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ADVANCED AT-LINE KAPPA MEASUREMENTS WITH NIR-SPECTROSCOPY FOR FIBRELINE OPTIMIZATION

FITNIR ANALYZERS INC.

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FIT NIR
The Next Generation of Process Analyzers

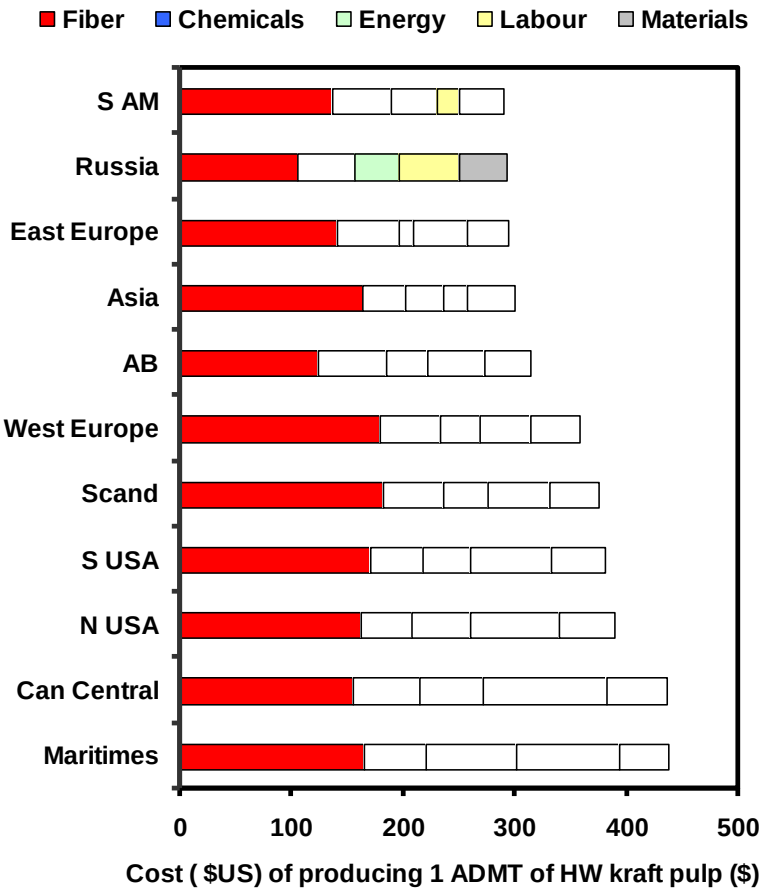
Realização: ABTCP

Correalização: ibá indústria brasileira de árvores, IPEF

Outline

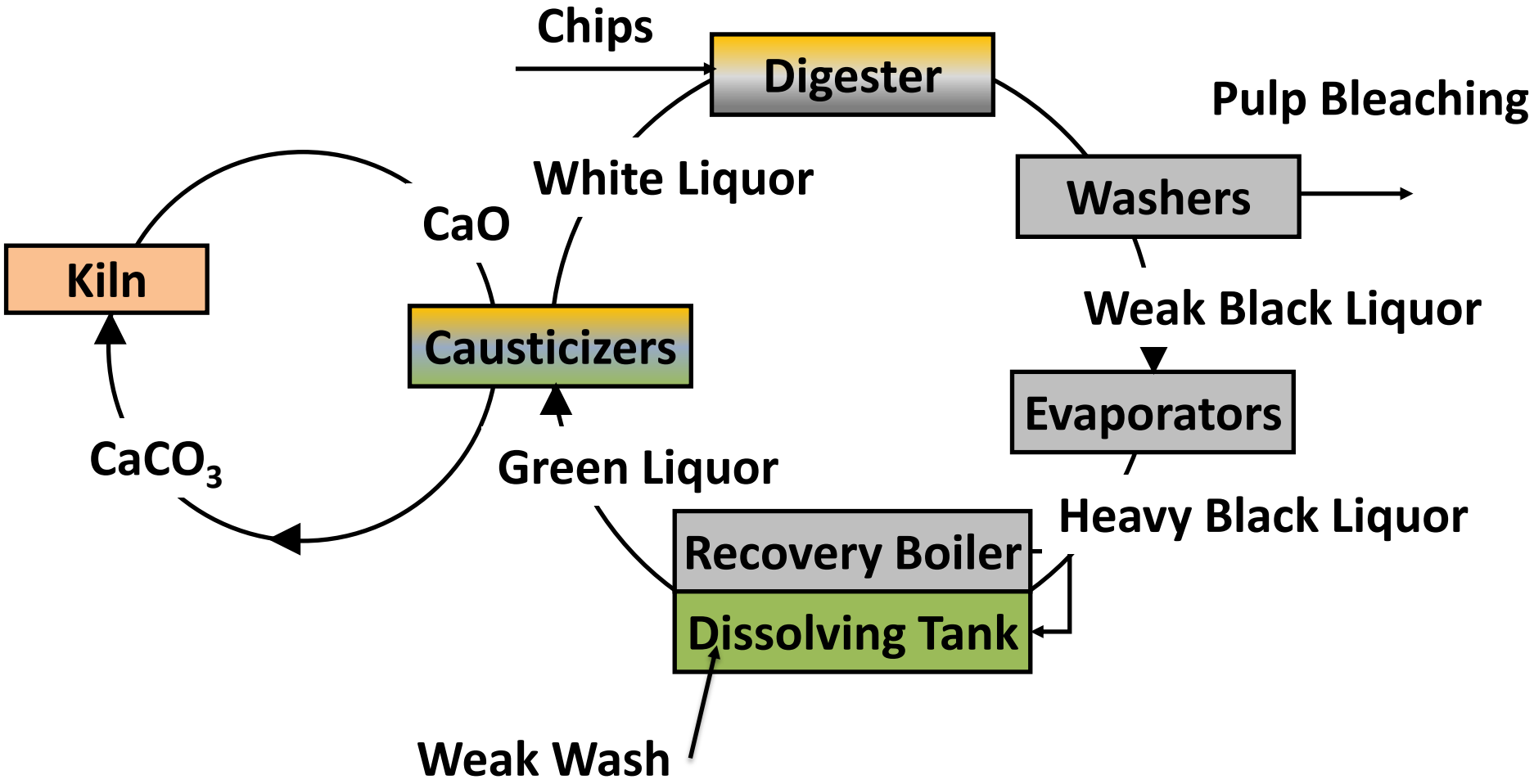
- Overview kappa measurements and it's importance for kraft mill
- Available technologies
- Installation and implementation of NIR analyzer
- Results from mill
  - Wet pulp kappa for stockline
  - Dry pulp sheet kappa for final product check/verification
  - Other applications
- Learnings and best practices
- Summary and conclusions

