

1221 **DETERMINATION OF ACID INSOLUBLE RESIDUE
IN LIMESTONE AND DOLOSTONE**
ASTM Designation D 3042 (Mn/DOT Modified)

1221.1 SCOPE

This method of test covers a procedure for determining the percent of acid insoluble residue in limestone and dolostone sedimentary rock. The procedure is intended to establish the amount of chert, sand, clay and other noncarbonate materials in limestone and dolostone (carbonate rock).

NOTE 1: ASTM D 3042 does not use vacuum filtration. Sample size used is 100 grams vs. 500 grams for ASTM D 3042.

1221.2 APPARATUS and SUPPLIES

- A. Sieve - A 76mm (3") diameter, 75 μ m (#200) sieve with a polycarbonate body and polyester screening.
- B. Vacuum Pump
- C. Polypropylene Buchner Funnels, 110mm in diameter.
- D. Bunsen Burner
- E. Glass Filter Flask, 1 liter.
- F. Balance - Conforming to AASHTO M 231, Class C with a readability, sensitivity and accuracy of 0.01 gram.
- G. Wash Bottle.
- H. Hydrochloric Acid, 6N.
- I. Oven - Capable of maintaining a temperature of 110 ± 5 °C (230 ± 9 °F) for drying samples.
- J. Sieve Shaker - Mechanical, Gilson-type.
- K. Sample Splitters
- L. Filter Papers - 110 & 150mm diameter, Whatman #1 filter paper (or equivalent).
- M. Large Iron Wire Triangle, 64mm (2 1/2").

- N. Pyrex Beakers, 1 liter, low form.
- O. Hot Plate
- P. Watch Glass, 125mm diameter.

1221.3 TEST SAMPLES

- A. Sieve bulk sample to obtain -9.5 to 4.75mm (-3/8" to #4) sized fraction.
- B. Wash the -9.5 to 4.75mm (-3/8" to #4) material.
- C. Oven dry sample to a constant weight at 110 ± 5 °C (230 ± 9 °F).
- D. By splitting, select four representative samples from 1221.3C, above. Each sample shall have a minimum weight of 100 grams.

1221.4 PROCEDURE

- A. Transfer the weighed sample ("**C**" in the calculations) to a 1000ml glass beaker and add enough deionized water to cover the sample.
- B. Slowly and carefully pour 500ml of 6N hydrochloric acid (**a small amount at a time**) over the sample. Stir or agitate sample and acid until all bubbling or effervescence has ceased. Continue to add acid until no reaction is noted. Then add an additional 100ml of acid to be sure that all carbonate is dissolved.
- C. After the reaction between the carbonate and acid has ceased, heat the beaker slowly over a burner to a gentle boil for at least 10 minutes and until all effervescence has ceased. Let cool.
- D. Weigh the filter papers (one piece of 110mm and one piece of 150mm) as a single weight ("**B**" in the calculations). Place the 150mm filter paper on top of the Buchner funnel and press into place by inserting a one liter beaker into the funnel. The 150mm filter paper will form a cup-shape in the bottom of the funnel. Center the 110mm filter paper in the bottom of the funnel, on top of the 150mm filter paper. Attach the Buchner funnel containing the filter papers to the filter flask and apply vacuum to seat the filter papers.
- E. Weigh a clean, dry 75 μ m (#200) sieve ("**E**" in the calculations) and insert into a support wire made from a large wire triangle. Place sieve on top of Buchner funnel. (**See Note 2**)

- F. Pour total residue and acid through the 75 μ m (#200) sieve. Wash the sieve with hot water until all the -75 μ m (-#200) residue has passed through the sieve. (**See Note 3**)
- G. Remove sieve and dry to a constant weight at 110 \pm 5 $^{\circ}$ C (230 \pm 9 $^{\circ}$ F) ("**D**" in the calculations).
- H. Wash filter papers with hot water until all trace of acid has been removed.
- I. Remove the filter papers and dry to a constant weight at 110 \pm 5 $^{\circ}$ C (230 \pm 9 $^{\circ}$ F) ("**A**" in the calculations).
- J. After the sieve and the filter papers have been dried, record the final weight.

1221.5 CALCULATIONS

- Where:
- A = The Final Weight of the Filter Papers with the Insoluble Residue
- B = The Initial Filter Papers Weight
- C = The Original Sample Weight
- D = The Final Weight of the Sieve with the Insoluble Residue
- E = The Initial Sieve Weight

- A. Percent Insoluble Residue, -75 μ m (#200) Fraction.

$$\frac{A - B}{C} \times 100$$

- B. Percent Insoluble Residue, +75 μ m (#200)

$$\frac{D - E}{C} \times 100$$

1221.6 REPORT

- A. Report both the minus 75 μ m (#200) and plus 75 μ m (#200) Percent Insoluble Residue for each sample and the average where triplicate samples are run.
- B. Unless otherwise specified, the average result shall be used to determine conformance to specifications.

NOTE 2: To facilitate filtering, large insoluble particles may be removed by pouring the sample through a 2.00mm (#10) sieve placed above the 75 μ m (#200) sieve. These are washed, dried and included in the fraction retained on the 75 μ m (#200) sieve.

NOTE 3: Visually inspect the residue retained on the 75 μ m (#200) sieve to determine whether or not particle agglomeration has occurred do to the presence of plastic clay fractions.

If agglomeration has occurred, physical reduction of these particles should be performed by gently rubbing the sample on 75 μ m (#200) sieve with a rubber-tipped pestle. Care should be exercised to prevent crushing of individual particle grains during this process and to prevent damage to the 75 μ m (#200) sieve.