

## DETERMINATION OF ACID INSOLUBLE RESIDUE OF AGGREGATES

LS-613 R33

### 1. SCOPE

- 1.1 This method covers the determination of the percentage of insoluble residue of aggregates by using hydrochloric acid to dissolve carbonate mineral content
- 1.2 The amount and size distribution of acid insoluble material in aggregates is of interest when conducting investigations on pavement frictional properties.
- 1.3 This test method may involve hazardous materials, operations and equipment, and does not purport to address all the safety concerns associated with their use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

### 3. APPARATUS

- 3.1 SAMPLE SPLITTERS: for fine and coarse aggregates. Refer to MTO LS-600..... \_\_\_\_\_
- 3.2 CRUSHER: An aggregate crusher adjusted to produce material passing the 4.75 mm sieve..... \_\_\_\_\_
- 3.3 SIEVES: Sieves with square openings conforming to ASTM E11 and of suitable sizes to furnish the information required by the specification covering the material to be tested..... \_\_\_\_\_
- 3.4 SCALE: A scale or balance accurate to 0.01 g..... \_\_\_\_\_
- 3.5 OVEN: An oven capable of maintaining a temperature of  $110 \pm 5^{\circ}\text{C}$ ..... \_\_\_\_\_
- 3.6 BUNSEN BURNER OR HOT PLATE..... \_\_\_\_\_
- 3.7 BOROSILICATE GLASS CONTAINER (Beaker): 2000 mL capacity..... \_\_\_\_\_
- 3.8 HYDROCHLORIC ACID (HCl): Aqueous solution of technical grade HCl prepared at 20 percent concentration by volume..... \_\_\_\_\_
- 3.9 FUME HOOD: Acid Digestion Laboratory Hood..... \_\_\_\_\_

### 4. PREPARATION OF TEST SPECIMEN

4.1 Test samples can be prepared from aggregate taken from stockpile or aggregates extracted from samples of asphaltic concrete, RAP or RST..... \_\_\_\_\_

4.2 Bulk samples may be reduced by splitting without separating into individual coarse and fine aggregate portions..... \_\_\_\_\_

When coarse aggregate and fine aggregate portions of a material need to be determined separately, separate the materials on the 4.75 mm sieve prior to splitting..... \_\_\_\_\_

*Note 1: Samples may need to be separated into individual fractions and reduced by splitting prior to crushing in order to meet specific requirements.*

4.3 Samples of coarse aggregate and bulk samples containing coarse aggregate should be crushed to reduce the particle size in order to improve the efficiency of acid digestion. Before crushing, reduce the sample size by splitting to the minimum masses shown below..... \_\_\_\_\_

Minimum sample mass is determined by the largest size through which all of the aggregate passes. For example, if the largest sieve through which all the aggregate passes is the 19 mm sieve, then the test sample size is 3000 g..... \_\_\_\_\_

Crush the sample so that all particles pass a 4.75 mm sieve..... \_\_\_\_\_

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Pass	Minimum Mass, g
37.5 mm	5000
26.5 mm	4000
19.0 mm	3000
13.2 mm	3000
9.5 mm	3000

*Note 2: For crushing, a representative sample of at least 3000 g is recommended in order to maintain feed through the crusher, which will increase crushing efficiency, reduce the overall time required to produce the required particle size and help reduce the generation of excessive fines. It is also recommended to run fine and coarse aggregates in bulk samples through the crusher as one in order to achieve similar benefits.*

4.3.1 Mix the crushed sample thoroughly and obtain a 100 to 125 g representative sample by splitting..... \_\_\_\_\_

4.4 For samples with 100 % passing the 4.75 mm sieve, reduce the sample by splitting to a minimum of 2000 g. Crushing is not required..... \_\_\_\_\_

