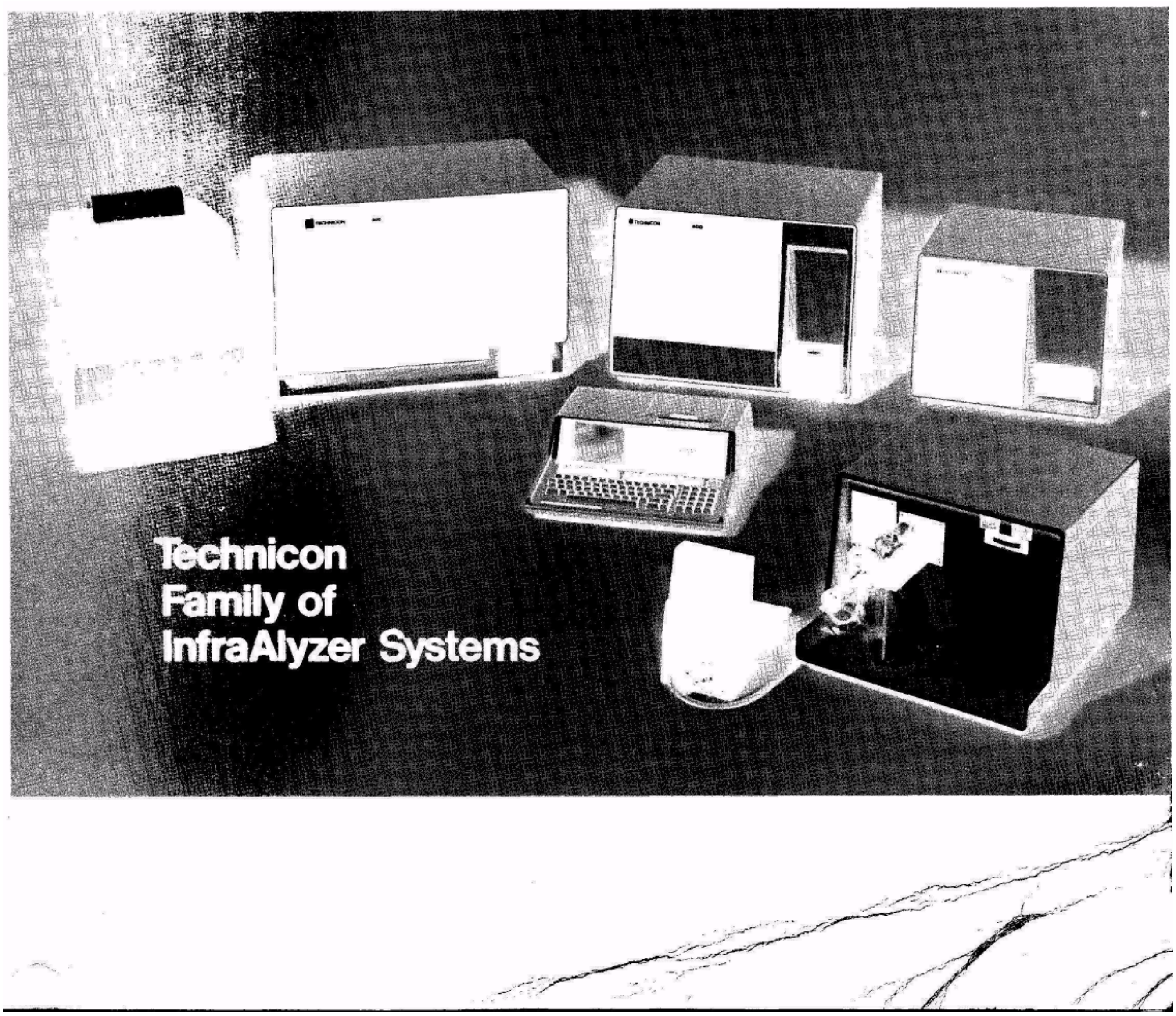
FOURTH INTERNATIONAL SYMPOSIUM ON NEAR INFRARED REFLECTANCE ANALYSIS (NIRA)

Abstracts of Proceedings



On July 19 and 20, 1983, the Fourth International Symposium on Near Infrared Reflectance Analysis (NIRA) was sponsored by Technicon Industrial Systems at the Science Center in Tarrytown. New York. With the rapid advances in NIRA technology and the resultant wide-ranging applications for process and analytical laboratories, this Symposium offered major industry leaders the opportunity to present and discuss the most recent developments.

