

ANALYSIS OF FEEDSTUFFS USING NEAR INFRA-RED SPECTROSCOPY

Introduction

Using near infra-red (NIR) spectroscopy to test feedstuffs has significant advantages for both testing labs and the customers. In just minutes, it can analyse for a wide range of analytes that would otherwise take many hours of work by conventional 'wet chemistry' methods. But the technique is not without pitfalls. At Hill Labs (HL), we have used NIR for over fifteen years and we understand the technology, including the limitations of the technique.

What is Near Infra-red Spectroscopy?

Near Infra-red is the region of light immediately adjacent to the visible range, between 700 and 3000 nanometers in wavelength. This region of light corresponds to the energy of molecular vibrations and will therefore selectively absorb NIR light, depending on the molecules present. The resulting absorbance spectra can be interpreted with high level chemometrics to determine the chemical composition of the sample. Calibration models are built by collecting many thousand spectra and relating these against reference chemistry values for the same samples – the models are then used to "predict" results for samples of the same type.

Unlike most laboratory tests, NIR testing does not measure the analytes directly. It is an indirect measurement technique that returns a test value based on the reference method calibration model.

Calibration

The use of NIR works well when the samples being measured are well represented in the calibration set. There is a high risk that erroneous results will be generated if quite different plant types are being scanned. At HL, the system quickly recognises any irregular spectra, and the samples are identified as having uncertain measurement statistics by notation on the lab report. Customers are offered the option to request reference method (wet chemistry) tests on the prepared sample retained by the laboratory.

From time to time, these new sample types are incorporated in to the calibration set, so they are no longer unusual and the system can process them routinely. NIR calibration at HL is a dynamic process that is continually being strengthened.

Hill Labs uses sophisticated calibration methodologies, surpassing most industry standard procedures. Where some labs may use only hundreds of samples, HL has calibration sets in the tens of thousands. When one has large sample calibration sets, more complicated algorithms which use local-weighted ('nearest neighbours') approaches can be employed. The procedure is, for a new sample, it's spectra is compared against all others in the calibration dataset, a selection of spectra that best match the new one are then selected and a calibration constructed 'on-the-fly', using only these selected spectra. This ensures the calibration samples will be similar in nature to the 'new sample', i.e. same sample type.

Quality Assurance

We run an expanded Quality Assurance program, where perhaps one in ten or twenty samples are re-analysed by the reference wet chemistry internally. The differences between these results and the predictions are continually monitored, to ensure acceptable accuracy of the NIRS system. The wet chemistry results are plotted against the NIR-predicted results and are statistically analysed to ensure the calibration is performing as expected, i.e. that the system is 'in control'. Should the results fall 'out of control', we revert immediately to 100% wet chemistry testing, and our chemometricians respond to resolve the problem.

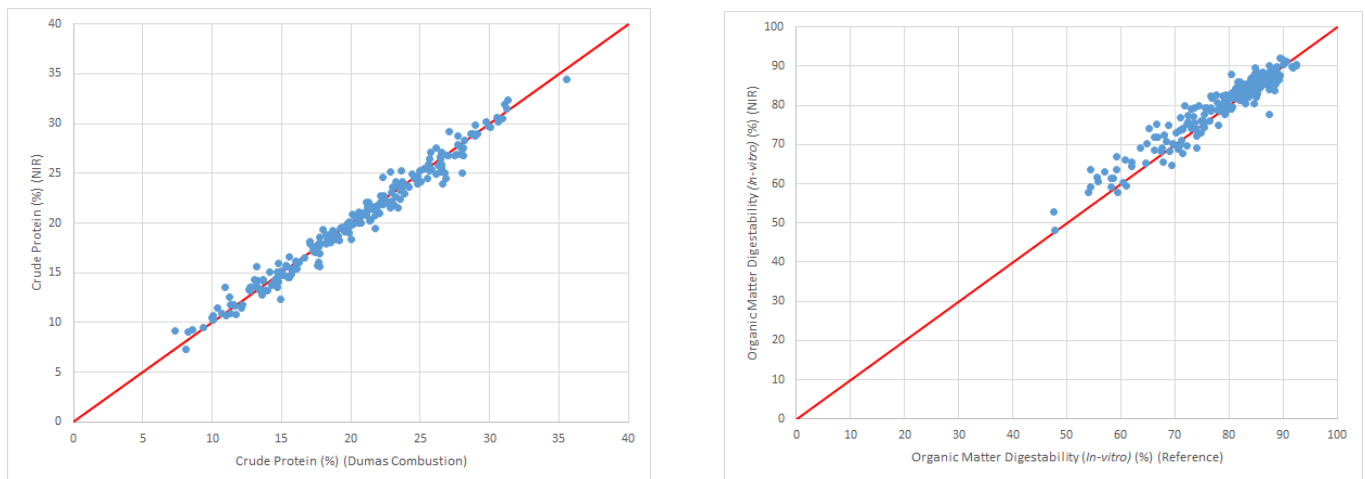


Fig 1: Plots showing correlation between NIR and conventional method analysis for Crude Protein and Organic Matter Digestibility for a sub-set of pasture samples.

Wet chemistry support is essential, and having ready access to this in-house ensures reference method test results are available if required and enables ongoing monitoring and maintenance of the NIR calibration.

Inter-Laboratory Comparison Programme (ILCP)

As well as the real-time and ongoing monitoring of the NIR prediction performance described above, the laboratory participates in both national and international inter-laboratory comparison programmes (ILCP) for the majority of the feed test reference methods. Performance is monitored and any outliers immediately investigated as to cause of variance. Samples are run through both the wet chemistry method of the ILCP programme and through our NIR instrument to observe performance by both techniques.

At the time of writing, IANZ accreditation is held for Crude Protein, Crude Fat, Acid Detergent Fibre, Neutral Detergent Fibre, Ash, Soluble Sugars, Starch, Lignin, Digestibility (DOMD) and Metabolisable Energy (ME) under the Conformance Standard NZS ISO/IEC 17025:2005. A copy of our accreditation schedule can be downloaded from our website or is available upon request to the laboratory.

The international ILCP programme the laboratory currently participates in, is administered by the Association of American Feed Control Officials (AAFCO) programme (USA).