4.4.1 Mix the sample thoroughly. Obtain a 100 to 125 g representative sample by splitting..... \_\_\_\_\_

4.4.2 Where testing is required on individual sieve fractions of a fine aggregate, e.g., the fraction passing 4.75 mm and retained 2.36 mm sieves, sieve sufficient material so as to obtain a minimum 100 to 125 g of material in the size range required to form the test sample. Reduce the sample size by splitting where necessary..... \_\_\_\_\_

4.5 For samples of asphaltic concrete, RAP or RST, extract the aggregate using LS-282, or as specified in the contract documents. To examine the coarse aggregate fraction, process sufficient material to obtain a minimum of 1000 g of clean, dry extracted aggregate retained on the 4.75 mm sieve..... \_\_\_\_\_

To examine the fine aggregate fraction, process sufficient material to obtain a minimum of 1000 g of clean, dry extracted aggregate passing the 4.75 mm sieve..... \_\_\_\_\_

*Note 3: Treat the coarse aggregate and fine aggregate portions as separate samples. Do not combine.*

4.5.1 Examine the material for residual asphalt cement and repeat the extraction process if necessary..... \_\_\_\_\_

4.5.2 Crush the coarse aggregate material so that all particles pass a 4.75 mm sieve..... \_\_\_\_\_

4.5.3 For extracted coarse aggregates, mix the crushed sample thoroughly and obtain a 100 to 125 g representative sample by splitting..... \_\_\_\_\_

4.5.4 For extracted fine aggregates, mix the sample thoroughly and obtain a 100 to 125 g representative sample by splitting..... \_\_\_\_\_

4.5.5 Where testing is required on individual sieve fractions, e.g., passing 4.75 mm and retained 2.36 mm fraction, process sufficient material in the extraction so as to obtain a minimum 100 to 125 g of material in the size range required to form the test sample. Reduce the sample size by splitting where necessary..... \_\_\_\_\_

**5. PROCEDURE**

5.1 Place a representative sample of the prepared aggregate weighing between 100 and 125 g in a pre-weighed beaker..... \_\_\_\_\_

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Record the mass of the beaker and the sample to the nearest 0.01 g..... \_\_\_\_\_

Report the mass of the sample (A) on the test data sheet in Figure 1 of the LS..... \_\_\_\_\_

5.2 Slowly, add approximately 100 ml of hydrochloric acid solution (HCl) to the container, taking care to allow for excessive effervescence or foaming to subside before further addition of acid solution. \_\_\_\_\_

*Note 4: Use extreme caution when adding HCl. Aggregates high in carbonate content may react vigorously during the initial stages. If needed, small amounts of distilled water may be added to the solution to slow the reaction. Addition of distilled water using a spray bottle has been found to be effective for this purpose.*

5.3 Gently agitate the solution between additions of HCl and observe for reaction. If a reaction is observed, continue agitation until the reaction subsides..... \_\_\_\_\_

*Note 5: A long glass stirring rod, approximately twice the height of the beaker in length, has been found to be effective for the purpose of agitating the mixture.*

5.4 Continue to slowly add HCl to the beaker in approximately 100 ml increments and agitating until the reaction appears to have subsided, or until a total of 1000 ml of HCl has been added to the container..... \_\_\_\_\_

*Note 6: The beaker should be of sufficient volume to fully contain the reaction. If there is insufficient space to contain the reaction and/or the acid is not added slowly enough, the reaction can be so vigorous that spattering out of the beaker can result in a loss of material. Sides of the beaker should also be periodically washed down with distilled water and/or scraped with the glass mixing rod so that any spatter or fines does not adhere to the glass.*

5.5 Allow the container to stand overnight or until the insoluble material settles. The insoluble material has settled sufficiently when the supernatant liquid is clear..... \_\_\_\_\_

*Note 7: Occasionally, during digestion of certain carbonate aggregates, a portion of the residue may present as floating on top of the supernatant liquid. This phenomenon has in particular been observed during digestion of certain types of shaley and/or organic-rich carbonates. Care must be taken not to lose this portion of the residue when pouring off the supernatant liquid. Use of pre-weighed filter paper to collect the floating residue may be an effective means of achieving this.*

