MaineDOT

Measuring the Chloride Content of Bridge Deck Core Using XRF

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XRF Advantages and Limitations

Advantages

- Pre-calibrated for a wide range of elements
- Automatic reading—no analysis experience required
- 1-3-minute testing time
- Little or no sample prep required
- No maintenance required—costs only associated with equipment acquisition ($35-$40K)
- Several applications possible in addition to the paint testing (more bang for your buck)

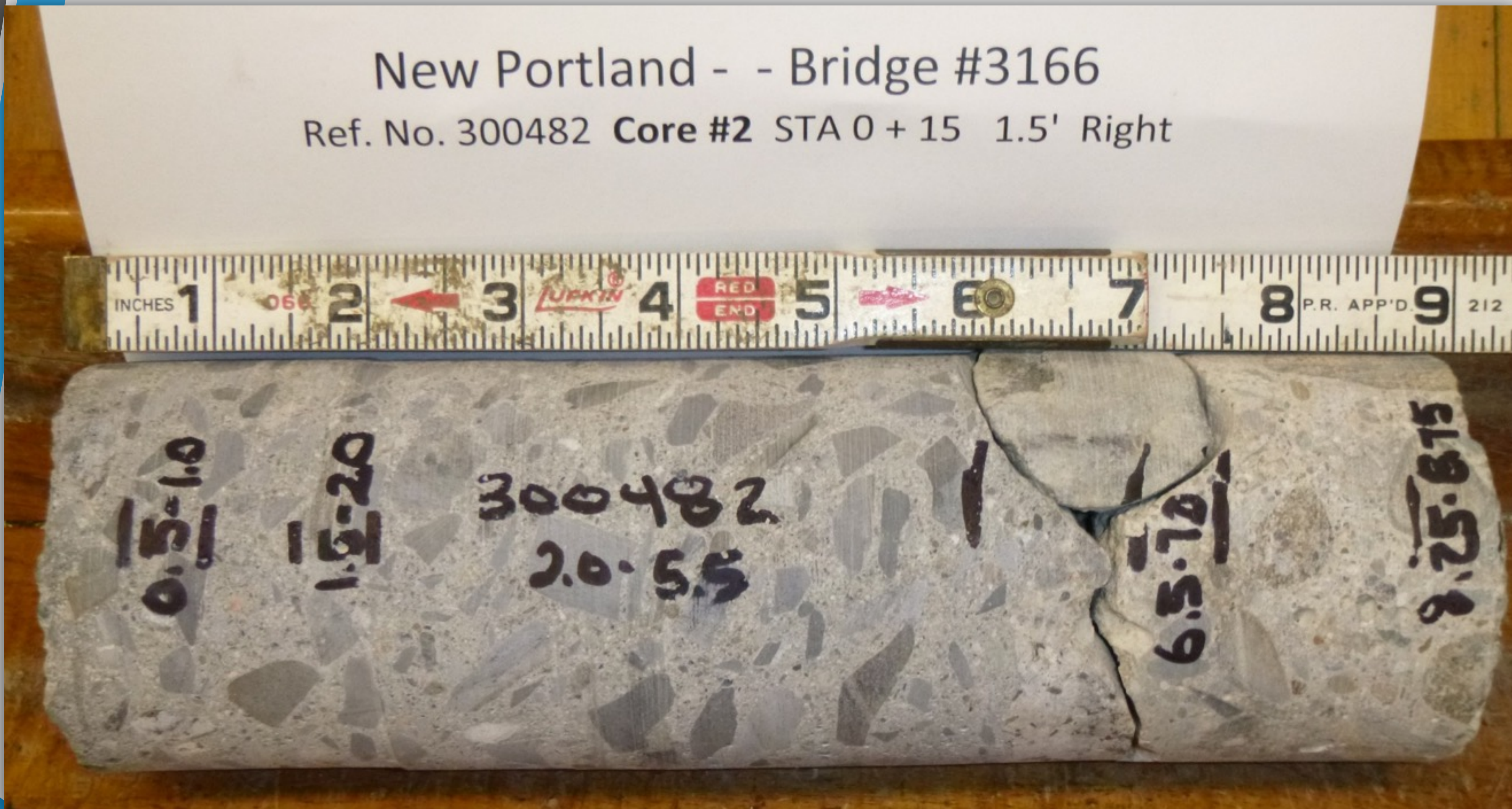
Limitations

- Can only be used by certified personnel
- Upper and lower limits—different calibrations needed for trace metals vs. ores (just a cost consideration)

SHRP2 R06B—MaineDOT

- MaineDOT goals for R06B:
  - Maximize non-destructive testing
  - Reduce test time and cost
  - Reduce incorporation of out-of-spec material into DOT work

