



BUREAU OF MATERIALS MATERIALS PROCEDURES

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APPROVAL: Edward Inman

INFRARED (IR) SPECTROPHOTOMETRY ANALYSIS OF CHEMICAL ADMIXTURES FOR USE IN CONCRETE

PURPOSE:

To establish standard procedures for Infrared (IR) Spectrophotometry Analysis of Chemical Admixtures for use in Concrete.

GENERAL REFERENCES:

NJ DOT Bureau of Materials Material Approval Procedures MAP#106.

NJDOT Standard Specifications Sections 903.02.02.

ASTM C494-19e1

AASHTO M194 (ASTM C494).

Rutgers State University of New Jersey Report FHWA-NJ-2011-007 "INFRARED SCAN OF CONCRETE ADMIXTURES AND STRUCTURAL STEEL PAINTS".

FORMS:

Site Manager (SM) NJDOT# M0194IR AASHTO M-194 Analysis of Infrared Spectrophotometry.

SM NJDOT# LB 417 Analysis of Liquid Concrete Admixtures.

TEST METHODS DESCRIPTION:

Quantitatively comparing the Chemical Analysis of the Test Sample to the Initially Approved Chemical Analysis for each Material Brand Name by the Correlation Coefficient (CC) to the Correlation Threshold (CT), based on ASTM C494 with exception and the Quantitative Test Method for Concrete Admixtures (FHWA-NJ-2011-007) described in "INFRARED SCAN OF CONCRETE ADMIXTURES AND STRUCTURAL STEEL PAINTS" research report.

- 1- ASTM C494 exception: Section 18.1 instead of "results should not be interpreted quantitatively" use "results could be used for quantitatively interpretation".

- 2- Use the Quantitative Test Method described in “INFRARED SCAN OF CONCRETE ADMIXTURES AND STRUCTURAL STEEL PAINTS”, design by Rutgers State University of New Jersey for NJ DOT. .
- 3- IR scan apparatus should be set for “Quantitative Analysis.”

TEST SPECIFICATION REQUIREMENT:

- A. Correlation Coefficient \geq Correlation Threshold.
- B. Correlation Threshold set for every Admixture Brand Name based on three years production data.
- C. Correlation Threshold for New Admixture set for every Manufacturer based on three years production data.

INSTRUCTIONS:

I. Assignment Procedures

- A. The Team Leader or Supervisor shall:
 1. Assure that the Team receives the following:
 - a. Copies of references and test methods pertinent to this MP.
 - b. Site Manager Sample ID Number.
 - c. Technical Data Sheet, MSDS and Production Report.
 - d. One-quart sample.
 2. Check the Sample Label against the paperwork.
- B. The Technician shall:
 1. Inspect the sample visually.
 2. Make the test preparation.
 2. Run the test.
 3. Put the test result in SM Form.
 4. Print out Crystal Report and file the hard copy.
- C. The Supervisor shall review the Crystal Report.
 - A. The Technician shall electronically distribute the Crystal Report to Inspection Group's Leader.
 - B. The Inspection Group's Leader shall immediately notify the RE of all failing test results.

II. Material Preparation for IR scan

- A. Preparation Procedure for Liquid Admixtures Type A to G & S (C 494/C 494M –19e1)
 1. Determine % solids by ASTM method C 494 / C 494M-19e1 Example:

114.5678 Weighing dish & Admix
110.1234 Weighing dish
4.4444

111.5678 Dish & Admix dried
110.1234 Weighing dish
1.4444 "Amount of loss"

2. Determine amount of water needed using the following formula.

$$(\text{"Amount of loss"}) / 0.015 - 4 = \text{"water needed"}$$

3. Measure "water needed" in a graduated cylinder and pour into appropriate size beaker. Use distilled water.

4. Pipette 4 ml of admixture into beaker with distilled water and stir with the pipette to incorporate.

5. Add 2.5g of KBr (Potassium Bromide) to a 15x100mm glass Petri dish.

6. Pipette 5ml of the admixture / distilled water mixture into the Petri dish.

7. Add 5ml of distilled water to the Petri dish.

8. Stir with metal spatula until KBr is dissolved.

9. Place Petri dish in oven at 105° C for 17 hours.

Continue with "**Pressed Pellet**" procedure (below).

