INFRARED SPECTROSCOPY SCANNING OF CONCRETE ADMIXTURES AND STRUCTURAL STEEL PAINTS

By

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And approved by

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Abstract of the Thesis

INFRARED SPECTROSCOPY SCANNING OF CONCRETE ADMIXTURES AND STRUCTURAL STEEL PAINTS

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This study evaluated correlation coefficients of concrete admixtures and structural steel paints by performing IR Scans in order to ensure adequate performance of the specimen using ASTM C494-05a specifications. In order to achieve this goal, numerous IR Scans were performed on the specimens from the manufacturer with different batches in order to ensure conformity. These scans were then analyzed and averaged to create a unique correlation coefficient for each admixture and steel paint. The correlation coefficients were used to quantitatively evaluate and interpret IR Scans of job samples. There were 23 of the most commonly used concrete admixtures tested by the NJDOT. These include air-entrainers, accelerators, retarders, water reducers, and other combinations of these agents including corrosion inhibitors. Their correlation coefficients were established by averaging 12 scans total over three different batches, 4 from each batch. In addition to the admixtures, 28 steel paints used by the NJDOT were also tested. These included primary and secondary coats, thin films, and resins. For the structural steel paints, the correlation coefficients were averaged with 6 scans from three different batches, 2 from each batch. In order to validate and establish an acceptance range, job samples were tested and compared to the correlation created. This study also includes the

effects of drying time on air-entraining admixture's correlation coefficients and the effects of different KBr types on correlation coefficients.

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Introduction

Concrete admixtures and structural steel paints are used all the time in civil engineering projects. However, these admixture and paints, by law, must be tested and approved for quality and identification. Some of these parameters can be found in the New Jersey Department of Transportation's Standard Specifications for Road and Bridge Construction sections 903.02.01, 903.02.02 and 912.01.01 (Najm et al., 2011). These tests are important to insure that the products have not been altered in any way to hamper their performance. One test method is to use an infrared spectrophotometry scan (IR scan) to verify that the job samples of admixtures or steel paints from construction projects match those established baseline samples that were originally submitted, approved, and listed on the Qualified Producer/Supplier List (Najm et al., 2011).

In general concrete admixtures are used to enhance the concrete performance and quality control process in the field. In this study, twenty three of the most commonly used concrete admixtures in NJDOT construction projects were chosen. These include air-entrainers, accelerators, retarders, water reducers, and other combinations of these agents including corrosion inhibitors (NJDOT Material Specifications, 2011). Admixtures can accelerate/slow the setting time, improve workability, enhance frost and sulfate resistance, and help control strength development. All of these effects have a place and purpose in concrete mix design. About 80% of concrete produced in North America contains one or more types of admixtures (Ramachandran, 1995).

It is often required by the government agencies (DOT's) to monitor the integrity of these admixtures so that they can guarantee the quality of materials that are used in

their projects. ASTM C494 requires testing of concrete admixture in accordance with Table 1 (ASTM 494) designated as Level 1 testing. It also requires Level 3 testing needed for uniformity and equivalency. Level 3 testing is established using the following requirements: 1) Infrared analysis, 2) Residue by oven drying, and 3) Specific gravity. The work done in this study focuses on the infrared analysis. ASTM C494/C494M-11 Section 6.1.1 requires that the absorption spectra of the initial sample and the test sample be essentially similar. This section does not provide specific criteria for acceptance or rejection of the test sample.

Objective

This study sought to establish acceptance criteria for correlations of concrete admixtures and steel paints based on a rigorous testing program and statistical analyses of IR scans. These criteria will be the basis for quantitative assessment of infrared scans for concrete admixtures and steel paints used in construction projects.

The objective of this investigation was to: 1) provide interpretations from the IR scans of the concrete admixtures and steel paints, including the factors that may influence them; 2) establish correlations coefficients and acceptable tolerances for standard manufacturer samples of the admixtures and paints; and 3) verify the established acceptance criteria by testing job samples. This thesis will present the findings from this study, discuss limitations and applications of developed correlation coefficients, and make recommendations for future tests or modifications to tests that can be preformed to better understand and identify what causes the nonconformity of the IR scans for concrete admixtures and structural steel paints.

Literature Review

Infrared spectroscopy is used both to gather information about a compound's structure and principal components and as an analytical tool for assessment for qualitative and quantitative analyses of the conformity of mixtures (Fernandez-Carrasco et al., 2012). These scans can be used to interpret both organic and inorganic compounds (Coates, 2000). Infrared radiation is absorbed by molecules and is converted into energy of molecular vibrations. When the radiant energy matches the energy of a specific molecular vibration, absorbance occurs (Fernandez-Carrasco et al., 2012). This absorbance would then hold unique information of a specific sample (spectrum).

Fourier transform infrared spectroscopy (FTIR) is a type of technique used to obtain an infrared spectrum of a solid, liquid, or gas sample. However, many materials are opaque to IR radiation and must be dissolved or diluted in a transparent matrix in order to obtain their spectra. Alternatively, it is possible to obtain reflectance or emission spectra directly from opaque samples (Settle, 1997).

This technique shines a beam of light, in a range of frequencies, at a sample all at the same time. Then an absorbance is measured at each wavelength. Mid-IR range includes wavelength of 2.5 to 25 µm that corresponds to a wave number (inverse of wavelength) range of 4000-400 cm-1 range, respectively. Because time and frequency are inversely proportional, a mathematical Fourier transform allows conversion of intensity versus time spectrum into intensity versus frequency spectrum that is typically used in FTIR analysis (Nasrazadani et al. 2010). Figure 1 shows the range of frequencies infrared spectroscopy operates in.

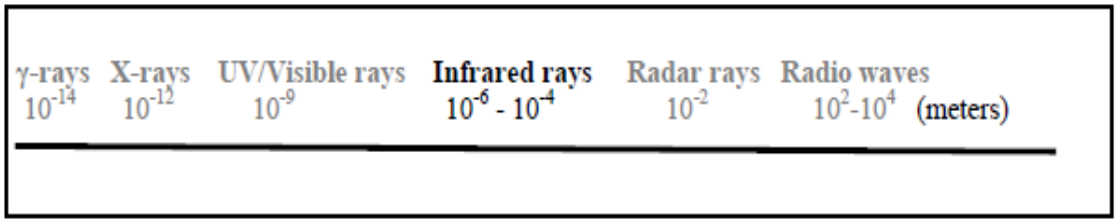

Figure 1: Electromagnetic spectrum (Nasrazadani et al. 2010).

FTIR analysis became the main used technique when specific analytical topics have to be addressed, mainly when non-destructive analysis is needed (Litescu et al. 2012). This is very important because we do not want to damage or compromise the infrastructure's structural integrity. In fact, many testing methods in the US are moving towards non-destructive testing and analysis.

"The preferred format for presenting spectral data for qualitative analysis is in the percentage transmittance format, which has a logarithmic relationship ($-\log_{10}$) with respect to the linear concentration format (absorbance). This format, which is the natural output of most instruments (after background ratio), provides the best dynamic range for both weak and intense bands. In this case, the peak maximum is actually represented as a minimum, and is the point of lowest transmittance for a particular band" (Coates, 2000).

The IR spectroscopy is typically used in cases where the sample (or spectrum) is a ''total unknown'' and an identification is required, the sample (or spectrum) is an unknown and it needs to be characterized or classified, and the sample is generally known but the existence of a specific chemical class needs to be determined (Settle, 1997). IR spectroscopy can also be used when the sample is a complete known and the interpretation is required to confirm the material composition and/or quality. This use of

IR spectroscopy would include product quality control of concrete admixtures and structural steel paints.

The Louisiana DOT outlines test methods for infrared spectrophotometric analysis (LADOT 1994). The method is used for a variety of materials such as paint, epoxy resin systems, anti-strip additives, concrete admixtures, thermo-plastics, solvents and other materials that occur as a solid, low volatile liquid, or highly volatile liquid. DOTD TR 610M-94 outlines sample preparation procedures for solid samples and liquid samples. The interpretation of results is qualitative based on a favorable comparison of the infrared spectrum to that of the original sample. According to LADOT memo DOTD TR 610M-94, a sample is considered rejected if its IR spectrum exhibits significant nonconformity to the IR spectrum of the original sample, i.e. if there are different absorption valleys in the two spectra or if an absorption valley in one spectrum is significantly displaced from that in the other one.

The California Department of Transportation (Caltrans) published tests methods for concrete admixtures in 2007. In their CA Test 416, Caltrans outlines the testing procedure for IR scan of concrete admixtures. This procedure is somewhat different from ASTM C494/C494M-11. According to the Caltrans criteria (2007), test results are used for comparison purposes only and each spectrum is compared with samples run previously. Two materials are considered similar if all of the absorption peaks match at wavelengths and relative magnitudes (Caltrans, 2007).

The Illinois Department of Transportation (IDOT) published a list of approved concrete admixtures and specifications that outlines the submittal process for the

approval of new concrete admixtures (IDOT, 2011). Among these specifications are those for the submittal of an infrared spectrophotometer trace (IR) of current production material, no more than five years old. The IR scan should be labeled with the date the scan was performed, the product name, and the manufacturer's name. However, the IDOT specifications do not provide information on quantitative methods for acceptance of IR scans of concrete admixtures.

The New Jersey Department of Transportation (NJDOT) currently uses a quality standard correlation coefficient equal to 0.975 for acceptance of job samples for all admixtures based on the manufacturer recommendation. Although this may seem like a fairly high and relatively safe correlation to abide by, every admixture possesses their own unique chemical and physical properties, and may not have the same acceptable correlation values. Furthermore, the basis for using this correlation coefficient for quantitative assessment of concrete admixtures quality control was not established.

Methodology

In order to accomplish the objectives of this study, a number of admixtures had to be selected for the experimental investigation. There are sixty one approved admixtures in Table 1 to choose from on the NJDOT Qualifier Producer/Supplier List (QPL) (Najm et al., 2011). Out of these sixty one admixtures, twenty three admixtures were identified as the most commonly used by the NJDOT in their construction projects. These can be seen shaded in yellow and green in Table 1.

