

Techniques to Fingerprint Construction Materials--R06B XRF and FTIR Spectroscopy

NESM EA

Atlantic City, NJ
October 16, 2018



R06B—Maine

- MaineDOT goals for R06B:
 - Maximize non-destructive testing
 - Reduce test time and cost
 - Reduce incorporation of out-of-spec material into DOT work
- XRF
 - Chlorides in bridge deck cores
 - Titanium in traffic paint
 - REOB in PG Binder
 - SS Rebar
 - Glass Beads – lead, arsenic
 - Presence of RAS in HMA?
- FTIR
 - Presence of polymer in asphalt
 - Asphalt binder library

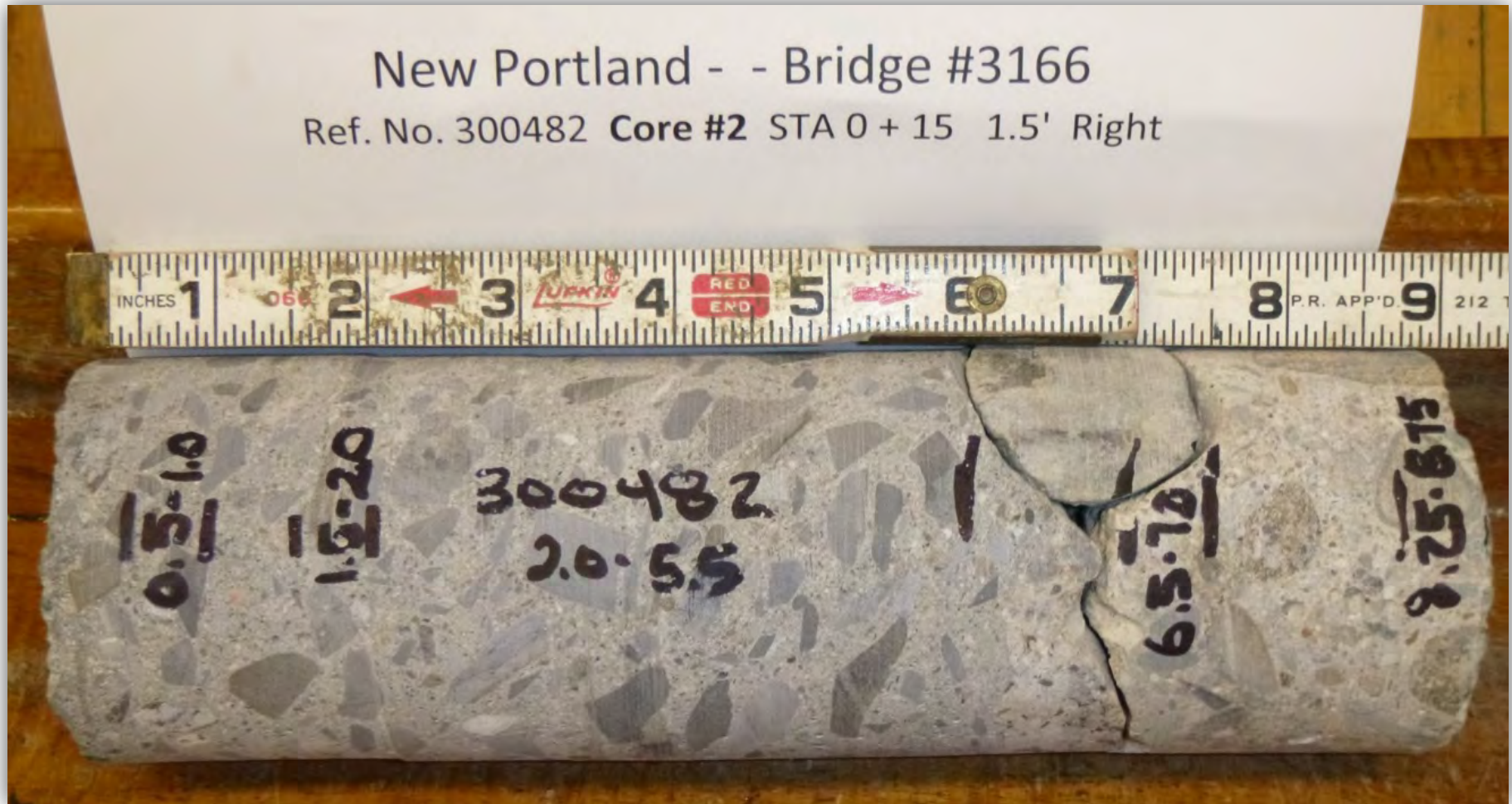


Stainless steel rebar



El	%	+/- 2σ
V	0.110	0.010
Cr	23.490	0.073
Mn	1.818	0.045
Fe	70.056	0.093
Co	0.123	0.045
Ni	3.758	0.044
Cu	0.347	0.014
Zr	0.004	0.001
Nb	0.018	0.001
Mo	0.253	0.004
W	0.017	0.005
Pb	0.007	0.002

Chloride Content – Bridge Deck Cores



- Concrete cores pulverized and analyzed for chloride content ~ rebar corrosion begins at 1.35lb/cy or 0.03%

Chloride Content – Bridge Deck Cores

- Current method: AASHTO T 260 (Gran Plot Method)
 - Requires nitric acid and silver nitrate
 - Numerous steps
 - 10 tests/day
- XRF method
 - No chemicals
 - 25+ tests/day
 - Less training required

