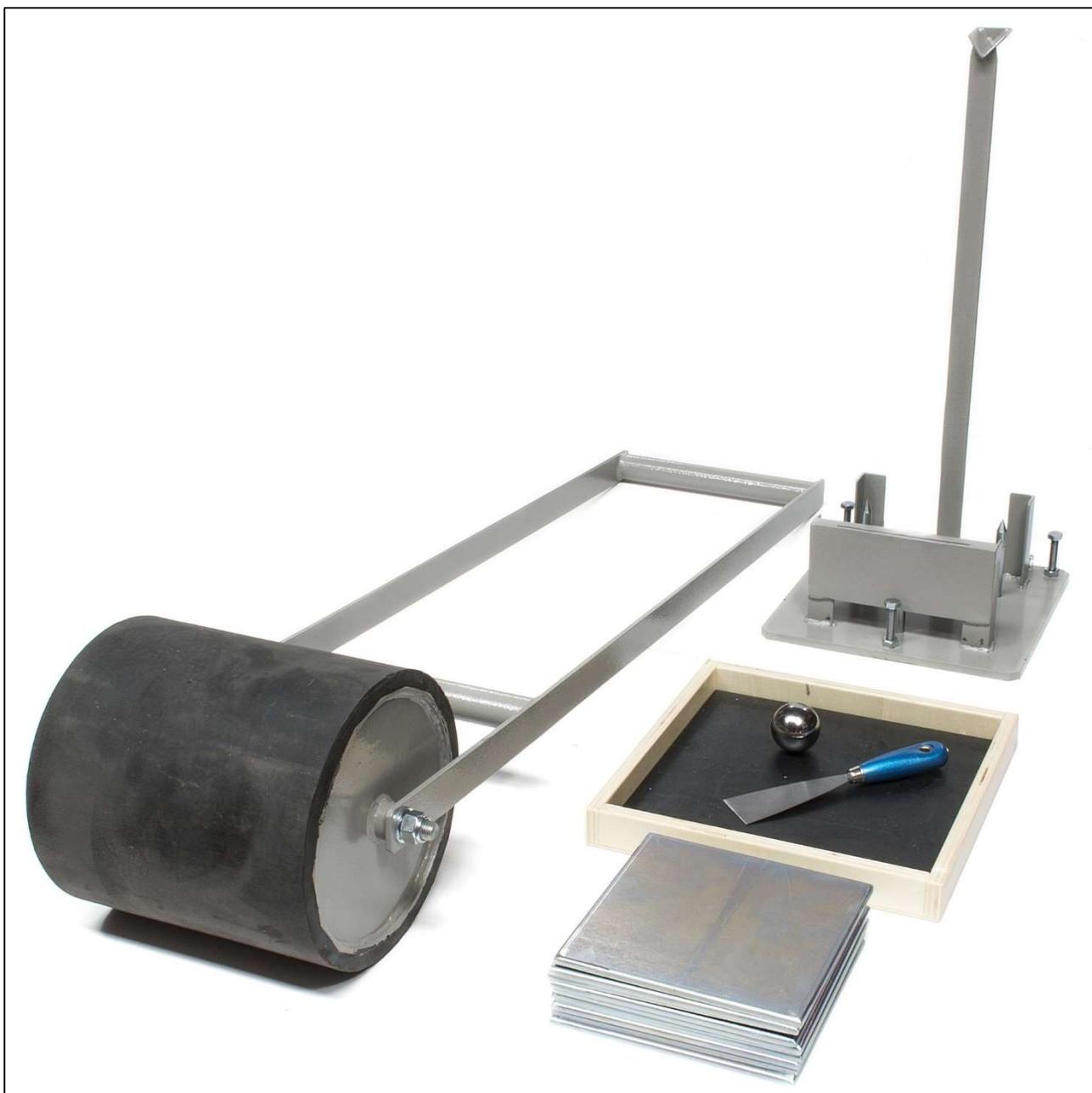


Figure 1: Drop Test Unit and Associated Equipment

4 Materials Preparation

4.1 Bitumen Grade

The test binder will normally be 180/200 grade bitumen compliant with NZTA M01 specification, of the type used for chip sealing work in New Zealand. Other grades of bitumen compliant with NZTA M01 may be used, but the plate conditioning temperature used in method A shall be adjusted to the temperatures in Table 1 below.