The following pages contain abstracts of the papers presented at this Symposium. A copy of the full proceedings from the 1983 Symposium is available at a charge of $125.00. (One complimentary copy of a single presentation is available free-of-charge.) A reply card is enclosed for easy order.

Whether you are presently using NIRA instrumentation, contemplating a purchase, or simply interested in the technology, the information on the next few pages will be of interest.

Abstracts

FOURTH INTERNATIONAL SYMPOSIUM ON NEAR INFRARED REFLECTANCE ANALYSIS

(NIRA) ***July 19-20,1933***

Technicon Science Center, Tarrytown, New York

Plenary Session I

Tomas Hirschfeld, presiding

Lawrence Livermore National Laboratory, Livermore, CA

Removing The Magic From NIRA

David Honigs, Indiana University, Bloomington, IN

Mr. Honigs holds a BS in Chemistry from Kansas State University and is presently a graduate student in Analytical Chemistry at Indiana University He has worked with NIRA and Fourier Transform Spectroscopy for four years, and has co-authored several publications in these fields.

Near Infrared Reflectance Analysis (NIRA) has become an extremely successful analytical technique. This is somewhat surprising since spectroscopically, the near infrared region is dominated by weak and overlapping combination and overtone absorptions. Rather than being a handicap, however, the weakness of the absorptions and their complexities are precisely why NIRA works so well.

The small absorptivities of the near infrared region allow NIRA sampling techniques to be simple and give the diffuse reflectance measurement a good depth of penetration. The small absorptivities also cause NIRA measurements to be linear with concentration over a wide dynamic range. The broad and overlapped peaks make wavelength selection in NIRA much less critical and allow relatively broad-band monochrometers and filters to provide the necessary resolution.

The complexity of the near infrared region overtone and combination bands allows NIRA to be used to monitor the secondary and tertiary effects of a sample, such as changes in hydrogen bonding, hydration state or bonding geometries.

NIRA Methods: What Can Be Done*

Dr. Hirschfeld, Senior Scientist, has approximately 300 published papers in spectroscopy, and is s a four-time winner of the Industrial Research 100 award

Tomas Hirschfeld, Lawrence Livermore National Laboratory, Livermore, CA

An enormous variety of problems come daily into a typical analytical laboratory. NIRA is applicable to a fairly substantial number of these, and will often be a preferred method for some.

In general, NIRA is appropriate for the analysis of sample components exceeding 0.1%, and for properties that are controlled by such major constituents. Medium complexity samples are optimal, since simple samples can be analyzed by conventional spectroscopy, and highly complex ones eventually are intractable. Practically all organic compounds, and about half of all inorganics, have characteristic NIR spectra.

NIRA can also be used to make other types of measurements. It can be used to measure minor constituents, or the properties they produce, if the minor constituent has an effect on a major constituent's spectrum. It can even allow determination of properties whose chemical compositional causes are not understood at all, and sometimes allow the deduction of these chemical causes. This measurement of a spectral correlation of a measurable sample property is a qualitative application of NIRA that is a new capability in industrial research.

Another qualitative NIRA application is the recognition or verification of a sample from within a set of possible candidates by rapid cross correlation, with very simple hardware. Qualitative analysis can be performed here with a simple filter instrument.

Finally, NIRA can be used for tolerance verification by factor analysis, ratio analysis, or simple subtraction. The value of these new technologies is discussed.

* Work performed under the auspices of the L .5. Department of Energy bv Lawrence Livermore National Laboratory under Contract W-7405-Eng-48.

Suggested Procedure for Testing NIRA Scan Instruments

K.H. Norris, U.S.D.A., Beltsville, Md. and R. Spies, U.S.D.A., Albany, CA.

Mr. Norris is Chief, Instrumentation Research Laboratory, Beltsville Agricultural Research Center. He has over 30 years' experience as leader of a research program to develop instrumentation techniques for predicting the quality of agricultural products. Mr. Norris originated the concept on which NIRA is based, and his work has been published extensively.