Variable-Costs in Pulp Production



- Five main components to variable costs:
  - Fiber
  - Chemicals
  - Energy
  - Labour
  - Maintenance
- To improve margins
  - Reduce internal variable costs
  - Improve efficiencies
  - Improve yield, product quality

Mill Optimization Opportunities



Top Factors Influencing Digester

- Pulping chemistry: WL EA/AA, sulfidity
- Wood species and anatomy
- Chip size distribution
- Chip quality (MC, others)
- Mill digester operations (time & temperature)
- Critical to measure the WBL REA for fast feedback
- All factors influence Kappa number:
  - Critical for feedback for digester as well as feedforward for bleach plant.

Current Standard Kappa Testing

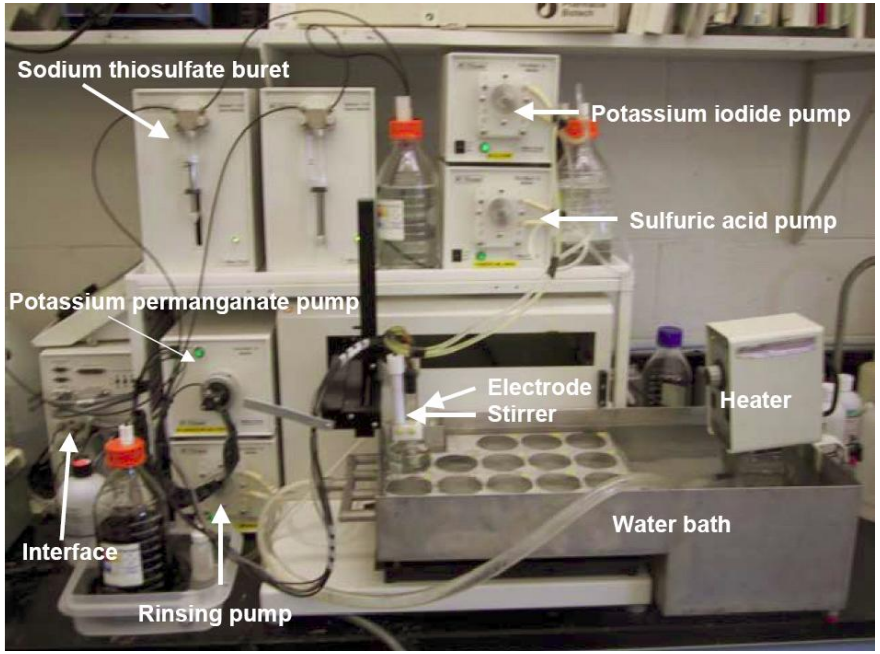
- **KMnO4 Titration:**
  - **First developed by Tasman and Berzins (1957)**
  - **Measures the residual lignin content of kraft and semi-chemical pulps**
  - **Kappa < 5 requires larger amount (>10g OD pulp)**
    - **Method requires 30 – 70% KMnO4 consumption**
  - **Kappa > 50 requires small amount (< 1g OD)**
    - **Higher shives, resinous bundles, uncooked fibres**
    - **Leads to greater error**

Pulp Kappa Measurements

- **Manual kappa titration suffers from many errors:**
  - **Shives, pins, and non-representative fibres**
  - **Inaccurate weight of samples**
  - **Liquor contamination**
  - **Chemical concentrations**
  - **Reaction time (10min.)**
- **Discrepancies can be observed between testers and between labs.**
- **Time consuming, resulting in low frequency of analysis**
- **To date, accurate and rapid kappa determination remains a challenge to the industry, especially with varying chip quality**

Benchtop Auto-titration

- Jiang, Audet, van Lierop, and Berry
  - Adapted an autotitrator technique to micro kappa
  - Automated sample tray for up to 17 samples
  - Improved accuracy and std. dev.
  - Still mimics standard titration with measurement time of ~ 20 min. per sample
  - Excellent lab technique – reference method