Table 1: NJDOT Qualified Producer/Supplier List of concrete admixtures

WR GRACE	EUCLID	BASF MB	GREAT EAST'N	SIKA	AXIM
DARAVAIR 1000	AEA-92	MB - AE90	Secton 6A	Sika Air	Catexol A260
MIRA 62	AIR MIX	MB-VR STD	Chemstrong A	Plasttolcrete 161	Catexol 1000 SP MN
RECOVER	EOCONWO	Pozzolit 200N	Chemstrong SP	Plastolcrete 161 FL	Allegro 122
ADVA140M	ACCELGUARD80	Glenium 7500	Chemstrong R		Catexol 1000 R
ADVA 190	ACCELGUARD90	Polvheed 900			Catexol 3000 GP
ADVA FLEX	AIR MIX 200	Glenium 7700			
ADVA CAST 555	EUCON 1037	Glenium 7710			
DARACEM19	EUCON CIA				
DARACEM100	EUCON MR				
DARASET 400	EUCON 75				
ADVA CAST 575	EUCON WR-91				
ADVA	EUCON WR-75				
WRDA-82	PLASTOL 341				
WRDA W/HYCOT	PLASTOL 5000				
POLARSET	PLASTOL 5500				
DARATARD17	AIR EXTRA				
DARAVAIR AT 60	EUCON 37				
DAREX II	EUCON 727				
LUBRICON NCA	PLASTOL 6000				
DCI	PLASTOL 6200 EXT				
DARACEM55					
Total = 21	Total = 20	Total = 8	Total = 4	Total = 3	Total = 5

Table 1 is arranged by supplier and shows a number of concrete admixtures they produce that have been approved on the NJDOT Qualified Product List (QPL). In this table, there are several different types of admixtures as listed below:

Type A	Water-reducing admixtures
Type B	Retarding admixtures
Type C	Accelerating admixtures
Type D	Water-reducing and retarding admixtures
Type E	Water-reducing and accelerating admixtures
Type F	Water-reducing, high range admixtures
Type G	Water-reducing, high range, and retarding admixtures
Air	Air-Entraining Admixture
VMA	Viscosity modifying agent
CI	Corrosion inhibitor

This is significant because some admixtures have different procedures than others, and therefore is important to know how each admixture is classified. Table 2 shows the

twenty three concrete admixtures chosen and their classifications according to their type and supplier (Najm et al., 2011). These are the concrete admixtures tested in this study.

Table 2: Classification of approved concrete admixtures based on supplier and type

Admixture	Supplier	Type
MB-AE 90 (A00181)*	Master Builder	AIR
MB-VR standard (A00180)*		AIR
Pozzolit 200N(A00174)		A
Glenium 7500(A00189)		A&F
AIR MIX (A00159)*	Euclid	Air
AEA92(A00158)*		Air
Eucon WR-91(A00166)		A&D
DARAVAIR 1000(A00215)*	WR GRACE	AIR
Daracem 55 (A00229)		A
WRDA with HYCOL (A00210)		A
Daracem 19 (A00203)		F
Secton 6A(A00226)*	Great Eastern	AIR
Chemstrong A (A00222)		A
Chemstrong SP (A00223)		F
Chemstrong R (A00221)		D
Sika Air(A00474)*	Sika	AIR
Plastolcrete 161(A00144)		A
Plastolcrete 161 FL(A00479)		C&E
Catexol AE 260(A00398)*	Axim	AIR
Catexol 1000 SP MN (A00400)		A&F
Allegro 122 (A00397)		A&F
Catexol 1000 R (A00402))		B&D
Catexol 3000 GP(A00394)		A

After the concrete admixtures were approved for testing, the structural steel paints had to be identified. The steel paints also have an approved QPL and can be seen in Table 3. There are thirty one steel paints on the NJDOT QPL for structural steel paints, however, only twenty eight paints were selected to analyze. The structural steel paints are also examined using IR scans, but have a different procedure compared to the concrete

admixtures. The twenty eight structural steel paints selected for this study are listed in Table 4.

Table 3: NJDOT Qualified Producer/Supplier List of structural steel paints

MAB	**International (Already tested)**
Ply-Tile Epoxy Zinc Rich Pt A	**OEU - Interzinc 315B Part A**
Ply-Tile Epoxy Zinc Rich Pt B	**OEU - Interzinc 315B Part B**
Ply-Thane 890 Urethane Pt A-Gray	**OEU - Interguard 475 HS Off White Part A**
Ply-Thane 890 Urethane Pt B	**OEU - Interguard 475 HS Converter Part B**
PLY- Mastic 650 Part B	**OEU - Interguard 475 HS Buff Part A**
PLY- TILE Part B	**OEU - Interthane 870 UHS Black Part A**
PLY-Thane 890 (Green)	**OEU - Interthane 870 CONVERTER Part B**
PLY-Thane 890 (Blue/Gray)	**Ameron/PPG**
PLY-Thane 890 Part A (Yellow)	**Amercoat 68 HC Cure**
PLY-Mastic 650 Part A (White)	**Amercoat 68 HC Resin**
PLY-Thane 890 Part B (HSG)	**Amercoat 450H Cure**
PLY-Thane 890 Part A (HSG)	**Amercoat 399 Pearl Grey Resin**
Sherwin-Williams	**Amercoat 399 Pearl Grey Cure**
Steel Spec Epoxy Red Pt A	**Carboline**
Steel Spec Epoxy White Pt A	**Carboguard 893 White 0800 Pt B**
Steel Spec Epoxy Pt B	**Carboguard 893 White 0800 Pt A**
Zinc Clad DOT Base Pt A	**Carbozinc 11 HS Base Green 0300**
Zinc Clad DOT Pt B	**Carbozinc 11 HS Activator**
Devon	**Urethane Converter 8800 0909**
Bar-Rust 235 Part A White	**Carbothane 133 LH Brown 2285**
Bar-Rust 235 Part B Converter	
Devthane 359 DTM White Tint Base Pt A	
Devthane 359 DTM Pt B Converter	

Table 4: Selected structural steel paints

International	**MAB**
OEU - Interzinc 315B Part A	PLY- Mastic 650 Part B
OEU - Interzinc 315B Part B	PLY- TILE Part B
OEU - Interguard 475 HS Off White Part A	PLY-Thane 890 (Green)
OEU - Interguard 475 HS Converter Part B	PLY-Thane 890 (Blue/Gray)
OEU - Interguard 475 HS Buff Part A	PLY-TILE Part A
OEU - Interthane 870 UHS Black Part A	PLY-Mastic 650 Part A (White)
OEU - Interthane 870 CONVERTER Part B	PLY-Thane 890 Part B (HSG)
Ameron / PPG	PLY-Thane 890 Part A (HSG)
Amercoat 68 HS Cure	**Carboline System**
Amercoat 68 Resin	Carboguard 893 White 0800 Pt. A
Amercoat 450H Cure	Carboguard 893 White 0800 Pt. B
Amercoat 399 Pearl Grey Resin	Carbozinc 11HS Base Green 0300
Amercoat 399 Pearl Grey Cure	Carbozinc 11HS Activator
Amercoat 450H Green Resin	Carbothane 133 LH Brown 2285
Amercoat 450H Blue	Urethane Converter 8800 0909

Once all of the concrete admixtures and structural steel paints have been identified, IR scans were performed on the manufacturer's samples to establish baseline data and acceptable tolerances and correlations. The general experimental procedure must parallel ASTM C 494/C 494M-05a which will be discussed later in this thesis under the research procedure section. To create the correlations coefficients for the selected admixtures and paints, it was decided that three different batches from each admixture and paint be tested at three different time intervals. This will ensure non-bias and uniformity of the IR scans. Within each batch there were four scans of each admixture and two scans for each paint. Thus a total of twelve scans were made for each admixture and six scans for each paint. Less scans were made of the paints because the correlations were so high it was decided that they did not need as many scans as those needed for the admixtures. With these scans, an extensive data library was established and had enough

scans to create acceptable correlation coefficients for all the concrete admixtures and structural steel paints.

After all of the samples have been scanned and correlation coefficients have been established, the job samples were tested to verify the established correlations and the acceptable tolerances. To verify the established correlations, five job sample admixtures were selected and tested. In order for the job samples to pass, their scans have to be equal to or higher than the established correlation coefficient of each admixture. Each job sample was scanned three times and only one of the scans had to be greater than the correlation coefficient for the job sample to pass. Only job samples of concrete admixtures tested to validate the established correlations. Job samples of steel paints were not tested because of the high correlation values of steel paints which made the verification process very sensitive and less conclusive.

Research Procedure

Testing admixtures and paints are important to ensure that they have not been adversely modified or altered. The most effective and timely method of testing these chemical admixtures and steel paints is by using an Infrared Spectrophotometry or IR Scan (Najm et al., 2011). The general procedure of conducting an IR scan test for a liquid concrete admixtures is described in the following section.

ASTM C 494/C 494M-05a:

Preparation of samples for liquid admixtures.

Determine the percent solids of liquid concrete admixtures:

1. Place 25 to 30g of standard Ottawa sand in a glass weighing bottle
2. Place the weighing bottle and stopper removed in drying oven and dry for 17± 1⁄4 hrs at 105 ± 3°C

3. Insert the stopper in weighing bottle, transfer to desiccators, cool to room temperature and weigh to the nearest 0.001g= M2
4. Remove the stopper and, using a pipette evenly distribute 4ml of liquid admixture over the sand. Immediately insert the stopper to avoid loss by evaporation and weigh to the nearest 0.001g= M1
5. Remove the stopper and place both the bottle and stopper in a drying oven
6. Dry for **17± 1⁄4 hrs @ 105 ± 3°C**
7. Insert the stopper in the weighing bottle, transfer to a desiccators, cool to room temperature and weigh to the nearest 0.001g.=M4
8. mass of sample M3=M1- M2
9. mass of dried residue M5 = M4-M2
10. Residue by oven drying% = 100 *35 MM

Determine the infrared spectra of materials:

1. Prepare a diluted liquid admixture (distilled water + liquid admixture) to yield a dissolved solid concentration of about 0.015g/mL
2. Pipette 5mL of the above solution and add it to a Petri dish with 2.5g of KBr IR grade and 5mL of distilled water
3. Stir and mix to dissolve
4. Place in a drying oven and dry for **17± 1⁄4 hrs @ 105 ± 3°C**
5. Cool and transfer the dried residue to a mortar and grind to a fine powder.
6. Weigh 0.1g of the powder and 0.4g of KBr IR grade
7. Mix in an electric amalgamator for 30s using stainless steel capsule and balls.

Preparation of a disc for the IR analysis:

1. Weigh 0.3g of the mixture prepared (part II , No.7) and transfer into a suitable die.
2. Apply vacuum for 2 minutes prior to pressing if an evacuable die is used
3. Continue vacuum and press at a suitable force for 3 min, producing a disc about 1mm thick.
4. Remove the disc from the die, insert into the infrared spectrophotometer and obtain IR absorption spectra.

Since ASTM C 494/C 494M-05a is the standard procedure, this study must stay within the boundaries of this ASTM Standard. However, parts of this standard are slightly unclear and had to be interpreted, such as how to prepare a diluted admixture to yield a dissolved solid concentration of 0.015 g/mL. This can be achieved by using the

specific gravity or by using a volumetric calculation. The exact procedure used in this study is described in the following section.

