FOR KARL NORRIS FROM JOHN SHENK

Concept Presented by John Shenk, Feb. 15-16, 1983
at the Third National NIR Workshop

NIR Equation Transfer

One of the objectives of NIR reflectance spectroscopy is to be able to derive master calibration equations that have broad application and use on many instruments. Presently NIR analysis is carried out by one of two means. Either an instrument is provided to the customer precalibrated or the instrument is purchased with a computer to accomplish the calibration. The latter approach is used primarily with forage and feeds. This works well for laboratories that have a larger volume of forage samples to be analyzed. These laboratories develop their own master calibrations but are not able to transfer these calibrations to other instruments. If equation transfer can be made possible, a more uniform analytical system can be established nationwide and the technology made available to more people.

Why is equation transfer a topic of research? Why is it not possible to transfer the equation directly from one instrument to another. The simple answer is that NIR instruments are not alike either between instruments manufactured by the same company or among instruments of the same model produced by the same manufacturers. These problems would seem to have a simple solution. Have the instrument manufacturers make all instruments meet a set of specifications. This, of course, has been done and for the most part the instrument companies are meeting these specifications.

The problem is that even with fairly tight specifications on the instruments, slight but important differences exist among instruments (Templeton et al. 1981). These differences must be corrected if two instruments are to give the same analysis on the same sample using a master calibration equation.

Equation transfer includes transfer within, among, and between instruments. A within transfer becomes important if the instrument has a major failure such that when repaired, old prediction equations developed for the instrument must be transferred to the repaired instrument. Among instrument transfers involves taking a prediction developed on one instrument model and using it on a second instrument of the same model. Between instrument transfer is similar to among instruments transfer but it is accomplished across instruments from different companies and different models within the company.

A second kind of equation transfer is possible. The transfer of an equation from one instrument to another followed by the update of the equation on the second instrument. This transfer could also be important with instrument failure. The old equation from the instrument must not only be transferred to the repaired instrument but needs to be updated with new calibration samples as well.

The purpose of this experiment is to determine the level of error attained by transferring a number of equations developed on one NIR reflectance monochromator to at least 7 other NIR reflectance monochromator instruments. These instruments will include 5 Neotec 6100, 1 Technicon 500, and 1 Carry 17 monochromator.

The following Procedure was agreed to on February 28, 1983 by Karl Norris, John Shenk, Franklin Barton, Mark Westerhaus and Gordon Martin.

Sixty samples of hay will be assembled for this study. Thirty samples will be obtained from Gordon Martin, St. Paul Minnesota and 30 samples from Franklin Barton, Athens, Georgia. These samples will consist of hay type forages of different species, mixtures of species, and possibly different drying methods. At least 10 g of each sample will be ground through a UDY mill, and sent to John Shenk at University Park, PA.

The sixty samples will be packed in sealed cells and allowed to equilibrate for 5 days. The University Park location will develop the equation to be transferred from the 30 samples used in an earlier study (Templeton et al., 1981). Equations will include protein, ADF, NDF, lignin, and IVDMD. The computer program has been developed at University Park to accomplish the equation transfer. The 60 samples will be analyzed for changes in their spectra by Karl Morris, Beltsville, MD at the beginning and end of the experiment. Each location will take part in the experiment according to the following procedure.

The 60 sealed samples will be sent to each participating laboratory. The package will contain the transfer program, equations to be transferred, and two files containing the spectra from 10 and 30 transfer samples obtained on the University Park instrument.

Spectra for the 60 samples will be collected in the following manner. The sample holder will be placed in the instrument and the spectra collected in the normal manner. The sample will then be rotated manually to 3 random positions for 3 additional scans. This will provide 4 complete spectra from each sample. When all samples have been scanned 4 times the program FILE will be used to average the 4 spectra into a single spectra for each sample. The spectra file will be split in two ways. First, 10 samples will be used to transfer the equation and tested with the 50 remaining samples. Second, 30 samples will be used to transfer the equation and the remaining 30 samples used to test the transfer.

The transfer will be accomplished as follows:

The computer program TRNSFR requires 3 input files.

1. The University Park equation file.
2. The University Park spectra file.
3. The spectra file from the second instrument.

It must be remembered that the exact same samples must be used in this transfer, that is, the 10 samples from University Park can only be used with the same 10 samples from the new instrument and the same is true for the 30 sample test. The output file from this computer program is a transfer equation for the second instrument.

The program PRE will be used to predict the composition of the test samples. Two equations will be developed, one from 10 and the other from 30 samples. A prediction file will be set up for each equation. The two prediction files properly identified will be copied to the University Park disk. Make sure a copy is maintained in your file. The samples will be returned to Karl Norris

for a final check of the spectra. University Park will conduct the statistical comparison of the 7 prediction files to determine the level of error associated with equation transfer. These findings will be coordinated with the results, obtained by Norris on spectral changes.

Carefully package the 60 samples back in the box and be sure to include the floppy disk with the University Park equation, transfer spectra, transfer program and the prediction file from your location. Send the package to the next location on the list. This work will require one person at each location at least 2 days to complete.

After all participating labs have completed the study the samples will be returned to University Park. The data collected at each location will be statistically analyzed at University Park and the samples returned to Karl Norris for verification of the sample spectra. The experiment outlined should be completed within 6 months.

In addition to the above experiment University Park will conduct studies and develop procedures and programs to evaluate the potential of transferring these equations to the Neotec 51 and 102 instruments. If this can be done satisfactory the method, procedures and programs will be provided to all Network locations desiring this capability.

Future research by Karl Morris will be directed toward the transfer of spectra from one monochromator to another. Areas of concerns are a) sensitivity differences among instruments b) and variation among ceramic standards. An additional factor of study will be the effects of sample and environment moisture on the transfer process.

Other comments to be added to the letter provided with this proposal.

1. BEST to be sent to all locations with 11/03's
2. Use BEST instead of CAL except for division.
3. Future conference call on or before May 1, 1983.