Table 1: Test Plate Conditioning Temperatures

NZTA M01 Bitumen Grade	Plate Conditioning Temperature (°C)
180/200	25
130/150	29
80/100	34
60/70	38

The bitumen sample should be heated, and subsamples of 300g or more poured into the 0.5L cans, which should then be sealed and stored at room temperature until needed for the test. The minimum possible amount of heating should be employed for sample preparation, and exposure of the binder to air while hot should also be minimised. These precautions minimise binder hardening.

4.2 Aggregate Preparation

Sieve the aggregate to obtain material of the required size range. Unless otherwise specified, NZTA M06 Grade 3 aggregate shall be sieved to pass a 13.2mm sieve and be retained on a 9.5mm sieve.

Obtain a representative sample of the test aggregate by quartering or use of a riffle box, of approximately 500 aggregate particles (approximately 1kg weight for typical Grade 3 chips).

4.3 Agent Preparation

For water dispersible adhesion agents, proceed as follows.

Shake the agent container, if necessary, to ensure uniformity of the product, and then prepare approximately 200g of water/adhesion agent mixture by mixing the agent with distilled or deionised water in an approximately 400mL plastic beaker. Agent concentration shall be that to be used in sealing practice. Quantities of water and agent used shall be measured to the nearest 0.01g and values noted. Mix the solution in the beaker with a stirring rod, and then transfer it to a clean plastic fog-spray bottle.

For solid agents in particulate form (eg, pellets or flakes) for which average particle weight is greater than 0.01g, grind together at least 30g of material, and mix into a uniform paste. Place in a sealed container.

Agents which are liquid or paste, or which consist of very small solid particles, require no special preparation.

4.4 Bitumen Preparation

Bring the heating oven to $140^{\circ}\text{C} \pm 5^{\circ}\text{C}$ and the hotplate to a temperature just sufficient to melt and spread evenly any binder on a test plate placed on it. The hotplate surface temperature shall be measured and recorded. The plate temperature shall not exceed 85°C for 180/200 grade bitumen, 90°C for grade 130/150 bitumen, 95°C for 80/100 grade bitumen or 100°C for 60/70 grade bitumen.

Choose a half-litre can of test bitumen and estimate the approximate weight of bitumen in it by weighing. Loosen the lid on the can, but do not remove it. Place the can in the oven for 2 – $2\frac{1}{4}$ hours (see note 10(c)). Ensure that the test plates are clean, using an appropriate solvent to remove any material adhering to them (note that new plates may have an undesirable oil film on their surfaces).

Proceed to clause 5 Method A, or clause 6 Method B depending on preference. In case of doubt Method A should be considered the reference method.

5 Test Procedure: Method A

5.1 Aggregate Conditioning

- (a) Bring the conditioning bath to the specified test temperature (see Table 1). Baths which will hold test plates must be refilled before each test with fresh distilled or deionised water to prevent contamination of the test binder surfaces.
- (b) Pour sufficient water into the conditioning vessel to give a saturated atmosphere when the vessel is sealed at the conditioning temperature. The water level should be at least 5 mm below the circular plate.
- (c) Clean the aggregate by placing the approximately 500 particle representative aggregate sample in the aggregate washing sieve and agitate thoroughly for at least 20 seconds in tap water 100mm or deeper. Repeat this procedure four times using fresh water each time. Drain the aggregate. Transfer it to an approximately 1L aggregate washing beaker and wash three times using at least 500mL of distilled or deionised water for each wash, whilst stirring the stone with a metal stirring rod. Rinse the washing sieve with distilled or deionised water, upend the aggregate on it, and leave approximately 20s to drain. Do not allow the stone to air dry before carrying out step (d) below.

- (d) Immediately make up four lots of 100 particles each from the washed aggregate, and place each of these lots in bags made from the non-absorbent wide-mesh material. Fasten the bags shut (rubber bands can be used for this), place them on the plate in the conditioning vessel and seal the vessel. No bag should rest on top of any other bag.
- (e) Place the conditioning vessel in a conditioning bath so that all but the top 25mm (or less) is immersed in the liquid. Use a weight to hold in place if necessary. Also place the fog-spray bottle (see step 4.3), if prepared, into a conditioning bath so that the top surface of the adhesion agent mixture is level with or below the bath liquid surface, with the spray mechanism above the liquid to prevent mixing of both liquid and adhesion agent solution. Leave in the bath/s for 25 hours \pm 2 hours immediately prior to starting to place aggregate on the test binder (step 5.3).
- (f) Proceed to step 5.2.1 if bitumen soluble adhesion agents are being tested or proceed to step 5.2.2 if water soluble adhesion agents are being tested.