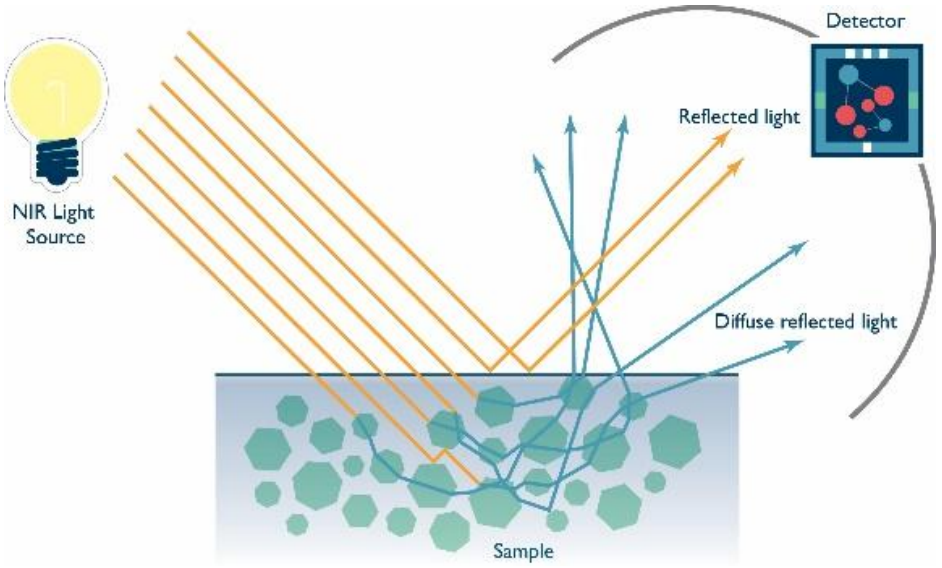


Online Kappa Analyzer

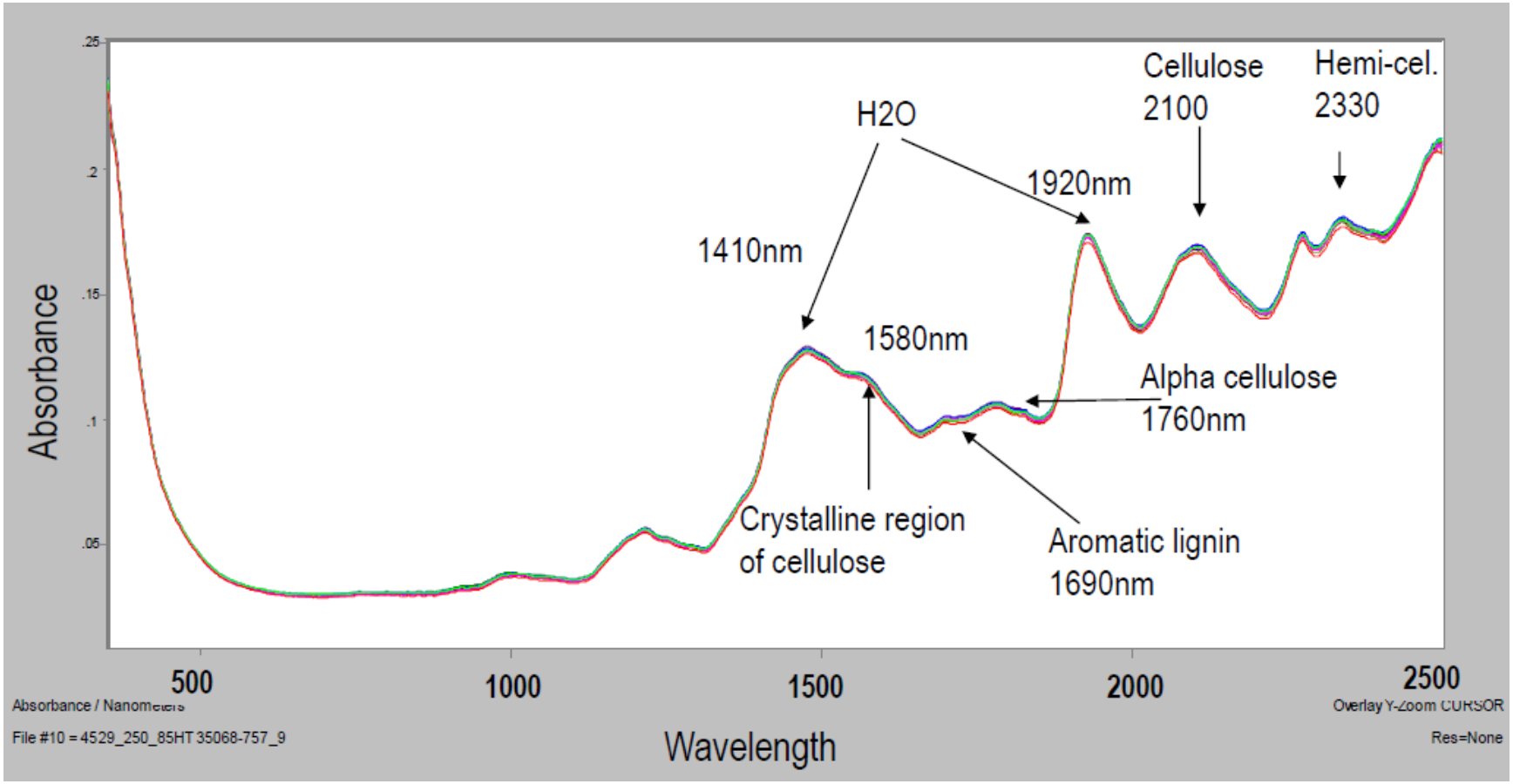
- **Photometric titrations – adapted based on KMnO4, KI absorbance peak**
  - **Monitors abs. loss with addition of thiosulphate.**
- **UV-based analyzer**
  - **Rely on UV and visible spectrophotometer**
  - **Multiple – discrete wavelength (species sensitive)**
  - **Well established and accepted**
  - **Reported to require higher maintenance**
  - **May not be well suited for high (>70) kappa due to low consistency requirements**
- **Insertion probe/single point**
  - **Promising technique and cost effective**
- **Industry still requires rapid, accurate benchtop kappa measurements**

Near-Infrared (NIR) Spectroscopy

- Energy from NIR light is absorbed by the lignin
- Remaining light is diffuse Reflected
- Optics collect diffuse reflected light
- Infrared detector registers absorption
- Spectrum contains unique features



Spectrum of Dry Pulp Sheet



NIR Kappa Analyzer Installation

Demin. Water Booster and Filter

- Booster pump to pump demin. water to 80 psi
- Wash pulp to reduce chemical residuals
- Floc pulp in suspension to allow good pad formation

Sample Preparation Device

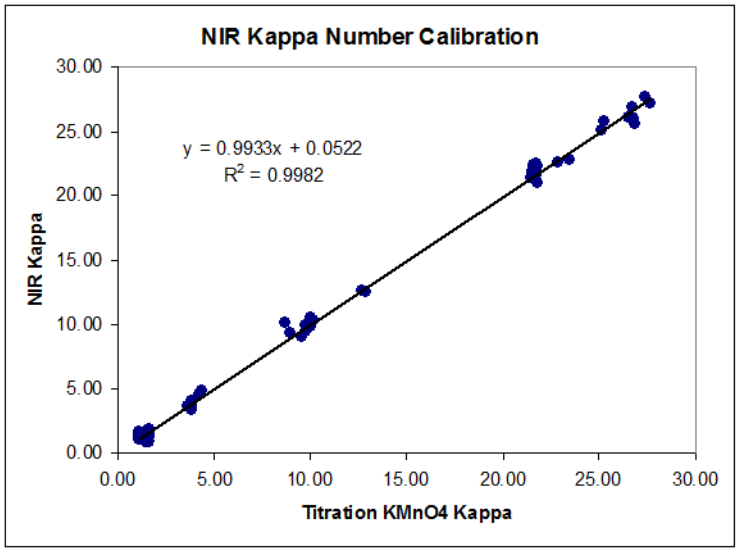
- Sample preparation station used to produce sample pad
- Pulp samples thoroughly washed
- Piston dewaters and presses to form pad, ~1/2” thick

