New Advancements In NIRS Technology, Quality and Minerals In Two Minutes?

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NIR has been in use for testing agriculture products for over 15 years based on Karl Norris' pioneer research at USDA Beltsville in 1965. In order to understand recent developments in NIRs, one must first understand the components of the NIR system. Those three components are a spectrophotometer, a computer, and a specific calibration approach to a given application. The sample preparation components of a complete forage testing system, for both wet and dry materials, include a microwave oven with a 600 watt setting and automatic timer, an electronic top loading balance with a 250 gram capacity and accuracy to .10 of a gram, and a grinder (either a UDY or Tecator mill with a .1 mm sieve) to grind all samples to a consistent particle size.

Some version of a scanning near infrared instrument must be used. These are either 4250, 6250, or a 5la. This is connected to a computer of an IBM variety, an Epson FX80 or 85 printer, and various combinations of software packages.

Karl Norris' original system had each of these components, however, the monochromator was a modified Cary-14 wavelength generator and the computer apparatus filled a whole room. If you were ever going to get this technology down to the point where it could be used on a practical basis, the instrument had to be made smaller, compact, more repeatable, faster and with less noise. The computer equipment had to be reduced to a table top model which we have today.

As many of you are aware, the current state of the art necessitates taking a sample, normally with a coring devise and that core sample then placed in a pouch and sent to a laboratory for analysis. Once it arrives at the NIR laboratory, wet samples are weighed on an electronic balance. Those wet samples are dried in a microwave oven for typically 3-4 minutes to reduce the sample moisture level to a point where one can grind it. The sample is re-weighed to calculate the volatiles driven off during grinding. Next the sample is ground through a cyclone mill and evacuated into a bottle, stirred thoroughly and spooned into a sample cup. A back is applied to the sample cup providing uniform compaction and forcing the powdered sample firmly against the quartz window for viewing by the instrument. After cleaning the front of the sample cup, it is inserted into the drawer and placed into the instrument. About a minute later you get the results displayed on the computer screen. This is the method currently employed with all of our models - most popularly the Model 4250 scanning instrument that you will see demonstrated later on today.

There are some limits to today's near infrared instrument designs. We are limited to working in reflectance only. Which means we must impinge a sample with near infrared energy and collect that absorbance information on detectors placed on the illumination side of the sample. Therefore, when we work in reflectance at the longer wavelengths, we can only work with dry samples, with moisture at a fairly low level. Dried forage samples also need to be ground to a very uniform, fine particle size to eliminate large baseline shifts and interferances in the spectra.

One of the biggest hopes that we have always had is that we can measure a sample in its as-received, unadulterated condition. The requirements to do that involve both software and hardware modifications. For today's purposes, we will look at the hardware requirements. We would like to work in transmission as well as reflectance. In order to make some of these measurements in very high moisture samples, the instrument must generate wavelengths that are below 1100 nm. Current instruments of today operate between 1900 and 2300 nm. Rather than look at a very small surface areas in cups that typically hold two grams of sample, we would like to look at as much as ten square inches of the material. We have already begun taking feasibility data on testing coarse wet haylage. The data is presented in Table 1. Each sample that we analyzed was tested 10 times for 80 equivalent samples. Our preliminary data shows us that it is possible to calibrate wet silage or wet hay material, and achieve accurate protein and moisture measurements one percent or lower in standard errors of prediction. There are various ways that we

can present these types of samples to the instrument optics. The traditional form of working with NIR was to take a finely ground substance and rotate it in a spinning sample cup. We have also worked with clam shell devices, flowable liquid and solid cells, and liquid cells capable of making measurements on liquids in true transmission.

The reason that we can work with these large materials is that we have an optical system that allows us to move the sample while we make the measurement. (Figure 1) Our systems are fast scanning, allowing us to move a large sample and take repeated measurements at all wavelengths over the entire surface area of the sample to average out any sampling or wavelength error. Some of our sample presentation methods allow us to work with whole grains. A transport mechanism is mounted onto the front of the instrument. When the operator tells the instrument to scan, the product will be moved through the optics of the system, and measurements will be taken over the entire surface of the sample. If we want to work in reflectance, the detectors are placed on the inside of the instrument; if we want to go all the way through the sample and make the measurement in transmission, the detector is placed in the door of the trasnport mechanism. In this manner the internal composition of the sample is measured, not the surface. Certain locations of the USDA Forage Research network are making use of this hardware advancement. In the case of grains, samples can be measured and upon the finished scan automatically dumped into a pan below the transport mechanism. The device used for wet forage material is a clam shell device where the haylage or silage is packed into a plastic bag, inserted into a sample cell and a window closes down forcing a fixed path length of the materials. Some of the types of sampling mechanisms that we looked at involve funnels of samples and taking quadrant measurements of silage or wet material. The sample is moved into the optics of the instrument and repeated measurements made for the other three portions of the sample and the data averaged.

We are also beginning work on milk analysis and other liquids so that we can actually measure a moving, flowing liquid that can be pumped or syringed into the sample compartment. So if we look back over the hardware advancements that have occurred since Karl Norris' original work, an entirely new instrument family has developed. Ease of operation and minimized sample preparation is emphasized in order to come up with the final measurement. So the answer to the question (the title of this talk), is it possible to take protein and mineral readings in less than two minutes? It is, if you reduce the sample preparation down to no drying or grinding. That is the future of our work.

TABLE 1

PRELIMINARY DATA – HAYLAGE PERFORMANCE CHART

CONSTITUENT	SEC	R^2	MATH TREATMENT
PROTEIN	.66	.92	FIRST DERIVATIVE
MOISTURE	.61	.99	FIRST DERIVATIVE

CONCLUSION: HIGH MOISTURE MATERIAL PROTEIN AND MOISTURE CONCENTRATION POSSIBLE TO MEASURE (SEP) TO 1%

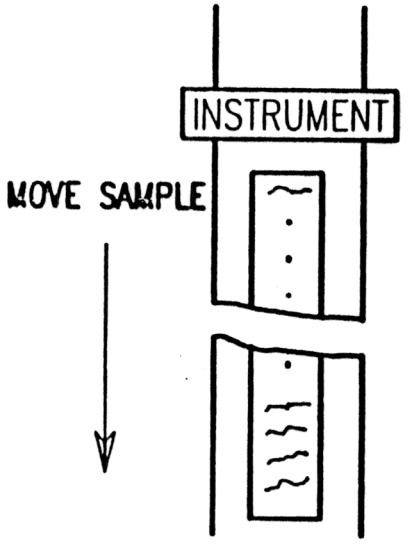
FIGURE 1

TRUE VALUE ± E

- ABSORBANCE
- WAVELENGTH
- SAMPLE

INCREASE MEASUREMENT AREA

(TO REDUCE SAMPLING ERROR)



FAST SCAN

(RESULTING SPECTRUM IS AVERAGE OF TOTAL SAMPLE)

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