5.6 Pour off the supernatant liquid, taking care not to lose any insoluble material..... \_\_\_\_\_

Add an additional 100 ml of HCl and gently agitate while observing for reaction..... \_\_\_\_\_

If a reaction is observed, repeat steps 5.4 to 5.5 until the reaction has gone to completion..... \_\_\_\_\_

*Note 8: If a white or yellowish precipitate is observed on the insoluble residue (especially on drying) it may be an indication that not enough acid was used during the digestion process.*

5.7 When no further reaction is observed, heat the container slowly to bring it to the boiling point..... \_\_\_\_\_

Continue gentle boiling for approximately 30 minutes to completely digest any remaining carbonate..... \_\_\_\_\_

5.8 After 30 minutes, cool the mixture, and allow the insoluble material to settle until the supernatant liquid is clear..... \_\_\_\_\_

Decant the acid solution and add a sufficient amount of water to the insoluble residue in the beaker to thoroughly dilute the remaining acid..... \_\_\_\_\_

Allow the mixture to settle until the supernatant liquid is clear, decant it, and repeat the procedure until the diluted solution is of neutral pH..... \_\_\_\_\_

*Note 9: The diluted solution may be checked for pH with pH strips or a pH meter to ensure neutrality of the rinse liquid.*

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- 5.9 Wash the insoluble residue material over a 75 µm sieve into a suitable sized container..... \_\_\_\_\_  
The use of a protection sieve (at least 1.18 mm sieve) is recommended – select the appropriate size depending on the dominant particle size of the insoluble residue..... \_\_\_\_\_
  - 5.10 Reserve the wash water containing the fraction of insoluble residue passing the 75 µm sieve. Allow the residue to settle, decant off the water and dry at 110 ± 5°C to constant mass..... \_\_\_\_\_
  - 5.11 Dry the retained 75 µm insoluble residue at 110 ± 5°C to constant mass..... \_\_\_\_\_
  - 5.12 Add 1 to 3 drops of the HCl solution to the dried residue of both the passing 75 µm and retained 75 µm fractions and observe for any reaction..... \_\_\_\_\_  
Evidence of reaction, e.g., foaming, effervescence, sulphurous odour, etc., is an indication of incomplete digestion of the carbonate minerals..... \_\_\_\_\_  
Repeat steps 5.2 through 5.8 until digestion is complete..... \_\_\_\_\_
  - 5.13 Sieve the retained 75 µm insoluble residue material through a nest of fine aggregate sieves including the 2.36 mm, 1.18 mm, 600 µm, 300 µm, 150 µm, and 75 µm sizes..... \_\_\_\_\_
  - 5.14 Weigh and record the mass of material retained on each sieve to the nearest 0.01 g..... \_\_\_\_\_  
Report the mass retained on each sieve in the area reserved for gradation of the insoluble residue on the test data sheet in Figure 1 of the LS..... \_\_\_\_\_  
Report the total cumulative mass of the retained 75 µm insoluble residue (B) on the test data sheet in Figure 1 of the LS..... \_\_\_\_\_
  - 5.15 Add any passing 75 µm obtained by sieving in Section 5.13 to the fraction passing 75 µm obtained in Sections 5.9 and 5.10..... \_\_\_\_\_  
Weigh and record the total mass of the passing 75 µm insoluble residue to the nearest 0.01 g.... \_\_\_\_\_  
Report the total mass of the passing 75 µm insoluble residue (C) on the test data sheet in Figure 1 of the LS..... \_\_\_\_\_
  - 5.16 Place each fraction of the insoluble residue obtained by sieving in Sections 5.13 to 5.15 in a separate bag and label each bag appropriately..... \_\_\_\_\_
- Note 10: The separated fractions of the insoluble residue may be saved for further analysis, e.g. detailed petrographic analysis to identify its composition and/or mineralogy.*