Chloride Content – Bridge Deck Cores



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

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

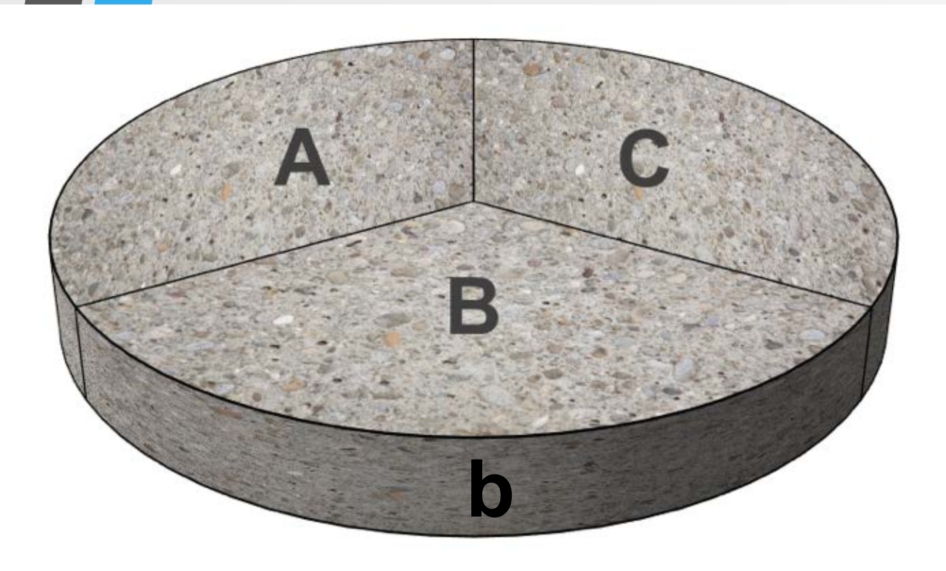
Chloride Content – Bridge Deck Cores

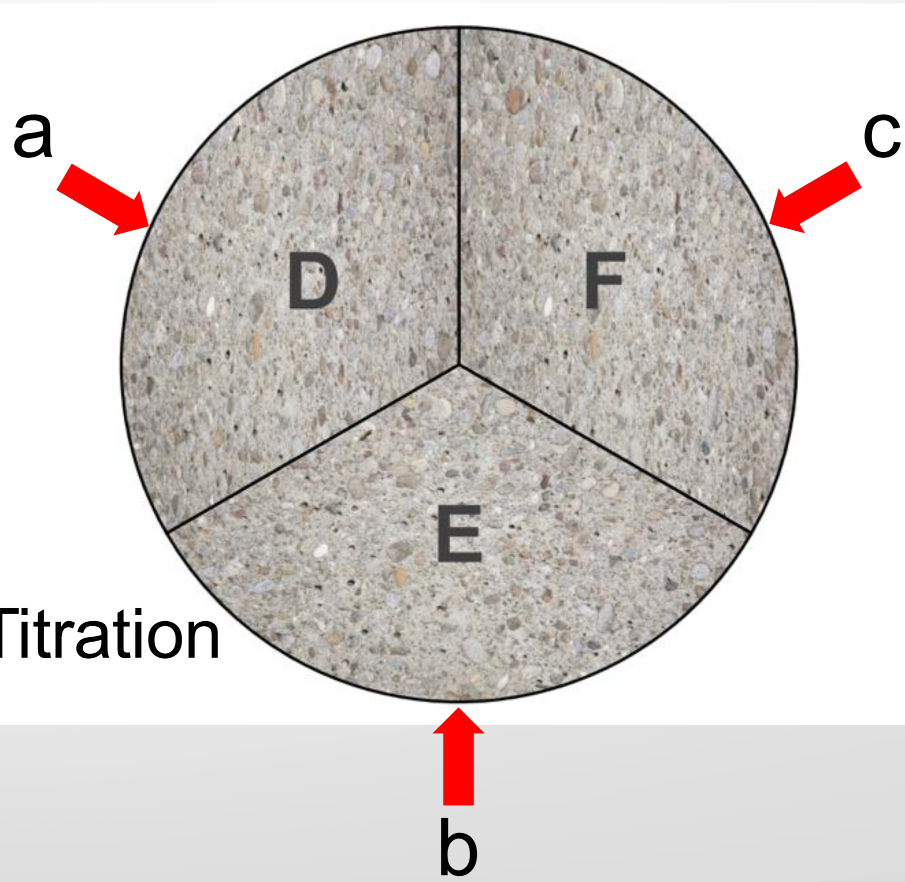
XRF for Chloride Content
Initial Experiment