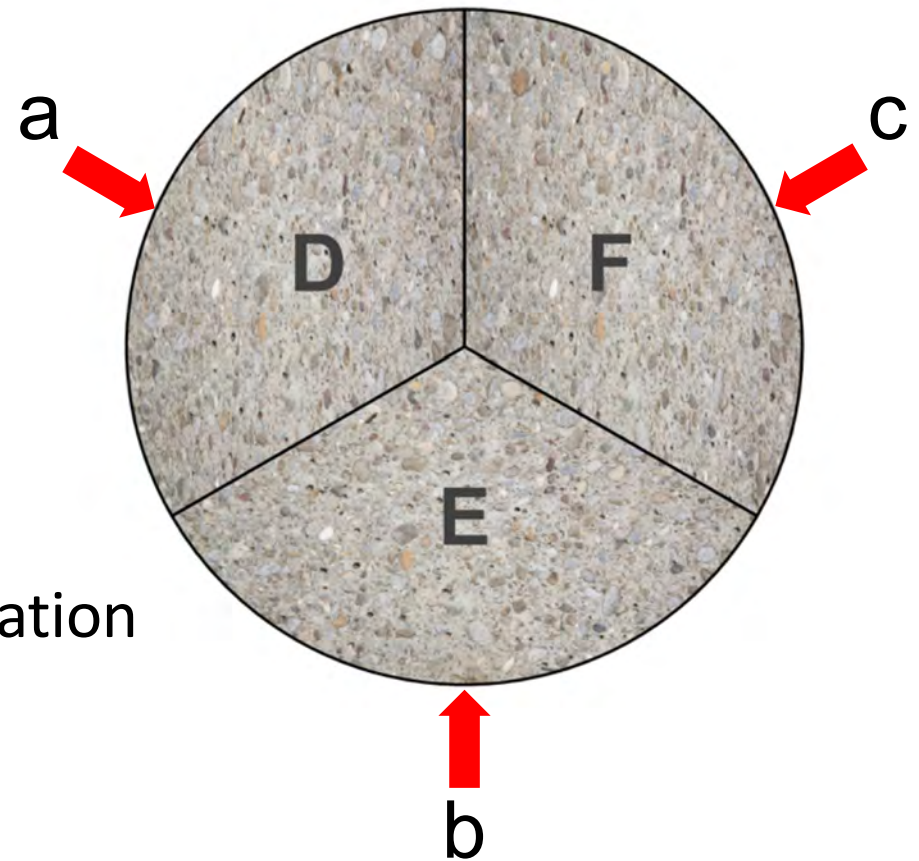
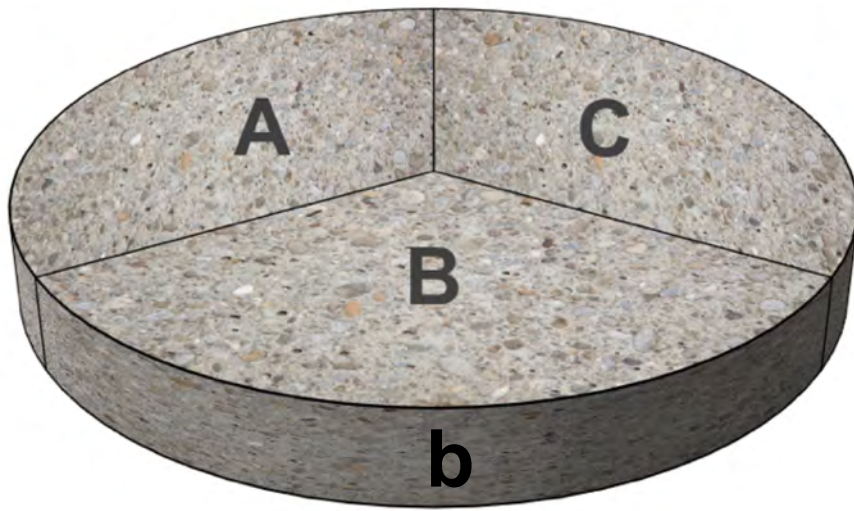


Chloride Content – Bridge Deck Cores

- Split-sample comparison on two types of samples:
 - Concrete Cores
 - Pellets from Pulverized Cores
 - Evaluated numerous binding agents for pelletized samples, XRF settings, direct measurement of concrete
 - Selected the settings that provided the best correlation on a limited amount of measurements vs. titration values
 - Expanded population of comparison

Item	Levels	Details
Analysis Mode	3	AllGeo and Two Mining Modes
Time Breakdown	2	5/5/5/45 & 15/15/15/15
Binding Agent	6	None and 5 recommended agents
Binding %	2	5% & 10%
Replicates	3	Three measurements on each pellet

