## **CUMBERLAND VALLEY ANALYTICAL SERVICES**

"Laboratory services for agriculture ... from the field to the feed bunk"



## EDXRF: A new technology application for determination of minerals in forages and feeds.

Understanding mineral content of feeds and rations is very important for maintaining long term health and production in cattle. The primary approach to routine forage analysis is by near infrared spectroscopy (NIRS). While this secondary analysis method works well for organic constituents where carbon, nitrogen, and OH bonding exists, most of the minerals that need evaluated are not bonded with these elements and as a result are not able to be analyzed with sufficient accuracy by NIR. It is possible to develop models for minerals with a reasonable correlation to NIR spectra, but the error around the prediction is high.

The lack of relationship between NIR spectra and minerals makes this a poor analytical approach. It may actually be more accurate to use good book reference values than to rely on NIR.

The most utilized laboratory approach is by use of inductively coupled plasma spectroscopy (ICP). This is a recognized reference method for mineral determination. The procedure is to digest the samples in acid and then inject a small portion into a hot argon plasma flame at over 5000 degrees Celsius. This causes the electrons in an element to emit light energy characteristic of that element that are used to identify presence and determine concentration.

ICP as an analytical approach requires costly equipment, significant labor, and a high level of technical training to perform well. That makes ICP applicable in larger labs running higher volumes of samples. There is another approach however that has recently gained acceptance as technology has improved and instrument cost has dropped.

Energy Dispersive X-ray Fluorescence (EDXRF) is a method for using X-ray fluorescence to detect and measure elements (minerals) in materials. EDXRF spectrometers use a burst of X-Ray energy to excite all the minerals in a test sample and a detector collects the subsequent fluorescence radiation emitted from the sample. It does not require the sample to be digested as the sample is presented to the instrument as a fine powder or pelletized powder.

The EDXRF approach does require sophisticated equipment that has to be correctly calibrated to a primary method such as ICP. However, the approach allows for routine labor efficient analysis and provides excellent results with high precision and accuracy. It has the advantage of being able to measure most elements (Na to Am). Where it traditionally takes two or three instruments in a lab to measure minerals of routine interest in dairy cattle nutrition, EDXRF can determine all from one sample scan.

Cumberland Valley Analytical Services has investigated this technology and worked with Bruker, one of the larger global laboratory instrument companies to implement calibrations. We have successfully established an instrument in our Madison WI lab and are implementing this technology with our partner labs in China, Spain, and Croatia. This technology will allow for cost effective analysis of forage, feed, and TMR samples often with day of receipt reporting of results.

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A view of the instrument configuration



An example relationship between intensity and concentration for calcium (r2 = .986)



A view of multiple sample spectra showing the peak for calcium at 3.696 keV.

Element	n	Range (in DM)	R <sup>2</sup>	SD	Precision
Са	58	0.01 to 2.65 %	0.986	±0.07 %	±0.01 %
Р	57	0.15 to 0.60 %	0.959	±0.02 %	±0.00 %
Mg	60	0.09 to 0.53 %	0.941	±0.03 %	±0.01 %
S	53	0.10 to 0.48 %	0.974	±0.02 %	±0.00 %
К	54	0.35 to 3.91 %	0.994	±0.07 %	±0.00 %
Cl	51	0.00 to 1.88 %	0.993	±0.026 %	±0.00 %
Na	60	0.01 to 0.72 %	0.942	±0.05 %	±0.01 %
Fe	57	20.8 to 1821 ppm	0.994	±26.0 ppm	±3.50 ppm
Mn	58	4.61 to 138.3 ppm	0.963	±7.11 ppm	±1.04 ppm
Zn	60	13.4 to 133.5 ppm	0.986	±3.47 ppm	±1.71 ppm
Cu	59	1.23 to 24.9 ppm	0.956	±1.35 ppm	±0.44 ppm

Example Calibration Model Statistics for Determination of Minerals in Forage