

METHOD OF TEST FOR DETERMINATION OF INSOLUBLE RESIDUE OF CARBONATE AGGREGATES

1. SCOPE

- 1.1 This method covers the determination of the percentage of insoluble residue in carbonate aggregates by using a hydrochloric acid solution to dissolve the carbonate minerals.
- 1.2 The amount and size distribution of insoluble material in carbonate aggregates is of interest when conducting investigations on pavement frictional properties.
- 1.3 This test helps distinguish carbonate aggregates that may polish excessively, and become slippery from those that will not.

2. RELEVANT DOCUMENTS

- 2.1 ASTM D 3042
- 2.2 MTO Test Method LS-600
- 2.3 MTO Test Method LS-602

3. APPARATUS

- 3.1 **SIEVES:** Sieves with square openings and of the following sizes conforming to OPSS specifications or ASTM specification E 11.

Coarse Series	Fine Series
19.0 mm	4.75 mm
16.0 mm	2.36 mm
13.2 mm	1.18 mm
9.5 mm	600 µm
6.7 mm	300 µm
4.75 mm	150 µm
	75 µm

- 3.2 **SIEVE SHAKER:** Any mechanical sieve shaker may be used which produces the thoroughness of sieving equivalent to the hand sieving described in MTO LS-602 Method of Test for Sieve Analysis of Fine and Coarse Aggregates.
- 3.3 **SCALE OR BALANCE:** A scale or balance accurate to 0.01 g.
- 3.4 **OVEN:** An oven of appropriate size capable of being maintained at $110 \pm 5^\circ\text{C}$.

- 3.5 BUNSEN BURNER OR HOT PLATE.
- 3.6 BOROSILICATE GLASS CONTAINER: 2 000 mL capacity.
- 3.7 CRUSHER: An aggregate crusher which may be adjusted to produce material passing the 4.75 mm sieve.
- 3.8 SPLITTER: Refer to MTO LS-600 - Method of Dry Preparation of Aggregates for Determination of Physical Constants.
- 3.9 HYDROCHLORIC ACID: This should be of technical grade, and prepared in 20 percent concentration by volume.

4. PREPARATION OF TEST SPECIMEN

- 4.1 Generally, the insoluble residue determination is performed on oven dried material passing the 19.0 mm sieve, and retained on the 4.75 mm sieve.
- 4.2 In order to obtain reliable results, at least 200 to 300 particles of each sieve fraction, representing more than 10 percent of the total sample, should be tested.
- 4.3 The 6.7 mm fraction is combined with the 4.75 mm fraction, to make a single fraction passing the 9.5 mm sieve, and retained on the 4.75 mm sieve.
- 4.4 Table 1 shows the recommended masses for each sieve fraction so that about 200 particles are tested:

Table 1

Passing	Retained	Approx. Mass g
19.0 mm	9.5 mm	1250
13.2 mm	9.5 mm	475
9.5 mm	4.75 mm	175

5. PROCEDURE FOR DETERMINING THE TOTAL INSOLUBLE RESIDUE

- 5.1 Reduce the prepared coarse aggregate test sample to sand size particles, using a crusher.
- 5.2 Place a representative crushed aggregate sample weighing between 100 and 150 g in the pre-weighed glass container. Record the mass of the container and sample. Slowly, add a 20 percent solution of hydrochloric acid, taking care to allow for excessive effervescence or foaming to subside before further addition of acid solution. Fill the container with solution, and allow it to stand overnight.
- 5.3 When the reaction appears to have subsided (no obvious bubbling), allow the insoluble material to settle, pour off the solution, and add about 1 000 mL of 20 percent acid solution. Take care not to lose any insoluble material.

5.4 If a reaction (bubbling) is observed, agitate until the reaction subsides, and add more 20 percent acid to the solution already in the container, and allow it to stand.

5.5 When no reaction is observed, heat the container gently to bring it to the boiling point. Continue gentle boiling for approximately 30 minutes to completely digest the carbonate.

