

### FITNIR KAPPA

FITNIR Kappa utilizes Near Infrared (NIR) spectroscopy to measure the kappa number of Kraft pulp. FITNIR Kappa represents a paradigm shift in how kappa determination can be done. In the past, consistent kappa testing has been difficult, varying from lab to lab. But, with its speed, consistency and accuracy, FITNIR Kappa makes standardization possible, reducing person-to-person and mill-to-mill testing errors. With FITNIR Kappa, mills can monitor and control pulp production from the beginning of the process to the final product, enabling quicker adjustments for improved efficiency. A simplistic yet effective approach to process control and optimization, FITNIR Kappa is the next generation of process analyzers.

### TRADITIONAL MEASUREMENT CHALLENGES

Kraft mills rely on kappa number (i.e., residual lignin content) of pulp to estimate the extent of delignification of the cooking process. To optimize cooking yield and product quality, kappa number is used as an input to automated control algorithms, allowing for adjustments in the liquor charge and temperature profile of the digester as well as the bleaching chemical charge in the bleach plant.

The standard method for kappa number determination is based on the consumption (50%) of potassium permanganate ( $KMnO_4$ ) by the lignin and then a back titration with thiosulfate. However, this method of analysis is slow and tedious and consumes a significant amount of chemicals as mills perform the analysis at regular intervals throughout the day, typically once every two hours. Although semi-automated or automated titration-based methods slightly reduce the time commitment, significant sample preparation and analysis time (approximately 15 to 20 minutes for each analysis) as well as chemical usage are still required.

With the shortage of fibre supply and greater fluctuations of chip quality and species mixtures, different cooking conditions are required resulting in higher kappa variability and demanding more kappa measurements. Analyzers using UV-visible light are strongly dependent on species' composition and can cause significant errors. Most importantly, UV-based methods are not well suited for measuring high kappa numbers of Kraft pulp due to their high absorption of lignin (i.e., high kappa number of Kraft pulp saturates the detector). Coupled with the very low sample consistency required for this method, the likelihood of inaccurate results is compounded.

#### INNOVATIVE SOLUTION

FITNIR Kappa utilizes NIR spectroscopy to measure the kappa number of Kraft pulp. The spectral absorption features of lignin allow for the direct quantification of residual lignin on the pulp. Consequently, FITNIR Kappa does not rely on other parameters for measurement (i.e., the strength of chemicals, precise sample dry weight, reaction time, or a subjective colour change indicating end-point), automates sample preparation, and eliminates all associated chemicals. FITNIR Kappa provides kappa number rapidly, with excellent accuracy and repeatability. Analysis time (scan) is less than 20 seconds and the entire time to prepare the sample is less than 4 minutes. More importantly, since FITNIR Kappa uses fundamental absorption peaks for calibration, the calibration models can be transferred from pulp-grade to pulp-grade without requiring major recalibration.

#### SAMPLE PREPARATION

FITNIR Kappa's sample preparation station is used to produce the sample puck. A pulp sample from the process line is collected, thoroughly washed with water to remove excess residual liquor, and then placed in the sample cup. The sample cup is positioned and secured onto the press of the sample preparation station (Figure 1). Dual-button operation engages the piston and the sample washing and pressing process begins. When complete, the pressed pulp is removed from the sample cup and is ready for analysis.

### **KEY FEATURES**

- Uses NIR spectrometry for rapid and accurate analysis of wet and dry Kraft pulp
- Does not rely on other parameters for measurement
- > No sample preparation and chemicals needed
- Allows operators to take numerous kappa number per hour across multiple process locations
- Analysis results in less than
   4 minutes, including sample
   preparation
- > Ability to measure high kappa numbers of Kraft linerboard pulp
- Calibration models can be transferred between pulp grades without requiring major recalibration



Figure 1: FITNIR Kappa sample preparation station



**Figure 2:** FITNIR Kappa analyzer/spectrometer showing the sample chamber.

# WET AND DRY PULP SAMPLE ANALYSIS

The analyzer or spectrometer conducts the sample analysis to determine kappa number. First, a reference scan is performed to zero the analyzer. Next, the pulp sample is placed in the sample chamber (Figure 2). Sample type or location is selected and collection time can be entered. Once the scan is activated, analysis time is approximately 20 seconds and the results are displayed on the screen.

FITNIR Kappa also has the capability of measuring dry pulp sheets at the finishing stage. A dry pulp sheet is cut into an approximately 4" diameter disc and mounted on a holder. As with a wet pulp sample, a reference is run and the sheet is analyzed. FITNIR Kappa is the first of its kind to directly measure kappa on the final product for procurement applications.