Rutgers IR Scan Procedure for Concrete Admixtures

In order to find the percent solids of an admixture, 25-30g of Ottawa sand was placed in a glass weighing bottle and moved to an oven for 8 hrs at a temperature of 105˚C. Then the stopper was inserted and the bottle was weighed to the nearest 0.001g (M2). The stopper was removed and 4mL of the admixture was added with a pipette. The bottle was again weighed with the stopper to the nearest 0.001g (M1). Next the stopper was removed and placed in an oven for 17 hrs at a temperature of 105˚C. After the time has elapsed, the bottle was taken out and weighed without the stopper to the nearest 0.001g (M4). Then M3 (M3 = M1 – M2), M5 (M5 = M4 – M2), and the percent solids of the admixture (% = 100 * M5/M3) were calculated.

For air-entraining admixtures, the weight was measured for a disposable aluminum weighing dish (M2). Then 1mL of the AE admixture was placed onto the weighing dish and weighed (M1). Next the weighing dish was placed into an oven for 25mins at a temperature of 125˚C. After that, the weight of the dried admixture was measured (M4). Finally M3 (M3 = M1 – M2), M5 (M5 = M4 – M2), and the percent solids of the admixture (% = 100 * M5/M3) were calculated.

To prepare the admixture for testing, 4mL of the admixture was placed into a beaker and mixed with the appropriate amount of distilled water to dilute the solution to 0.015g/mL. This can be found by the following equation:

For non Air-Entraining admixtures:

$$\text{Amount of water (mL)} = (M5 / 0.015) - 4 \qquad \text{Eq (1a)}$$

For Air-Entraining admixtures:

$$\text{Amount of water (mL)} = (4*M5 / 0.015) - 4 \qquad \text{Eq (1b)}$$

Then 5mL of the diluted solution was placed into a petri dish with 2.5g of KBr and 5mL of distilled water. The solution was mixed until all the KBr had dissolved. Then the petri dish went into an oven for 17hrs at 105˚C.

To test the admixture, the sample must first be cooled in a desiccator. Then the dried sample was scraped off into a mortar and pestle and grinded to a fine powder. Next 0.1g of the powder and 0.4g of KBr were weighed out and put in a stainless steel capsule with a stainless steel ball. The capsule was placed into an electric amalgamator for 30 seconds. Next 0.3g of the shaken powder was taken and pressed with 10 tons of force for 3 minutes with a 1 minute release time. This will create the pellet needed for the infrared spectrometer. The final step is to place the admixture pellet into an infrared spectrometer for scanning.

These tests were performed using the Perkin-Elmer infrared spectrometer shown in Figure 2A. The individual pellets are placed carefully inside the spectrometer and the machine passes a beam of infrared light through the sample. Figure 2B shows several pellets after being tested in spectrometer.

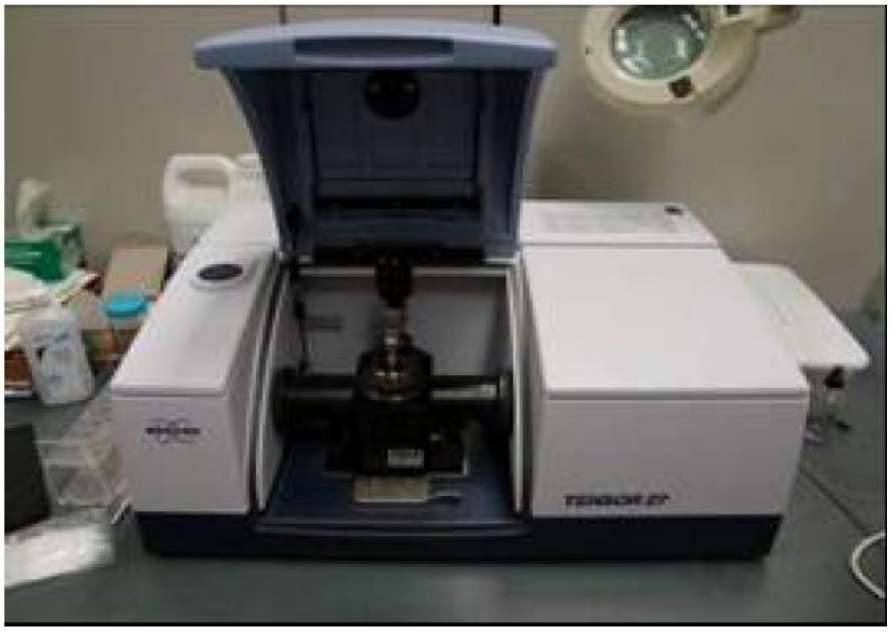

Figure 2A: The Perkin-Elmer IR spectrometer used in this study

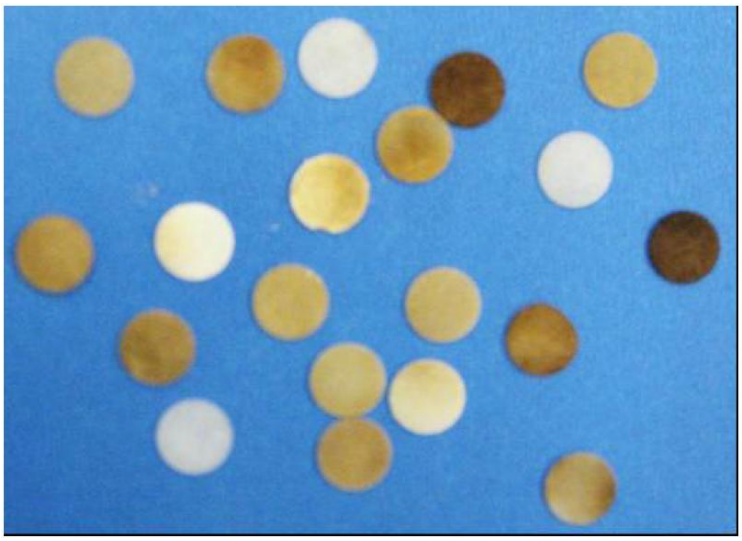

Figure 2B: Several sample pellets after being tested for IR scan.

The spectrometer analyzes the amount of transmitted through the pellet and records the energy absorbed at each wavelength. A computer connected to the spectrometer records the digital data and generates a graph representation of the sample's transmittance (or absorbance) spectrum versus its wavelength. A typical graph of the transmittance versus the wave length of admixture DARACEM 55 from various batches is shown in Figure 3.

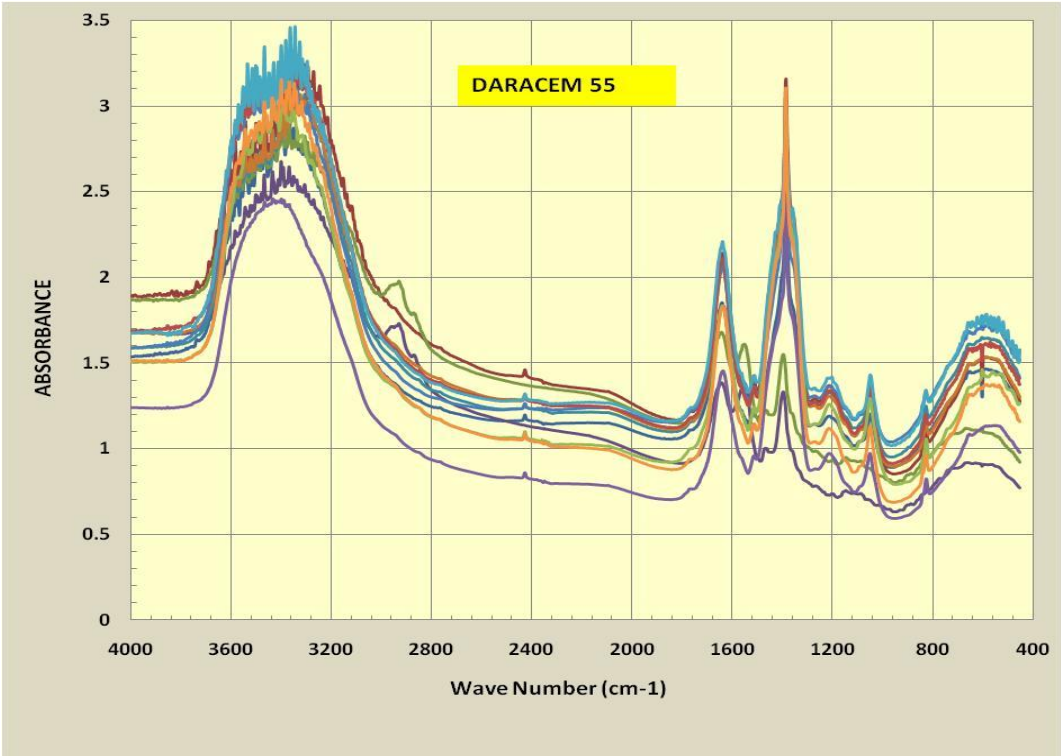

Figure 3: Typical absorbance versus wave length spectrum for DARACEM 55

These absorption characteristics provide information about the molecular structure of the sample and its principal components. Therefore, each scan is unique to that particular type of admixture.

The IR scan procedure for the structural steel paints was simpler than the concrete admixtures. First, the paint sample was placed into a mechanical mixer for approximately 3 minutes and briefly hand mixed to ensure uniformity. Then a background scan was run on the clean lens before a layer of paint was applied evenly over the entire lens using a pipette. It is essential that the entire lens is covered to prevent light from affecting the scan. Finally, the infrared scan was run to obtain the absorbance versus wave length data as shown in Figure 4.