5.6 After 30 minutes, or when the reaction has stopped, cool the mixture, and allow the insoluble material to settle. Decant the acid solution, and add water to the aggregate in the container to thoroughly dilute the remaining acid. Allow the mixture to settle, decant it, and repeat the procedure, if necessary.

5.7 Oven dry the material at $110 \pm 5^{\circ}\text{C}$ to a constant mass, and weigh the dried sample to the nearest 0.01 g.

5.8 Record the data on the Insoluble Residue Test Data Sheet, Figure 2.

6. PROCEDURE FOR DETERMINING THE RETAINED 75 μm FRACTION OF INSOLUBLE RESIDUE

6.1 Perform the steps in 5.1 through 5.7.

6.2 Sieve the insoluble residue material through the fine aggregate sieve series.

6.3 The mass of material retained on each sieve is weighed, and recorded cumulatively down to the 75 μm sieve.

7. CALCULATIONS

7.1 Calculate the total percentage of insoluble residue expressed as a percentage of the total original sample mass.

7.2 Calculate the cumulative percentage of insoluble residue retained on the 75 μm sieve expressed as a percentage of the original sample mass.

8. REPORTING OF RESULTS

8.1 Report the total percentage of insoluble residue and the percentage of insoluble residue retained 75 μm sieve to the nearest 0.1 percent.

9. PRECISION AND ACCURACY

9.1 The precision and accuracy for measuring insoluble residue in carbonate aggregates has not yet been established.

9.2 Testing of a 9.5 mm stone from one source by one operator, over a period of four years, showed the following variability:

	Total insoluble residue	Insoluble residue retained 75 μ m
Average	64	54
Range	59-72	50-58
Tests	8	8

10. GENERAL NOTES

10.1 Care should be taken when reducing coarse material that an excess of passing 75 μ m is not produced. A recommended fine aggregate gradation is shown in Figure 1.

10.2 If agglomeration has occurred following the oven drying of the insoluble residue, physical reduction of the agglomerated particles should be performed by use of a mortar and rubber-tipped pestle, prior to sieve analysis. Take care to prevent the crushing of individual particle grains during this process.

10.3 Acids are diluted on a volumetric basis. To prepare a 20 percent acid solution, use 4 parts of water to 1 part of acid. ALWAYS add the acid to the water.

11. PRECAUTIONS

11.1 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.

11.2 The addition of concentrated acid solutions to limestone aggregates produces violent foaming and effervescence. Proper safety apparel should be worn (i.e. goggles, gloves, apron).

