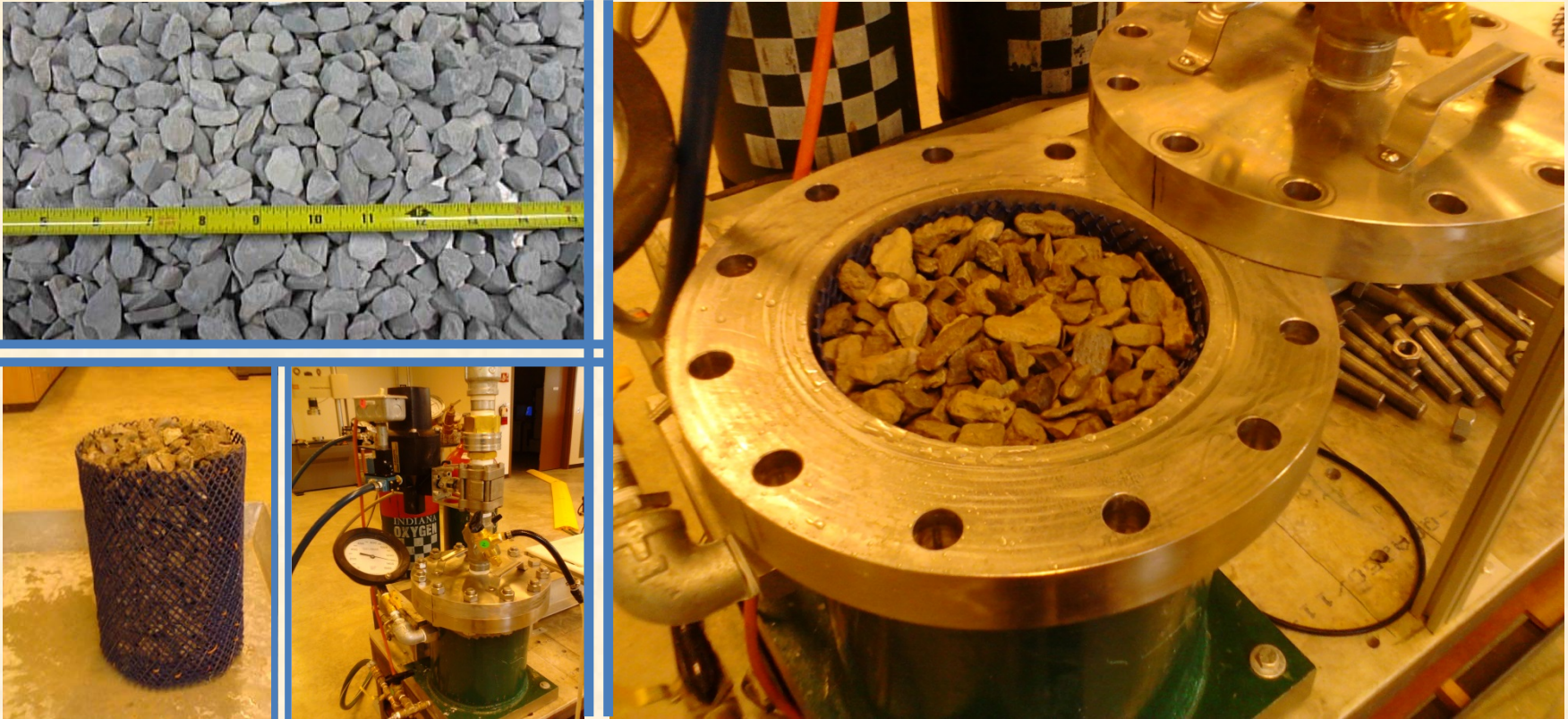


Accelerated Test Method to Identify Freeze-Thaw Durability of Aggregates



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General Outline

Part I: Study Findings & Background

Part II: Natural & Recycled Concrete Aggregates (RCA)

Part III: Highlights of the Ongoing Tests

Part I

Study Findings & Background

- Study Findings
 - Natural Aggregates
 - RCA
- Background
 - Freeze-Thaw Related Damages in Concrete
 - The Certified Aggregate Producers (CAP) Program
 - ASTM C666 (ITM 210)
 - History and Protocol of the HFT
 - INDOT HFT Equipment (History, Design, Features Calibration)

Study Findings

- The Hydraulic Fracture Test (HFT) equipment and refined procedures developed at INDOT accurately predicted the ITM 210 90-day freeze-thaw test results of nearly all aggregates in 8 days.
- The RCA testing showed:
 - All 5 aggregate sources made from older INDOT concrete pavement passed the ITM 210 FT test
 - RCA is responsive to testing in the HFT
 - Testing of additional RCA sources is expected to lead to a reliable model to predict FT durability of RCA in 8 days

Aggregate-Related Freeze-Thaw Damages



Identification of Freeze-Thaw Durability of Aggregates

Rapid Freezing and Thawing -ASTM C666/ AASHTO T161

INDOT's ITM 210

- Commonly used test
- Evaluates concrete beams exposed to freeze-thaw cycles
- Determines % dilation and DF of beams
- Requires expensive equipment



Freeze-Thaw Chamber



- Can take months to complete

Certified Aggregate Producer (CAP) Program

The Indiana Department of Transportation (INDOT) :

- Tests (ITM 210) all their concrete aggregate sources every 1-3 years
- Frequency of test depends on source variability and historical test results

Why Accelerated Test Method?

- The natural variability within aggregate sources may require frequent testing
- Confirm quality for sources that frequently go in and out of spec
- A quick approval or confirmation of approval of certified sources just before construction



Objective

To develop a **reliable, quick test method** for determining the freeze-thaw resistance of carbonate aggregates quarried in Indiana **using the Hydraulic Fracture Test (HFT) equipment.**

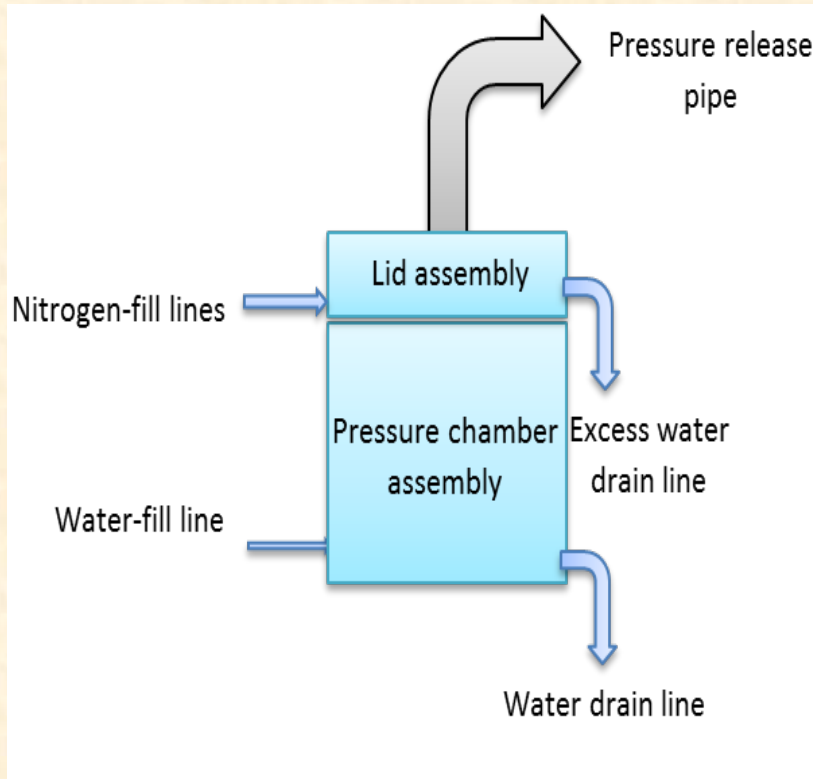


The HFT

- The Hydraulic Fracture Test (HFT) was developed under SHRP Program in the early 1990's
- AASHTO developed a provisional standard TPI2-93 modified and reapproved as TPI2-96 *“Method for Determining the Hydraulic Fracture of Coarse Aggregate”*
- The equipment available and method for interpreting the results at that time had marginally acceptable accuracy

HFT Test Protocol

- Oven-dry aggregates are placed in chamber (~28lb)
- Chamber is filled with water

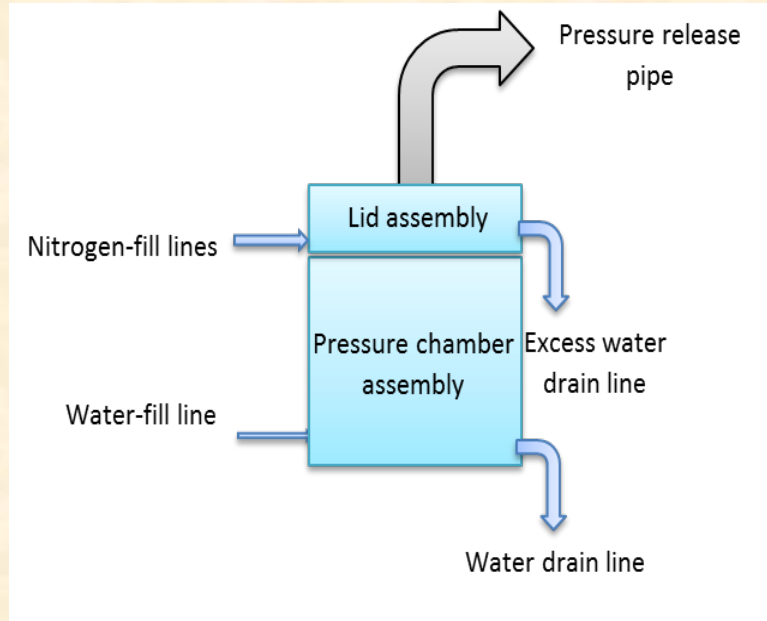


Schematic diagram of INDOT HFT equipment

- Apply pressure (1300 psi) using nitrogen to force water into the pores of the aggregate
- Pressure is then released **rapidly** causing compressed air trapped within the aggregate pores to expand, expel water from pores creating internal stresses

Test Protocol

- If the pore structure of the aggregate cannot allow the water to exit rapidly and the resulting hydraulic pressure exceeds the strength of the aggregate then it will fracture aggregate particles.