III. Pressed Pellet Procedure

1. After time has elapsed, remove Petri dish from oven and place in desiccators and allow cooling for 30 minutes.

2. Using a metal spatula, carefully scrape the dried material from the Petri dish into a mortar and pestle.

3. Grind it to a fine powder.

4. Weigh 0.1g of the powder in a weight dish and carefully pour into a stainless-steel capsule (weight between 0.1000 & 0.1009).

5. Weigh 0.4g of KBr (Potassium Bromide) and carefully add to stainless steel capsule. (weight between 0.4000 & 0.4009).

6. Add steel mixing ball to capsule.

7. Take capsule to Spex Sample prep mixer/mill and insert capsule into a plastic vial.

8. Put lid on the vial.

9. Insert vial into #8011 adapter along with an empty vial for balance.

10. Place adapter in mixer/mill and tighten securely in place.
11. Close the lid and fasten the latch. Set timer to 30 seconds and press start.
12. Remove capsule and return to scale.
13. Remove lid from capsule.
14. Using a magnet, remove the metal mixing ball.
15. Weigh 0.3g (between 0.3000 & 0.3009)
16. Carefully pour into the center of the metal die.
17. Gently shake the die to evenly distribute the powder.
18. Insert the metal plug on top of the powder, shiny side down.
19. Put cover on die and squeeze the halves together.
20. Invert the die and place in the center of the Spex X-Press.
21. Turn the hand wheel on the top of the press to lower the adjustment screw snugly on the top of the die.
22. Close the sliding door of the X-Press and press "Run".
23. The press is set to apply 10 tons of pressure for 3 minutes with a 1 minute release time.
24. The press will beep loudly when the time has elapsed.
25. Loosen the adjustment screw by turning the hand wheel.
26. Remove the die.
27. Invert the die.
28. Remove the cover.
29. Place the metal "C" ring on the top of the die.
30. Return the die to the press and lower the adjustment screw until the pressed pellet is released.
31. The pellet may stick slightly to the die or the metal plug.
32. Use a razor blade to carefully free the pellet and slide it onto the pellet holder.
33. DO NOT TOUCH the pellet with your fingers.

IV. IR scan Test

- A. Run the Test:
 1. Turn on the computer.
 2. Click the NJDOT Icon.
 3. Open the Assure ID Analyzer Program.

4. Click OK.
5. At the top of the box, identify the sample by name from the drop-down menu.
6. Click "Analyze".
7. Click "Next".
8. Make sure there is nothing blocking the scan position.
9. Click "Start" to perform background scan.
10. After background, throughput, and noise check, Click "Next".
11. Enter the sample serial number.
12. In the "Material Type" window, identify the sample by name.
13. Click "Next".
14. Click "Next".
15. Insert the sample holder with the pressed pellet into the scan position; make sure the pellet remains in an upright position.
16. Click "Start".
17. Wait for the scan to be completed.
18. Click "Next".
19. Click "View Report", maximize, and enlarge screen to view result.
20. Scroll down slightly so both the material scan and the fingerprint scan are visible.
21. Take a screen shot by pressing "Control and Print Screen" at the same time.
22. Open the WordPad program from the Start Menu.
23. Click the "Paste" icon in the upper left corner.
24. Click the "Save" icon in the upper left corner.
25. Click on "Desktop".
26. Find the file folder for the admixture being scanned and Click on it.
27. Locate "File Name" and enter the sample serial number.
28. Click "Save".
29. Close WordPad program.
30. Close "View Report" window.
31. Click "Next".
32. Click "Finish".
33. Close Assure ID window.

B. IR scan Retest:

If the Test failed, make another pellet (see Section III. Making Pressed Pellet above) from prepared material (see Section II. IR scan Preparation above) and test (see Section IV. IR scans Test above).

IV. Reporting Test Result

- A. Put the test result into SM Form # M0194IR AASHTO M-194 Analysis of Infrared Spectrophotometry.

B. For New Material:

- Set the IR Scan Standard for Brand Name Admixture.
- Report to the Admixture Spec Manager.

V. Authority and Responsibility

The Technician should bring to the attention of the Supervisor any uncertainties regarding the quality of material, equipment, methods of operation or sampling. Document the discussion and the corrective action taken.

V. Distribution of Forms

It shall be the Team Supervisor's responsibility to ensure forms are distributed as follows:

FORM

DISTRIBUTION

LB417 The Technician shall electronically distribute the LB417 Analysis of Liquid Concrete Admixtures Report to Inspection Group's Leader.