Chemical Testing of Manufactured Sand and Other Fine Aggregates

CHEMICAL LAB. PROCEDURE ; M/T C-9.0

** * This procedure does not purport to address all the safety concerns associated with its use. It's the responsibility of the user of this procedure to establish appropriate health and safety practices and dispose of hazardous materials in an approved manner.*

Test: Chemical Analysis of Manufactured Sand and Other Fines Aggregates

(Fine aggregates defined as blasting abrasives, concrete cores, soil, etc.)

Equipment: Standard Lab and Safety Equipment, Substantial Degree of chemical skill using relatively elaborate instrumentation.

Reference : NCDOT Chemical M/T C-9.0

Procedures:

I. Determination of Silicon Dioxide

II. Sulfate Analysis on Blasting Abrasive, etc.

---------Some Abbreviations used: fp= filter paper, ppt=precipitate, hp=hotplate, Pt=Platinum,

---------HOH= distilled water

Sample Preparation;

1. Samples are received from the Field Section. They are then logged in and a part is received on HiCAMs representing the sample.
2. Sample containers are labeled with Chem. Lab internal identification.

Testing Procedures:

Any crucible should be fired at the temperature it will be used at.

Dessicate Platinum crucibles for 30 minutes ± 5 minutes.

Dessicate CEM quartz fiber crucibles 10 minutes ± 2 minutes.

Dessicate Porcelain or quartz crucibles 90 minutes ± 5 minutes.

Label all reagent containers and glassware during analyses.

I. Determination of % SiO2 in Manufactured Sand and other fine aggregates.

1. Grind blasting abrasive in mortar and pestle then….
2. Dry a small amount of the sand in an oven at 110ºC for 8 hours or overnight. Do not attempt sodium carbonate fusion with a sand.
3. Weigh out 1.0000 grams on the analytical balance and transfer to a 400 mL Phillips beaker.

4. Add 30 mL of 1:1 HCl to the beaker while swirling.
5. Bring the sample to a boil and let boil 30 seconds.
6. Remove, cool, and filter through a #41 Whatman filter (12.5 cm). A waste beaker may be used unless Ca, Mg, or Fe is desired. Use M/T C-1.1.
7. Rinse the paper and sample at least 8x with hot HOH.
8. Transfer the filter paper to a weighed Pt crucible that has been brought to a constant weight.
9. Place the crucible on a mat over low heat and burn off until the filter paper has turned black.
10. Transfer crucible to a 950ºC muffle furnace for 20 minutes.
11. Move crucible to dessicator and allow to cool for 30 minutes.
12. Weigh crucible and record weight. Let this be weight A.
13. To the crucible add 1 mL HOH and 4 drops of 1:1 H_2SO_4.
14. Place crucible on hot plate only under hood and immediately fill it half full of HF(conc).
    **Much care should be used when dispensing hydrofluoric acid(50%). Use adequate ventilation so as not to breath its fume and wear rubber gloves.
15. Evaporate contents of crucible to dryness on a 99ºC hot plate.
16. Remove from hot plate and allow to cool to room temperature.
17. Again fill the crucible about 1/3 full of HF acid and evaporate to dryness on the hot plate.
18. Place the crucible on a mat over low heat and gradually increase heat to drive off the residual acid. Heavy fumes will be burning off for several minutes. Move to a triangle over low heat and gradually increase heat. After no more "smoke" occurs move to muffle furnace at 950ºC for 5 minutes.
19. Remove, dessicate and weigh. Let this be weight B.
20. (A – B) x 100 = % SiO_2
21. Specification is 25 % minimum.

II. Sulfate Analysis on Blasting Abrasive and Similar Materials

(Use of a microgram analytical balance is required)

1. Check conductivity before starting test. If zero do not analyze.
2. Grind sample in large mortar and pestle until a fine powder (20 mesh) is obtained.
3. Dry sample in 105ºC oven for 24 hours.
4. Dessicate dried sample 90 minutes and then weigh out 10.00000 grams into a 400 mL Phillips beaker.
5. It is recommended duplicates be done on samples of environmental concern.
6. Add 25 mL HOH while swirling the beaker.

7. While swirling quickly add 5-8 mL HCl(conc) and then boil 30 seconds.
8. Place on a 99ºC hot plate and digest for 3 hours.
9. Filter through a #41 inside a #40 Whatman (12.5 cm) fp into a 400 mL beaker.
10. Rinse with hot HOH 6 x and police beaker.
11. Dilute filtrate to 225-260 mL and place on a Fisher burner.
12. Place a stirring rod and bumper in the beaker also. Cover with a watch glass.
13. When solution starts boiling lower heat slightly and add 10 ml $BaCl_2$(100g/L) dropwise.
    Add 10 mL BaCl2 reagent to 125 mL HOH. Digest. Run as a reagent BLANK.
14. Let the solution boil for 2 more minutes and remove and digest on a 99ºC hp 8-12 hours.
15. Filter through a #40 Whatman(12.5 cm) fp and rinse with hot HOH 6x and police well. Scour the beaker with a piece of filter paper.
16. Place filter into a weighed Pt crucible and char off slowly.
17. Place crucible into 850ºC muffle furnace for 45 minutes.
18. Dessicate and weigh.
19. Equation for calculation is (34.3 / 10) x grams= % SO_3 as $BaSO_4$. Multiply by 10000 to get parts per million(ppm).
20. Specifications= 100 ppm maximum.