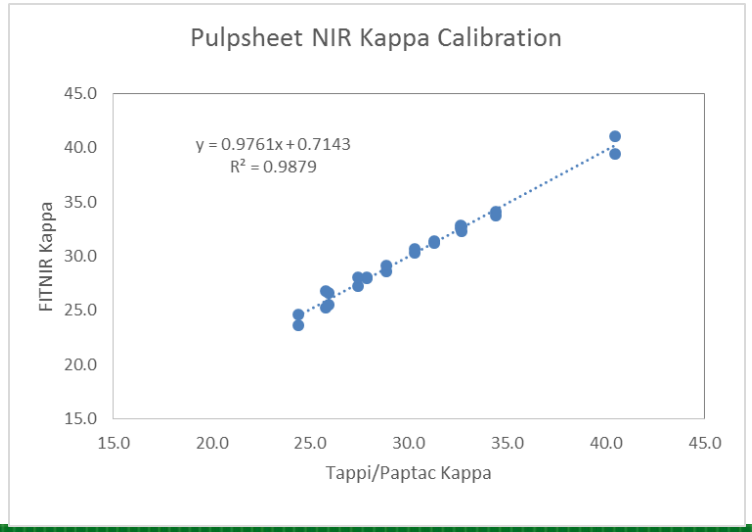
Sample Analysis

- Blank is scanned first run to zero the analyzer
- Wet (or dry) pulp sample is placed in sample chamber
- Sample type/location and collection time entered
- Analysis time ~ 15 secs
- Results displayed on screen or DCS

Analyzer Calibration and Performance

- Spectral data from analyzer is then correlated with reference values
  - Kappa of wet pulp
  - Kappa of dry pulp sheet
  - Possibility of other properties:
    - Pulp viscosity
    - Airdry content
    - S10 and S18
    - Etc.



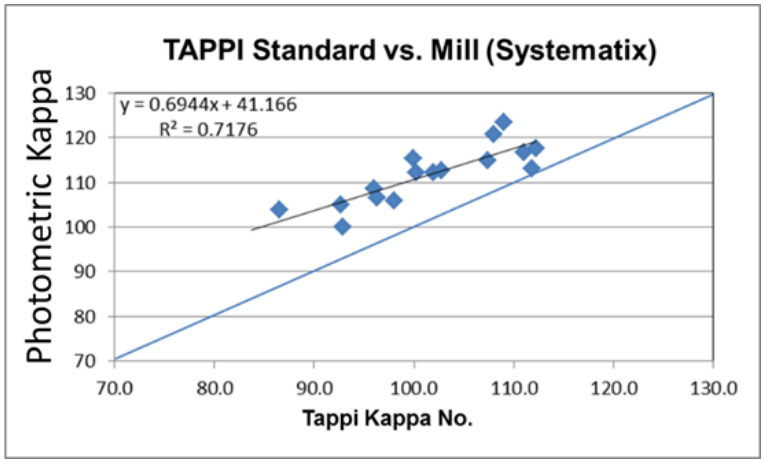


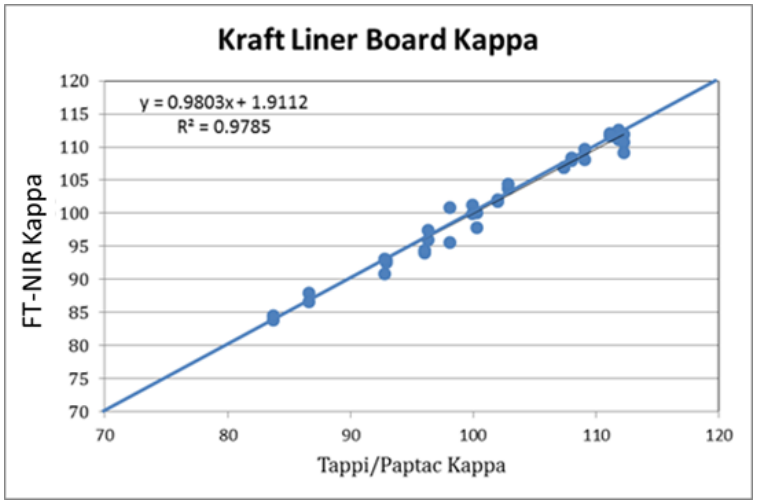
KRAFT LINERBOARD APPLICATION

Wet Pulp Kappa Analysis

Kraft Linerboard Application

- Mill-A produces approx. 1200 t/d of linerboard pulp
- Has been utilizing an obsolete photometric kappa analyzer at the digester testing lab.
  - Results showed large variations shift-to-shift
- Comparison with TAPPI method showed ~10 point offset
- NIR data showed excellent linearity with TAPPI method