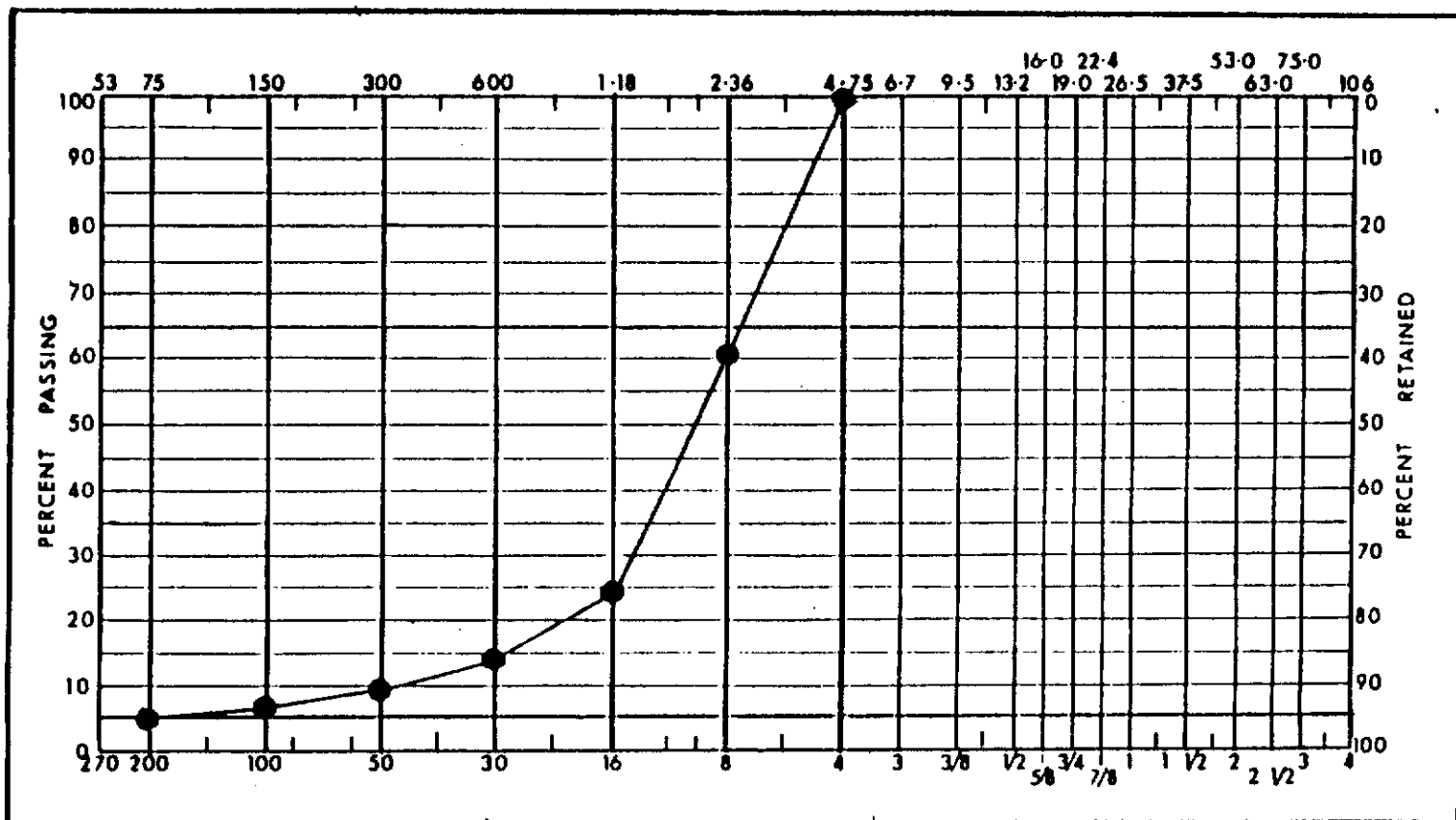


Figure 1 Recommended Gradation



DETERMINATION OF INSOLUBLE RESIDUE
BY DIGESTION IN HYDROCHLORIC ACID

SAMPLE NUMBER _____	DATE _____
SAMPLE LOCATION _____	
INTENDED USE _____	
BEAKER NUMBER	<input type="text"/>
MASS OF BEAKER AND SAMPLE	<input type="text"/>
MASS OF BEAKER	<input type="text"/>
MASS OF SAMPLE (A)	<input type="text"/>
MASS OF BEAKER AND INSOLUBLE RESIDUE	<input type="text"/>
MASS OF BEAKER	<input type="text"/>
MASS OF INSOLUBLE RESIDUE (B)	<input type="text"/>
PERCENT INSOLUBLE RESIDUE $\left[\frac{B}{A} \times 100 \right]$	<input type="text"/>
MASS OF INSOLUBLE RESIDUE (B)	<input type="text"/>
MASS OF INSOLUBLE RESIDUE RETAINED 75 μm (C)	<input type="text"/>
PERCENT INSOLUBLE RESIDUE RETAINED 75 μm $\left[\frac{C}{A} \times 100 \right]$	<input type="text"/>
TOTAL INSOLUBLE RESIDUE, PERCENT _____	
RETAINED 75 μm INSOLUBLE RESIDUE, PERCENT _____	
REMARKS : _____	

Figure 2 Test Data Sheet