- 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

XRF for Chloride Content
Surface Testing of Core Slices

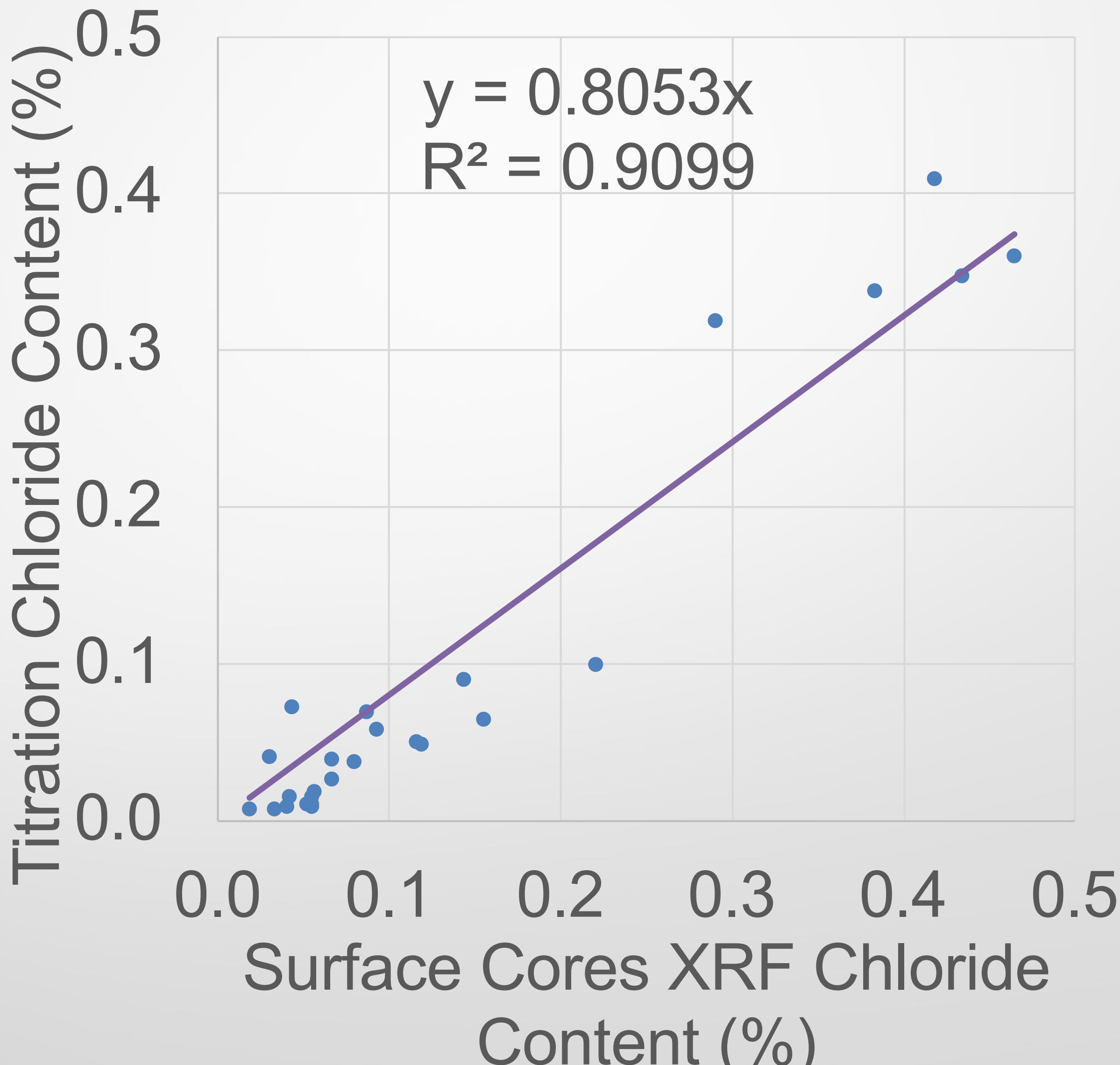




- Top, bottom, edge of slice
- Average of all readings v. Titration

XRF for Chloride Content
Surface Testing of Core Slices

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



Pulverized & Pelletized Specimens

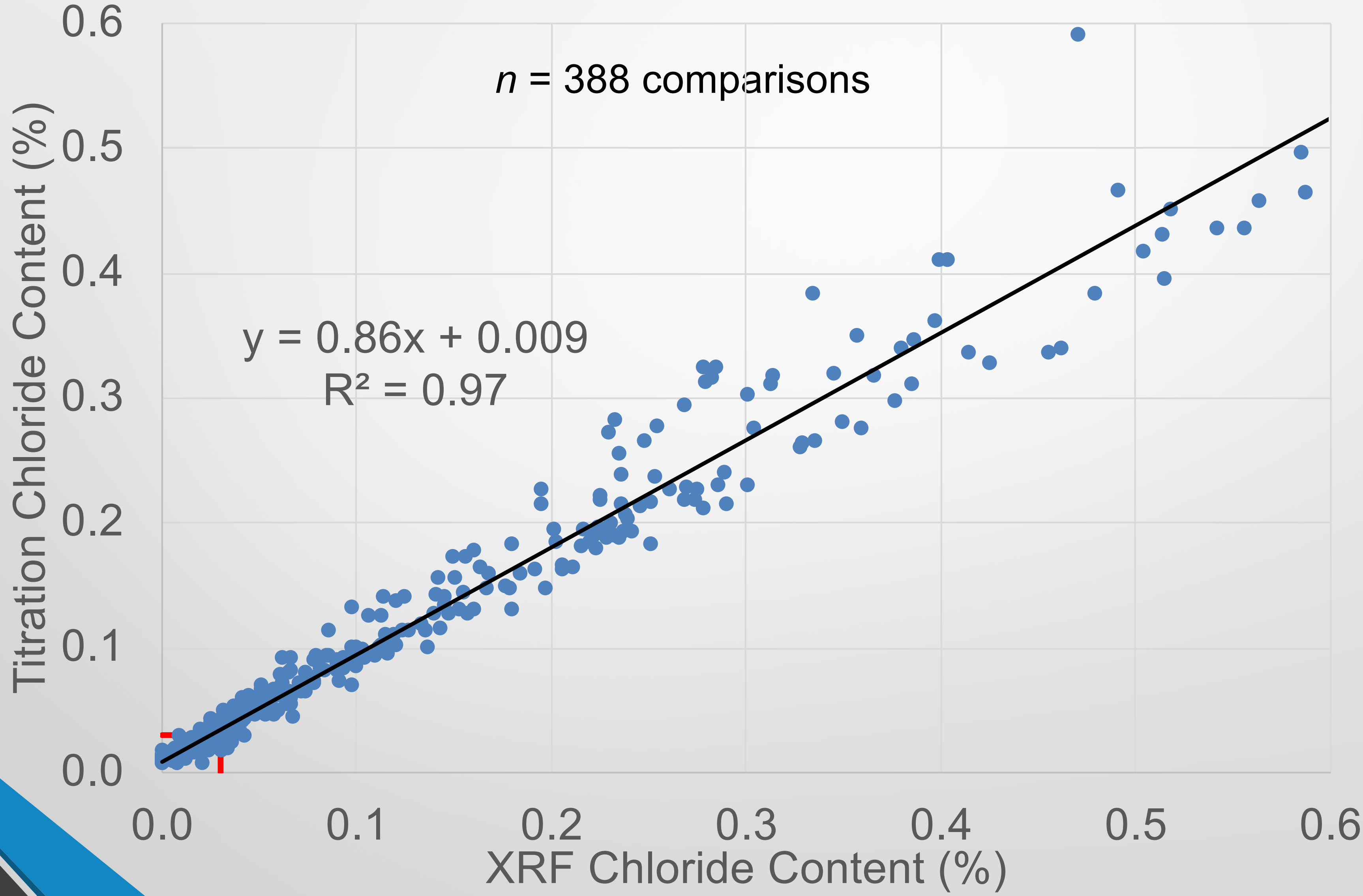
Pulverized & Pelletized Specimens