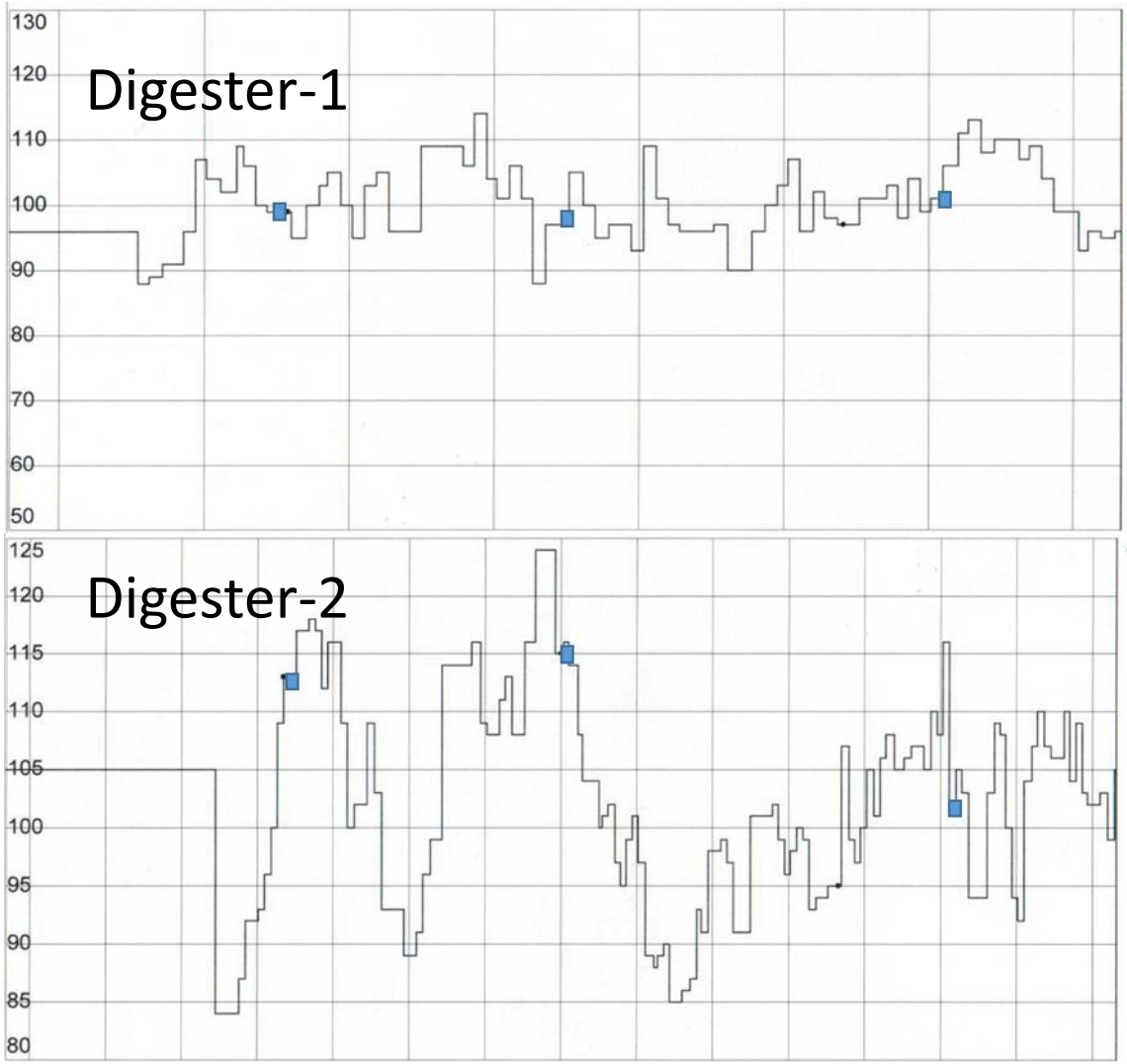


Crosschecks with Mill TAPPI Titrations

Sample #	Digester	Standard Kappa*	Analyzer Kappa
S1	A	112.9	112
S 2	B	111.1	111
S 3	B	114.4	113
S 4	B	114.6	114
S 5	B	115.6	115
S 6	B	115.1	115
S 7	A	104.7	105
S 8	A	109.3	110
S 9	A	91.7	91
S 10	B	102.9	103