Traditional kappa testing proves to be too tedious and time consuming to reliably provide frequent data to ensure the



**MEASUREMENT PRINCIPLES** 

FITNIR Kappa uses diffuse reflectance NIR spectroscopy as the basis of measurement. Energy from the NIR light penetrating the sample is absorbed by the lignin. Optics collect the diffused reflected light, registering its absorption on the infrared detector and generating the spectrum with its unique features (Figure 5). The spectral data from the analyzer is then correlated with a previously built calibration model to determine kappa number. product stays within acceptable quality limits, as required by the customer. The ability to measure the final pulp kappa also enables product segregation. FITNIR Kappa has demonstrated strong correlations with both auto-titration and manual titration methods for dry pulp.

# PROVEN REPEATABILITY AND ACCURACY

FITNIR Kappa can be used for rapid kappa number measurement of wet pulp with reproducibility and accuracy. FITNIR Kappa is calibrated according to **TAPPI and PAPTAC standards. Figures 3** and 4 show the correlations between FITNIR Kappa and auto-titrations for Kraft pulp and Kraft linerboard pulp  $(R^2 \sim 0.98)$ . The ability to measure kappa directly on wet pulp simplifies measurements and allows for greater testing frequency. By simplifying and reducing the sample preparation steps, which critically affect titration results, FITNIR Kappa significantly improves kappa repeatability and accuracy.

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### **KEY BENEFITS**

- > Simplified analysis for greater testing frequency
- > Significantly reduces
   operator-to-operator
   variability, improving
   accuracy and repeatability
- Faster results for quicker adjustments, improving efficiency and profitability
- Eliminates hazardous testing chemicals and reduces process chemicals by up to \$150,000 per year
- Improves customer satisfaction by adhering to quality standards and enabling product segregation



Figure 4: FITNIR vs. standard kappa measurements for linerboard mill. Excellent correlation is observed for pulp with kappa number beyond 110 kappa.



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### FITNIR KAPPA: INSTALLATION & RESULTS

**Installation:** A mill installation of FITNIR Kappa incorporates the sample preparation station and the analyzer located at the fibre line testing lab. Samples are collected from the process

and brought back to	the testing lab for
analysis.	

**Results:** Table I shows the comparison between the TAPPI Kappa titration results and FITNIR Kappa for a Kraft linerboard mill. Excellent correlation is observed for a wide range of kappa values. Figure 6 illustrates results from FITNIR Kappa measuring dry pulp sheets performed at 15 minute intervals over a period of 8 hours. The trend shows swings in kappa number over time and suggests that better measurements could lead to improved control and subsequently better product quality and efficiency. Testing final pulp quality allows for product segregation and valuation.

SAMPLE #	DATE	DIGESTER	tappi Kappa	FITNIR KAPPA
FITNIR I	07-Aug	А	112.9	112
FITNIR 2	07-Aug	В	3.	111
FITNIR 3	07-Aug	В	115.4	113
FITNIR 4	07-Aug	В	114.6	114
FITNIR 5	07-Aug	В	115.6	115
FITNIR 6	07-Aug	В	115.1	115
FITNIR 7	26-Sept	А	104.7	105
FITNIR 8	26-Sept	А	109.3	110
FITNIR 9	26-Sept	А	91.7	91
FITNIR 10	26-Sept	В	102.9	103



**Figure 6:** FITNIR Kappa on pulp sheet, performed every 15 minutes in machine direction. Validation was done with lab hand-titrations.

**Table I:** Validations showing the accuracy of the FITNIR Kappa

 versus TAPPI's auto-titration analyzer for kappa measurement.

# ROBUST, USER-FRIENDLY EQUIPMENT

The sample preparation station has a 304 brushed stainless steel construction for exceptional corrosion resistance. The sample preparation station measures 36.3"H x 18"W x 16"D (92.2 cm x 45.7cm

x 40.6 cm) and weighs 145 lbs (65.66 kg). The analyzer unit measures 15.5"H x 22.5"W x 15"D (39.4 cm x 57.2 cm x 38.1 cm) and weighs 67.6 lbs (30.7 kg).

The sample station incorporates dualbutton operations as a safety measure and position indicator to ensure the sample cup is in the correct position. The piston is pneumatically operated with an emergency stop button to ensure safe operations. The analyzer incorporates a touch-screen computer for ease of operation. Data is stored locally with current measurement results displayed onscreen as a historical data trend for the last 20 measurements.

#### FITNIR SUPPORT

At FITNIR, we understand your business. Our expertise in both the lab and in the field goes into every aspect of our product development. Our innovations, process knowledge and dedication are focused on supporting your business success.

FITNIR offers a wide range of customer support services, including project coordination, application engineering (including kickoff meeting, system configuration calibration and validation), system verification and testing, application documentation, training and after-sales support.

Contact FITNIR Analyzers Inc. to find out how we can partner with you to optimize your pulp production.



The Next Generation of Process Analyzers

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