XRF for Chloride Content
Pellets from Cores

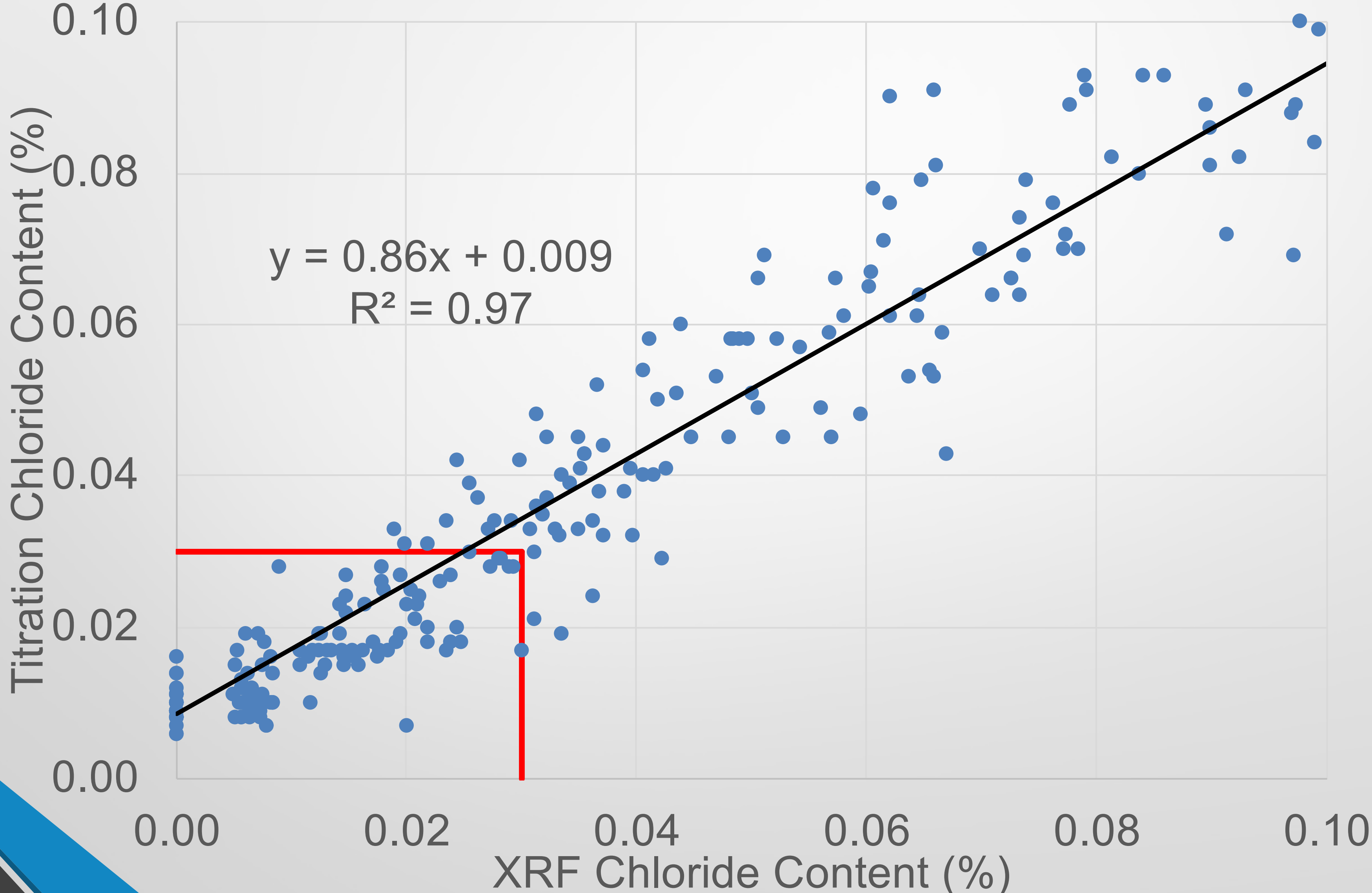
Mode/Range @ 60 Sec.	Binding Agent	% Binding Agent	R^2	Coefficient
Mining Ta/Hf 5/5/5/45	A	5	0.996445	1.091516
AllGeo 5/5/5/45	B	5	0.996009	1.142771
Mining Cu/Zn 5/5/5/45	A	5	0.995589	1.078925
AllGeo 5/5/5/45	None	---	0.99518	0.993099
Mining Ta/Hf 5/5/5/45	B	5	0.994987	1.145006
AllGeo 5/5/5/45	A	5	0.99459	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