Epolene is a polyethylene wax which, when ground, provides a white powder with several sharp absorption bands. It is suggested that a reflectance sample of Epolene be measured five times in succession at the beginning of each day to test noise, wavelength accuracy and wavelength precision. Wavelengths for the absorption bands at 1394, 2310 and 2350 nanometers are computed to a very high precision with a 9-point quadratic curve-fitting procedure (Savitzky-Golay) and the mean and standard deviations of these peaks are computed for the five scans.

The standard deviation of the log 1/R value at each wavelength is computed in three spectral regions (1300-1500, 2050-2250, and 2250-2450 nanometers) to test for noise.

Typical data are presented for three different scanning NIRA spectrophotometers. Short-term wavelength precision varied from 0.002 to 0.02 nanometers. Day-to-day wavelength precision varied from 0.03 to 0.33 nanometers. The noise (standard deviation of log 1/R) varied from undetectable levels to 1.0×10^{-3} log 1/R values, depending on spectral region and the instrument being tested.

Measurements of two samples of Epolene over a two-year span, with temperature variations of 15° C, showed no detectable changes in wavelength (less than 0.5 nanometers).

The three instruments show wavelength differences as much as 3.9 nanometers.

Linearity and Specular Component Effects in NIRA

Edward Stark, Technicon Industrial Systems, Tarrytown, NY

Mr. Stark, Principal Scientist, has 28 years' experience in infrared, electro-optics and information processing, the last 13 with Technicon. Since 1976, he has been associated with NIRA technology and development of the InfraAlyzer product line.

NIRA is based on the assumption of a linear relationship between the optical data and constituent concentration. Data transformations such as log 1/R or Kubelka-Munk are used to linearize the reflectance data. This paper discusses the causes and effects of nonlinearity in NIRA systems, including the specular reflection from the sample surface and the nonlinear relationship between transformed reflectance and constituent concentration.

Data is presented showing that the specular component effect is minimized by use of an integrating sphere. The relatively smaller nonlinearity of log 1/R compared to difference equations (derivatives) is also analyzed. Kubelka-Munk data is reviewed to demonstrate the variation of nonlinearity as a function of sample variables. It is also shown how multilinear regression compensates for this nonlinearity and how range splitting can be used to further extend the analytical range.

Beginning in the 1950's, the U.S. Department of Agriculture's Instrumentation Research Laboratory, under the direction of Mr KarlN orris, identified and developed a new technique for the analysis of food and agricultural products. ! This new technique utilized the spectral absorption characteristics of a sample to quantitate the level of the constituent of interest: e.g., the percentage of protein in wheat.

In the early 1970's, manufacturers began providing commercial instruments utilizing the wavelengths which had been identified by the USDA. These early instruments required tedious on-site calibration. As these early instruments evolved, the Technicon InfraAlyzer 2.5A Near Infrared Reflectance Analysis (NIRA) system was introduced. Although its temperature-controlled, sealed optics module improved

FOOD & DAIRY SESSION

W. Carew, presiding
The Quaker Oats Company

Lipid Structures and NIRA

David Wetzel, Kansas State University, Manhattan, KS

Dr. Wetzel is a Professor in the Department of Grain Science and Industries. An analytical chemist. his research interests include the application of modern separation and spectroscopic techniques including HPLC and NIRA, to the evaluation of grain and cereal product quality.

A chromatographic characterization of triglycerides in hydrogenated soybean oil is presented. This characterization attempts to quantify the chemical phenomena that affect the solid fat index (SFI) of hydrogenated oils. The chromatographic description of the triglyceride exhibits good correlation to SFI data and is fairly well correlated to NIRA spectral data. It is hoped that this approach may lead to an alternate method of determining oil functionality, other than the time-consuming manual SFI procedure.

The Use of NIRA to Determine Iodine Value in Oils

Adele Preston, Technicon Industrial Systems, Tarrytown, NY

Ms. Preston joined Technicon seven years ago, and she has been in the NIRA Applications Department of TIS for the past year, concentrating on applications in the processed food and edible oil industries She holds a BS in Biology from the College of Notre Dame Maryland, and her experience includes work in clinical *pharma*cology, immunology, chromatography and radioimmunoassay.

