NIR Technology Systems

Technical Note 22: Developing a NIR Method

Introduction

NIR spectroscopy is a secondary or correlative technique i.e. the spectral data collected is correlated through statistical means to some reference (laboratory) data. Regression techniques are used in order to develop a model, which is subsequently downloaded into an NIR analyser for use as a prediction tool.

The following steps, also presented in the following flow chart, outline the general steps in developing a calibration:

- Optimise sampling method.
    - Pathlength considerations (Cropscan 2-4abs, NIT 0-2, 2-4abs)
    - Homogenisation procedures (FOP and NIT)
    - Reproducibility of chosen sampling technique

- Sample collection
    - Successful calibrations usually contain around 100 or more samples.
    - Should contain most of the variability expected for "real" samples.
    - Constituent concentrations ideally should be evenly distributed (box car distribution).

- Reference Analyses
    - Should be performed using accepted reference method.
    - Should be performed in duplicate (for error estimation in final method).
    - Should not differ by more than 5% (if so, repeat).

- Spectral Data
    - Samples should be run in duplicate.
    - Store spectral data along with reference data (Excel).

- Regression Analysis
    - Use chemometrics software package to perform:
        - Partial Least Squares (PLS) Analysis (Multiple Wavelengths).
- Download the Model
    - Validate the model on "real" samples
    - Evaluate temperature stability.
    - Evaluate parameters such as repeatability and reproducibility.

Flow Chart for Calibration Development