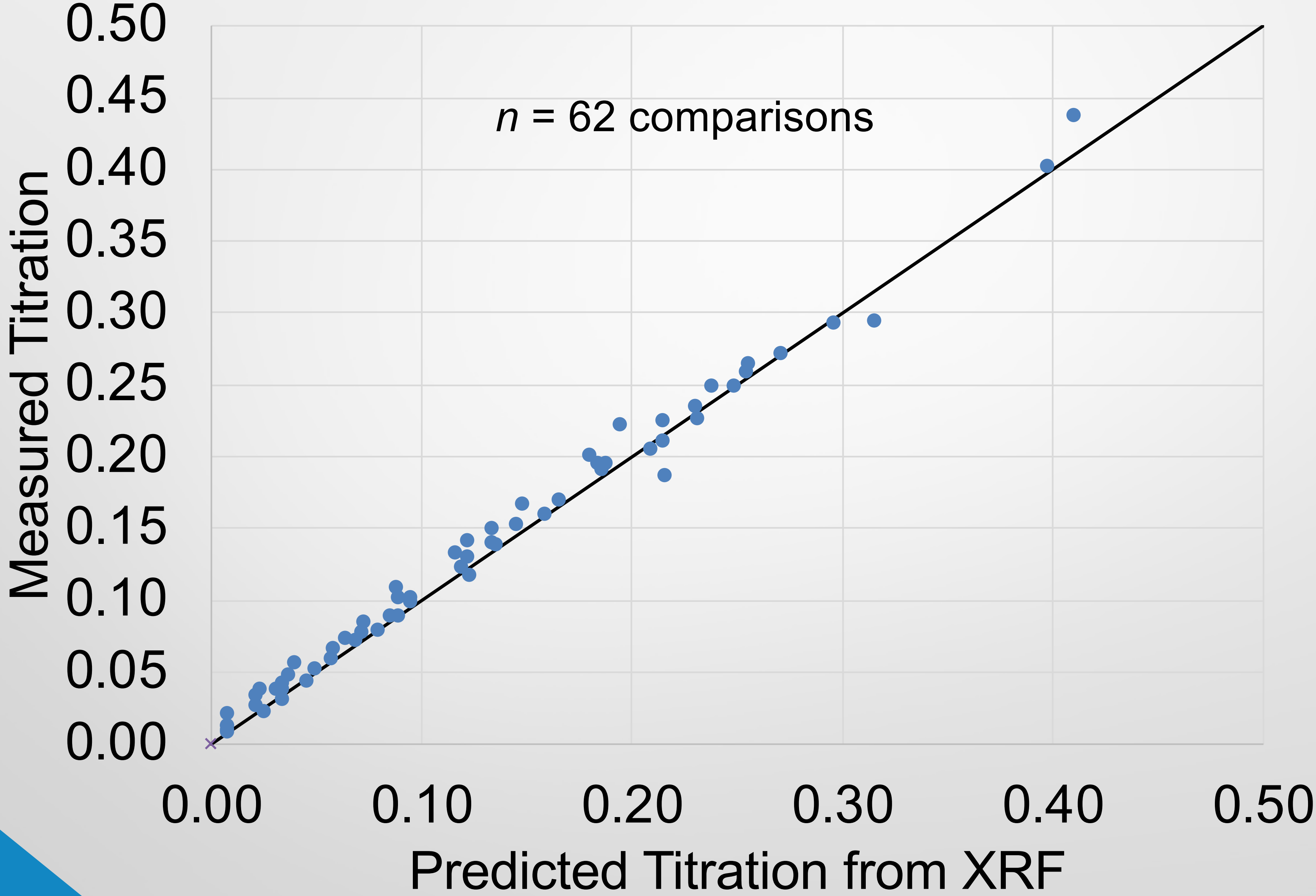
Split Sample Comparison



Split Sample Comparison

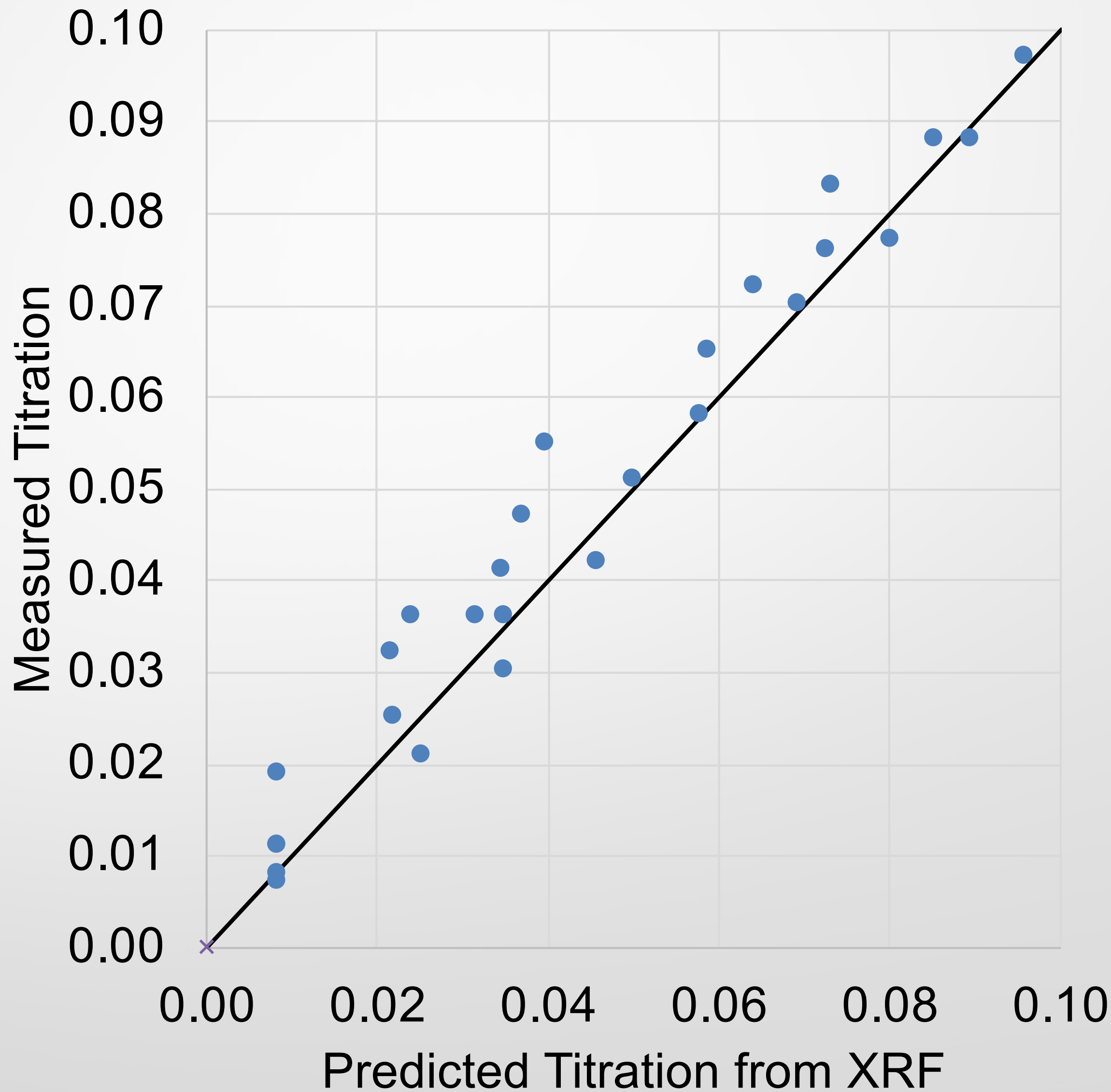


Model Validation



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	



XRF for Chloride Content
Initial Findings & Challenges

- Pellets of pulverized material superior to surface readings of slices
- No binding agent required
- Correlation between titration and XRF reading excellent