The determination of Iodine Value, a measure of the average number of double bonds, is important for the characterization of oils following hydrogenation. The NIRA method for Iodine Value has significant advantages over the time-intensive manual method, including elimination of the need for glassware and dangerous reagents. Accuracy of the NIRA method is equivalent to that of the manual method.

A NIRA method for SFI is also being investigated.

Process Control Applications for Cereal and Dehydrated Vegetables

Ann Miners, General Foods Corporation, Modesto, CA

Ms. Miners is Laboratory Manager at General Foods Modesto plant. She has been with General Foods for five yeaʳs. working in areas of raw material microbiological and analytical chemistry testing

At General Foods' Modesto plant, NIRA is used to analyze moisture, total sugars, reducing sugar and fat in cereals, and moisture in dehydrated vegetables. Technicon InfraAlyzer 400R systems have been placed in the cereal and vegetable areas to permit production personnel to run their own in-process analyses rather than having samples analyzed by laboratory personnel. We feel that use of NIRA systems can save time and free up people for other duties.

For cereals, ranges may be up to 36% moisture, 46% total sugars, 16% reducing sugar and 9% crude fat. For dehydrated vegetables — spinach, celery, green peppers, beets and carrots — moisture contents may range up to 13%.

stability and reliability, difficult calibration techniques still plagued this and other NIRA systems.

In the late 1970's, advances in the microprocessor industry contributed significantly to the introduction of improved NIRA instrumentation. The routine availability and sophistication of personal computers simplified the development of instrument calibration constants. Users could now generate their own "custom calibrations," and practical calibrations were largely reduced to button-pushing. Tighter component specifications and internal referencing procedures resulted in more stable instruments without the drift problems inherent in earlier systems.

Now, Technicon offers calibrations for a wide range of applications in food, dairy, meat, pharmaceutical, chemical, textile, agricultural and many other areas of interest.

InfraAlyzer 400D Dairy System: Experimental Data

Karen Luchter, Technicon Industrial Systems, Tarry town, NY

Dr. Luchter, Project Scientist for TIS, has directed the InfraAlyzer Applications Laboratory for four years. Dr. Luchter's work includes research in the fundamentals of NIRA, methods development and publications in the fields of mechanistic and synthetic organic chemistry.

A brief outline of the steps necessary for developing a constituent calibration was presented. This was followed by a typical HP-85 data printout on prediction results for fat and total solids in white and chocolate ice cream mixes. The calibrations used were developed for one ice cream mix, and then transferred to another instrument and used to predict a different supplier's ice cream mix formulation. This was done on both white and chocolate mixes.

Data from mini-studies on various process conditions for fat and total solids assays in milk was also presented. Specifically, data on preserved, pasteurized, raw, homogenized and water-diluted milk was shown. The unique hardware features engineered into the Technicon InfraAlyzer 400 D system to accomplish this performance were described.

NIRA Spectra of Celite at Low and High Humidity, With and Without Oil

George Anderson, Pillsbury Company, Minneapolis, MN

Dr. Anderson holds a Ph.D. in Physical Chemistry from the University of Iowa and is involved with spectroscopic analysis of food products at Pillsbury.

Celite (diatomaceous earth), a powder used in the food industry as a filtering aid, was analyzed using a Technicon InfraAlyzer 500 Scanning Spectrophotometer. The analyses were carried out on dry and moist celite, with and without oil, and differences in spectra were noted.

Apparently, oil does not "wet" moist celite as effectively as dry celite. Moist celite/oil was more reflective over the 1100 to 2500 nm range than dry celite/oil. It may be that the differences in spectra came about because of varying penetration depths. These "negative" shifts in log (1/R) due to water adsorption must be accounted for in NIR analysis of mixtures, diluents and complex food systems.

Field Applications of the InfraAlyzer 400D System in a Cheese Processing Plant

Gregg Roemhild, Twin Town Cheese Factory, Inc.,Almena, WI

Mr. Roemhild, Production Manager of Twin Town Cheese Factory since 1975, is responsible for total whey and cheese plant production. He has had extensive experience with agricultural and dairy products, and has been involved with NIRA since 1981.