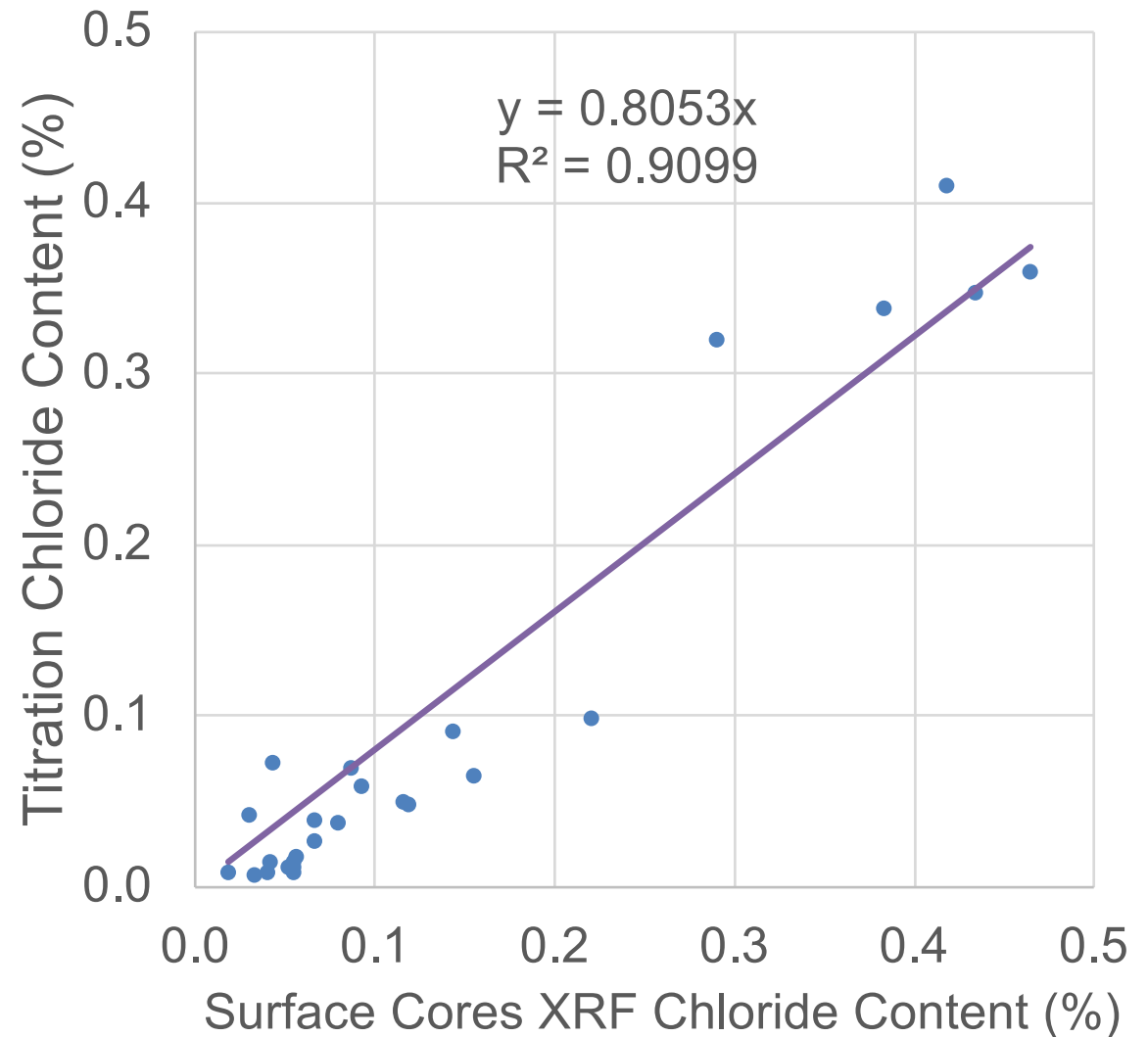
Surface Testing of Core Slices



- Top, bottom, edge of slice
- Average of all readings v. Titration

Surface Testing of Core Slices

- General trend exists but significant drawbacks
- Technician discretion to avoid exposed aggregate
- Higher variability in measurements



Chloride Content – Pellets from Cores

Mode/Range @ 60 Sec.	Binding Agent	% Binding Agent	R ²	Coefficient
Mining Ta/Hf 5/5/5/45	A	5	0.996445	1.091516
AllGeo 5/5/5/45	B	5	0.996009	1.142771
Mining Cu/Zn 5/5/5/45	A	5	0.995589	1.078925
AllGeo 5/5/5/45	None	---	0.99518	0.993099
Mining Ta/Hf 5/5/5/45	B	5	0.994987	1.145006
AllGeo 5/5/5/45	A	5	0.99459	1.084792
AllGeo 5/5/5/45	C	10	0.994295	1.082809
Mining Ta/Hf 5/5/5/45	A	10	0.994101	1.065355
Mining Cu/Zn 5/5/5/45	None	---	0.993977	0.985461
AllGeo 5/5/5/45	A	10	0.993585	1.061301
Mining Cu/Zn 5/5/5/45	A	10	0.993433	1.06045
AllGeo 5/5/5/45	C	5	0.993298	1.031429
Mining Ta/Hf 5/5/5/45	D	10	0.992926	1.008566
Mining Cu/Zn 15/15/15/15	A	5	0.992883	1.129886
Mining Cu/Zn 5/5/5/45	B	5	0.992812	1.144496
Mining Cu/Zn 15/15/15/15	E	5	0.992806	1.053816
Mining Cu/Zn 5/5/5/45	E	5	0.992745	1.045713
Mining Ta/Hf 5/5/5/45	None	---	0.992719	0.973055
Mining Cu/Zn 15/15/15/15	C	10	0.992453	1.051661
Mining Ta/Hf 5/5/5/45	C	10	0.992397	1.102904
Mining Cu/Zn 15/15/15/15	A	10	0.992358	1.034796

- Nearly all combinations showed excellent correlation
- Selected the simplest configuration with no binding agent