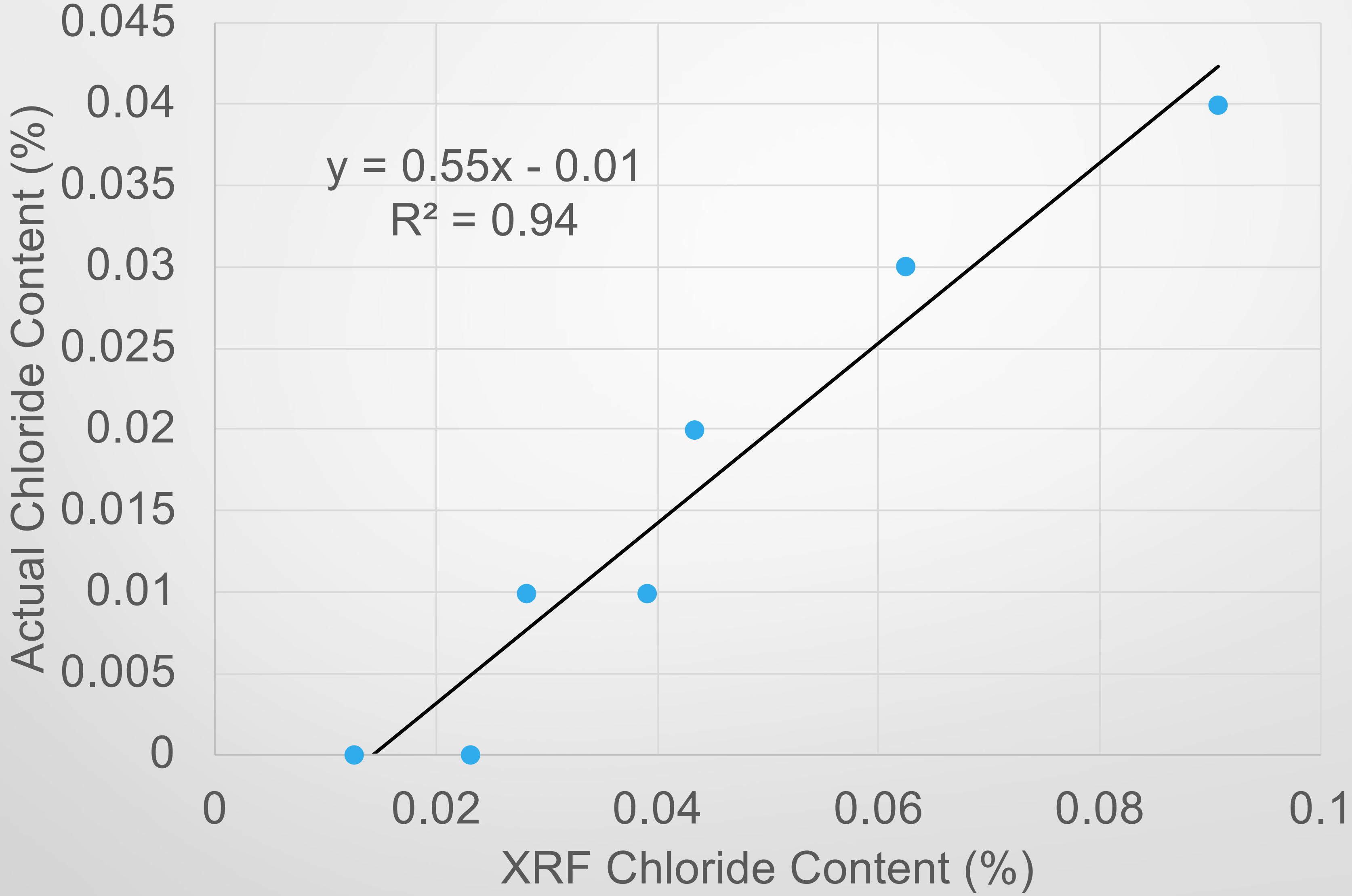
Next Steps in Investigation

- Testing of lab-prepared reference samples
- Investigate the stability of measurement of chloride content with time due to concerns about “drift”

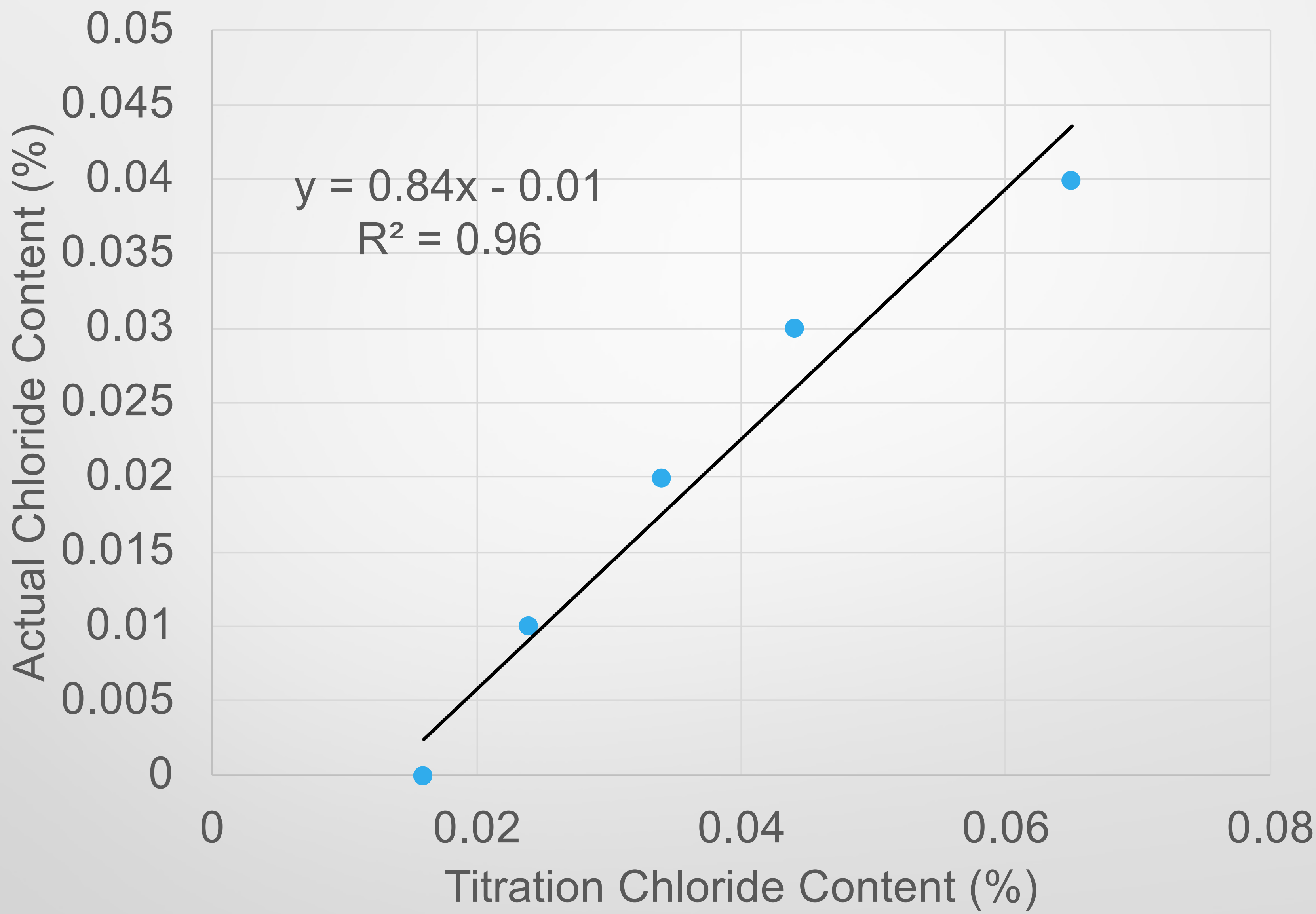
XRF for Chloride Content
Lab-Prepared Reference Samples