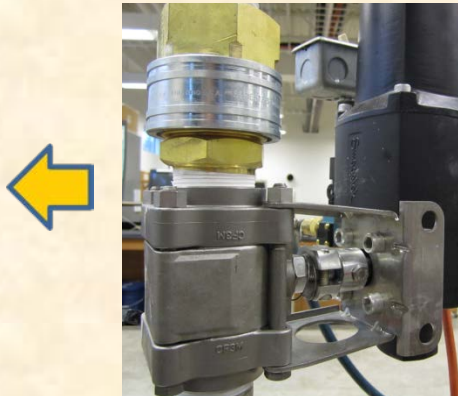
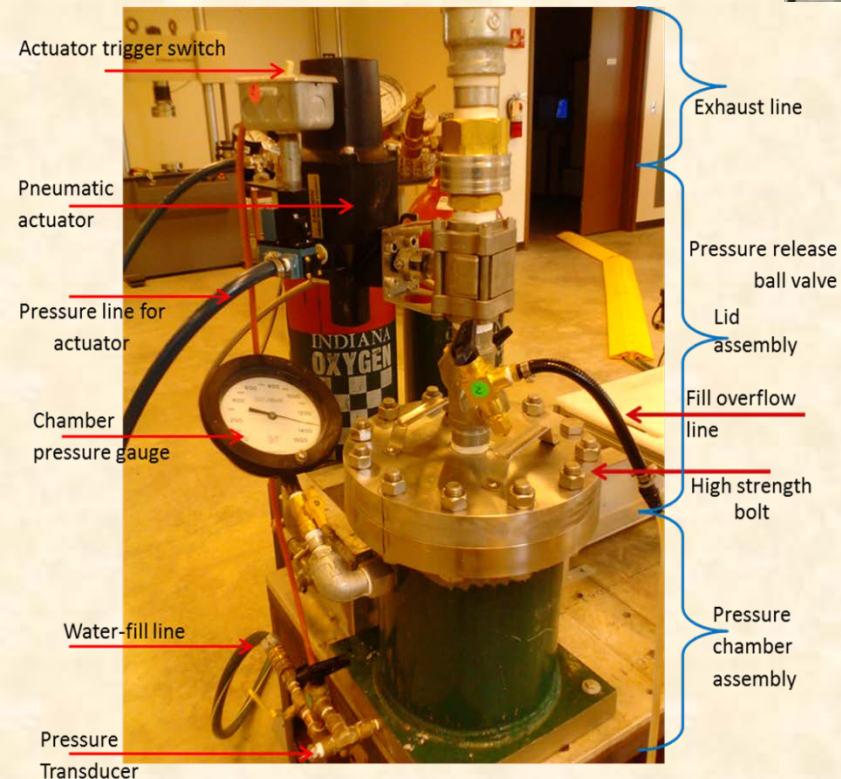
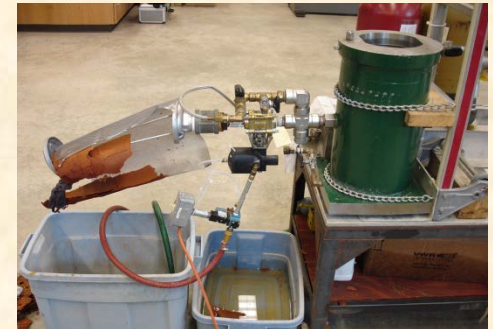
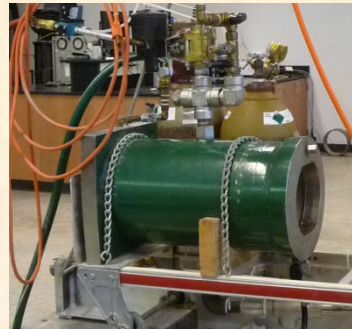


Schematic diagram of INDOT HFT equipment



Fractured aggregate after subjected to 50 HFT cycles (example)

Evolution of INDOT HFT Equipment

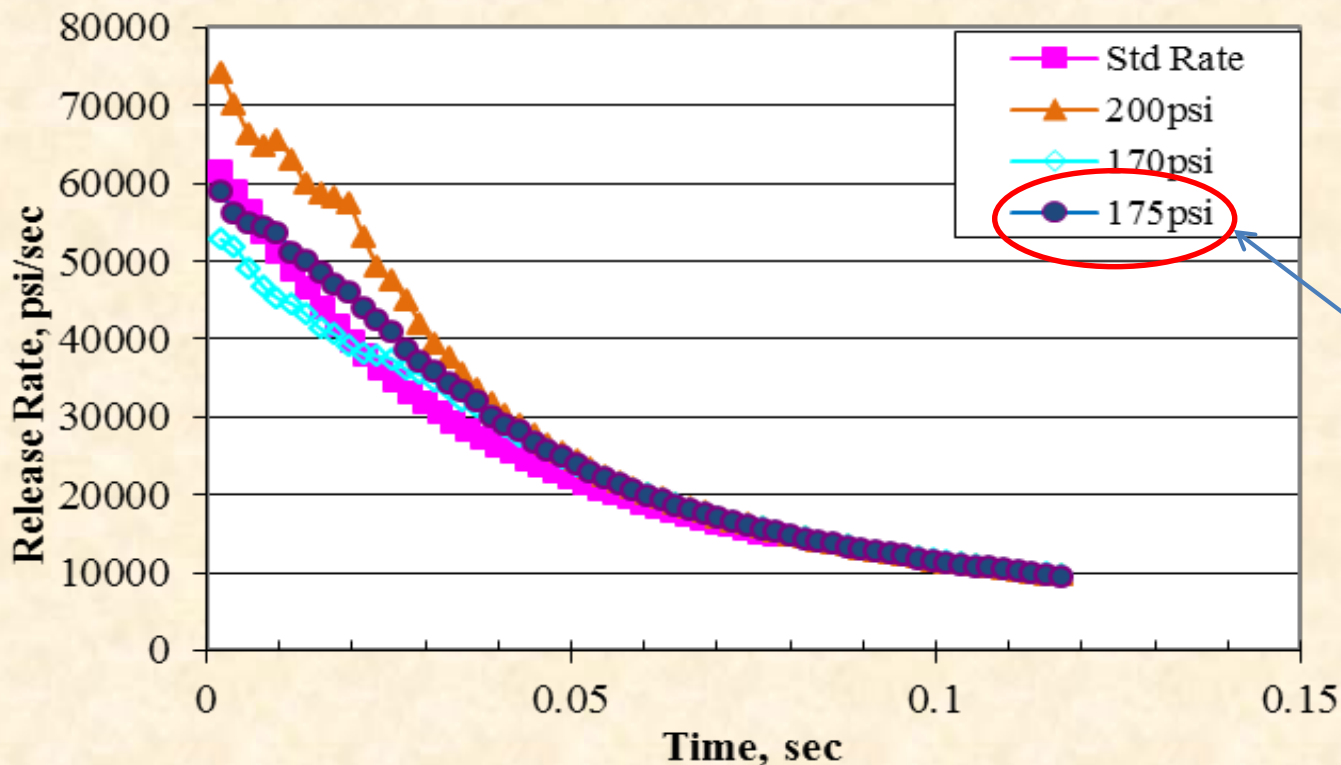


The HFT Chamber Calibration

- The amount of fracturing caused by HFT is directly related to the pressure release rate
- The goal of calibration was to establish the combination of actuator pressure and the chamber pressures that consistently produced release rate curves similar to the “standard” that was adopted in previous studies

The HFT Chamber Calibration

The INDOT HFT equipment was calibrated by running a series of pressurizing and depressurizing cycles at different pressures combination



A Chamber pressure of 1300 psi, & actuator pressure of 175psi selected for further testing

Pressure release profiles for chamber pressure of 1300psi and different actuator pressure

Part II

Quarried and Recycled Concrete Aggregates

- Quarried Aggregate
 - Selection
 - Testing
 - Analysis
 - Model development
 - Conclusions
 - Implementation
- Recycled Concrete Aggregate (RCA)
 - Testing
 - Analysis
 - Model development
 - Conclusions
 - Implementation