5.2 Test Plate Preparation

5.2.1 Bitumen Soluble Adhesion Agents

- (a) Heat the 0.5L can containing bitumen in the heating oven set to 140°C until it is sufficiently liquid to pour. Heating shall not exceed 2½ hours to minimise binder hardening.
- (b) Place a 250mL can on the balance. Accurately weigh approximately 200g of bitumen into the can and record the mass to the nearest 0.01g. Calculate the mass of adhesion agent required for the specified dosage and accurately weigh the adhesion agent into the can. Record the mass to the nearest 0.01g. Immediately mix the bitumen and adhesion agent for 2 minutes using the high shear stirrer and return the can to the heating oven for no more than 15 minutes.
- (a) Remove the 250mL can from the oven and thoroughly stir the binder with the (cleaned) binder-stirring rod.
- (b) Place a test plate on the balance and pour approximately 40g of binder on to the plate. The test plate may be preheated to aid spreading of the binder, but its temperature must at no time be greater than that of the hotplate.
- (c) Cover the can and replace it in the oven until required for the next plate.
- (d) Place the binder-covered plate on the hotplate and, using the flexible filling knife, spread the binder over the test plate as uniformly as possible. Leave the test plate on the hotplate until the binder surface just levels, as indicated by reflection of light off the surface. Do not proceed to step (e) until levelling is complete.
- (e) Remove the binder-covered test plate from the hotplate and reweigh it to ensure total binder cover is $40.0\text{g} \pm 1.0\text{g}$. If not, reject the plate and prepare another.
- (f) Repeat steps (b) to (e) until four satisfactorily coated plates have been obtained.
- (g) The following steps (h) and (i) inclusive shall be carried out under an ambient temperature between 20°C and 30°C inclusive.
- (h) Place each coated test plate on a level surface for 20 ± 0.5 minutes in air. Twenty minutes after taking each test plate off the hot plate, immerse it in the conditioning bath to a depth of at least 25mm for 20 ± 0.5 minutes.
- (i) At the end of the 20 minute period remove the test plate from its bath, hold it binder surface downwards, and shake vigorously to remove water. Ensure that as much water as possible has drained from the test binder surface, but do not wipe the surface. Proceed to step 5.3.

5.2.2 Water Dispersible Adhesion Agents

- (a) Calculate the weight of the water dispersible adhesion agent solution that will be required to cover a test plate of area 0.040m^2 .
- (b) Remove the can of test bitumen from the oven and thoroughly stir the bitumen with the binder-stirring rod.

- (c) Place a test plate on the balance and pour $40\text{g} \pm 1\text{g}$ of binder from the open-top can on to the plate. The test plate may be preheated to aid spreading of the binder, but its temperature must at no time be greater than that of the hotplate.
- (d) Cover the open-top can and replace it in the oven until required for the next plate.
- (e) Place the binder-covered plate on the hotplate and, using the flexible filling knife, spread the binder over the test plate as uniformly as possible. Leave the test plate on the hotplate until the binder surface just levels, as indicated by reflection of light off the surface. Do not proceed to step (f) until levelling is complete.
- (f) Remove the binder-covered test plate from the hotplate and reweigh it to ensure total binder cover is $40.0\text{g} \pm 1.0\text{g}$. If not, reject the plate and prepare another. Repeat steps (c) to (e) until four test plates have been prepared.
- (g) The following steps (h) to (k) inclusive shall be carried out under an ambient temperature between 20°C and 30°C inclusive.
- (h) Place each coated test plate on a level surface for 20 ± 0.5 minutes in air.
- (i) Twenty minutes after taking each test plate off the hot plate, immerse it in the conditioning bath to a depth of at least 25mm for 20 ± 0.5 minutes.
- (j) At the end of the 20 minute period remove the test plate from its bath, hold it binder surface downwards, and shake vigorously to remove water. Ensure that as much water as possible has drained from the test binder surface, but do not wipe the surface.
- (k) Place the test plate, binder surface up, on the polystyrene pad on the balance. Ensure that the plate completely overlaps the pad. Remove the plastic fog-spray bottle from the conditioning bath and wipe it dry. Apply the adhesion agent solution to the binder surface, using the fog-spray bottle, moving the nozzle parallel to one edge of the test plate and alternatively at right angles to this, to give an even coating of the solution on the binder surface of weight as close as possible to that calculated above. The final weight of the coating shall be recorded to 0.01g. Take care to ensure that no adhesion agent solution is sprayed on to the balance pan. Return the fog-spray bottle to the conditioning bath.