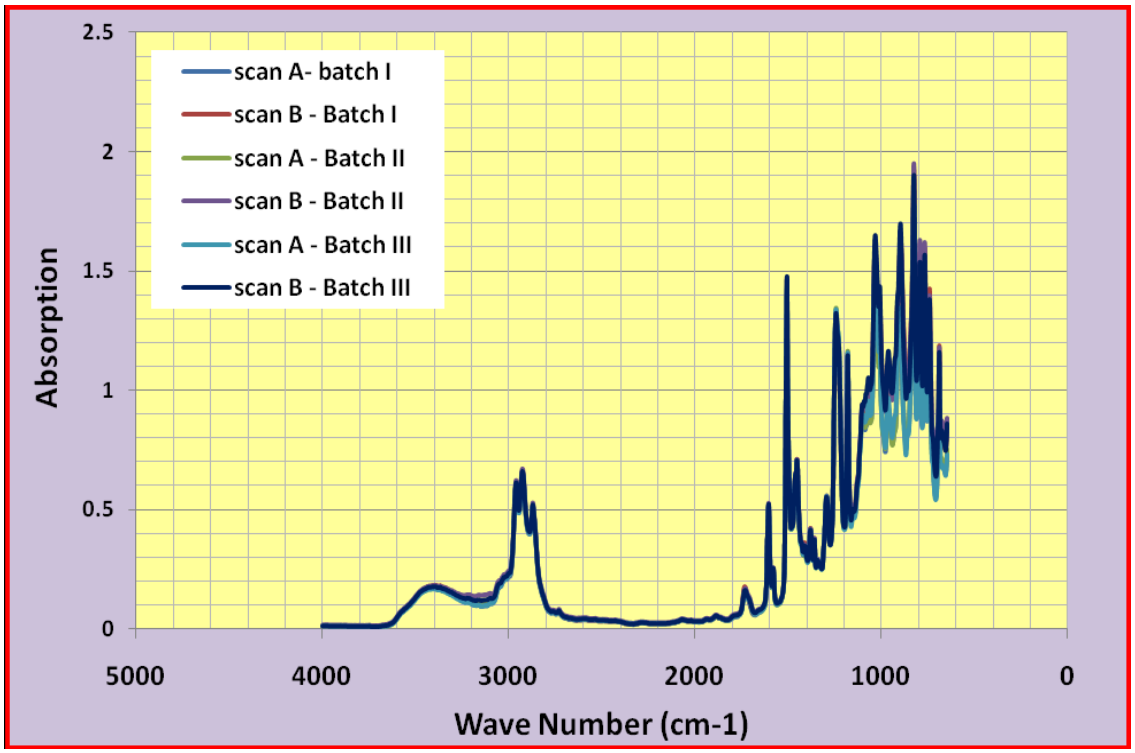

Figure 4: IR scans for steel paint OEU Interzinc 315B Part A from three batches

One can see in Figure 3 that all six scans are very consistent. The scans were taken from three different batches of paints and their consistency shows that the twelve scans were not necessary in order to achieve and establish a high-quality correlation for the paint samples.

Analysis and Results

One way to interpret the scan data obtained from the infrared spectrometer was to establish correlation coefficients between all of the batches in order to determine the acceptable tolerances for the structural steel paints and concrete admixtures. Therefore, if the samples stay within these limitations, one can have confidence that not only the admixture or paint has not been altered in any way, but also predict sufficient

performance when using these specimens. The general formula for determining the correlation coefficient of a typical admixture or paint is stated below.

$$r = correl\ (X, Y) = \frac{\sum(X-\bar{X})(Y-\bar{Y})}{\sqrt{\sum(X-\bar{X})^2(Y-\bar{Y})^2}} \qquad \text{Eq (2)}$$

Where,

X = absorbance values of scan A of admixture/paint

$\bar{x}$ = average of the absorbance values of scan A of admixture/paint

Y = average absorbance values of all scans from all three batches of admixture/paint

$\bar{Y}$ = average of the average absorbance of all scans from all three batches of admixture/paint

Correlation Coefficients for Concrete Admixtures

To determine the target correlation coefficient for each admixture, the Fisher's r-to- Z transformation technique was used. Fisher realized that this transformation makes the variability of correlations which are close to ±1.00 comparable to those of mid-range correlation values (Garcia, 2011). The Fisher r-to-Z transformation method is one of several procedures available to transform the correlation coefficients into additive quantities. In this method, a transformation parameter Z is calculated using the following equation:

$$Z = \frac{1}{2} ln\left(\frac{1+r}{1-r}\right) \qquad \text{Eq (3)}$$

The standard error in Z is given by Eq. (4)

$$SE_Z = \frac{1}{\sqrt{n-3}} \qquad \text{Eq (4)}$$

The arithmetic mean of the Z values is obtained using Eq. (5):

$$\overline{Z} = \left(\frac{1}{n}\right) \sum_{i=1}^{n} Z_i \qquad \text{Eq (5)}$$

The Fisher weighted mean correlation coefficient of the 12 scans from the three batches is determined using Eq. (6):

$$r_w = tanh\bar{Z} = \frac{e^{\bar{Z}} - e^{-\bar{Z}}}{e^{\bar{Z}} + e^{-\bar{Z}}} \qquad \text{Eq (6)}$$

The correlation coefficients obtained from Eq. (2) for the concrete admixtures from each batch are given in Table 5. These correlations coefficients associate the average absorbance values of all twelve scans to the individual absorbance values of each scan. The arithmetic mean correlations (r_{avg}) showed in the fourth column of Table 6 are the values of the concrete admixtures when all twelve scans are considered. These correlation coefficients are very close to 1.0 as expected. The weighted mean correlation coefficients (r_w) from Eq. (6) are shown in the first column of Table 6. Since these samples were delivered directly from the manufacturer and were stored in lab conditions until tested, it was expected that they will achieve high correlations. Using these developed correlation coefficients, one can establish target correlations and acceptance criteria for job samples. The high correlation also verifies the accuracy, consistency, and care taken in performing the IR scan tests. Using the weighted mean correlation is recommended especially for cases when the individual correlations are not high.

Table 5: Correlation coefficients of all admixtures from all batches

Admixture	Correlation coefficient between mean absorvanse and each scan.											
	Batch _ I				Batch _ II				Batch _ III			
	Scan_A	Scan_B	Scan_C	Scan_D	Scan_A	Scan_B	Scan_C	Scan_D	Scan_A	Scan_B	Scan_C	Scan_D
AEA-92*	0.98310	0.97932	0.99554	0.99559	0.96255	0.97173	0.95924	0.95797	0.99096	0.99271	0.96369	0.97775
AIR MIX *	0.99060	0.98967	0.98742	0.99218	0.96462	0.95547	0.99289	0.91205	0.99177	0.99501	0.92930	0.90000
Eucon WR-91	0.98818	0.99280	0.99847	0.99590	0.99319	0.99777	0.99755	0.99228	0.99775	0.99756	0.99411	0.99298
MB-VR standard *	0.94894	0.94431	0.96918	0.89841	0.99290	0.99458	0.99240	0.97899	0.99167	0.99813	0.97963	0.98184
MB-AE 90 *	0.97359	0.97651	0.91430	0.94170	0.99428	0.99292	0.99287	0.98980	0.99673	0.99245	0.99742	0.99051
Pozzolit 200N	0.98316	0.97605	0.93357	0.91774	0.99338	0.99168	0.98666	0.99288	0.99008	0.99635	0.97845	0.99444
Glenium 7500	0.97332	0.98750	0.96369	0.90907	0.99406	0.99066	0.99377	0.99019	0.99059	0.98724	0.99575	0.99372
Daracem 55	0.99349	0.98156	0.98242	0.99589	0.90675	0.91322	0.98451	0.98679	0.98776	0.99122	0.98662	0.99212
WRDA with HYCOL	0.99204	0.98877	0.99483	0.96995	0.95371	0.95501	0.97644	0.99592	0.97941	0.95937	0.99698	0.99222
ARAVAIR 1000(A00215)	0.97846	0.98664	0.99425	0.99316	0.95600	0.98123	0.97285	0.86774	0.99523	0.99701	0.99652	0.99531
Daracem 19	0.99162	0.99268	0.99587	0.96570	0.98036	0.95795	0.99826	0.99739	0.99723	0.95828	0.99327	0.98780
Secton 6A*	0.97236	0.94886	0.87012	0.92054	0.98781	0.98065	0.98788	0.98103	0.98920	0.97570	0.91925	0.91164
Chemstrong A	0.98165	0.97771	0.96748	0.92071	0.98240	0.98230	0.95930	0.93586	0.98134	0.97999	0.98366	0.98999
Chemstrong SP	0.98027	0.98107	0.98015	0.94295	0.98027	0.97784	0.98422	0.95306	0.99744	0.99547	0.95701	0.97587
Chemstrong R	0.97634	0.98966	0.99723	0.99782	0.99555	0.99313	0.90430	0.90700	0.98760	0.99040	0.99454	0.99124
Sika Air*	0.94308	0.94187	0.96079	0.96890	0.96458	0.96516	0.97995	0.98260	0.96262	0.86390	0.95744	0.94597
Plastolcrete 161	0.91355	0.93069	0.99413	0.98323	0.94565	0.91742	0.97270	0.94722	0.97886	0.96491	0.96286	0.94246
Plastolcrete 161 FL	0.93942	0.95225	0.99372	0.99431	0.97396	0.98917	0.98769	0.98434	0.98597	0.97983	0.98659	0.98692
Catexol AE 260*	0.99371	0.98910	0.98844	0.99276	0.93069	0.96467	0.98305	0.98845	0.99683	0.99250	0.98409	0.99226
Catexol 1000 SP MN	0.95934	0.98512	0.94906	0.93108	0.91099	0.88520	0.97735	0.95325	0.99484	0.98973	0.96238	0.97225
Allegro 122	0.97526	0.97162	0.98467	0.98160	0.90964	0.93359	0.98208	0.97186	0.98835	0.98298	0.96808	0.92662
Catexol 1000 R	0.99690	0.99765	0.99776	0.99840	0.98424	0.98961	0.99539	0.98151	0.99392	0.99628	0.99683	0.99782
Catexol 3000 GP	0.98987	0.97430	0.98852	0.93328	0.98634	0.97259	0.97329	0.94194	0.99351	0.99843	0.99494	0.99269

Table 6: Weighed mean correlations r_{bar}, R^2 and arithmetic mean correlations r_{avg}, R^2 of all admixtures.