Aggregate Sources

18 quarried carbonate sources

Group A

A1
A2
A3
A4
A5
A6

Group B

B1
B2
B3
B4
B5
B6

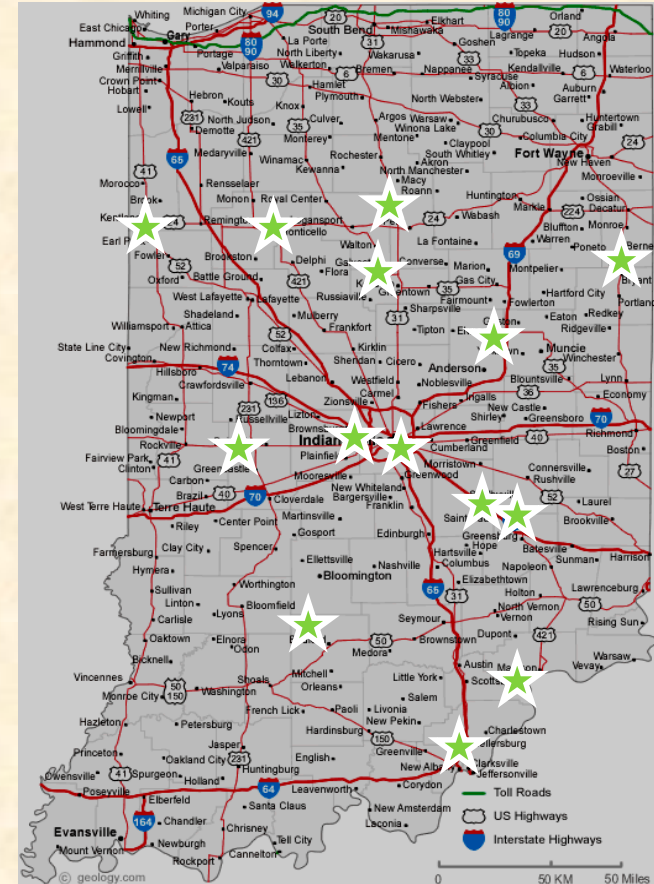
Group C

C1
C3
C4
C5
C6
C7

Group A: Freeze-thaw durable sources

Group B: Freeze-thaw nondurable sources

Group C: Variable or unknown freeze-thaw performance



Aggregate Sample Preparation

Representative samples from each aggregate source were separated by sieving into three size ranges:

3/4 to 1 in. (19.0 to 25 mm)

5/8 to 3/4 in. (16.0 to 19.0 mm)

1/2 to 5/8 in. (12.5 to 16.0 mm)

Aggregate Sample Preparation

Individual fractions were recombined into an HFT test sample (about 28lb) using the gradation shown in the table.

Sieve Size	% Passing
1 in (25 mm)	100
$\frac{3}{4}$ in (19 mm)	89
$\frac{5}{8}$ in (16 mm)	40
$\frac{1}{2}$ in (12.5 mm)	0

Aggregate Sample Preparation

Aggregate samples:

- Washed and oven dried
- Submerged in a water-based silane solution for 60 seconds
- Drained and oven dried



Double boiler used to soak aggregate sample

Aggregate Sample Preparation

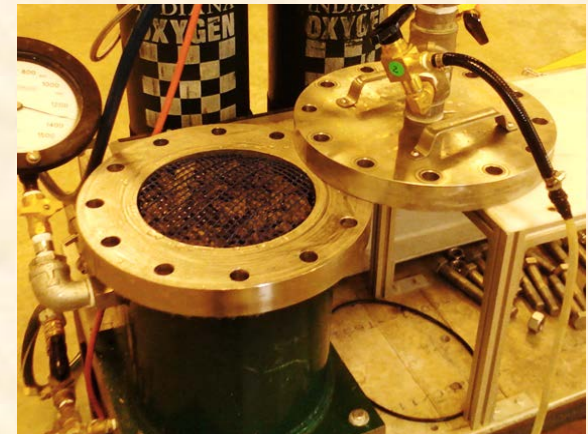
Aggregate samples:

- Tumbled in a rock tumbler



A rock tumbler

- Sieved over the $\frac{3}{4}$ ", $\frac{5}{8}$ " and $\frac{1}{2}$ " sieves, any piece passing the $\frac{1}{2}$ " sieve discarded



Aggregate Sample placed in HFT chamber 21

HFT Sample Sieving

After each 10 HFT cycles, the samples removed from the chamber, oven-dried, tumbled and sieved.



HFT Result Example (B3)

Sieve size	Mass (g)						PCMR*	
	0 Cycle	10 Cycles	20 Cycles	30 Cycles	40 Cycles	50 Cycles		
3/4"	1462.5	1462	1412.8	1363.6	1375.9	1263.8	-13.59	P34
5/8"	6344.0	6338.4	6328.8	6270.2	6099.8	6266.7	-1.22	P58
1/2"	5187.0	5180.5	5193.7	5281.2	5355.7	5243.8	1.10	P12
3/8"	0.0	0.0	13.7	9.4	44.7	78.9	0.61	P38
5/16"	0.0	0.0	0	0.0	1.3	4.0	0.03	P516
1/4"	0.0	0.0	0.5	0.0	0.0	0.0	0.00	P14
#4	0.0	0.0	0.6	0.0	0.0	0.0	0.00	P4
Pan	0.0	12.6	24.3	26.6	25.3	22.2	0.85	P0
Total Mass	12993.5					12875.4		

*Percent Change in Mass Retained (after 50 cycles)

Freeze-Thaw Test - ITM210

Beams were fabricated from concrete mixtures that were produced using each aggregate source

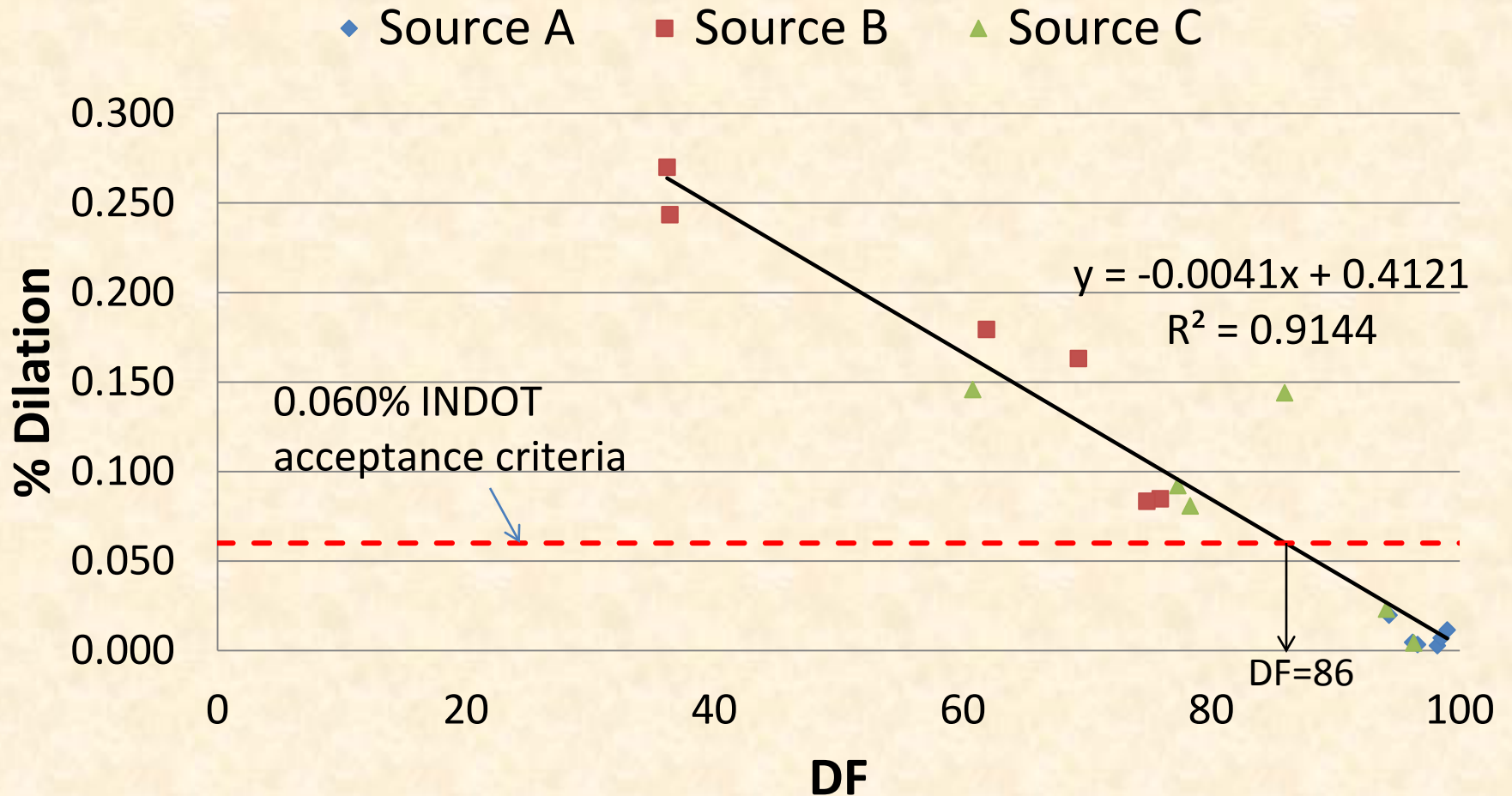
- $w/c = 0.43$
- $6.5 (\pm 1.5) \% \text{ air}$
- Beams (3 in. X 4 in. X 15 in.)
- % dilation & durability factor (DF) measured

