WEST VIRGINIA DEPARTMENT OF TRANSPORTATION
DIVISION OF HIGHWAYS
MATERIALS CONTROL, SOILS AND TESTING DIVISION

MATERIALS PROCEDURE

# DETERMINING PERCENT QUARTZ IN CARBONATE AGGREGATES BY INSOLUBLE RESIDUE

## 1. PURPOSE

1.1 To provide a standard procedure for determination of the Plus No. 200 (75 μm) quartz content within the insoluble residue of carbonate aggregates.

## 2. SCOPE

2.1 This procedure is designed to exclusively determine the percentage of Plus No. 200 (75 μm) quartz content found within the insoluble residue of carbonate aggregates through chemical digestion using 6 *N* hydrochloric acid (HCl) solution.

## 3. REFFERENCED DOCUMENTS

3.1 *ASTM Standards:*

- C 136M-14
- C 702M-11
- D 75M-14
- D 3042
- E 11-16

3.2 *Other Standards:*

- Comparison chart for estimation of percentage composition (Terry and Chilingar 1955), Attachment 1
- West Virginia Department of Highways Materials Procedure, MP 700.00.06

## 4. APPARATUS

4.1 Sieves, U.S. standard 8 in. (203.2 mm) diameter, conforming to Specification ASTM E 11-16 in the following sizes:

No. 16 (1.18 mm)
No. 30 (600 µm)
No. 50 (300 µm)
No. 100 (150 µm)
No. 200 (75 µm)

4.2 Aggregate grinder, laboratory size and capable of reducing the coarse aggregate down to appropriate sieve size fractions.

4.3 Balance or scale having a capacity of 500 g and a sensitivity of at least 0.1 g for weighing fine aggregates.

4.4 Hot Plate capable of maintaining 230°F ± 9°F (110°C ± 5°C).

4.5 2000 ml Pyrex (Borosilicate glass) beaker for performing the acid test.

4.6 Wash Bottle (1000 mL) tap water.

4.7 Wash Bottle (500 mL) HCl.

4.8 Corrosion resistant 2 in. (3 oz.) tins.

4.9 Safety Eyeglasses, Rubber Gloves, Apron and Respirator.

4.10 Chemical Fume Hood

## 5. REAGENTS

5.1 Hydrochloric Acid (HCl); 6 *N* solution of 37% (concentrated) reagent grade HCl.

## 6. SAMPLES

6.1 Field samples shall be representative of the sources from which they are obtained according to MP 700.00.06.

6.2 Dry sample aggregates to constant mass at 230°F ± 9°F (110°C ± 5°C).

6.3 Prepare one 200 g minimum test portion for each aggregate field sample, per stockpile. A clean oven-dry sample having a minimum mass of 200 g shall be used for the test.

## 7. PROCEDURE

7.1 This section provides a procedure for the determination of Plus No. 200 (75 µm) size insoluble residue. The insoluble residue shall be separated through a series of sieves (Section 4.2) to ease the determination of quartz content.

7.2 Reduce aggregate particles to specified size utilizing a clean aggregate grinder. The aggregate used in the test sample shall pass the No. 16 (1.18 mm) sieve and be retained on the No. 30 (600 µm) sieve.

7.3 Weigh oven-dried sample, 200 g minimum, and place in 2000 mL borosilicate glass beaker. Slowly add small amounts of 6 *N* HCl and allow any excessive effervescence or foaming to subside before further addition of acid solution. Agitate the contents gently using a glass stirring rod until excessive effervescence has subsided.

7.3.1 **WARNING:** Hydrogen chloride gas is released during this procedure. Exercise extreme care to prevent corrosive gas from entering the laboratory atmosphere. Hydrogen chloride gas produced during the chemical reaction is destructive to laboratory equipment and is a definite safety hazard to laboratory personnel. The test shall be performed at all times under a ventilated hood. Personnel shall wear the appropriate safety equipment when performing the procedure.

7.4 Check the container periodically. When the reaction appears to have subsided (no obvious bubbling), neutralize the solution with water and decant the solution over a No. 200 (75 µm) sieve, taking care not to lose fines. Rinse the No. 200 (75 µm) sieve back into the beaker.

7.5 Add approximately 300 mL 6 *N* HCl, observe for reaction. If a reaction (bubbling) is observed, continue agitation until the reaction subsides and repeat the procedure in section 7.4.

7.6 When the reaction has stopped, heat the container gently on a hot plate. Heat the sample up to a temperature of approximately 230°F (110°C). Maintain this temperature for approximately 1 hour.