Admixture	Weighted Mean Correaltion r bar	Weighted Coefficient of Determination R2	Percent of Data Remaining Unexplained (1-R2)	Arithmatic Mean Correlation r avg	Arithmatic Coefficient of Determination R2	Percent of Data Remaining Unexplained (1-R2)	% Diff between r bar and r avg
AEA92(A00158)*	**0.96621**	0.93357	**6.6%**	**0.97751**	0.95553	**4.4%**	**1.16%**
AIR MIX (A00159)*	**0.96206**	0.92556	**7.4%**	**0.96675**	0.93460	**6.5%**	**0.48%**
Eucon WR-91(A00166)	**0.99154**	0.98314	**1.7%**	**0.99488**	0.98978	**1.0%**	**0.34%**
MB-VR standard (A00180)*	**0.96821**	0.93742	**6.3%**	**0.97258**	0.94591	**5.4%**	**0.45%**
MB-AE 90 (A00181)*	**0.97720**	0.95492	**4.5%**	**0.97942**	0.95927	**4.1%**	**0.23%**
Pozzolit 200N(A00174)	**0.97267**	0.94608	**5.4%**	**0.97787**	0.95623	**4.4%**	**0.53%**
Glenium 7500(A00189)	**0.97570**	0.95200	**4.8%**	**0.98080**	0.96196	**3.8%**	**0.52%**
Daracem 55 (A00229)	**0.96989**	0.94069	**5.9%**	**0.97520**	0.95101	**4.9%**	**0.54%**
WRDA with HYCOL (A00210)	**0.97247**	0.94570	**5.4%**	**0.97955**	0.95953	**4.0%**	**0.72%**
DARAVAIR 1000(A00215)*	**0.97655**	0.95365	**4.6%**	**0.97620**	0.95297	**4.7%**	**-0.04%**
Daracem 19 (A00203)	**0.98202**	0.96437	**3.6%**	**0.98470**	0.96964	**3.0%**	**0.27%**
Secton 6A(A00226)*	**0.93671**	0.87743	**12.3%**	**0.95375**	0.90964	**9.0%**	**1.79%**
Chemstrong A (A00222)	**0.95173**	0.90579	**9.4%**	**0.97020**	0.94129	**5.9%**	**1.90%**
Chemstrong SP (A00223)	**0.96360**	0.92853	**7.1%**	**0.97547**	0.95154	**4.8%**	**1.22%**
Chemstrong R (A00221)	**0.97880**	0.95805	**4.2%**	**0.97707**	0.95466	**4.5%**	**-0.18%**
Sika Air(A00474)*	**0.92189**	0.84987	**15.0%**	**0.95307**	0.90835	**9.2%**	**3.27%**
Plastolcrete 161(A00144)	**0.92912**	0.86327	**13.7%**	**0.95447**	0.91102	**8.9%**	**2.66%**
Plastolcrete 161 FL(A00479)	**0.96853**	0.93805	**6.2%**	**0.97951**	0.95945	**4.1%**	**1.12%**
Catexol AE 260(A00398)*	**0.97648**	0.95352	**4.6%**	**0.98305**	0.96638	**3.4%**	**0.67%**
Catexol 1000 SP MN (A00400)	**0.93749**	0.87889	**12.1%**	**0.95588**	0.91371	**8.6%**	**1.92%**
Allegro 122 (A00397)	**0.94472**	0.89249	**10.8%**	**0.96470**	0.93064	**6.9%**	**2.07%**
Catexol 1000 R (A00402))	**0.99118**	0.98244	**1.8%**	**0.99386**	0.98776	**1.2%**	**0.27%**
Catexol 3000 GP(A00394)	**0.97297**	0.94668	**5.3%**	**0.97831**	0.95709	**4.3%**	**0.55%**

Table 6 also shows the difference between using the weighted mean correlation and the arithmetic mean correlation. There is not a big difference between the two methods, however the weighted mean correlation is suppose to be more accurate than the arithmetic mean correlation. This is because correlation coefficients are not linear distributed and cannot be averaged normally. However, the Fisher Transformation is the best known technique for transforming the coefficient r values into additive quantities (Garcia, 2010). Therefore the more scattered the correlation coefficient data is, the bigger the error the arithmetic mean method would generate. The same method was used to calculate the steel paint correlation coefficients.

Correlation Coefficients for Steel Paints

The structural steel paints used the same correlation coefficient equation as the concrete admixtures. However, only six scans were used to find the paint's correlation coefficients. This is because their scans were very close to each other that six scans were enough to create correlation coefficients. These correlation coefficients can be found in Table 7.

Since these are samples straight from the manufacturing paint, the goal was to achieve high correlations in order to set an acceptable standard of adequacy. The high correlation also verifies the precision and care taken in performing the IR scan experiments.

Table 7: Established weighted and mean correlation coefficients (r_{bar} and r_{avg}) for steel paints

PAINT	Correlation coefficient between mean absorbance and each scan									
	Batch _ I		Batch _ II		Batch _ III		Z bar	r bar	ravg	% Diff
	Scan_A	Scan_B	Scan_A	Scan_B	Scan_A	Scan_B	(4)	(5)		
OEU - Interzinc 315B Part A	0.99812	0.99805	0.99820	0.99865	0.99820	0.99861	3.4838	**0.9983**	**0.9983**	**0.002**
OEU - Interzinc 315B Part B	0.99973	0.99971	0.99963	0.99975	0.99954	0.99928	4.4489	**0.9996**	**0.9996**	**0.003**
OEU - Interguard 475 HS Off White Part A	0.99918	0.99814	0.99843	0.99822	0.99790	0.99671	3.9025	**0.9982**	**0.9981**	**0.015**
OEU - Interguard 475 HS Converter Part B	0.99920	0.99905	0.99969	0.99965	0.99856	0.99881	3.9102	**0.9993**	**0.9992**	**0.012**
OEU - Interguard 475 HS Buff Part A	0.99865	0.99844	0.99910	0.99878	0.99877	0.99881	3.6506	**0.9988**	**0.9988**	**0.002**
OEU - Interthane 870 UHS Black Part A	0.99937	0.99993	0.99853	0.99949	0.99798	0.99766	4.0297	**0.9993**	**0.9988**	**0.044**
OEU - Interthane 870 CONVERTER Part B	0.99986	0.99987	0.99995	0.99995	0.99996	0.99995	4.8015	**0.9999**	**0.9999**	**0.001**
PLY- Mastic 650 Part B	0.99975	0.99975	0.99979	0.99974	0.99974	0.99989	4.4962	**0.9998**	**0.9998**	**0.001**
PLY- TILE Part B	0.99952	0.99959	0.99969	0.99968	0.99964	0.99954	4.1667	**0.9996**	**0.9996**	**0.001**
PLY-Thane 890 (Green)	0.99982	0.99986	0.99986	0.99985	0.99963	0.99967	4.6523	**0.9998**	**0.9998**	**0.002**
PLY-Thane 890 (Blue/Gray)										
PLY-TILE Part A	0.99745	0.99852	0.99902	0.99879	0.99948	0.99934	3.3311	**0.9989**	**0.9988**	**0.016**
PLY-Mastic 650 Part A (White)										
PLY-Thane 890 Part B (HSG)	0.99744	0.99738	0.99913	0.99912	0.98767	0.98771	3.3289	**0.9970**	**0.9947**	**0.223**
PLY-Thane 890 Part A (HSG)	0.99985	0.99987	0.99991	0.99986	0.99997	0.99997	4.7502	**0.9999**	**0.9999**	**0.002**
Amercoat 68 HS Cure	0.98892	0.98896	0.96914	0.96752	0.99863	0.99886	2.5953	**0.9924**	**0.9853**	**0.710**
Amercoat 68 Resin	0.97687	0.97674	0.99065	0.99070	0.99005	0.99015	2.2240	**0.9871**	**0.9859**	**0.126**
Amercoat 450H Cure	0.99924	0.99935	0.99652	0.99635	0.99637	0.99567	3.9394	**0.9979**	**0.9973**	**0.060**
Amercoat 399 Pearl Grey Resin	0.99699	0.99941	0.99668	0.99900	0.99905	0.99841	3.2484	**0.9986**	**0.9983**	**0.030**
Amercoat 399 Pearl Grey Cure	0.99907	0.99945	0.99979	0.99945	0.99917	0.99950	3.8351	**0.9995**	**0.9994**	**0.006**
Amercoat 450H Green Resin	0.99809	0.99841	0.99860	0.99870	0.99989	0.99990	3.4770	**0.9994**	**0.9989**	**0.045**
Amercoat 450H Blue	0.99813	0.99767	0.99914	0.99924	0.99875	0.99943	3.4878	**0.9989**	**0.9987**	**0.015**
Carboguard 893 White 0800 Pt. A	0.99886	0.99815	0.99859	0.99854	0.99738	0.99744	3.7363	**0.9982**	**0.9982**	**0.009**
Carboguard 893 White 0800 Pt. B	0.99943	0.99945	0.99717	0.99631	0.99783	0.99858	4.0852	**0.9985**	**0.9981**	**0.040**
Carbozinc 11HS Base Green 0300	0.99925	0.99885	0.99705	0.99902	0.99847	0.99883	3.9472	**0.9987**	**0.9986**	**0.014**
Carbozinc 11HS Activator	0.99235	0.99330	0.97077	0.96709	0.99152	0.99296	2.7813	**0.9880**	**0.9847**	**0.338**
Carbothane 133 LH Brown 2285	0.99984	0.99967	0.99989	0.99994	0.99973	0.99971	4.7088	**0.9998**	**0.9998**	**0.003**
Urethane Converter 8800 0909	0.99930	0.99443	0.99915	0.99980	0.99867	0.99993	3.9801	**0.9994**	**0.9985**	**0.083**

Acceptance Criteria

As mentioned in the introduction, state DOT's are using different methods for acceptance of IR scan test results of concrete admixtures from jobsites. Few DOT's have a quantitative assessment procedure in place for infrared analysis. The NJDOT is currently using a target correlation coefficient of 0.975 for acceptance criteria for all admixtures (NJDOT, 2010). This value was recommended by the manufacturer of the IR

spectroscopy system; however, the basis of this target value was not established (Najm et al., 2011). The coefficient of determination (r^2 or R^2) of the correlation coefficient provided by the manufacturer is 0.9506. This means about 95% of the total variation in absorbance can be explained by the linear relationship. Accepting a correlation coefficient of 0.975 thus means accepting that the other 5% of the total variation remains unexplained or determined by other variables or by chance. These unexplained data can be also looked at as an "error" in R^2. Examining the correlation data for r and R^2 in Table 6 indicates that the coefficient of determination R^2 will vary from 0.98314 to 0.84987 using the weighted mean correlations. The average error in R^2_w for all admixtures is 6.9% or a value of 0.931 for R^2_w and a correlation value $r_w = 0.965$. Thus using the weighted mean correlations, the target value of all admixtures tested in this study, r_w will be 0.965 resulting in an error of about 6.9% on the average. To be more specific, we can establish a target correlation for individual admixtures using the data in Table 6. This will prove to be more accurate since each scan holds unique information of a specific spectra as stated previously.

Job Samples

The established correlation coefficients for all admixtures evaluated in this study were tabulated in Table 6 earlier for the weighted mean correlation r_w and the coefficient of determination R^2_w. The established weighted mean correlations will be used to quantitatively assess job samples from road and bridge construction sites. Five job samples of admixtures were tested against the established target correlations to observe the applicability and the reliability of these correlations in providing quantitative quality assurance and quality control.

The five job samples were designated as follows:

1. ADMX 1
2. ADMX 2
3. ADMX 3
4. ADMX 4
5. ADMX 5

These admixtures were actual job samples supplied by the NJDOT from several of their construction projects. Three IR scans from each job sample were prepared and compared to the target correlation coefficient for each admixture. Comparison of the individual correlations of the three job samples (total 15 scans) to the target weighted means are shown in Table 8.The criteria for a job sample to pass inspection is for the job sample to have the same correlation coefficient or higher when compared to the established or target correlations from the data base collected. The job sample will be approved if one out of three scans passes; otherwise it will be rejected (fail). Table 8 shows that when using the target weighted mean correlation for comparison with the individual scans, 10 out of 15 scans passed (or 4 out of the 5 job samples).