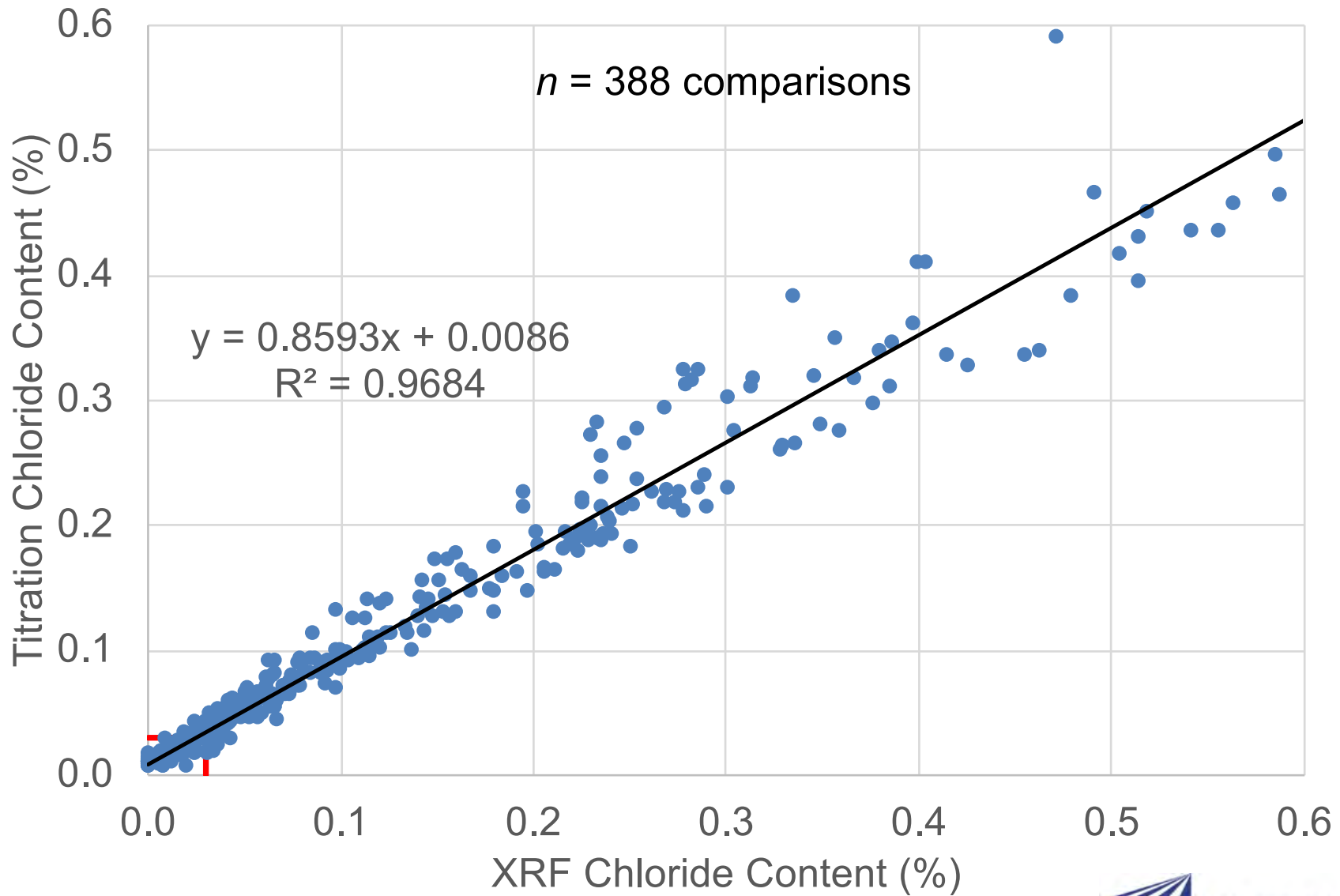
Pulverized & Pelletized Specimens



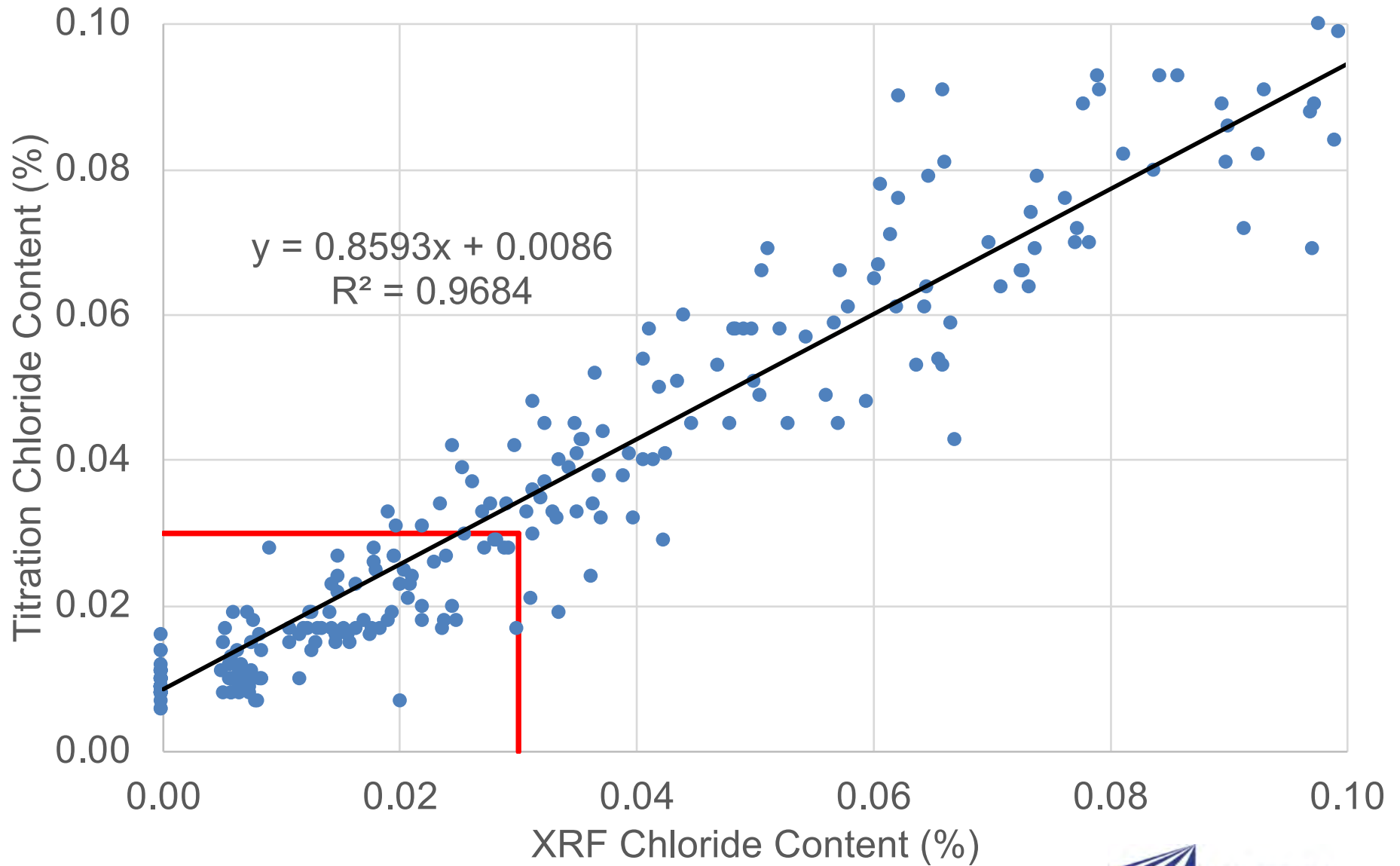
Pulverized & Pelletized Specimens



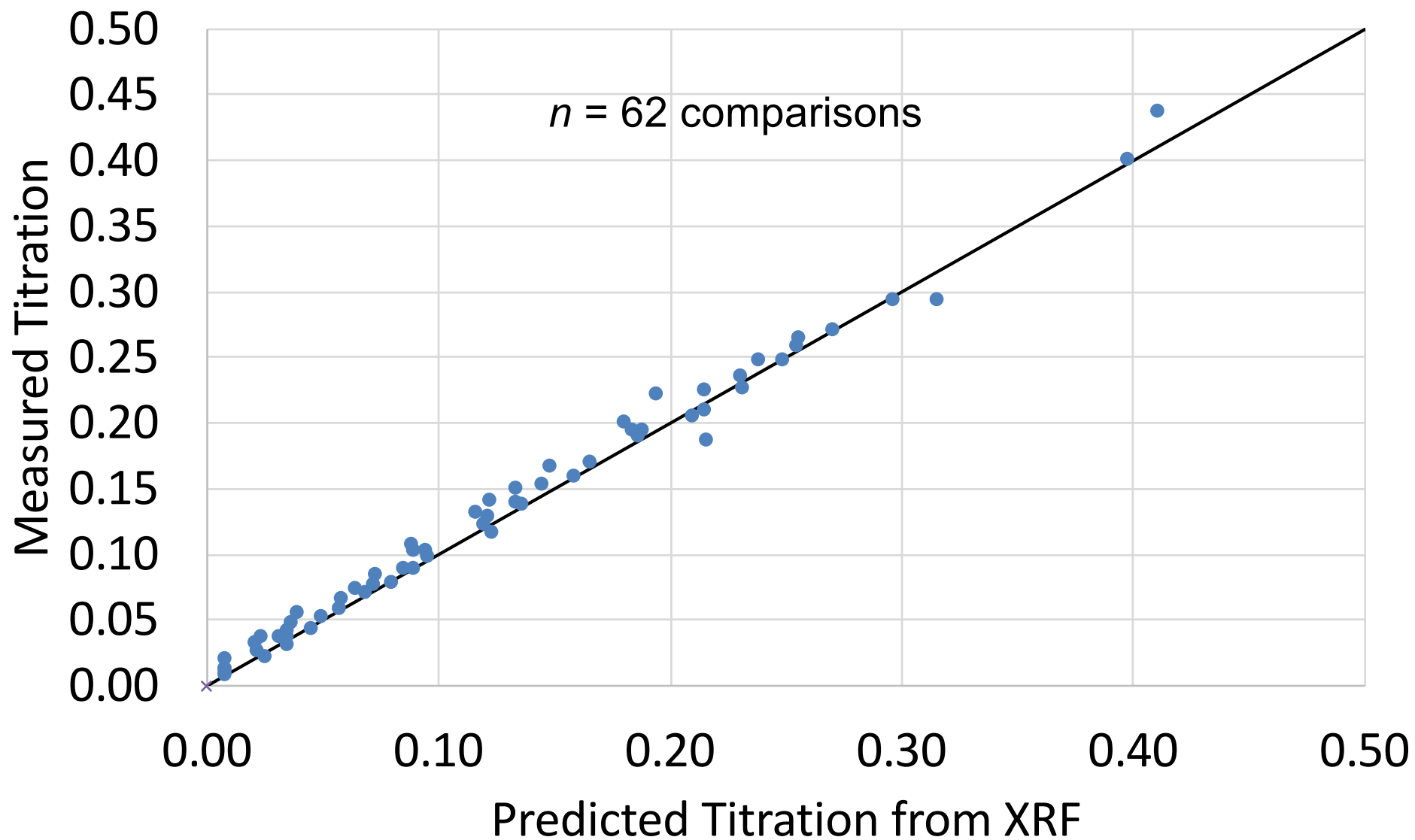
Split Sample Comparison