- Most elements detected with XRF have known standards and references – used as a quality check
- No known available standards for chlorine since it is a lighter element
- All data comparisons have been between XRF and titration – **but how does it predict actual chloride content?**
- Reference concrete samples with known chloride contents fabricated in the lab (0%, 0.01%, 0.02%, 0.03%, 0.04%)
  - Tested via XRF and AASHTO T 260

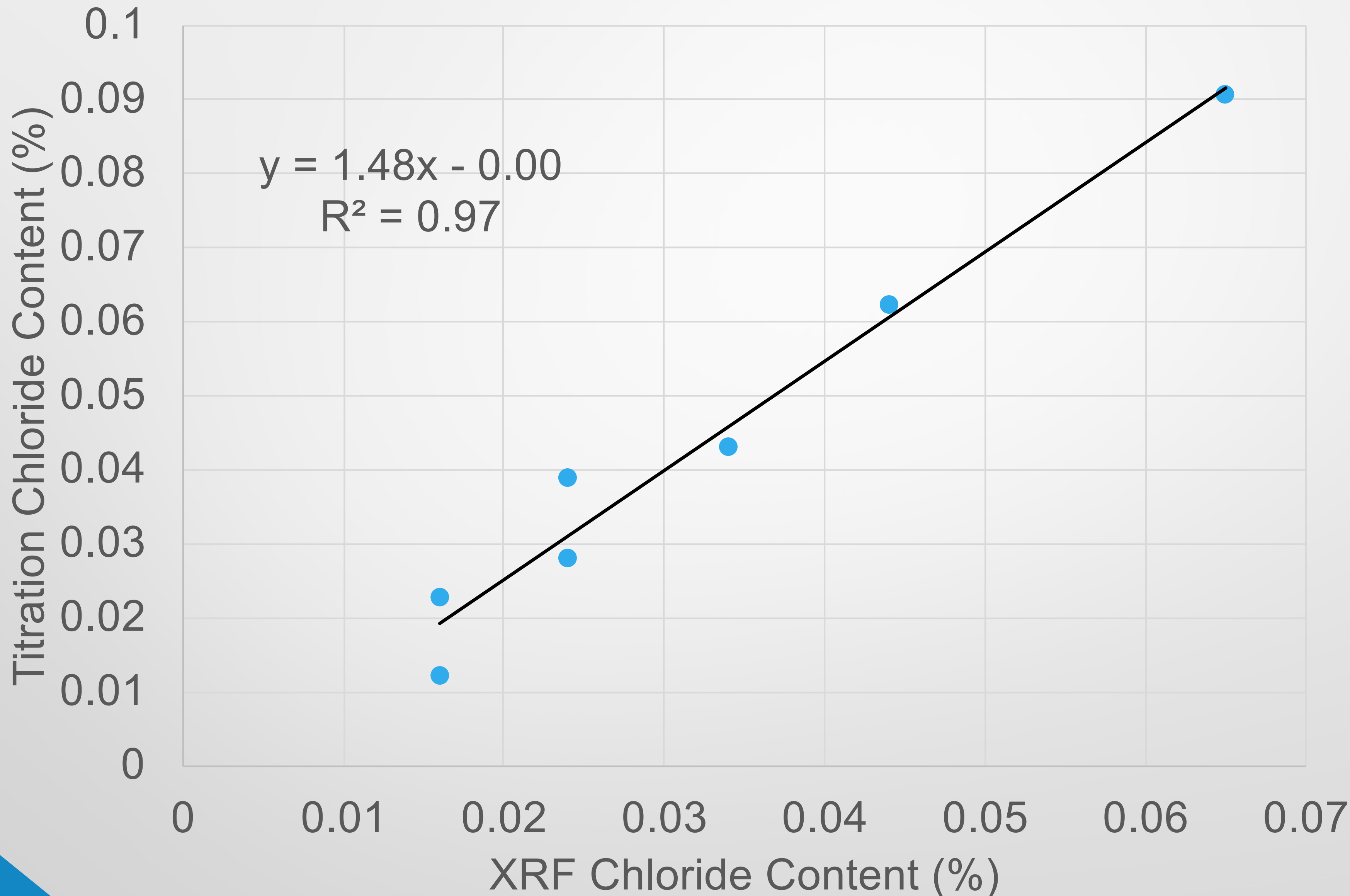
Actual vs. XRF



Actual vs. Titration



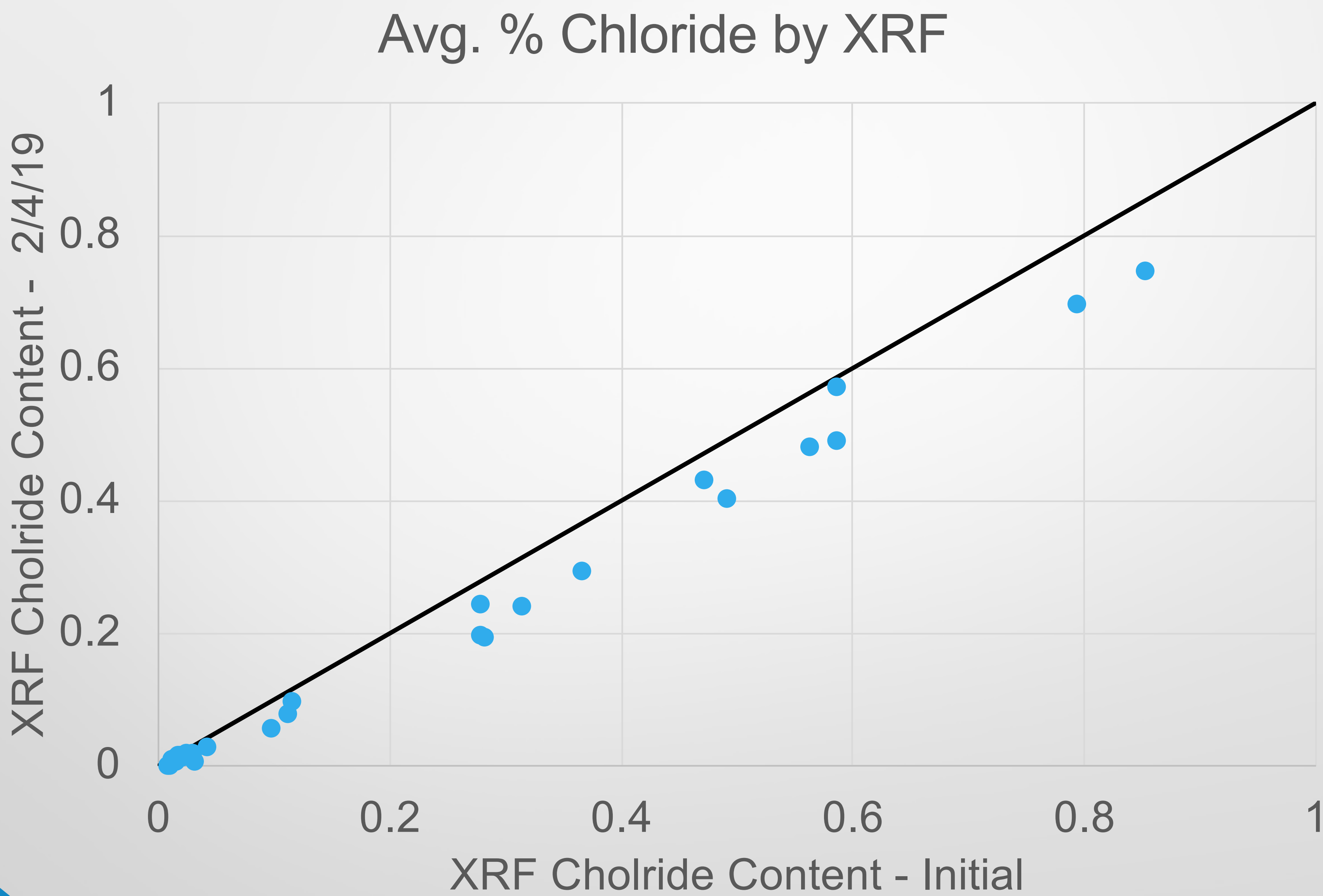
Titration vs. XRF



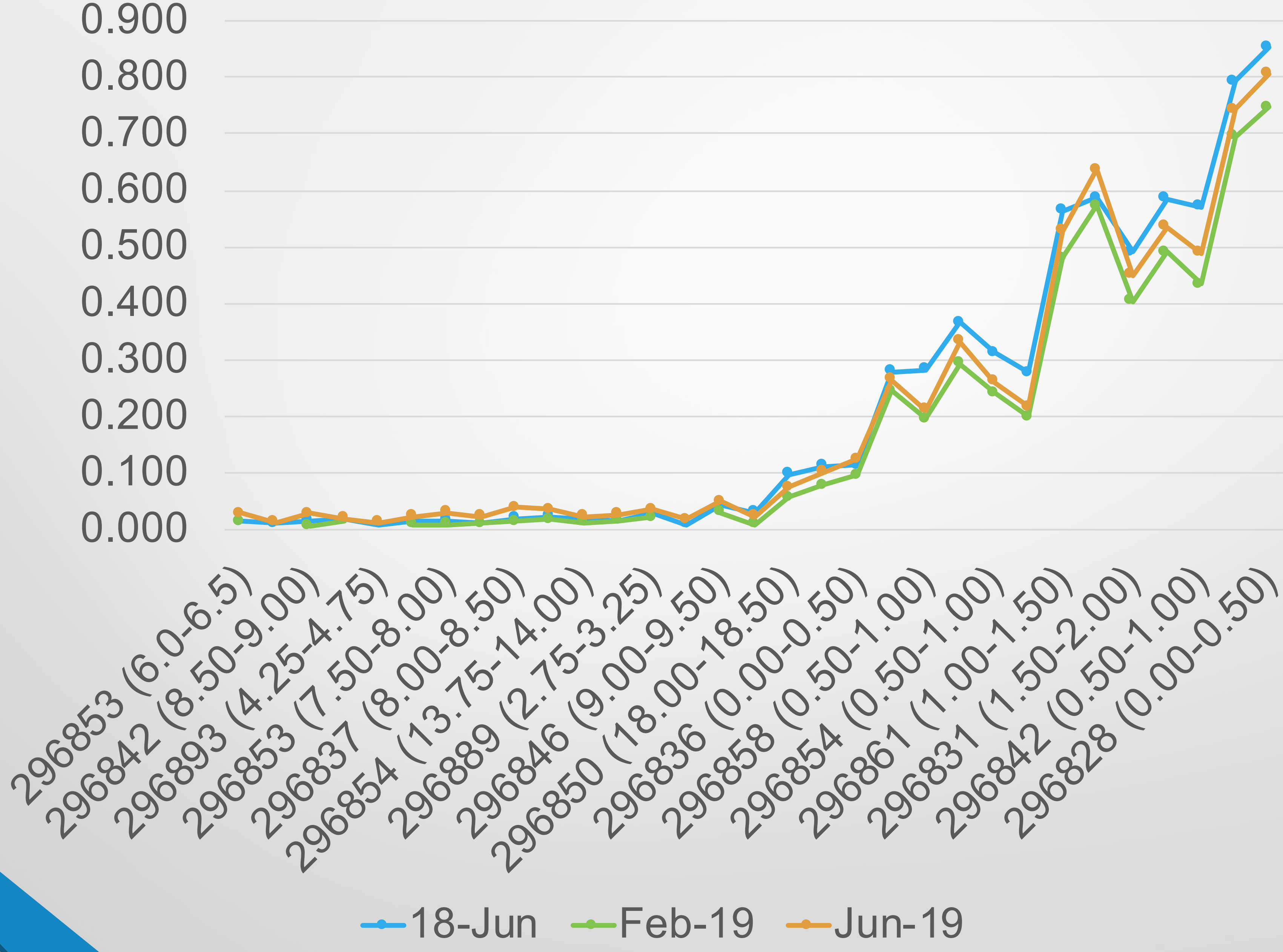
XRF for Chloride Content
Repeatability of Measurements Over Time

- All data comparisons have been between XRF and titration – **but how stable is the measurement of the pellet?**

Chloride Content Over Time



Chloride Content Over Time



XRF for Chloride Content
Where are we now?

- Need to resolve the time dependence of the pellets
  - May need to develop guidance on a time limit to test pellets within
- Looking into higher grade equipment with better resolution for the lighter elements
  - Action limit is so low compared to typical measurements
  - Hand-held model may not be the best piece of equipment for the use
- Still running titrations and XRF in parallel but would like to transition away from titrations soon

Questions?