RMSEP = 0.68 Kappa

Analyzer Measurements @ Start-up



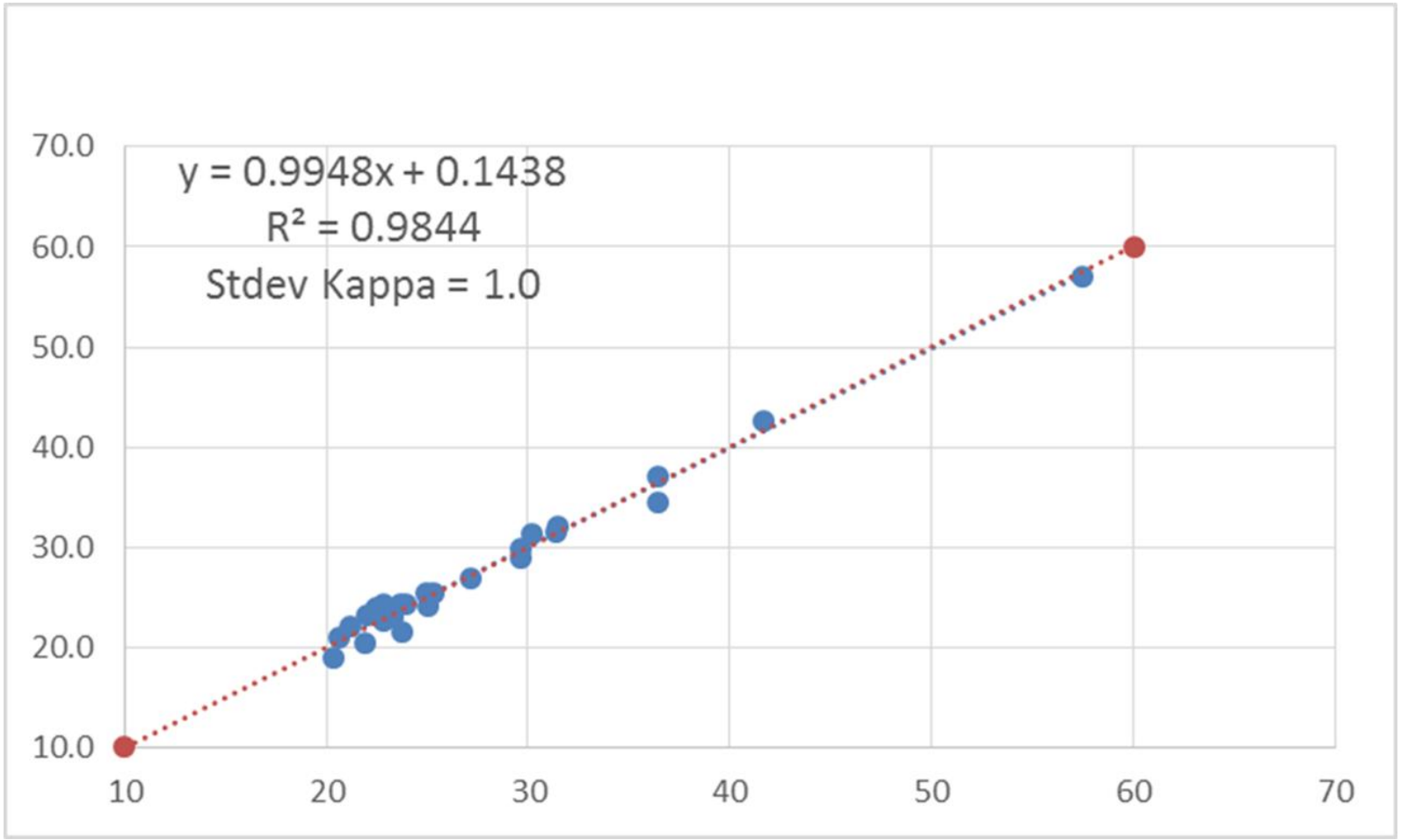
CONVENTIONAL KAMYR APPLICATION

Wet Pulp Kappa Analysis

Conventional Kamyr Kappa Testing

Sample No.	Location	Tester 1	Tester 2 (Control)	NIR Kappa
1	Blow-line	39.6	38.2	38.15
2	Upper Ex	41.4	40.5	40.4
3	Decker	37.7	36.4	36.1
4	Blow-line	27.7	29.6	30.2
5	Upper Ex	35.0	31.4	31.5
6	Blow-line	25.4	25.5	25.8
7	Blow-line	22.8	22.6	22.3

Mill Validation Results



NIRS Kappa Analyzer

- Accurate measure of true kappa number
- Method reduced tester-to-tester variability
- No chemical requirement
- Measurement can be completed in < 5min.
- Allow for higher frequency testing

FINAL PULP INSPECTION

Pulp Sheet Kappa Analysis

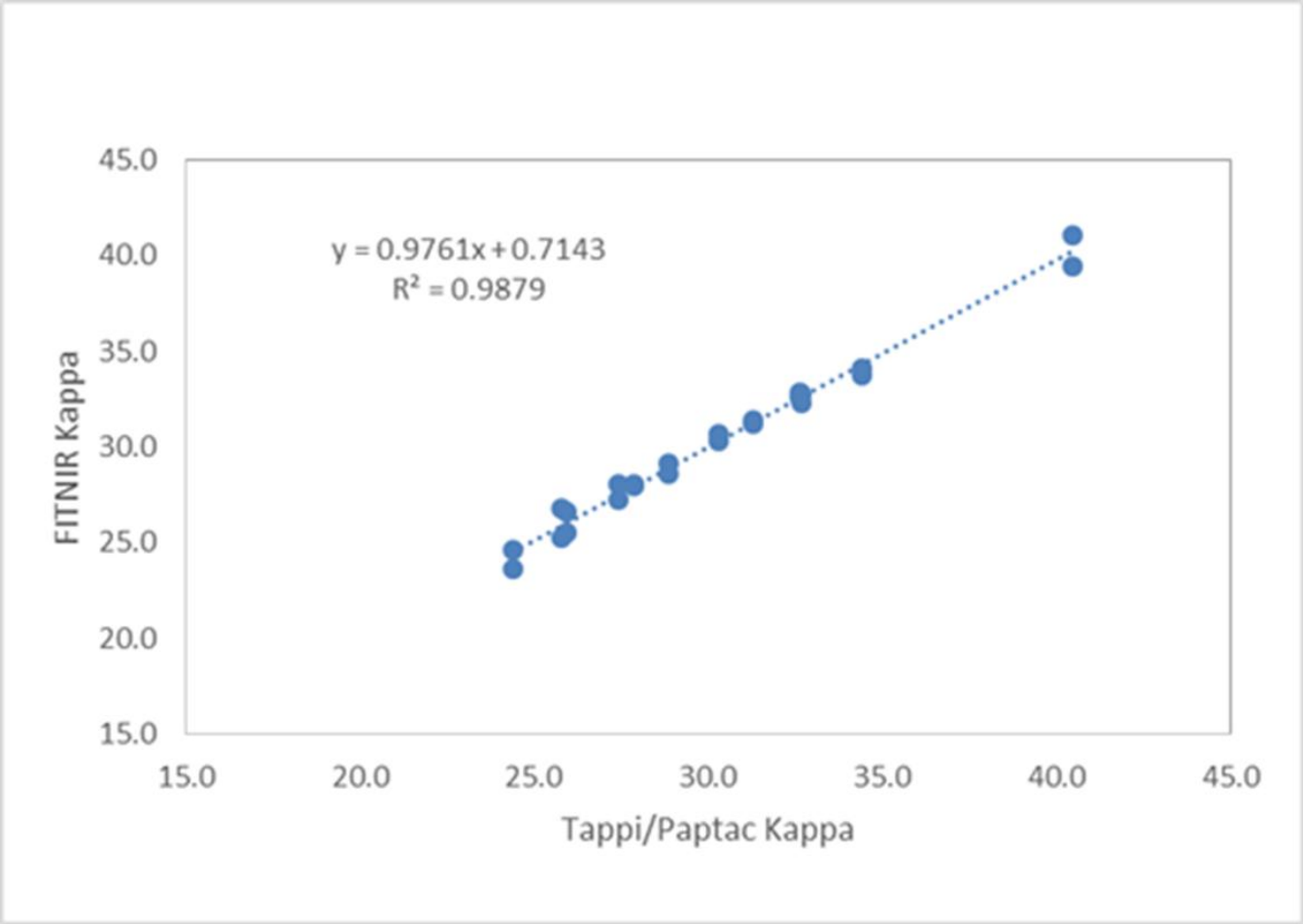
Finishing Line Quality Control

- Quality control at the finishing line requires onerous testing, especially when it comes to chemical and physical testing
- For mills producing brown pulp for specialty applications (cement board), customer requires kappa number of pulp with shipment
- Tedious and time consuming manual kappa means that product could be out of compliance and shipment could be rejected or called back