Table 8: Quantitative assessment of job samples using mean correlations

ADMIX	Weighted Mean Correlation	JOB SAMPLE CORRELATIONS					
		Scan A	P/F	Scan B	P/F	Scan C	P/F
ADMX 1	**0.96206**	0.92503	FAIL	0.92505	FAIL	0.93099	FAIL
ADMX 2	**0.99154**	0.99336	PASS	0.92132	FAIL	0.93668	FAIL
ADMX 3	**0.97720**	0.99673	PASS	0.99370	PASS	0.99639	PASS
ADMX 4	**0.97267**	0.98759	PASS	0.99612	PASS	0.99498	PASS
ADMX 5	**0.93671**	0.98497	PASS	0.95242	PASS	0.96969	PASS

Comparison of the individual correlations of the five job samples (15 scans) to the target correlation value of 0.965 computed earlier is shown in Table 9. The comparison in Table 9 shows 9 out of 15 scans pass (still 4 out of the 5 job samples) when the target weighted

mean correlation coefficient equals 0.965. Finally, comparing the weighted mean correlations of the three scans of each the 5 job samples to the individual weight mean correlations in Table 10 shows that 3 out of 5 samples pass.

Table 9: Quantitative assessment of individual job samples using target correlations

ADMIX	Target Weighted Mean	JOB SAMPLE CORRELATIONS					
		Scan A	P/F	Scan B	P/F	Scan C	P/F
ADMX 1	0.965	0.92503	FAIL	0.92505	FAIL	0.93099	FAIL
ADMX 2	0.965	0.99336	PASS	0.92132	FAIL	0.93668	FAIL
ADMX 3	0.965	0.99673	PASS	0.99370	PASS	0.99639	PASS
ADMX 4	0.965	0.98759	PASS	0.99612	PASS	0.99498	PASS
ADMX 5	0.965	0.98497	PASS	0.95242	FAIL	0.96969	PASS

Table 10: Quantitative assessment of job samples correlations compared to target correlations

ADMIX	Weighted Mean Correlation	Job Sample Weighted Mean Correlation	PASS/FAIL
ADMIX 1	0.96206	0.92708	FAIL
ADMIX 2	0.99154	0.96761	FAIL
ADMIX 3	0.97720	0.99580	PASS
ADMIX 4	0.97267	0.99377	PASS
ADMIX 5	0.93671	0.97210	PASS

Quantitative assessments using the weighted mean correlations of the job samples and a target mean weighted correlation of 0.965 seems to be an acceptable criteria for most admixtures, however, the average level of error of was 7% based on 276 performed IR scans for a total of 23 admixtures. As shown in Table 6, error levels vary for different admixtures and for certain admixtures lower correlation values may be used based on observations from job sample tests. The results in Table 6 can be used to select different

target correlation values for these admixtures. More testing of job samples is needed to verify the consistency of the test results and to have more confidence in using individual target correlations instead of using an overall target correlation coefficient equal to 0.965. Also further testing from additional manufacturer samples and batches is needed for further investigation and justification of the target correlations and for continuous improvement of the adopted correlations.

Factors Influencing IR Scan Testing

The procedure for testing concrete admixtures and steel paints with infrared spectrophotometry scanning is very precise and well established, however, at times it can vary in results if care is not taken in performing these tests and as reported by NJDOT. Therefore, it is important to identify and evaluate the factors that affect these IR scans and propose ways how to minimize their influence. Two factors this study investigated were the effects of drying time compared to moisture content and the effects of potassium bromide (KBr).

Effect of Drying Time on Correlation Coefficients

The presence of water significantly alters the IR scans which can skew data. Therefore to observe how much water could affect correlation coefficients, the drying times of several admixtures were investigated. The ASTM Standards require 17 hour drying time for the admixtures. Based on the results obtained, it was noticed that the air-entraining admixtures, because of their properties, still retained some moisture after 17 hours of drying. To evaluate the effect of extended drying time for air-entraining admixtures on its correlation, eight commonly used air–entraining admixtures were

scanned using a 24 hour drying time compared to 17 hours. These seven admixtures are shown in Tables 11 and 12 for 17 hr and 24 hr drying time respectively. These tables also show the weighted mean correlation coefficients from all batches for the 17 and 24 hour drying periods respectively.

Table 11: Correlation coefficients for the 17 hour drying time

Admixture	17 Hour Study						CORRELATION COEFFICIENT
	Batch _ I		Batch _ II		Batch _ III		
	Scan_A	Scan_B	Scan_A	Scan_B	Scan_A	Scan_B	
AEA92	0.99265	0.99077	0.97934	0.98729	0.98608	0.99439	**0.98943**
AIR MIX	0.99550	0.98899	0.98126	0.98141	0.99043	0.98979	**0.98908**
MB-VR standard	0.97839	0.97607	0.99649	0.99533	0.97221	0.98642	**0.98784**
MB-AE 90	0.98590	0.98845	0.99634	0.99339	0.99132	0.98416	**0.99097**
DARAVAIR 1000	0.98266	0.98915	0.96051	0.98435	0.99314	0.99523	**0.98748**
Secton 6A	0.99535	0.98284	0.98984	0.98771	0.99027	0.99456	**0.99101**
Catexol AE 260	0.99559	0.98616	0.94839	0.97677	0.99430	0.98778	**0.98684**

Table 12: Correlation coefficients for the 24 hour drying time

Admixture	24 Hour Study						CORRELATION COEFFICIENT
	Batch _ I		Batch _ II		Batch _ III		
	Scan_A	Scan_B	Scan_A	Scan_B	Scan_A	Scan_B	
AEA92	0.99419	0.99418	0.97812	0.98037	0.93761	0.94468	**0.98071**
AIR MIX	0.97652	0.9879	0.9796	0.99189	0.99425	0.99763	**0.99071**
MB-VR standard	0.9941	0.95964	0.99612	0.99741	0.96886	0.99864	**0.99315**
MB-AE 90	0.99758	0.99624	0.99865	0.99313	0.99137	0.99704	**0.99641**
DARAVAIR 1000	0.99866	0.98221	0.99594	0.99833	0.99025	0.99874	**0.99645**
Secton 6A	0.99693	0.99891	0.99742	0.99553	0.9955	0.99897	**0.99763**
Catexol AE 260	0.99596	0.99669	0.99558	0.99372	0.98982	0.99209	**0.99443**

Table 13: Summery of correlation values between the 17 and 24 hour drying periods

Admixture	Correlation Coefficient (17 hrs)	Correlation Coefficent (24 hrs)
AEA92	0.98943	0.98071
AIR MIX	0.98908	0.99071
MB-VR Standard	0.98784	0.99315
MB-AE 90	0.99097	0.99641
DARAVAIR 1000	0.98748	0.99645
Secton 6A	0.99101	0.99763
Catexol AE 260	0.98684	0.99443

Table 13 compares the weighted mean correlation coefficients for the 17 hour and the 24 hour drying times. Most of these admixtures benefited from the additional drying time and were noticeably drier in appearance. However, because of the longer drying period, most of the samples were burned to some extent. In order to not skew the data, the unburned parts of the admixture were used to create the test sample. Although the selection of scan material from unburnt areas if the sample may cause bias and is not recommended, it was done here because we needed to evaluate the effect of additional drying time which caused most of the samples to be burnt. However, the results in Table 13 show that the burning of the samples did not affect the scans because high correlations were still obtainable. Even though most of the admixtures correlation coefficients increased, more research is needed to evaluate the effect of drying times for air-entraining admixtures.

Effect of Potassium Bromide on Correlation Coefficients

The potassium bromide (KBr) has an important effect in the scanning results due to its volume in the sample and its purity. The effect of potassium bromide was a factor in

this study observed originally because the NJDOT and the Rutgers research team were using slightly different types of KBr from two different suppliers due to availability and delivery times. Furthermore, some of the KBr used by NJDOT was dried in an oven prior to its use. One KBr type was delivered in a crystallized form and the other was in powder form. Therefore, in order to evaluate the effects of the KBr on the correlation coefficients, KBr from three different suppliers were tested with five different concrete admixtures. Tables 14 show the KBr types and suppliers while Table 15 shoes the five concrete admixtures used in the KBr investigation.

Table 14: KBr products used in KBr investigation

Product Name	Supplier	Size
KBr 1	Spectrum Chemicals and Lab Products	125 g
KBr 2	Acros Organics (Fischer Scientific)	100 g
KBr 3	VWR Inc. (EMPX1378-1)	25 g

Table 15: Concrete admixtures used in KBr investigation

Admixtures	Supplier	Type
AIR MIX (A00159)	Euclid Chemicals co.	AIR
DARAVAIR 1000 (A00215)	WR Grace	AIR
Daraset 400 (A00208)	WR Grace	C
Eucon 1037(A00162)	Euclid Chemicals co.	CI
Pozzolith 100-XR(A00183)	BASF Admixtures inc	B , D

The KBr investigation followed the same procedure as the original IR Scan for concrete admixtures discussed in previous sections. However, only two scans were taken

from each batch making a total of six scans to create a correlation coefficient. Figure 5 shows IR scans of the chemical admixture AIR MIX with each of the different sources of KBr for one batch. This figure shows that the effect of the potassium bromide on the IR Scan results. This trend was also observed with the other four concrete admixtures. The effects of the KBr source on the admixture's correlation coefficients can be seen in Tables 16. Table 16 shows that using one type of KBr, resulted in high correlation coefficients as expected. However, when mixing the KBr from different suppliers, the correlation coefficients tend to decrease, also shown in Table 16. For example, admixture EUCON WR-91 has a weighted mean correlation from the six scan using the same KBr (KBr-1) equal to 0.98350. When determining the weighted mean correlation using the 18 scans from all KBr, the weighted mean decreases to 0.95018.