Expansion $< 0.060\%$ after 350 cycles of F/T

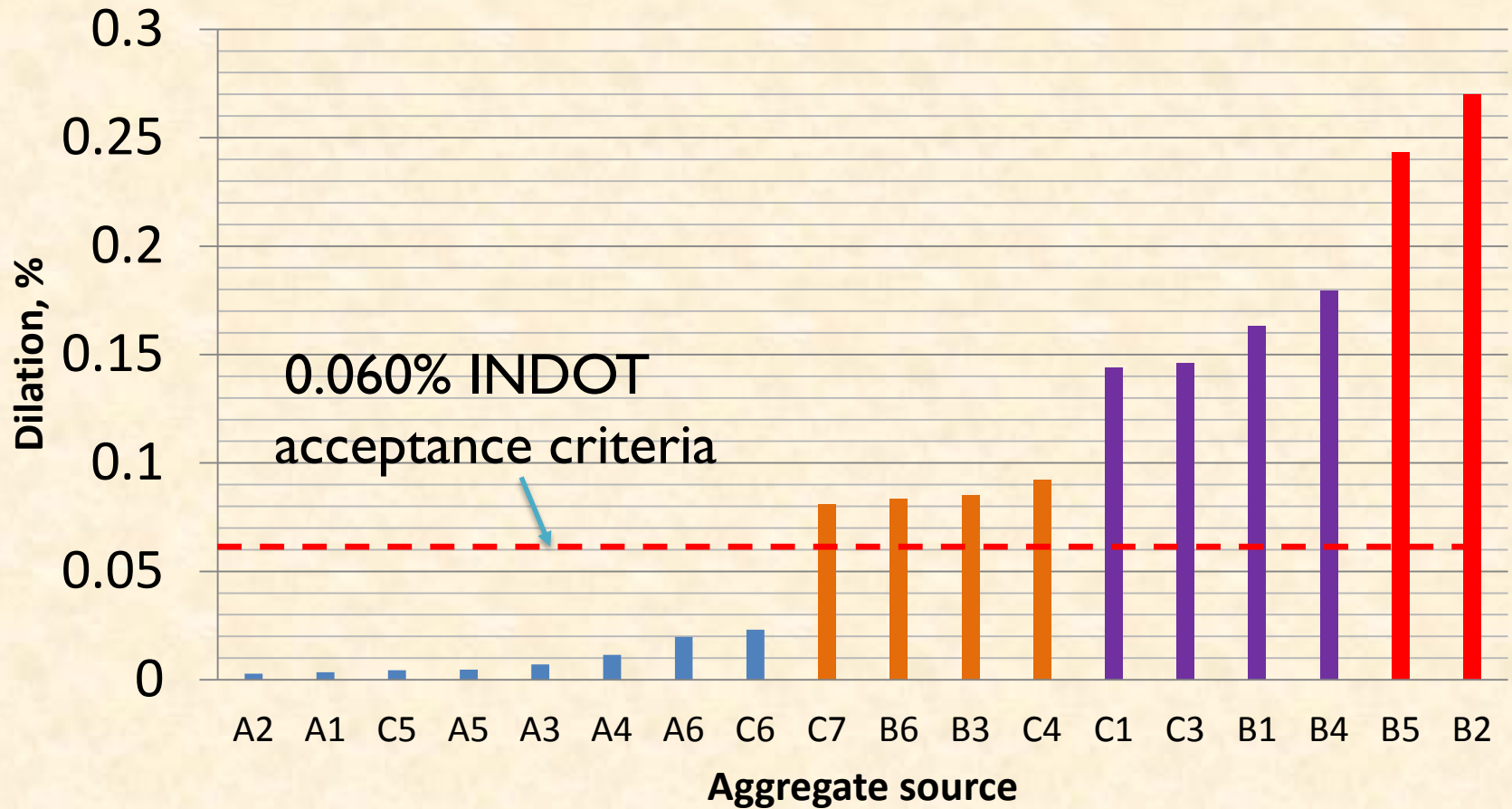


INDOT

Analysis



Analysis



Model Development

A linear regression model was developed to predict the average % dilation of freeze-thaw test beams using parameters obtained from HFT results

$$\% \text{ Dilation} = 8.25\text{E-}2 + 6.33\text{E-}3*\mathbf{P34} + 9.64\text{E-}2*\mathbf{P38} - 3.12*\mathbf{P14} + 4.3*\mathbf{P4}$$

Model statistics:

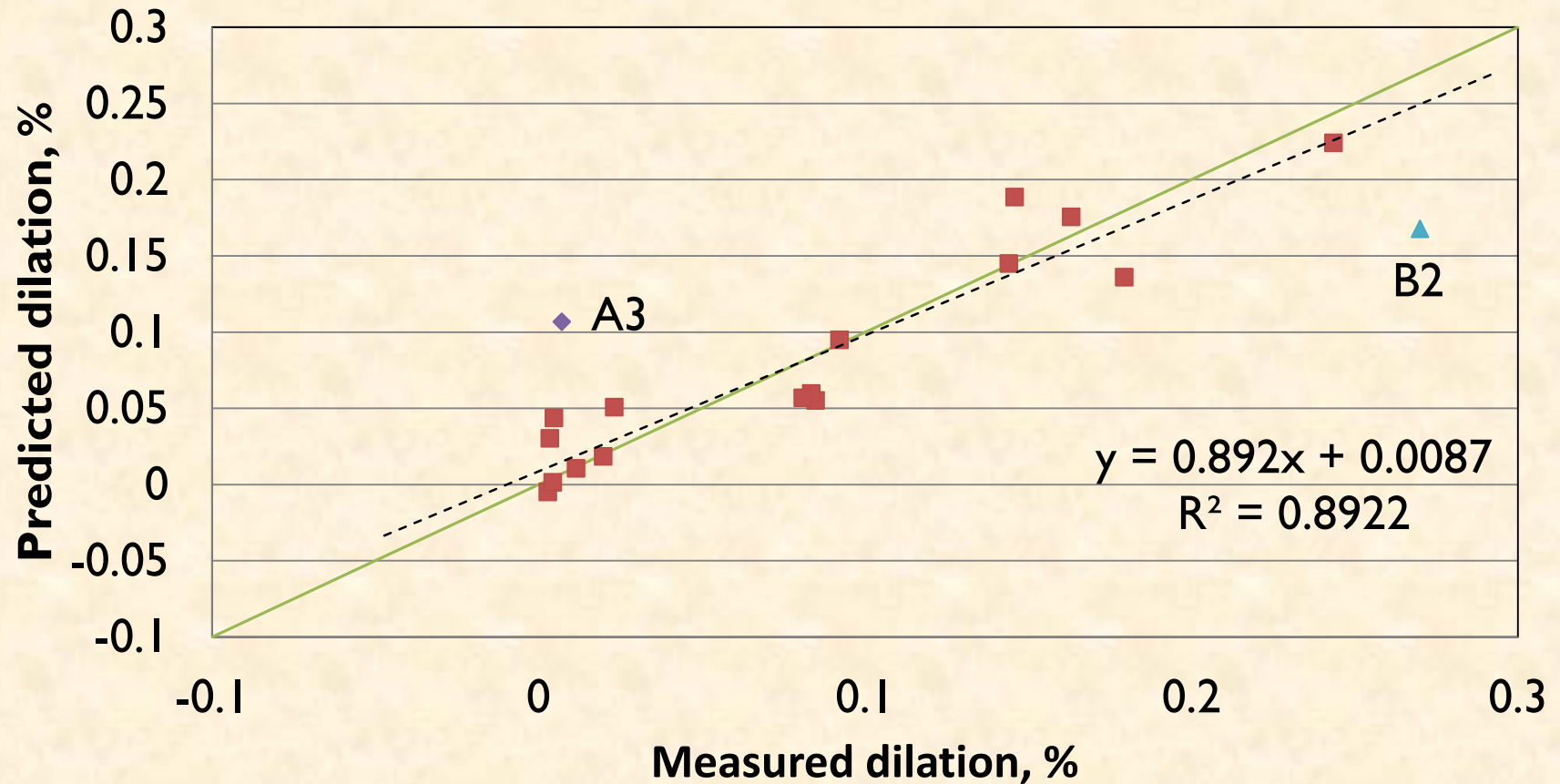
$$R^2 = 0.892,$$

$$R^2 \text{ (adj.)} = 0.853,$$

$$SEE = 0.029, n = 16,$$

$$\text{Model } P\text{-value} < 0.0001$$

Measured vs Predicted Dilations



— Ideal 1:1
relationship of x
and y axes

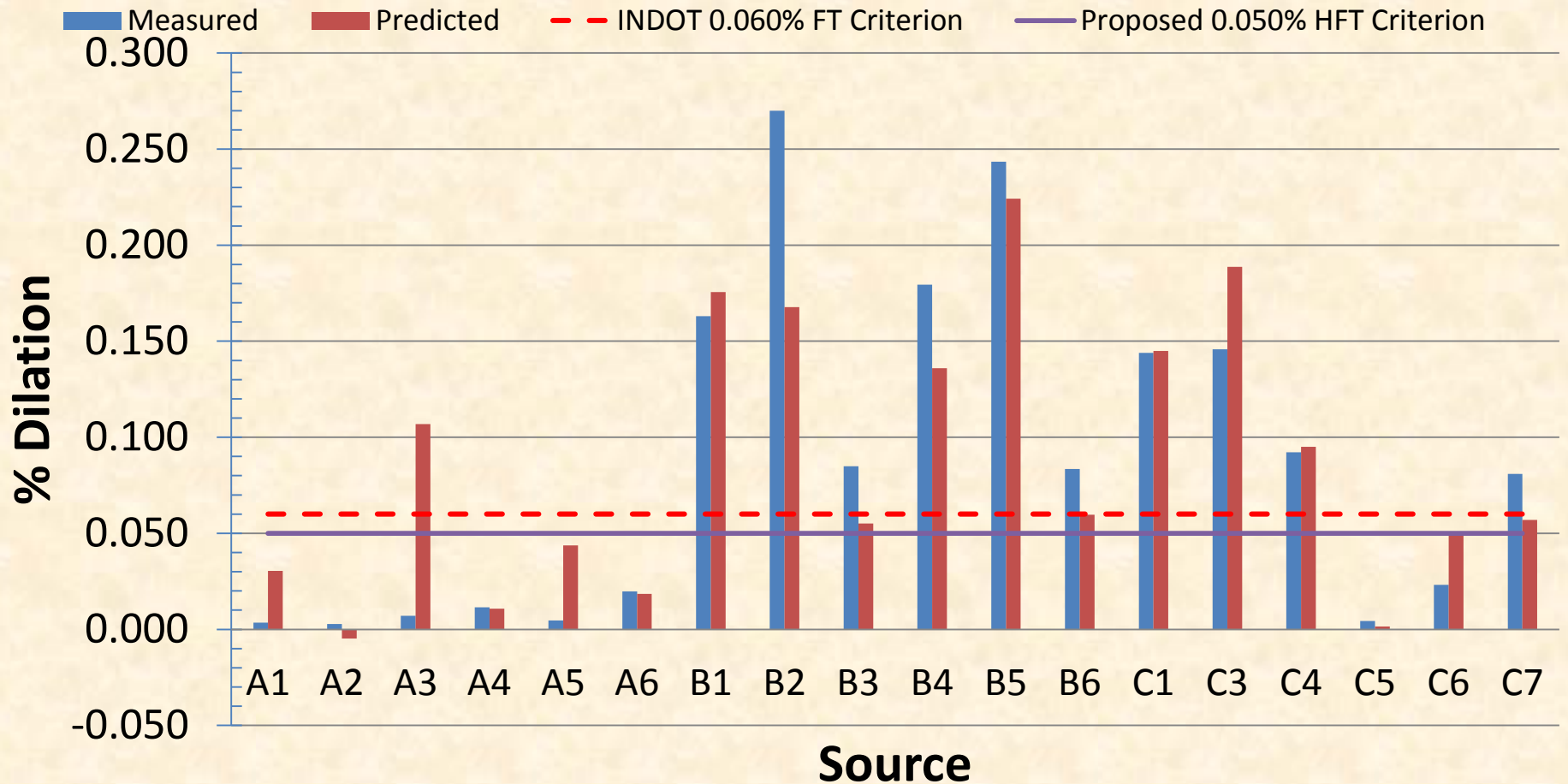
---- Data Trend line
excluding A3 and B2

Measured vs Predicted Dilations

The developed dilation model correctly predicted the freeze-thaw durability for all sources tested except for the four sources shown in the table (14 of the 18 sources).

Source (Group)	Measured Dilation, %	Predicted Dilation, %
A3	0.0071	0.1068
B3	0.0849	0.0551
B6	0.0835	0.0597
C7	0.0809	0.0569

Measured vs Predicted Dilations



Conclusions

- The refined INDOT HFT equipment, procedures and analysis appear to provide a quick method to evaluate the freeze-thaw resistance of carbonate aggregates quarried in Indiana predicting the 90-day FT test results in 8-days.
- Testing of additional aggregate sources, and possible refinement of the regression model, the reliability of this test is expected to increase

Significance

- ITM 210 - INDOT plans to continue testing and certifying their aggregate sources using ITM 210 every 1 to 3 years as part of their Certified Aggregate Producers (CAP) program.
- The HFT will be used as a **quick approval**, or **confirmation of approval of certified sources**. This is especially useful for quarry sources that have ledges with questionable or variable performance.
- HFT results will be a **good check** that INDOT receives for construction similar quality material that was tested during the certification process.

Implementation

At this time, it is recommended that the developed regression model be used as a screening test with the following criteria:

Predicted Dilation	Expected Durability
<0.050%	Durable
Between 0.050% and 0.060%	ITM210 is required to determine the aggregate durability
> 0.060%	likely nondurable

Recycled Concrete Aggregate (RCA)

RCA Sources

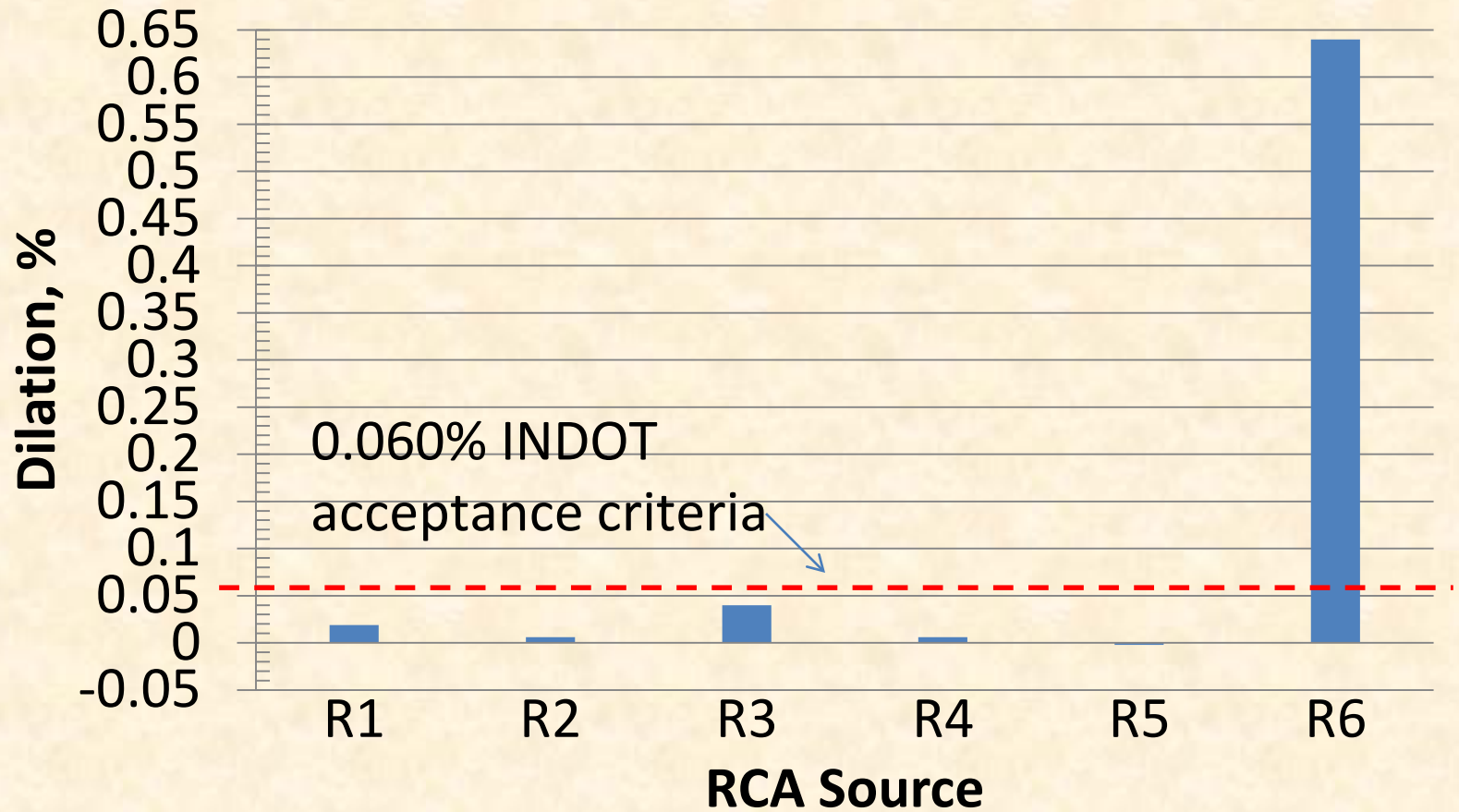
Source No.	Structure	Original Aggregate Type
R1	SR26 INDOT pavement	Gravel
R2	US 52 INDOT pavement	Gravel
R3	I-65 INDOT pavement	Gravel
R4	SR19 INDOT pavement	Gravel
R5	SR912 INDOT pavement	Crushed carbonate rock
R6	Misc. structures from Indianapolis Dept. of Public Works	Primarily gravel, some crushed carbonate rock