7.7 If a reaction is observed after heaing for approximately 1 hour, repeat the procedure beginning with step 7.5.

7.8 When the reaction has stopped, thoroughly dilute the liquid with tap water. Decant the solution over a No. 200 (75 µm) sieve.

7.9 Rinse the diluted solution and sample over a No. 200 (75 µm) sieve using tap water.

7.10 Thoroughly rinse all the material on the No. 200 (75 µm) sieve to remove any remaining acid residue. Cover the sieve with a loose fitting lid and place in a pan. Dry the sample to a constant mass at 230°F ± 9°F (110°C ± 5°C).

7.11 When the sieve and sample have cooled to room temperature, sieve the sample by hand over a No. 200 (75 µm) sieve to obtain the weight of the total amount of Plus No. 200 (75µm) insoluble residue.

7.12 Visually inspect the sample, with magnification if necessary, to determine if there are particle agglomerations due to the presence of plastic clay fractions.

7.13 If agglomerations are present, physically reduce the agglomerated particles with use of a mortar and rubber-tipped pestle. Exercise care to prevent the crushing of individual particle grains. If no agglomeration has occurred, proceed with the remainder of the test procedure.

7.14 To facilitate the Petrographic examination dry sieve the sample over the following series of nested sieves:

No. 30 (600 µm)
No. 50 (300 µm)
No. 100 (150 µm)
No. 200 (75 µm)

7.15 Weigh the material retained on each sieve and place each sample fraction in separate, clean, 2 in. tins. Cover the tins and mark the lids with the corresponding sieve sizes. Record each mass retained in the appropriate space provided on the Insoluble Residue form. (Attachment 2)

7.16 Place all tins for each sample into a single pan with the original sample card for identification. Document the mass of each sample fraction on the sample card.

## 8. PETROGRAPHIC EXAMINATION

8.1 Under a 45X power microscope, observe each tin's content by spreading a thoroughly mixed, representative sample onto an 8 oz. tin lid and shake to spread into a single layer. Determine the percent of total quartz material (only quartz crystals having transparent and hexagonal properties will be counted) through comparison to Attachment 1. Observing multiple viewing areas, note the percentage in each tin of the Insoluble Residue that is not quartz and write down in the appropriate area on the Insoluble Residue form, Attachment 2.

## 9. CALCULATION

9.1 Calculate the total percent of insoluble residue obtained in 8.1 by noting the percentage in each tin of Insoluble Residue that is not quartz, sum up the totals of the non-quartz Insoluble Residue and subtract from the Original Sample Weight. Report as a percentage of the total original aggregate sample mass.

9.2 To obtain the total quartz insoluble residue content, calculate the cumulative percent of quartz insoluble residue retained on each of the sieves in the indicated series, expressed as a percentage of the total original aggregate sample mass.

Example Form

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Lab No. 1451401 | Sampled: | 9/29/2015 | Size Mesh | Weight | Non-Quartz | | Quartz |
| | Test Run: | 10/27/2015 | | g | vol % | g | g |
| Field No. M124S0304 | Source: | Quarry | +30 | 6.9 | 45 | 3.11 | 3.80 |
| | Location: | | +50 | 8.1 | 33 | 2.67 | 5.43 |
| Agg. Size & Rk. #8 Limestone | Sample Type: | Extracted | +100 | 10.7 | 10 | 1.07 | 9.63 |
| | Sample Wt., g: | 184.7 | +200 | 21.6 | 8 | 1.73 | 19.87 |
| Tested by: Tech | Insoluble Residue Wt.,g: | 47.6 | -200 | 0.3 | n/a | +200 Qtz., g | 38.73 |
| | Sieving Loss/Gain, %: | 0.0 | Total: | 47.6 | | **+200 Qtz.,%** | **21** |