Split Sample Comparison



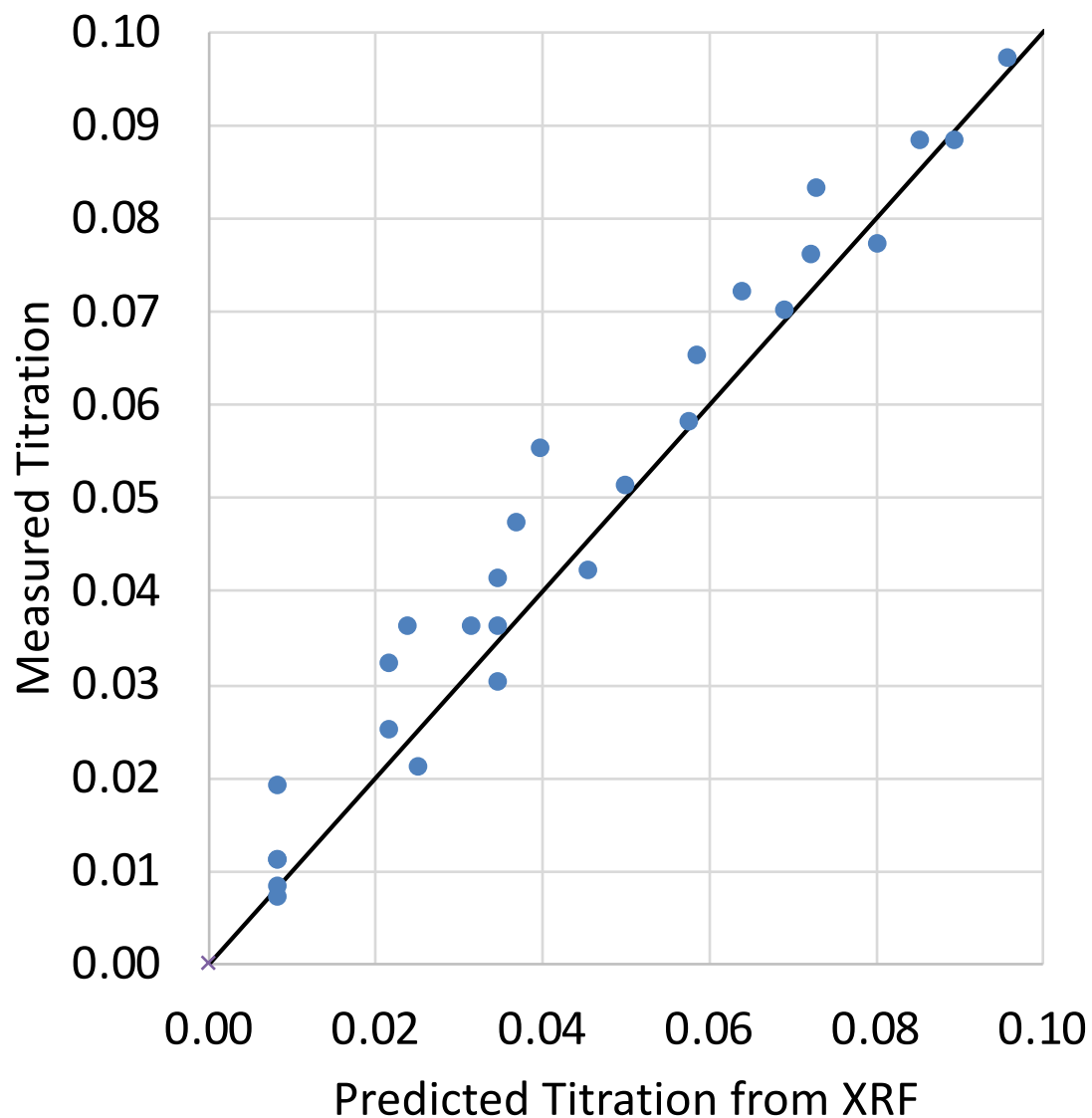
Model Validation



Model Validation

t-Test: Paired Two Sample for Means

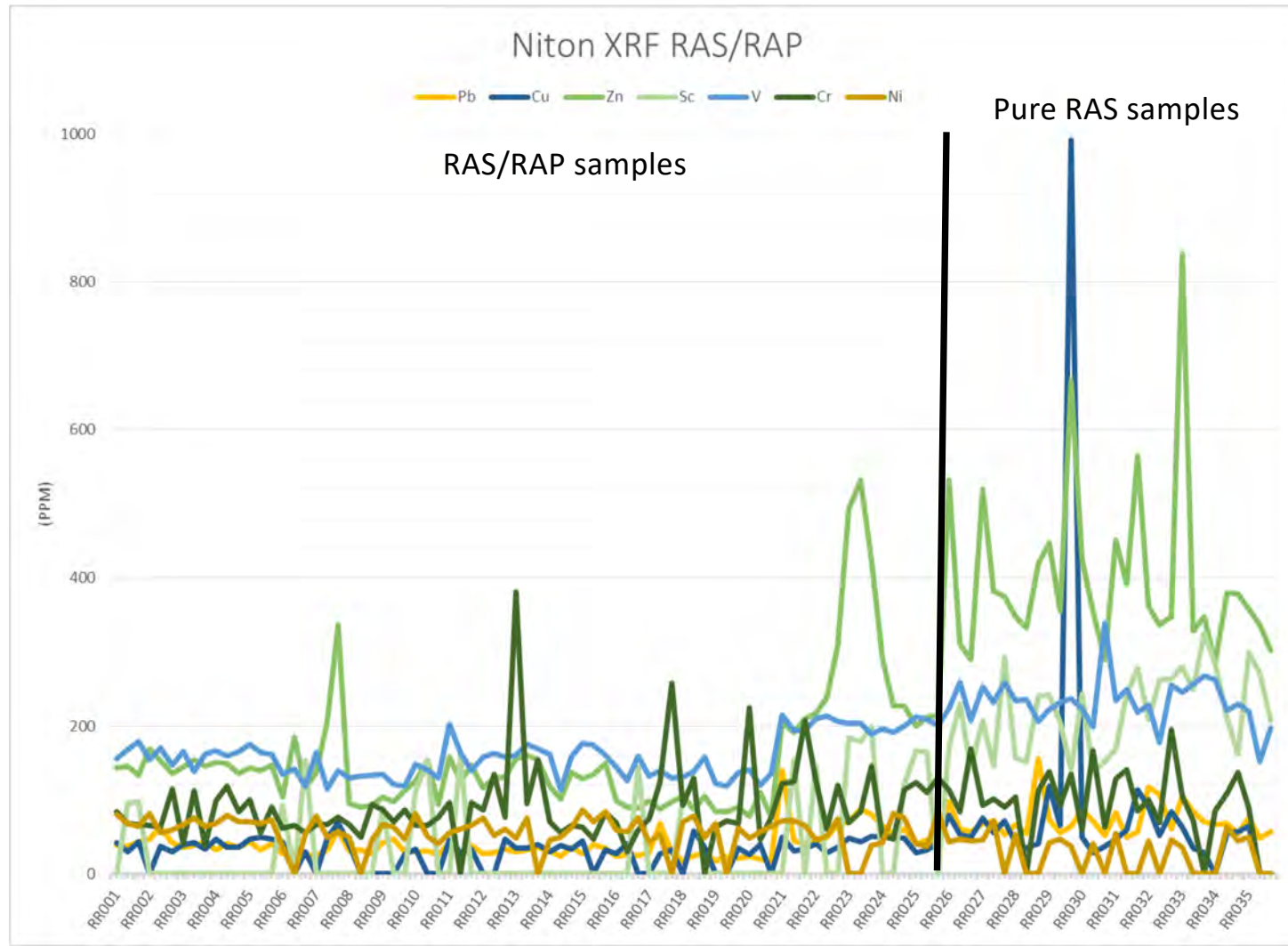
	Validation Titration (%)	Titration (%)
Mean	0.1298	0.1336
Variance	0.0095	0.0095
Observations	62	62
Pearson Correlation	0.995	
df	61	
t Stat	3.136	
P(T<=t) one- tail	0.0013	
t Critical one- tail	1.670	
P(T<=t) two- tail	0.0026	
t Critical two- tail	1.999	