Analysis

Summary of HFT and freeze-thaw test results

	Response Variables		Predictor Variables							
	FT test result		HFT test result (PCMR Values)							
RCA Source	Dilation	DF	P34	P58	P12	P38	P516	P14	P4	P0
R1	0.019	98.4	-14.39	-8.27	5.60	1.39	0.07	0.08	0.06	1.51
R2	0.006	99.6	-27.46	-7.82	7.68	1.24	0.09	0.14	0.06	2.49
R3	0.040	94.2	-10.41	-10.29	6.38	1.21	0.15	0.09	0.06	1.80
R4	0.006	88.4	-19.24	-15.55	6.01	2.44	0.08	0.10	0.07	2.27
R5	-0.002	96.0	-17.60	-5.50	6.03	1.03	0.05	0.03	0.04	1.67
R6	0.640	30.0	-21.56	-11.60	8.74	1.75	0.18	0.17	0.12	2.82 ³⁶

Analysis



Previous Models

The model developed for predicting freeze-thaw durability of the carbonated quarried aggregates cannot be used to predict the freeze-thaw durability of RCA

Source	Dilation	
	Measured	Predicted
R1	0.019	0.134
R2	0.006	-0.151
R3	0.04	0.110
R4	0.006	0.185
R5	-0.002	0.149
R6	0.64	0.100

New RCA Model

A linear regression model was developed to predict the average % dilation of freeze-thaw test beams using parameters obtained from HFT results

$$\% \text{ Dilation} = -1.447 + 4.65\text{E-}02\text{P34} + 1.21\text{E-}01\text{P58} + 3.29\text{E-}01\text{P12} + 9.24\text{E-}01\text{P38}$$

Model statistics:

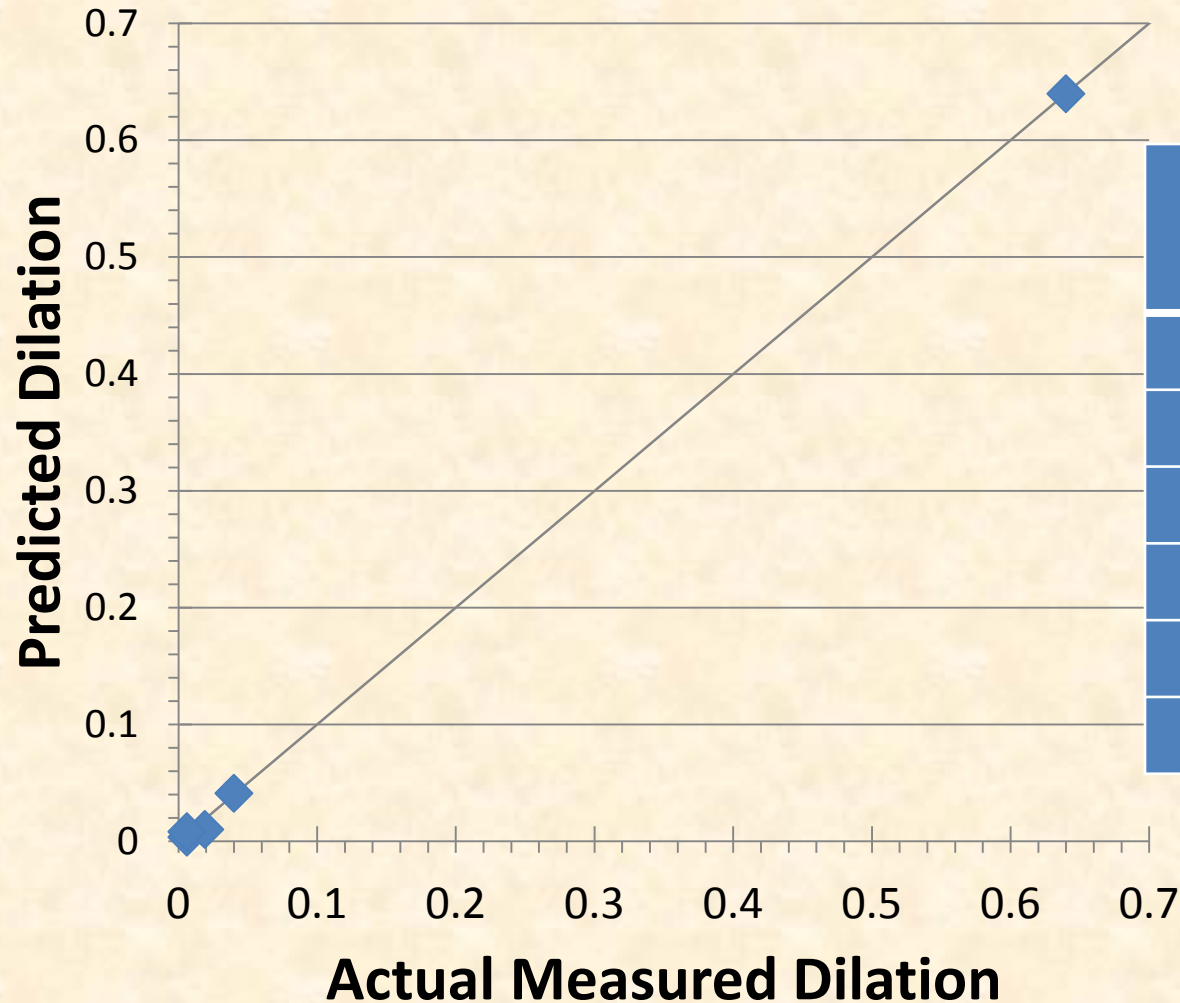
$$R^2 = 0.999,$$

$$R^2 (\text{adj.}) = 0.997,$$

$$\text{RMSE} = 0.012, n=6,$$

$$\text{Model } P\text{-value} < 0.0316$$

Measured vs Predicted



	Dilation	
	Measured	Predicted
R1	0.019	0.010
R2	0.006	0.004
R3	0.04	0.041
R4	0.006	0.008
R5	-0.002	0.006
R6	0.64	0.640

Conclusions

- RCA is responsive to testing in the HFT
- The models developed for predicting freeze-thaw durability of the carbonated quarried aggregates cannot be used to predict the freeze-thaw durability of RCA

Implementation

- It is recommended that the dilation model developed be used for **research and preliminary screening purpose only**.
- Further HFT and FT testing of additional RCA sources that represent **a greater spectrum of FT performance is needed** to improve these models prior to using as part of the acceptance criteria for RCA as coarse aggregate in INDOT concrete paving projects.

Part III

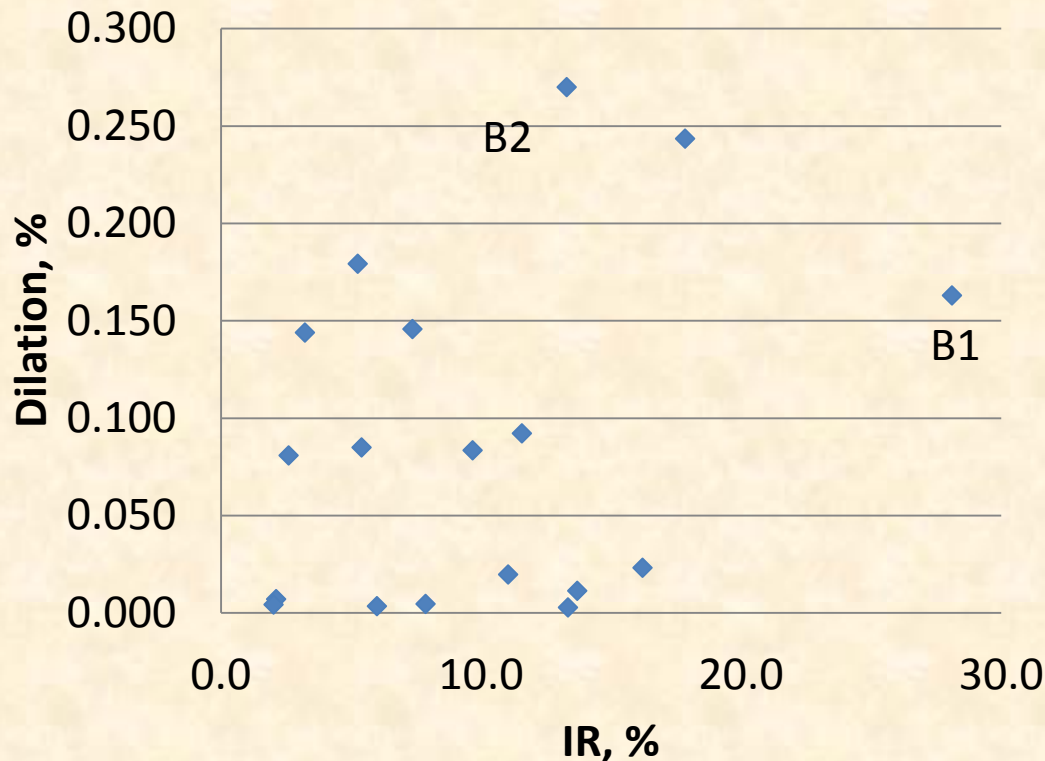
Highlights of the Ongoing Tests

- Highlight of the ongoing tests
 - Objective
 - Testing
 - Analysis
 - Preliminary Conclusions/Observations
- Acknowledgements