**6. CALCULATIONS**

- 6.1 Calculate the cumulative percentage of insoluble residue retained on the 75 µm sieve (D) expressed as a percentage of the original test sample mass (A) ..... \_\_\_\_\_
- 6.2 Calculate the total percentage of insoluble residue passing the 75 µm sieve (E) expressed as a percentage of the original test sample mass (A) ..... \_\_\_\_\_
- 6.3 Calculate the total percentage of insoluble residue expressed as a percentage of the total original test sample mass (D + E) ..... \_\_\_\_\_

**7. REPORTING OF RESULTS**

- 7.1 Report the total percentage of insoluble residue and the percentage of insoluble residue retained on the 75 µm sieve to the nearest 0.1 percent..... \_\_\_\_\_

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7.2 Report the gradation of the insoluble residue. The percentage of insoluble residue retained on individual sieves may also be calculated and reported as a percentage of the original test sample mass, where required..... \_\_\_\_\_

### 9. GENERAL NOTES

9.1 Care should be taken when reducing coarse material than an excess of passing 75 µm is not produced during crushing (see Note 2) ..... \_\_\_\_\_

9.2 Ensure that all aggregate splitters and metal containers used for splitting are CLEAN prior to use. Remove aggregate particles and other foreign matter and wipe any dust coatings prior to splitting of different samples..... \_\_\_\_\_

Prior to crushing, make sure that all parts of the crushing equipment are clean..... \_\_\_\_\_

Use acetone or alcohol to clean crusher plates and contact surfaces before crushing each sample \_\_\_\_\_

9.3 In previous versions of this test method (R16), the retained 75 µm insoluble residue was determined by sieving of the dried total insoluble residue over a nest of fine aggregate sieve series. However, agglomeration of the passing 75 µm portion frequently occurred during drying of the total residue, particularly if clay minerals were present. These agglomerations can lead to erroneously high values for the retained 75 µm insoluble residue. Physical reduction of agglomerations through use of a mortar and rubber-tipped pestle was performed prior to sieve analysis; however care was needed to prevent the crushing of individual particle grains. Crushing of individual particle grains can lead to erroneously low retained 75 µm insoluble residue values. For this reason steps 5.9 through 5.16 of this test method are implemented..... \_\_\_\_\_

9.4 Acids are diluted on a volumetric basis. **ALWAYS add the acid to the water.** An example calculation for the dilution of concentrated acid to the appropriate concentration is provided below..... \_\_\_\_\_

9.4.1 For preparation of 2000 ml of 20 % HCl solution from a 37 % concentration (assay) HCl solution:

$$C1V1 = C2V2$$

Where

**C1** = Initial concentration of the stock solution of acid, e.g., 37 % HCl, (see manufacturers' label for the assay)

**V1** = Volume of concentrated stock solution of acid required

**C2** = Final concentration of acid required, e.g., 20 % HCl

**V2** = Final volume of acid required, e.g., 2000 ml

$$(0.37)(V1) = (0.20)(2000 \text{ ml})$$

$$V1 = 1081 \text{ ml}$$

This means that 1081 ml of the concentrated 37 % HCl solution should be diluted with 919 ml of distilled water to bring the total volume to 2000 ml of 20 % HCl solution.

### 10. PRECAUTIONS

10.1 Exercise extreme care to prevent corrosive gas from entering the laboratory atmosphere. Hydrogen chloride gas may be destructive to laboratory equipment, and is a definite safety hazard to personnel. Under no circumstances should the test be performed without proper and adequate ventilation. \_\_\_\_\_

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10.2 The addition of concentrated acid solutions to limestone aggregates produces violent foaming and effervescence if appropriate care is not taken. Addition of acid to the beaker containing the sample should always occur in small volumetric increments in a relatively slow and measured manner (see Section 5.2). Proper safety apparel should be worn, e.g., goggles, face shield, rubber gloves, apron, etc..... \_\_\_\_\_

### **COMMENTS**