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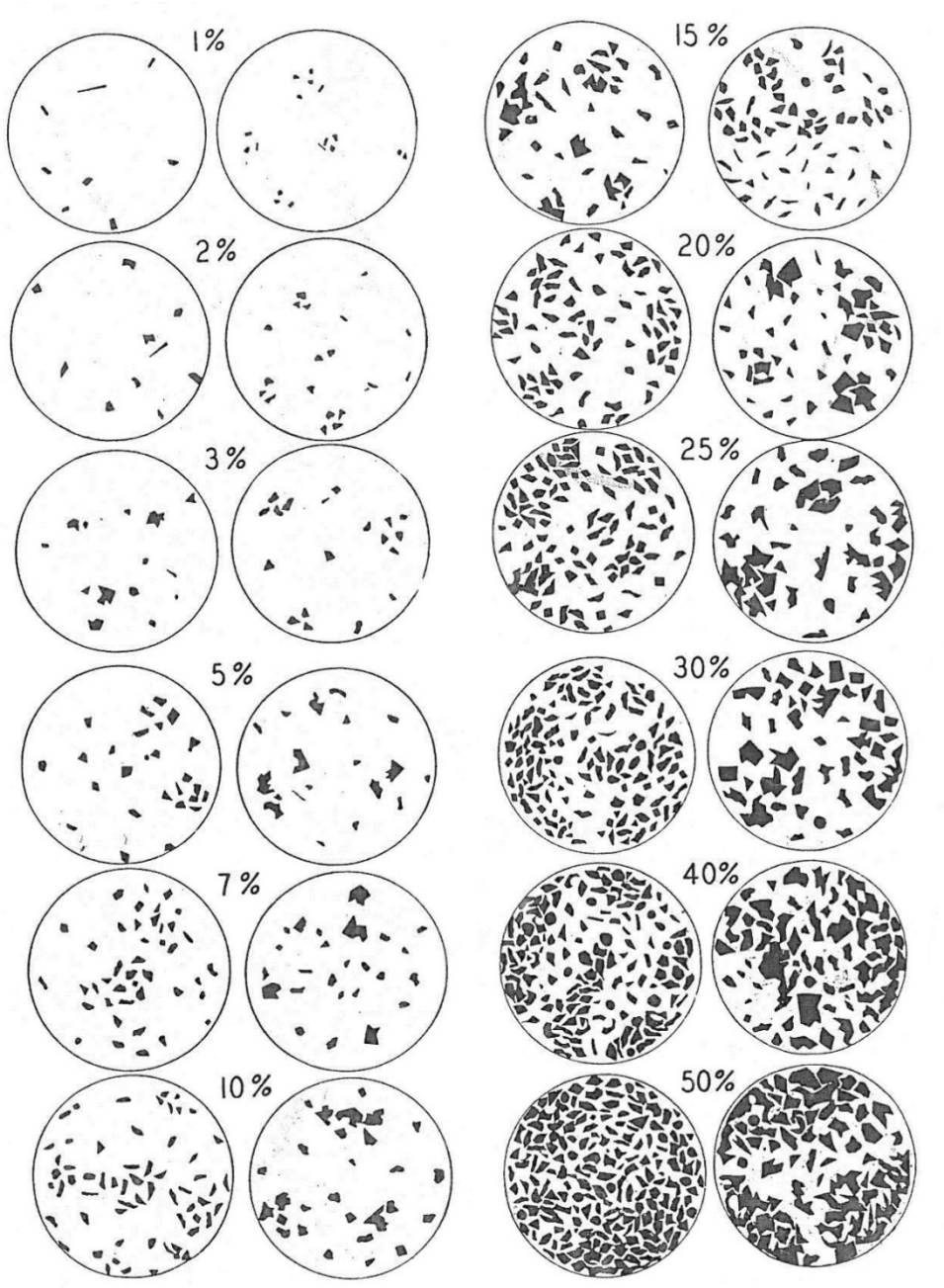
**ATTACHMENT 1**
**COMPARISON CHART FOR ESTIMATION OF PERCENTAGE COMPOSITION**




"Comparison chart for estimation of percentage composition," *from*: Terry, Richard D. and Chilingar, George Varos, 1955, Summary of "Concerning some additional aids in studying sedimentary formations" by M. S. Shvetsov. *Journal of Sedimentary Research*, vol. 25, no. 3, 229-234.

## ATTACHMENT 2
## BLANK DATA COLLECTION AND CALCULATION FORM

| Lab No. | Sampled: | | Size | Weight | Non- Quartz | | Quartz |
|---|---|---|---|---|---|---|---|
| | Test Run: | | Mesh | g | Vol % | g | g |
| Field No. | Source: | | +30 | | | | |
| | Location: | | +50 | | | | |
| Agg. Size | Sample Type: | | +100 | | | | |
| | Sample Wt., g: | | +200 | | | | |
| Tested by: | Insoluble Residue Wt., g: | | -200 | | N/A | +200 Qtz.,g: | |
| | Sieving Loss/Gain,%: | | Total: | | | **+200 Qtz.,%:** | |