Objectives

- To investigate the role of aggregate's mineralogy, microstructure on the HFT result & freeze-thaw performance
- To further refine the HFT test procedure, data analysis and models developed
- To explain why some sources did not fit in the model

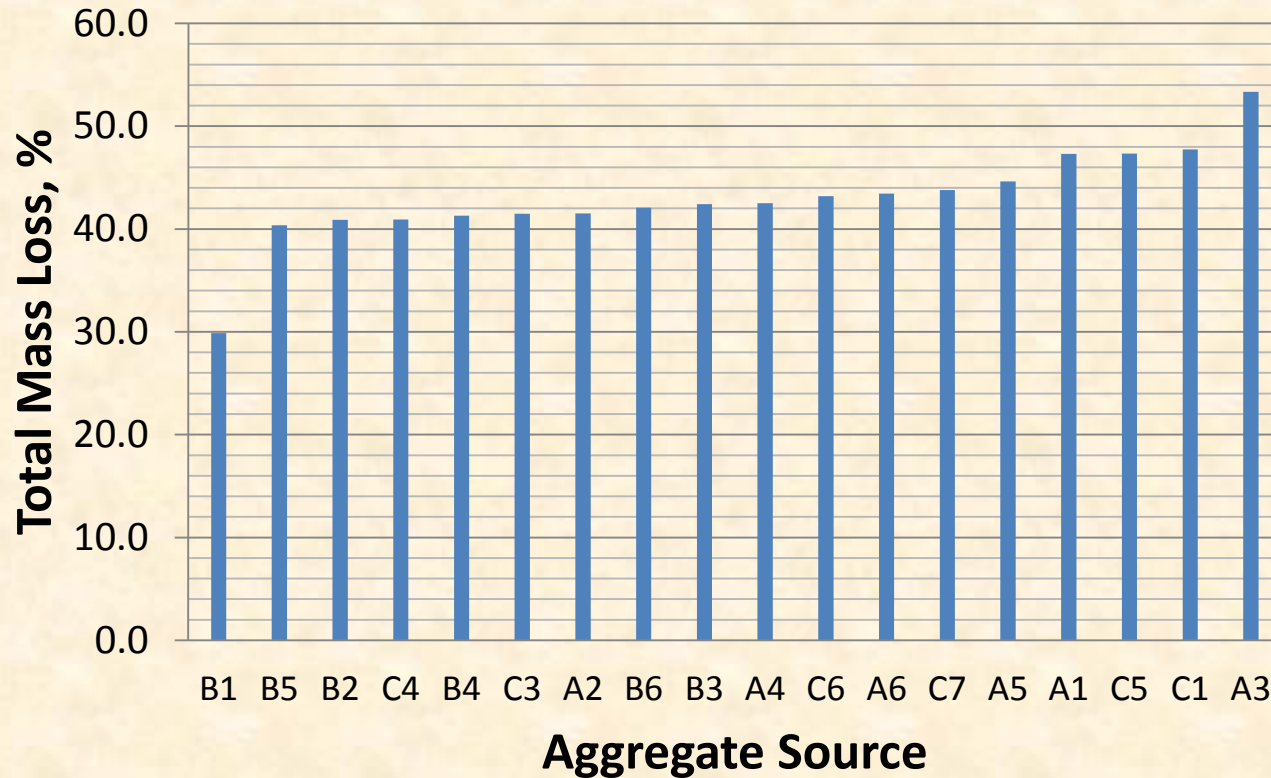
Acid Insoluble Residue (IR)- ASTM D3042



IR is % by weight of silt & clay size particles when the aggregate is dissolved in 6N HCl

- No correlation between IR and dilation or DF observed
- Source B1 had the highest IR, and percent fracture in HFT

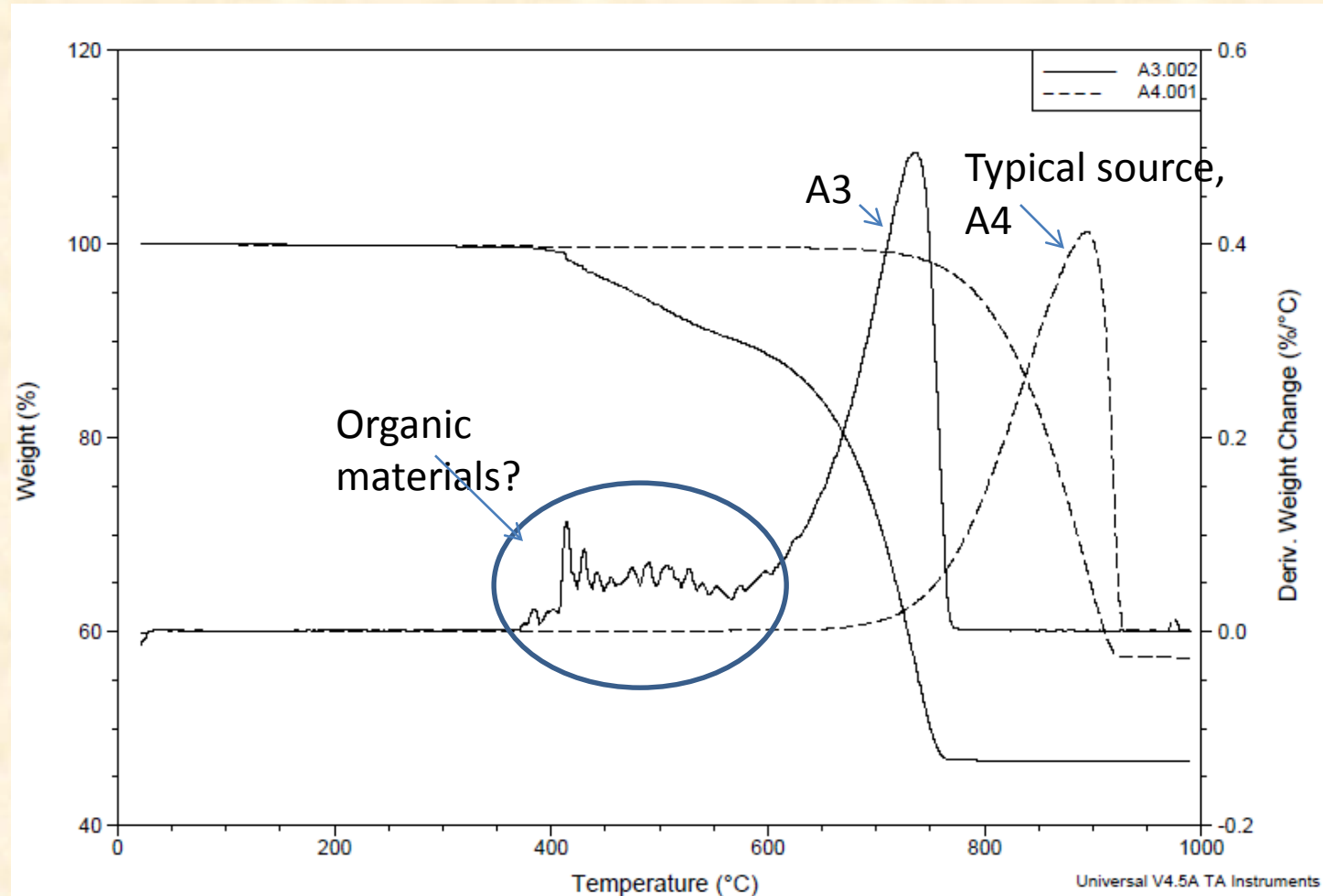
Thermal Analysis (TGA)



Mass Loss:
Min. 30% ,B1
Max. 53%, A3
Avg. 43%

- B1 had the highest amount of fracture in HFT
- A3 did not fit in the model

TGA



Comparison of A3 with a particular source, A4

Literature on Critical Pore Size Range for Freeze-Thaw Durability

Shakoor 1982	10nm-10 μ m
Salcedo 1984	45nm-10 μ m
Kaneuji, Winslow and Dolch 1980	4.5nm-5 μ m
Dubberke and Marks 1985	40nm-0.2 μ m

Pittenger and West, 1995

Sorption Analysis

- Three sources were considered
- Dynamic Vapor Sorption (DVS) analysis conducted on about 50mg, 0.8mm thick specimen
- Mass loss was recorded by changing the RH from 97% all the way to 0%