5.3 Aggregate Placement

- (a) Place each test plate, binder surface up, with the polystyrene pad beneath, on a flat level surface. Remove a bag of aggregate from the plastic conditioning vessel. Reseal the vessel and leave it in the conditioning bath. Place all the aggregate particles from the bag on the binder, adjusting their position by hand so that no particle touches any other. Do not press any particle into the binder to any extent whatsoever.
- (b) Place the low thermal conductivity board on the floor with the test plate on top, binder surface up, taking care not to move any of the aggregate particles. Roll the rubber-covered roller over the plate three times in one direction (e.g., twice forward and once reverse), then turn the plate through 90° and repeat.
- (c) Replace the plate in the conditioning bath for $2\frac{1}{2}$ hours ± 5 minutes. The total period the plate has been out of the bath shall be less than 5 minutes.
- (d) Repeat steps (a) to (c) for the other three plates.
- (e) Following the conditioning period of $2\frac{1}{2}$ hours ± 5 minutes proceed to clause 7.

6 Test Procedure: Method B

6.1.1 Preparation of Test Plates

- (a) Wash the aggregate subsample obtained in clause 4.2 and dry overnight in the drying oven.
- (b) Preheat the test plates in the heating oven.

Note: this test procedure is only suitable for test plates that have a lipped edge.

- (c) Heat the 0.5L can containing bitumen in the heating oven set to 140°C until it is sufficiently liquid to pour. Heating shall not exceed 2½ hours to minimise binder hardening.
- (d) Place a 250mL can on the balance. Accurately weigh approximately 200g of bitumen into the can and record the mass to the nearest 0.01g. Calculate the mass of adhesion agent required for the specified dosage and accurately weigh the adhesion agent into the can. Record the mass to the nearest 0.01g. Stir the bitumen and adhesion agent mixture for 2 minutes and return the can to the heating oven for 2 hours.
- (e) Place a preheated test plate on the balance suitably protected by an insulated pad. Ensure that the test plate is level and pour 40g ± 1g of bitumen and adhesion agent mixture from the can on to the plate. Place the test plate in the heating oven for no more than 5 minutes to allow the bitumen to level.
- (f) Repeat step (e) until four satisfactorily coated plates have been obtained.
- (g) Condition the washed and dried aggregate subsample and the four test plates in the climate chamber set to 23°C and 50% ± 5% relative humidity. Proceed to step (h) after 24 hours but no more than 48 hours conditioning in the climate chamber.
- (h) Remove test aggregate sample and test plates from the climate chamber. Lightly place 100 aggregate particles (chips) on each test plate with no particle touching another. Return the test plates to the climate chamber for 17 hours ± 0.5 hours (i.e. overnight).
- (i) Bring the conditioning bath to 23°C (note that this temperature is independent of binder grade). Baths which will hold test plates must be refilled before each test with fresh distilled or deionised water to prevent contamination of the test binder surfaces. Place the test plates in the conditioning bath and leave for 24 ± 0.5 hours (i.e. overnight).

7 Adhesion Testing

Place the drop-test unit on a rigid, non-flexible, surface (see note 10(a)). Using the levelling screws, adjust the unit until the steel ball-bearing will roll off the sloped trough to strike any test plate placed in position on the unit within 15mm of the centre of the plate (see note 10(b)). Do not move the drop-test unit after this adjustment has been made.

At the end of the set-up period remove each plate from the water bath, invert so that stones and binder are underneath, and place on the three-point support on the drop-test unit.

Place the ball-bearing on the trough and allow it to roll off to strike the back of the test plate. Repeat two more times. The three impacts should occupy a total time of less than 10 seconds, and the time between removing the plate from the bath and completion of the impacts should be no greater than 20 seconds.

Remove the tray from the drop-tester and count the number of stones still adhering to the binder. Inspect the stones which have been detached to see if they retain any significant amount of binder (see note 10(d)).