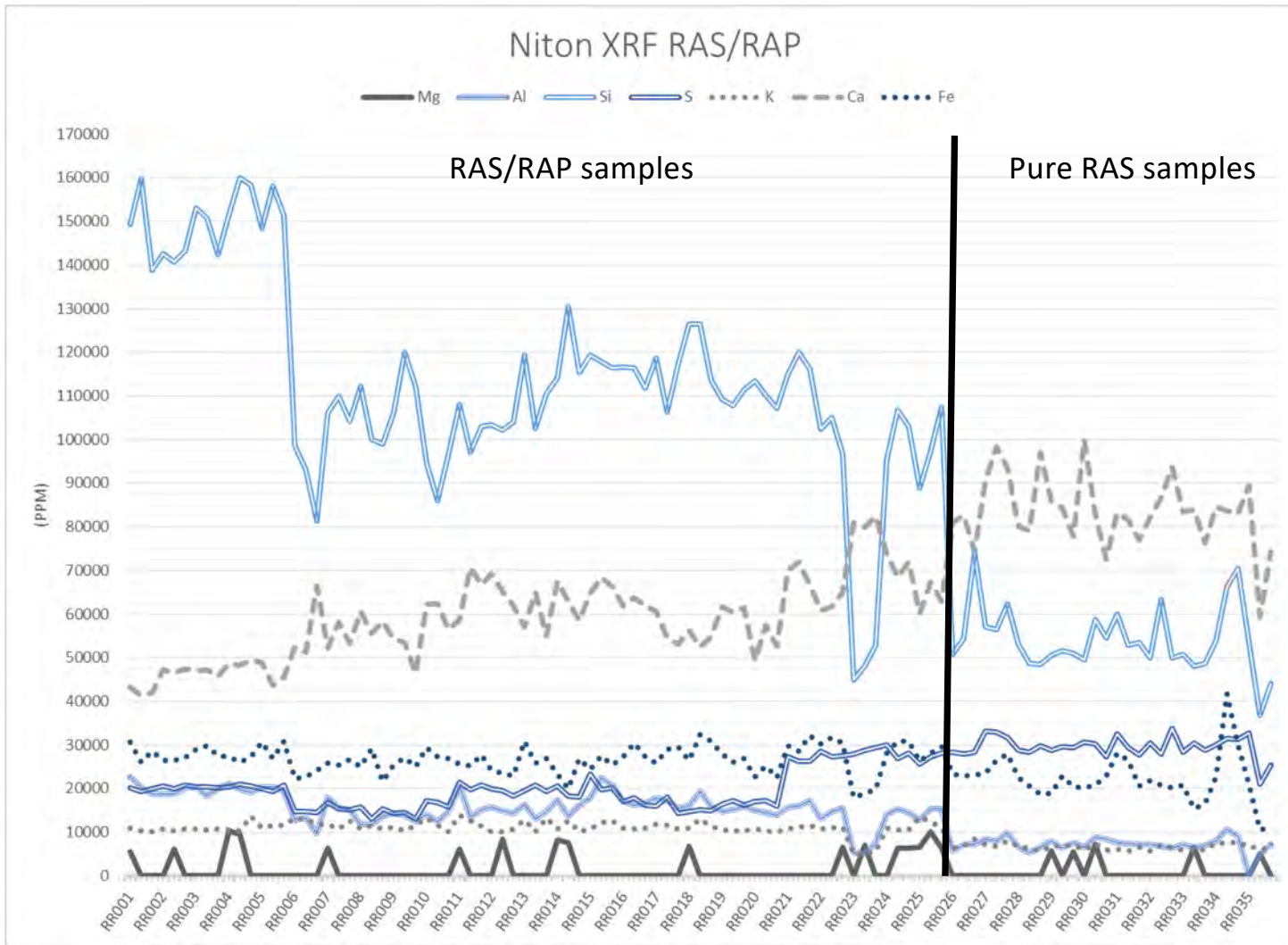
Chloride Content – Bridge Deck Cores

- Conclusions from study
 - Pellets of pulverized material superior to surface readings of slices
 - No binding agent required
 - In process of testing lab-prepared reference samples
 - In process of validating correlation with independent split-sample comparisons