Source	Dilation	DF
A1	0.0035	96.6
B1	0.1631	69.3
B2	0.27	36.2

Kelvin –Laplace Equation

The Kelvin–Laplace equation used to approximate the pore radius that corresponds to a specific relative humidity

$$RH = \exp\left(\frac{-2\gamma \cos(\theta)}{r} \cdot \frac{V_m}{RT}\right)$$

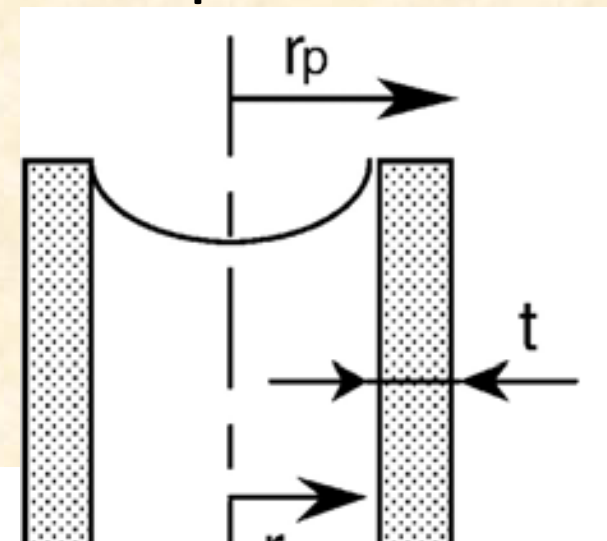
where

R = universal gas constant (8.314 J/mol·K),

T = temperature (K),

RH = internal RH, and

V_m = molar volume of pore solution ($\approx 18 \times 10^{-6} \text{ m}^3/\text{mol}$)

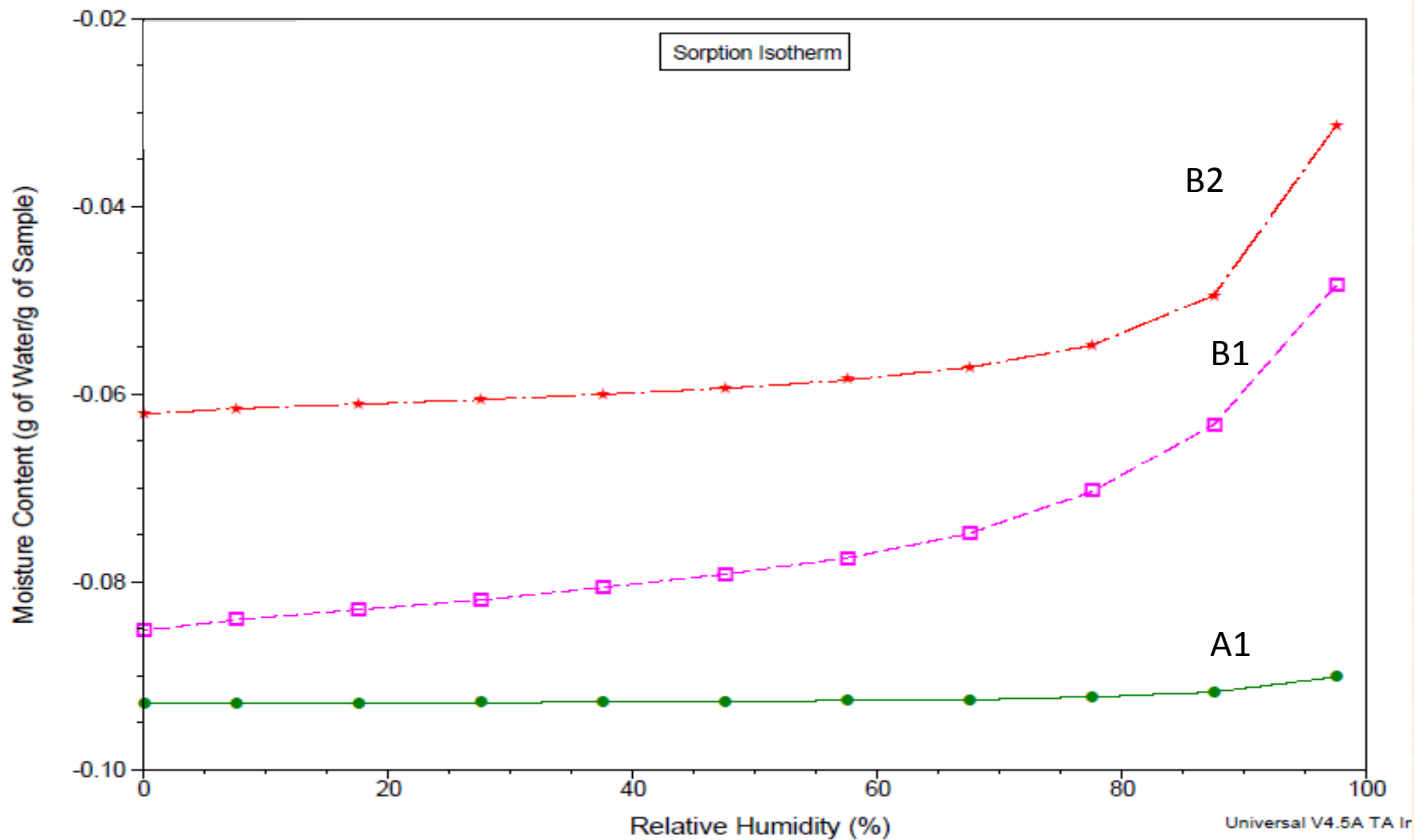


r_p : pore radius

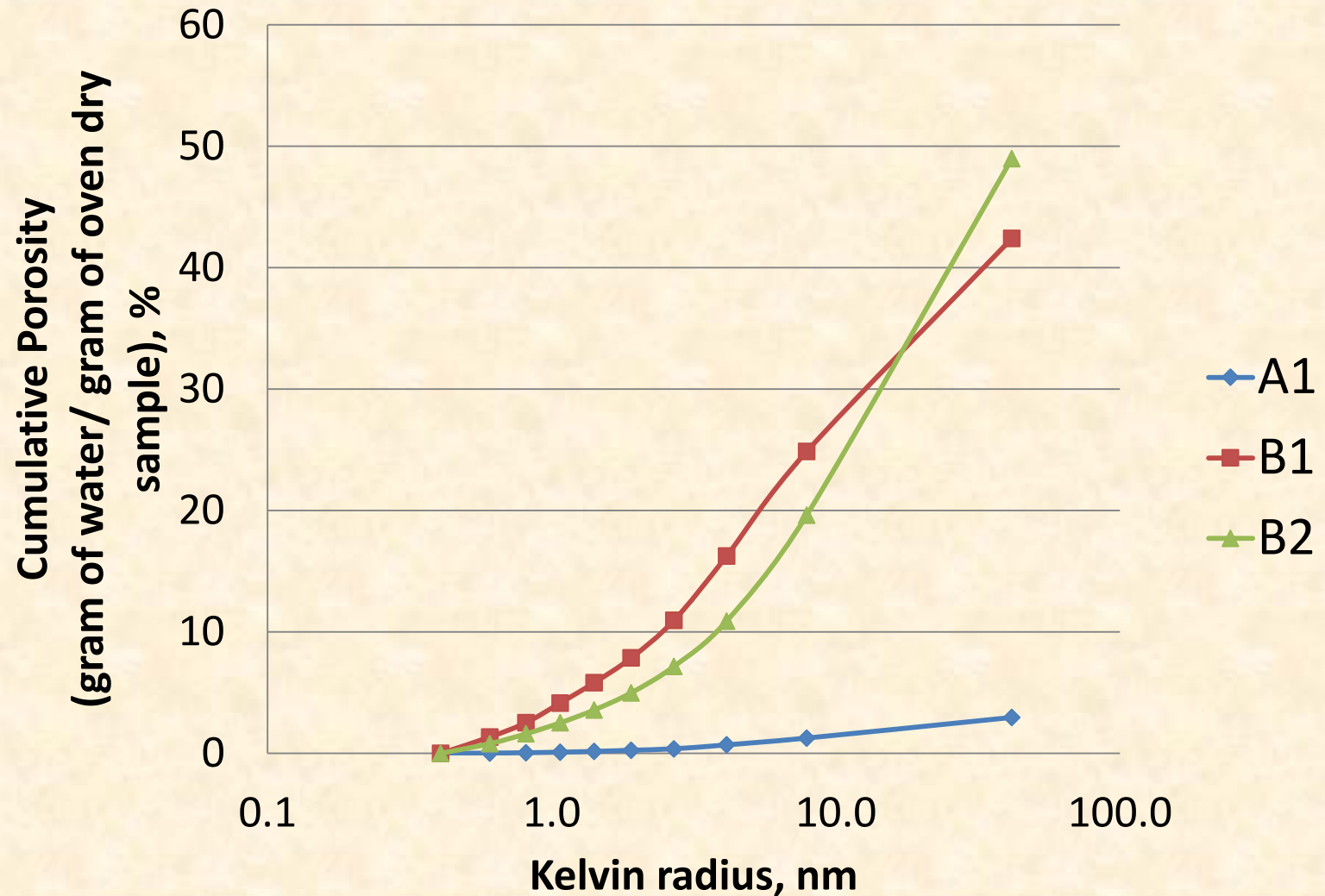
r_k : Kelvin radius

t : adsorbed layer thickness

Desorption Isotherm



Cumulative Porosity



Preliminary Conclusions

- No correlation observed between IR and dilation or DF observed
- Incorrectly predicted source had the highest mass loss in TGA
- Cumulative porosity (pore size < 80nm) determined by DVS identified durable from nondurable sources

Acknowledgements

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Full report on Part I and II is available online:

<http://docs.lib.purdue.edu/jtrp/I565/>