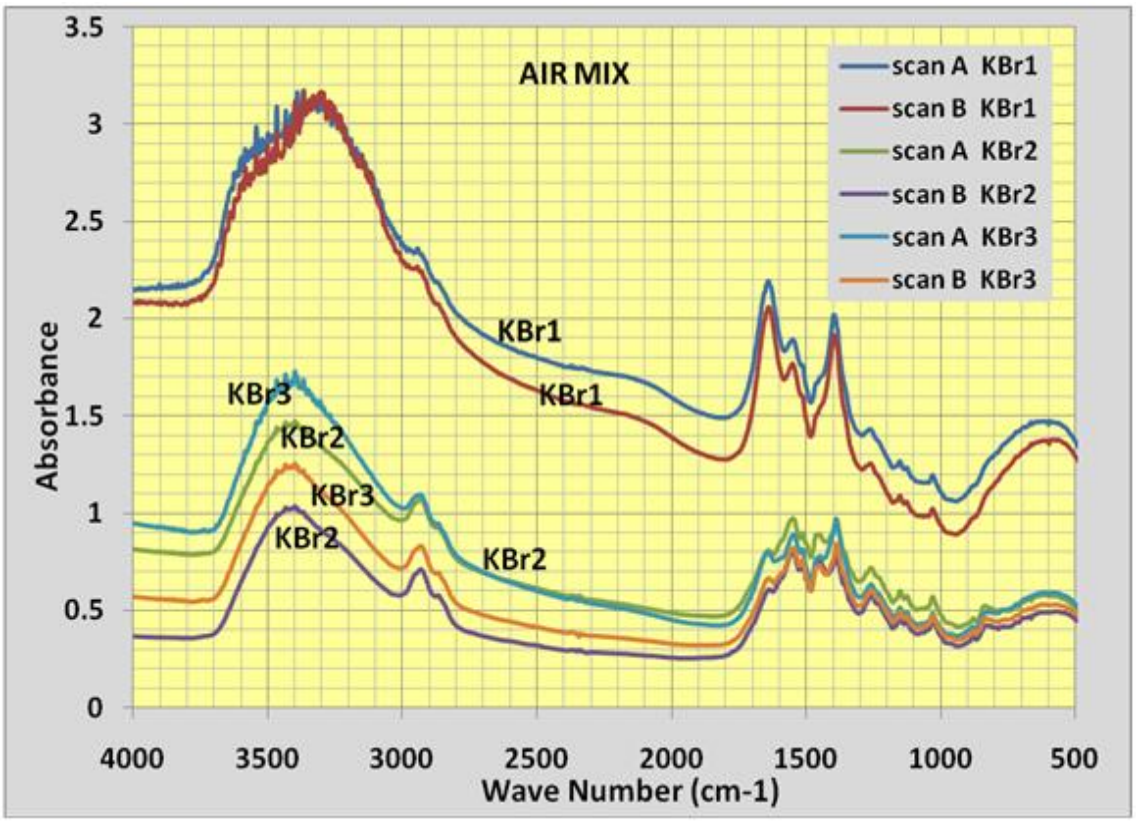

Figure 5: AIR MIX IR scan of one batch with three different KBr's

The same trend was observed for all five samples. These results show that using different KBr's from different sources to obtain correlation coefficients may lead to lower correlations and poor results. Moreover, testing job samples with a KBr from different sources will result in poor comparisons. Therefore, any KBr that follows the ASTM specifications for IR scan tests can be used to perform these tests, set up a library, and test job samples. However, once a potassium bromide is chosen, the tests then must be performed using the same type of KBr and same manufacturer to establish correct correlations and to accurately evaluate job samples. All of the work done in this study was performed with KBr_1, supplied by the Spectrum Chemicals and Lab Products.

Table 16. Correlation coefficients for each individual source of KBr

Admixture		Correlation coefficient between mean absorvanse and						Arithmatic Mean Correlation	Weighted Mean Correaltion
		Batch _ I		Batch _ II		Batch _ III			
		Scan_A	Scan_B	Scan_A	Scan_B	Scan_A	Scan_B		
AIR MIX	KBr_1	0.99602	0.98777	0.98108	0.99597	0.99599	0.99804	**0.99248**	**0.99445**
	KBr_2	0.92824	0.93368	0.97729	0.94103	0.88601	0.94646	**0.93545**	**0.94160**
	KBr_3	0.80071	0.81516	0.99156	0.97917	0.91420	0.98971	**0.91509**	**0.95676**
Mean Correlations from all three KBr's together =								***0.94767***	***0.91206***
Eucon WR-91	KBr_1	0.93538	0.95819	0.99183	0.99192	0.99169	0.98669	**0.97595**	**0.98350**
	KBr_2	0.99847	0.99770	0.99810	0.99517	0.99841	0.98811	**0.99599**	**0.99709**
	KBr_3	0.99416	0.99463	0.99846	0.99900	0.99354	0.99332	**0.99552**	**0.99643**
Mean Correlations from all three KBr's together =								***0.98915***	***0.95018***
Daracem 55	KBr_1	0.99757	0.98952	0.99546	0.98027	0.99328	0.99751	**0.99227**	**0.99419**
	KBr_2	0.99166	0.99550	0.98988	0.98819	0.98658	0.98115	**0.98883**	**0.98978**
	KBr_3	0.95826	0.88033	0.98637	0.99847	0.99583	0.96465	**0.96398**	**0.98406**
Mean Correlations from all three KBr's together =								***0.98169***	***0.98335***
DARAVAIR 1000	KBr_1	0.99952	0.99917	0.99921	0.99734	0.99604	0.99901	**0.99838**	**0.99878**
	KBr_2	0.97605	0.99162	0.99725	0.98428	0.97471	0.99794	**0.98697**	**0.99122**
	KBr_3	0.97548	0.89556	0.97338	0.96541	0.99832	0.98861	**0.96613**	**0.98100**
Mean Correlations from all three KBr's together =								***0.98383***	***0.97541***
Catexol 1000 R	KBr_1	0.99938	0.98749	0.99702	0.98701	0.99817	0.99891	**0.99466**	**0.99709**
	KBr_2	0.99723	0.92215	0.89925	0.90578	0.96812	0.97940	**0.94532**	**0.96640**
	KBr_3	0.94225	0.92150	0.99382	0.95925	0.97626	0.96608	**0.95986**	**0.96868**
Mean Correlations from all three KBr's together =								***0.96661***	***0.97189***

Conclusions and Recommendations

Correlation coefficients for concrete admixtures and steel paints were obtained using three different batches from the manufacturer supplied at three reasonable separated time intervals. The reliability of this quantitative approach to test job samples was also analyzed and evaluated by testing several randomly selected job samples using target correlations for acceptance criteria. The quantitative approach seems to be a more reliable method for determining whether or not concrete admixtures and structural steel paints are acceptable and will perform the tasks required of the material. The quantitative approach can be used to support assessments made by the qualitative method. The limitation of this method is bound by the library created from the admixtures and paints obtained. This library of scans and correlation values need to be updated whenever the concrete admixture or the steel paint is altered by the manufacturer. The library can be improved by testing more samples from the manufacturer. The level of accuracy of this approach is also dependant on the acceptance criteria and the target error level for admixtures. More testing of job samples are needed to verify selected target correlation. Also further testing of manufacturer samples and batches may be needed for further investigation and justification of the target correlations. IR scans for steel paints were easier to perform and the correlation values had less variations compared to concrete admixtures. Future research is needed for identifying unknown samples using modeling programs with the data collected. Another method to test and identify samples would be to conduct a principal component analysis. This can be a more reliable and mathematically rigorous way of comparing the relevant parts of the spectra.

For the influencing factors, it was found that the drying time can be increased to 24 hour for air-entraining admixtures, but more testing should be done in order to confirm this drying time will not result in burning samples and create bias in IR scans. The potassium bromide had a significant effect on the correlation coefficients and must be consistent throughout the entire library and job sample testing process. Additional research is needed to find what other factors, in addition to the KBr, that may influence the IR analysis such as sample mixing time, press time, volume of KBr, and sample volume.

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Appendix A

How to check job samples

First, follow the Rutgers IR Scan Procedure for Concrete Admixtures and one for Steel Paints explained earlier in this report. Once the pellet has been scanned, follow the following steps:

1. In the spectrometer computer, change the graph so that it records absorbance versus wave number.
2. Save the scan as an ascii file
3. Open the .ascii file in notepad and save only the absorbance numbers into the Rutgers excel spreadsheet under the **Job Scans** tab in the appropriate column.
4. In the excel spreadsheet, check the **Correlation ceoff._all batch** tab to see if the job sample has passed.
5. Repeat these steps for each of the three scans of the job sample.
6. Only one out of the three scans needs to pass in order for the admixture to be accepted.

How to add/update admixtures library

In order to add/update an admixture to the library, the following steps must be followed:

1. Contact manufacturer/supplier to obtain three separate batches over a period of 2-3 months between batches
2. Perform four IR scans from each batch of the admixture following the Rutgers IR scan Procedure for Concrete Admixtures.
3. All scans must be saved as absorbance versus wave number in .ascii file format.
4. Open the ascii file in notepad and save only the absorbance values into the Rutgers excel spreadsheet in the appropriate tab and the correct column
5. When adding an admixture to the library, one will have to add extra four columns to the first, second, and third batch tabs for the new admixture.
6. Also, another column must be added into the **average_ALL BATCH** tab so that all the numbers from the first, second, and third batches are averaged in the column. N
7. Then add one more row to the **Correlation ceoff._all batch** tab and make sure all of the cells in the row are programmed correctly.

Appendix B

This section contains a sample of raw data taken from the IR scan to complete this study. The following is data collected from the concrete admixture Master Builder MB-AE 90.

MASTER BUILDER	
Wave Number	MB-AE 90 (A00181)
	A00181_RU_A_2
	06/04/10
4000	1.884107
3999	1.884645
3998	1.885196
3997	1.885487
3996	1.885466
3995	1.885342
3994	1.885273
3993	1.885045
3992	1.884219
3991	1.882686
3990	1.881043
3989	1.880278
3988	1.88098
3987	1.882871
3986	1.885028
3985	1.886471
3984	1.886643
3983	1.885641
3982	1.884153
3981	1.883149
3980	1.883392
3979	1.884945
3978	1.886963
3977	1.88806
3976	1.887216
3975	1.884588
3974	1.881377
3973	1.878798
3972	1.877261
3971	1.876461
3970	1.876023
3969	1.87589
3968	1.876194
3967	1.877007
3966	1.878314
3965	1.880081
3964	1.88221
3963	1.884389
3962	1.886109
3961	1.88691
3960	1.886623
3959	1.885368
3958	1.883458
3957	1.881354
3956	1.879608
3955	1.878665
3954	1.878569
3953	1.878856
3952	1.87893
3951	1.878621
3950	1.878358
3949	1.878683
3948	1.879586
3947	1.880477
3946	1.8808
3945	1.880448
3944	1.879625
3943	1.878749
3942	1.878355
3941	1.87864
3940	1.879259
3939	1.879843
3938	1.880416
3937	1.881349
3936	1.883047
3935	1.885478
3934	1.88778
3933	1.8885
3932	1.886828
3931	1.883546
3930	1.880263
3929	1.877974
3928	1.876796
3927	1.876451
3926	1.876581
3925	1.877122
3924	1.878504
3923	1.881001
3922	1.88414
3921	1.886932
3920	1.888428
3919	1.888152
3918	1.886399
3917	1.884012
3916	1.881548
3915	1.879006
3914	1.876426
3913	1.874253
3912	1.873073
3911	1.873241
3910	1.874738
3909	1.877314
3908	1.880567
3907	1.883979
3906	1.886962
3905	1.88928

3904	1.891597
3903	1.894654
3902	1.897892
3901	1.900098
3900	1.900613
3899	1.899243
3898	1.895757
3897	1.890337
3896	1.884475
3895	1.880425
3894	1.879455
3893	1.88084
3892	1.8828
3891	1.884429
3890	1.88595
3889	1.888303
3888	1.892999
3887	1.899967
3886	1.905921
3885	1.906905
3884	1.902502
3883	1.895794
3882	1.890171
3881	1.887037
3880	1.88578
3879	1.884694
3878	1.882331
3877	1.879224
3876	1.877078
3875	1.876497
3874	1.876835
3873	1.877448
3872	1.877855
3871	1.877686
3870	1.877501
3869	1.878323
3868	1.879868
3867	1.881293
3866	1.882372
3865	1.882746
3864	1.882238
3863	1.881453
3862	1.880683