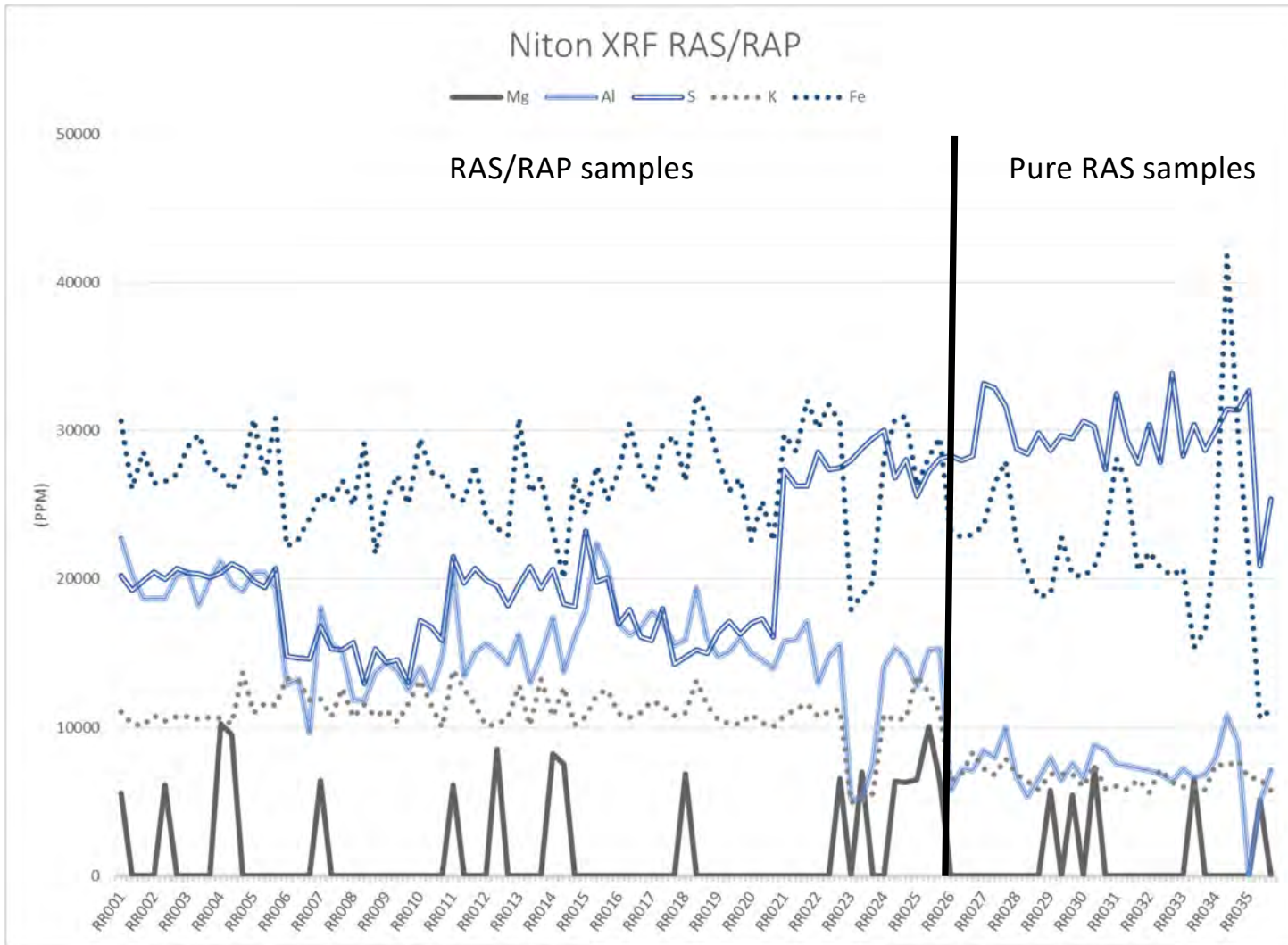
XRF RAS



XRF RAS

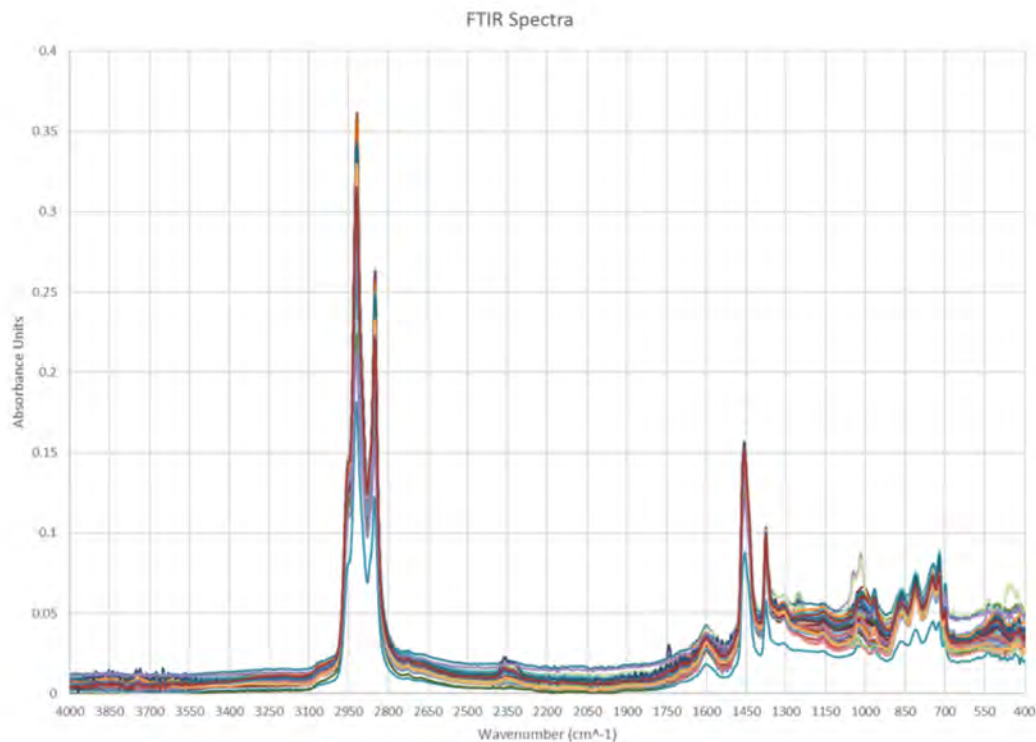


XRF RAS



Fourier Transform Infrared (FTIR)

- Identifies compounds
- Simple testing process
- Analysis more difficult than XRF

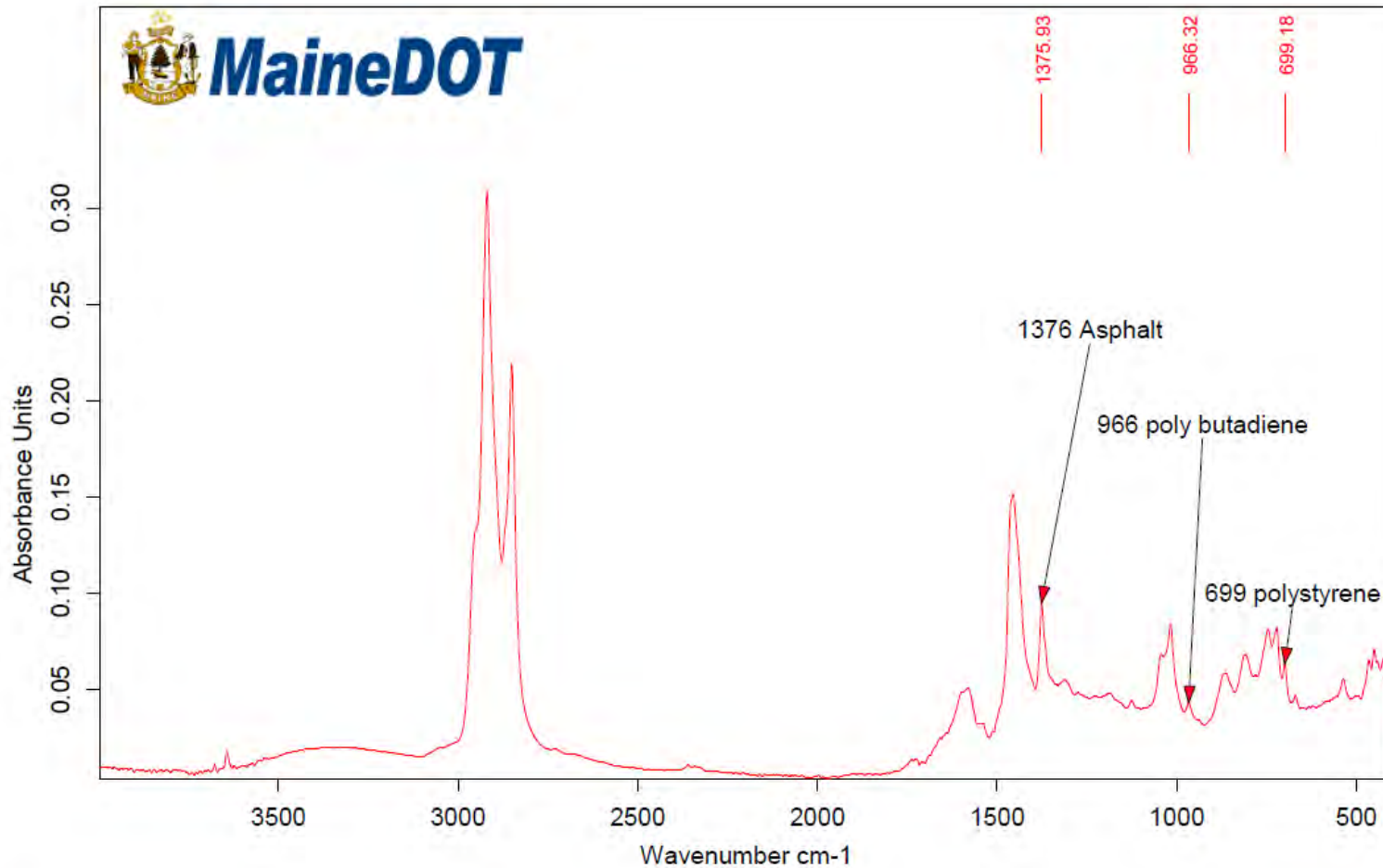


FTIR & XRF Testing of PGAB

- Conducting FTIR and XRF on every PGAB verification sample taken
 - FTIR for polymer identification and library
 - XRF for detecting presence of REOB



FTIR Testing of PGAB



C:\Alpha FTIR Data\HMA with 2%SBS.0

HMA with 2%SBS

Temp Controlled ATR

9/6/2017

Challenges

- Further verification for chloride content needed?
- Will need to develop procedures for chloride content determination
- Analyze FTIR PGAB data
- Others?

For More Information on R06B

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R06B Product

Page

Coming soon



MaineDOT