Pulpsheet Kappa with NIRS

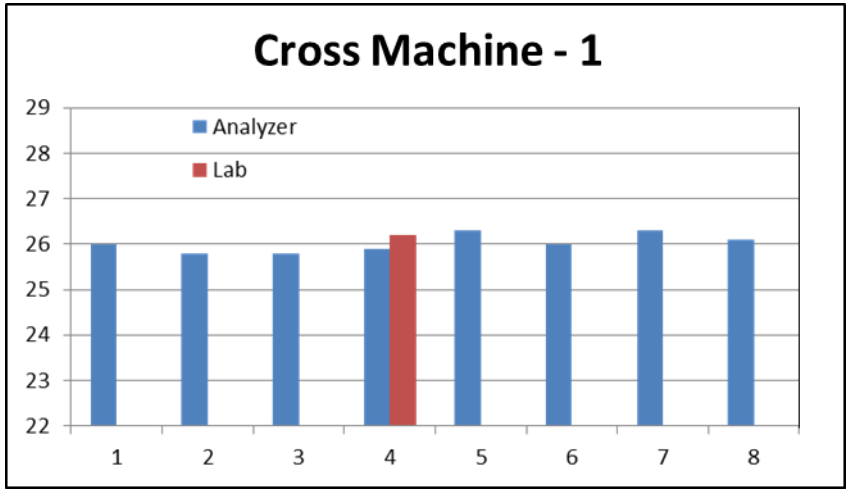
- Based on the same principle of operation, applied NIRS to measure pulp sheet kappa
- Pulp sheets taken at pulp machine, cut into ~6” discs
- System zeroed and sample is measured as is.