3861	1.879368
3860	1.877332
3859	1.875499
3858	1.875199
3857	1.876772
3856	1.878573
3855	1.877788
3854	1.875262
3853	1.87772
3852	1.88482
3851	1.887485
3850	1.884361
3849	1.880644
3848	1.880012
3847	1.882847
3846	1.887165
3845	1.890008
3844	1.889374
3843	1.885828
3842	1.881904
3841	1.879637
3840	1.879485
3839	1.881939
3838	1.887997
3837	1.896428
3836	1.902931
3835	1.904058
3834	1.900069
3833	1.893615
3832	1.887306
3831	1.882322
3830	1.878435
3829	1.875245
3828	1.873046
3827	1.872386
3826	1.873553
3825	1.876546
3824	1.88099
3823	1.885557
3822	1.888018
3821	1.887779
3820	1.886738
3819	1.885495

3818	1.883771
3817	1.882335
3816	1.881894
3815	1.882074
3814	1.881505
3813	1.879593
3812	1.877651
3811	1.877339
3810	1.879089
3809	1.881859
3808	1.883834
3807	1.883994
3806	1.882797
3805	1.880816
3804	1.878078
3803	1.873969
3802	1.868533
3801	1.864947
3800	1.865847
3799	1.869429
3798	1.873274
3797	1.876274
3796	1.877838
3795	1.878176
3794	1.878173
3793	1.878798
3792	1.880602
3791	1.883124
3790	1.885124
3789	1.885652
3788	1.884632
3787	1.882607
3786	1.880309
3785	1.878594
3784	1.878448
3783	1.880698
3782	1.885614
3781	1.892308
3780	1.898254
3779	1.900506
3778	1.898164
3777	1.892898
3776	1.887291

3775	1.883399
3774	1.882181
3773	1.883596
3772	1.886853
3771	1.890573
3770	1.893448
3769	1.894924
3768	1.895031
3767	1.894208
3766	1.892991
3765	1.891527
3764	1.889868
3763	1.888605
3762	1.888678
3761	1.890175
3760	1.891859
3759	1.892574
3758	1.892405
3757	1.89195
3756	1.891649
3755	1.89171
3754	1.891445
3753	1.888879
3752	1.882859
3751	1.877107
3750	1.877341
3749	1.881816
3748	1.884946
3747	1.885896
3746	1.887202
3745	1.891956
3744	1.901933
3743	1.913048
3742	1.919892
3741	1.924025
3740	1.929448
3739	1.936805
3738	1.943759
3737	1.94712
3736	1.945313
3735	1.940635
3734	1.936731
3733	1.93372

3732	1.929523
3731	1.92339
3730	1.916568
3729	1.911085
3728	1.907901
3727	1.906395
3726	1.905695
3725	1.905745
3724	1.906786
3723	1.908611
3722	1.91084
3721	1.913211
3720	1.91572
3719	1.918548
3718	1.921527
3717	1.924218
3716	1.926415
3715	1.928063
3714	1.92858
3713	1.926844
3712	1.923458
3711	1.922326
3710	1.926056
3709	1.93191
3708	1.935858
3707	1.936736
3706	1.935896
3705	1.935278
3704	1.935983
3703	1.938078
3702	1.941488
3701	1.946326
3700	1.952091
3699	1.95788
3698	1.963337
3697	1.968295
3696	1.972275
3695	1.975025
3694	1.977364
3693	1.981285
3692	1.989179
3691	2.002585
3690	2.020375

3689	2.036629
3688	2.043303
3687	2.038278
3686	2.026697
3685	2.01508
3684	2.00688
3683	2.002372
3682	2.000509
3681	2.00013
3680	2.000233
3679	2.000218
3678	2.000584
3677	2.003686
3676	2.013864
3675	2.031648
3674	2.04695
3673	2.049956
3672	2.04242
3671	2.033038
3670	2.031257
3669	2.041147
3668	2.056182
3667	2.067687
3666	2.074327
3665	2.079501
3664	2.086627
3663	2.097869
3662	2.113731
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995	0.687395
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942	0.663712
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938	0.664037

937	0.664222	**894**	0.677976	**851**	0.715571
936	0.664382	**893**	0.678458	**850**	0.716957
935	0.664511	**892**	0.678994	**849**	0.718504
934	0.664643	**891**	0.679611	**848**	0.720095
933	0.664729	**890**	0.680401	**847**	0.721704
932	0.664718	**889**	0.681313	**846**	0.723366
931	0.664682	**888**	0.682193	**845**	0.724995
930	0.664693	**887**	0.682995	**844**	0.726441
929	0.664676	**886**	0.683758	**843**	0.727712
928	0.66454	**885**	0.684443	**842**	0.728941
927	0.66438	**884**	0.684956	**841**	0.730148
926	0.664358	**883**	0.685328	**840**	0.731225
925	0.664473	**882**	0.685669	**839**	0.73216
924	0.664613	**881**	0.685999	**838**	0.73307
923	0.664766	**880**	0.686295	**837**	0.73402
922	0.664993	**879**	0.686641	**836**	0.734969
921	0.665267	**878**	0.68715	**835**	0.735918
920	0.665525	**877**	0.687778	**834**	0.736936
919	0.665801	**876**	0.688389	**833**	0.738039
918	0.666149	**875**	0.688968	**832**	0.739185
917	0.666509	**874**	0.68959	**831**	0.740404
916	0.666822	**873**	0.690244	**830**	0.741784
915	0.667196	**872**	0.690885	**829**	0.743324
914	0.667784	**871**	0.691602	**828**	0.744947
913	0.668553	**870**	0.692542	**827**	0.746642
912	0.669371	**869**	0.693709	**826**	0.748429
911	0.670205	**868**	0.695002	**825**	0.750212
910	0.671076	**867**	0.696385	**824**	0.751843
909	0.671897	**866**	0.69785	**823**	0.753306
908	0.672552	**865**	0.699301	**822**	0.754671
907	0.673077	**864**	0.700656	**821**	0.755895
906	0.673578	**863**	0.701987	**820**	0.756871
905	0.674044	**862**	0.703393	**819**	0.757631
904	0.674426	**861**	0.704798	**818**	0.758327
903	0.6748	**860**	0.706064	**817**	0.759035
902	0.675275	**859**	0.707226	**816**	0.759754
901	0.675796	**858**	0.708395	**815**	0.760544
900	0.676236	**857**	0.709523	**814**	0.761508
899	0.676596	**856**	0.71048	**813**	0.762644
898	0.67695	**855**	0.711316	**812**	0.76386
897	0.677258	**854**	0.712216	**811**	0.765124
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895	0.677642	**852**	0.714366	**809**	0.767827

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807	0.770542	**764**	0.82647	**721**	0.871362
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805	0.773636	**762**	0.827948	**719**	0.872037
804	0.77526	**761**	0.828701	**718**	0.872006
803	0.776934	**760**	0.829371	**717**	0.87194
802	0.778696	**759**	0.830019	**716**	0.871974
801	0.780413	**758**	0.830791	**715**	0.872245
800	0.781889	**757**	0.831696	**714**	0.872822
799	0.783135	**756**	0.832628	**713**	0.8736
798	0.784349	**755**	0.83358	**712**	0.87439
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796	0.786992	**753**	0.8359	**710**	0.875845
795	0.788383	**752**	0.837203	**709**	0.876529
794	0.789867	**751**	0.838515	**708**	0.877122
793	0.791402	**750**	0.839822	**707**	0.877668
792	0.792865	**749**	0.841047	**706**	0.878245
791	0.794218	**748**	0.842108	**705**	0.878807
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786	0.801398	**743**	0.84733	**700**	0.881324
785	0.803065	**742**	0.848226	**699**	0.881593
784	0.804643	**741**	0.849187	**698**	0.881856
783	0.806116	**740**	0.850188	**697**	0.882237
782	0.807556	**739**	0.851191	**696**	0.882734
781	0.808957	**738**	0.852183	**695**	0.883362
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779	0.811494	**736**	0.853973	**693**	0.885146
778	0.812704	**735**	0.854887	**692**	0.886155
777	0.813868	**734**	0.85602	**691**	0.887127
776	0.814951	**733**	0.857355	**690**	0.888046
775	0.816044	**732**	0.858755	**689**	0.888831
774	0.817255	**731**	0.860175	**688**	0.889378
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772	0.819719	**729**	0.863112	**686**	0.889948
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676	0.895408	**633**	0.909928	**590**	0.914344
675	0.895833	**632**	0.910405	**589**	0.91412
674	0.896567	**631**	0.910765	**588**	0.914096
673	0.897708	**630**	0.911163	**587**	0.913991
672	0.899398	**629**	0.911568	**586**	0.913807
671	0.901993	**628**	0.911839	**585**	0.913741
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667	0.905394	**624**	0.911432	**581**	0.913846
666	0.901333	**623**	0.910962	**580**	0.91401
665	0.899535	**622**	0.910796	**579**	0.91405
664	0.899574	**621**	0.911014	**578**	0.914012
663	0.90015	**620**	0.911536	**577**	0.913866
662	0.900749	**619**	0.912309	**576**	0.913462
661	0.901311	**618**	0.913121	**575**	0.912948
660	0.901791	**617**	0.913558	**574**	0.912759
659	0.902175	**616**	0.9135	**573**	0.912974
658	0.9026	**615**	0.91337	**572**	0.913221
657	0.903142	**614**	0.91356	**571**	0.913274
656	0.903653	**613**	0.913919	**570**	0.913288
655	0.904006	**612**	0.914113	**569**	0.913281
654	0.904315	**611**	0.914111	**568**	0.91293
653	0.90466	**610**	0.914014	**567**	0.912233
652	0.905013	**609**	0.913829	**566**	0.911752
651	0.905421	**608**	0.913657	**565**	0.911858
650	0.905905	**607**	0.913628	**564**	0.912244
649	0.906245	**606**	0.913504	**563**	0.912476
648	0.906173	**605**	0.913341	**562**	0.912503
647	0.905821	**604**	0.914872	**561**	0.91238
646	0.905584	**603**	0.920573	**560**	0.911971
645	0.905588	**602**	0.929938	**559**	0.911315
644	0.905652	**601**	0.937549	**558**	0.910812
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640	0.906122	**597**	0.915362	**554**	0.909254
639	0.906483	**596**	0.914226	**553**	0.9086
638	0.907035	**595**	0.914504	**552**	0.908001
637	0.907653	**594**	0.914699	**551**	0.907283

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