8 Calculations

- (a) Calculate the adhesion value for each test plate:

$$A_n = 100 - N$$

Where: A_n = The adhesion value for test plate n

N = the number of aggregate particles that fall off and do not retain a significant amount of binder on a face (see note (d))

- (b) Calculate the average adhesion value \bar{A} :

$$\bar{A} = \frac{\sum A_n}{n}$$

Where: n = The number of test plates used in the test (normally 4)

- (c) If \bar{A} is not equal to 0 or 100, calculate the standard deviation S_A of the adhesion values:

$$S_A = \sqrt{\frac{\sum(A_n - \bar{A})^2}{n - 1}}$$

(d) Calculate the test criterion T :

$$T = \frac{S_A}{\sqrt{\frac{\bar{A}(100 - \bar{A})}{100}}}$$

If T is greater than 2.0 repeat the test. If the repeat test has $T \leq 2.0$ accept the repeat value. Otherwise quote both binder aggregate adhesions and their respective T -values and report "abnormally large spread of results".

Calculate actual dose rates of adhesion agents and added diluents (if any) used in the test from weights recorded during the test procedure.

9 Reporting

Report the following:

- (a) Agent type and dose rate in g/m² of agent (excluding diluting water) for water dispersible agents or parts per hundred by mass or volume of bitumen for other agents.
- (b) Bitumen source (supplier and terminal if known) and grade.
- (c) Any diluents used, in parts per hundred by mass or volume of bitumen.
- (d) Aggregate information including:
 - i. Supplier and quarry.
 - ii. Geological name of parent stone if known.
 - iii. Original NZTA M06 Grade, with a description of any sieving done in the laboratory to prepare the test sample.
 - iv. Any other information on aggregate condition which might be considered capable of affecting the result of the test.
- (e) The method variant used and any departure from the standard methods and quantities specified in this method.
- (f) Test temperature in degrees Celsius.
- (g) Binder aggregate adhesion and its T -value.

10 Notes

- (a) If the drop-test unit is placed on a flexible surface, erratic adhesion values can result. Avoid flexible thin metal bench or sink surfaces and wooden bench surfaces without support immediately below the test unit.
- (b) The place at which the ball bearing strikes the test plate can be determined by placing a thin piece of adhesive plasticine or a similar material on the upper side of the plate at the centre and noting the position of the indentation the ball produces. Other methods which will determine the strike point with sufficient accuracy are acceptable.
- (c) The time of the warming procedure is specified so that all samples receive the same treatment, and different degrees of hardening which would affect the adhesion results are avoided.
- (d) Of bond failures occurring in the impact test, only those at the interface of stone and bitumen are of interest, since it is these that indicate the failure of an adhesion agent. Thus, stones dislodged by rupture within the bulk of the binder (and thus still having bitumen adhering to a face) are not counted as lost.

A "significant amount of binder" shall be binder which has, in the view of the operator, remained adhering to more than 5% of that face of a detached stone which was formerly in contact with the binder layer on the test plate. Binder adhering to the edges of this face shall not be considered in this assessment, as such binder may be there because it caught on protrusions as the portion of the stone near the bonded face was pulled through the binder upon detachment.

11 Annex A: Method for Assessing the Hardness of the Rubber on the Test Roller

11.1 Apparatus

- (a) Two steel or brass rods, diameter 6.0 ± 0.1 mm, length at least 580 mm.
- (b) One pair of Vernier callipers.
- (c) One meter rule.
- (d) White blackboard chalk.

11.2 Procedure

- (a) Rule four parallel lines at least 590mm long on a flat horizontal surface. The distances of the left-most line from the other three should be 60mm, 200mm, and 260mm respectively, each within ± 2 mm.
- (b) Cover the two rods with chalk to within 50mm of each end, first of all roughening the rod surfaces with emery cloth if this is necessary to make the chalk dust adhere to them.
- (c) Lay the two rods along the two inner lines.
- (d) Using the two outer lines as guides to the position of the edges of the roller surface, draw the roller by its handle slowly along the rods, for a distance of at least 400mm over the chalked surfaces. Ensure that the rods do not move during this process and that both rods are as near as possible equidistant from the respective nearer edges of the roller.
- (e) Measure the widths of the chalk lines produced on the rubber surface of the roller by the rods. Take three readings for each line at three different positions, each position at least 100mm from either end of the line. Average the six readings.
- (f) The rubber on the test roller shall be accepted as being of a satisfactory hardness provided the average chalk line width is between 1.9mm and 3.6mm inclusive.

Note: Rubber of approximately 80 International Rubber Hardness Degrees is expected to pass the above test. This information is provided only as a guide to roller manufacture. The roller must satisfy the test criterion described above to be acceptable for use in the binder aggregate adhesion test method.