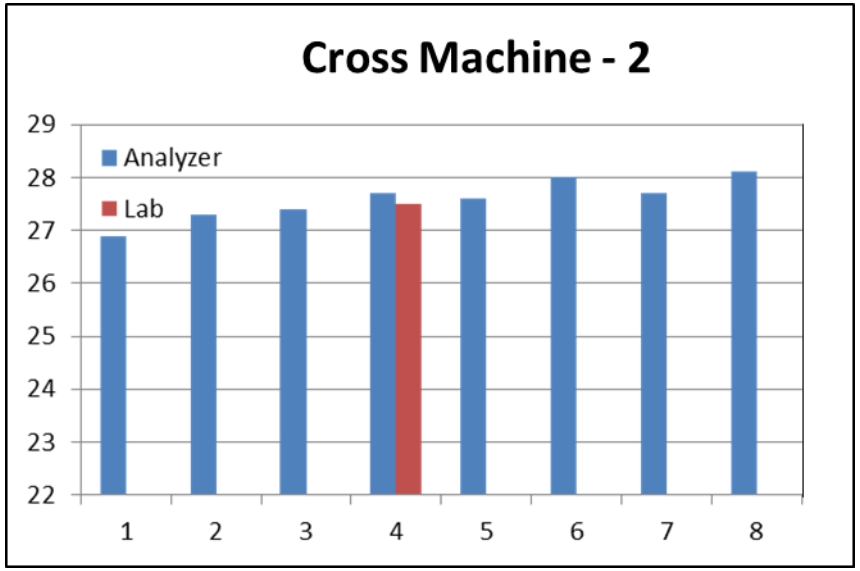
Pulpsheet Kappa Performance



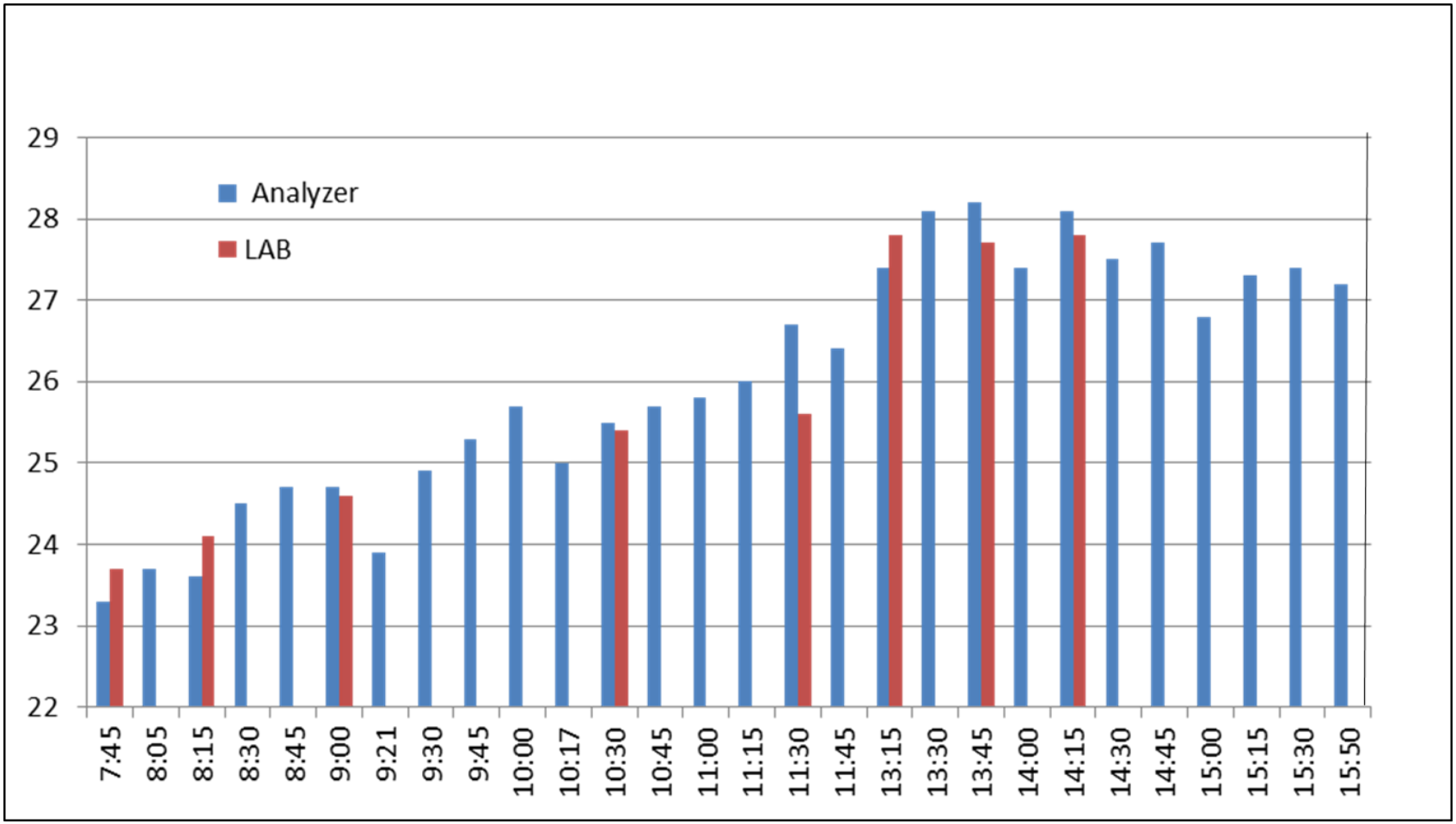
Cross Machine Variability Analysis

- Pulpsheet samples from all bales across the machine were sampled
- Analysis done for all 8 samples
- Drop #4 chosen to perform crosscheck
- Cross machine direction data shows that kappa is consistent, within 1-kappa unit

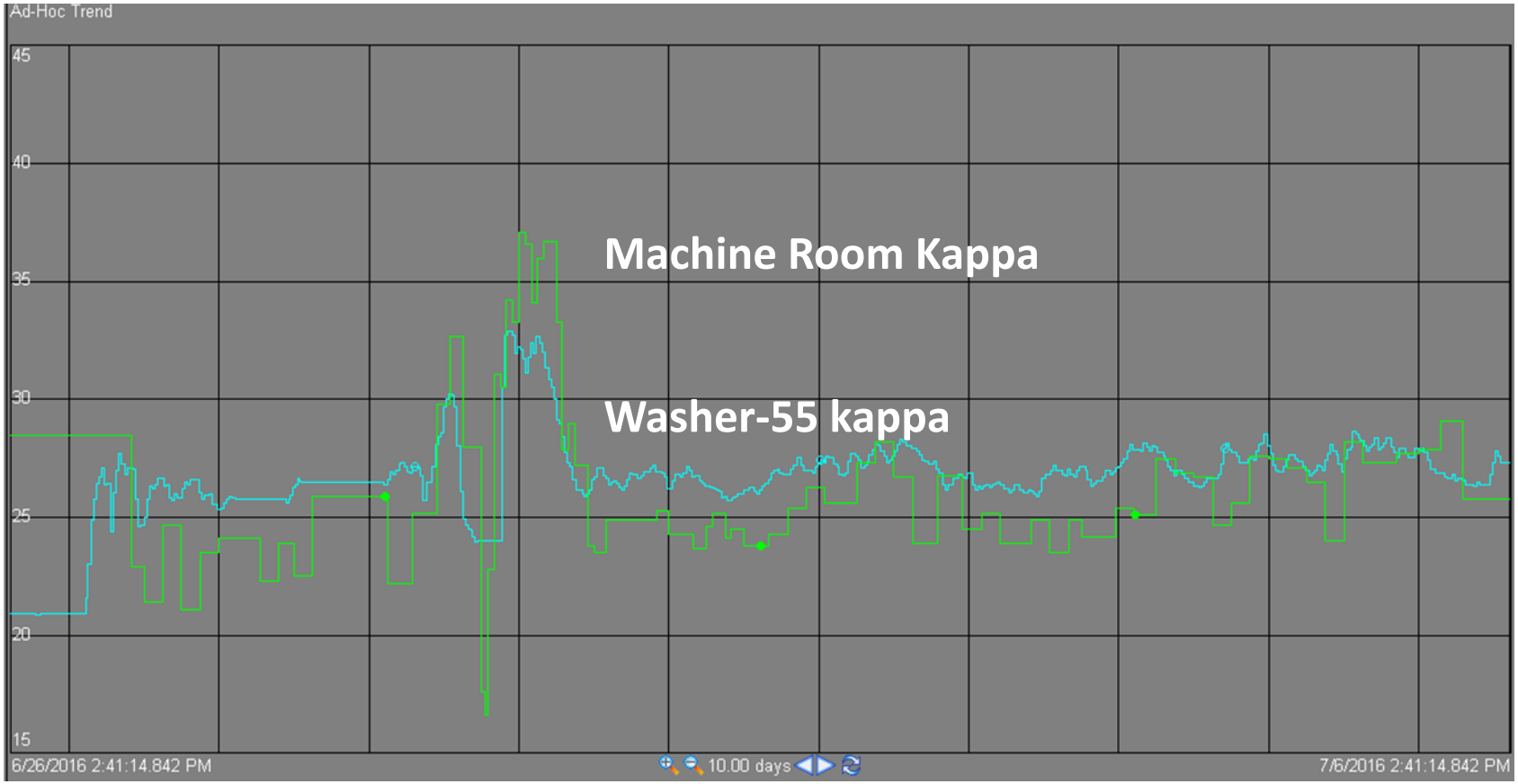




Machine Direction Kappa



Machine and Washer-55 Kappa



Summary

- **NIR technology, as applied, has been successfully implemented for mill's day-to-day kappa testing:**
  - **Simplified analysis and removed sources of errors**
  - **Allowed for greater frequency of testing**
  - **Is currently being used to track digester operations, allowing for optimization**
  - **Eliminates hazardous chemicals**
  - **Savings on chemical upwards of $100K/year, based on purchasing of KMnO4 and acid**
- **Can also be used for other applications:**
  - **Pulpsheet kappa and other properties**
  - **Pulpsheet viscosity**

Learnings and Best Practices

- **Manual testing of kappa:**
  - **Must follow standard TAPPI or PAPTAC procedure to ensure accurate and meaningful values**
  - **Pulp should be washed thoroughly to ensure no contamination from dissolved lignin**
  - **Sample dry weight is critical**
  - **If chemical strengths are suspect, discard all and restart with blank titration**
  - **Avoid shives and pins which could highly impact analysis**
- **NIR Measurements:**
  - **Run blank (15s) every hour or before each test**
  - **Provide ~sufficient sample size to produce pad**

Theme: Industry of the future:

"Chemical-Free Lab"

50 anos ABTCP

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THANK YOU

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FIT NIR
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