The InfraAlyzer 400D system is currently used to analyze the three major products of our plant: raw milk, raw whey and dry whey. Raw milk is analyzed for fat, solids, protein and lactose. Raw whey is analyzed for fat, solids and protein. Dry whey is analyzed for moisture and protein.

All our laboratory personnel can operate the 400D with little training. The laboratory supervisor reprograms and does some troubleshooting. The 400D system is operated daily. With this system, we are able to run at a rate of 50 samples per hour compared with 35 to 40 samples per hour when the operator performs tests manually.

The cost-per-sample averages 18^{C} for liquids and even less for dry products. This cost includes labor but does not include the initial cost of the analyzer.

With regard to accuracy, bias adjustments are made about twice a month on milk fats, and four times a month on protein and solids. Raw whey calibrations are more stable; some months, no bias adjustments are made. The dry whey calibration, which was the most complicated to develop, has been so stable that we have gone more than two months without bias adjustments.

The most recent development in NIRA instrumentation is the introduction of a high-speed, computerized scanning spectrophotometer: the Technicon InfraAlyzer 500 system. Designed specifically for laboratories interested in methods development and non-routine analysis, the Technicon InfraAlyzer 500 system combines high-resolution NIR spectral measurements with multilinear regression transforms, to allow users to develop calibrations for an extensive range of materials including those which exhibit poor spectral resolution. Once these methods have been developed, the unique UTC (User-Transferable Calibration) feature of all Technicon InfraAlyzer systems, is used to directly transfer calibrations to less-costly dedicated instruments.

Our 400D was purchased to save us time in analyzing and to help monitor our products to increase profit and give better product control.

We had some initial problems with setting up and calibrations, but with the help of the Technicon Applications staff, these problems were solved. We now obtain good results in our product analysis.

Future applications of the 400D for measurement of other constituents in cheese and other products are expected to result in further increases in efficiency and profitability.

New Directions for NIRA in Food and Dairy

James J. Psotka, Technicon Industrial Systems, Tarrytown, NY

Mr. Psotka, Marketing Manager of Food and Dairy Industries, was previously in the NIRA Applications Dept. at TIS. Prior to joining Technicon in 1981. he was with The Quaker Oats Company for 13 years, most recently as Chief Chemist at the Cedar Rapids, Iowa plant.

As many instrument users have discovered, the non-traditional applications of NIRA have opened new avenues of approach for the solution of so-called "unsolvable" process control problems. The challenge is given for NIRA users that once the pressing needs of methods development for traditional process control applications have been satisfied, imaginative approaches should be sought to these "unsolvable" problems. Mr. Psotka used as an example the potential ability of cheese processors to determine the best time to change brining mixture for the preservation of product quality.

PLENARY SESSION II

R. Spies, presiding
U.S.D.A., Albany, CA

From Measurements to Analysis: The Calculation of NIRA'

(Biographical information on Dr. Hirschfeld is included with Abstract of paper in Plenary Session I)

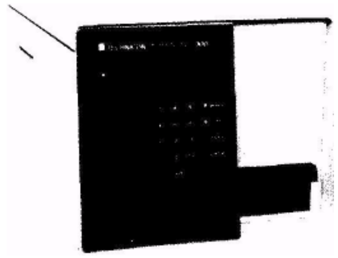
Tomas Hirschfeld, Lawrence Livermore National Laboratory, Livermore, CA

After collecting a reference sample data set, a number of calculations are used to produce the NIRA analytical procedure:

1. The spectra are examined to decide which ones are useful enough to require lab analysis. The value of this step depends on the logistics involved.
2. After lab analysis, the extreme spectra are examined, and "absorbance-subtract" is used to locate "must" wavelengths to force selection of measured wavelengths and calibration coefficients. The selection of wavelength range and resolution, or of a set of filters for this selection, can be done by a few simple rules of thumb.
3. The selection procedure is then run in the computer, using one of three procedures: row reduction, sequential wavelength search, or the "all-possible combinations" search. The latter is more accurate but much slower, and is, therefore, hard to improve further by repeated trying. For this search one may specify the type of function desired (log 1/R = absorbance, differences, derivatives, corrected absorbance). Again, a few rules of thumb can be used for this choice, and also for the choice of the number of wavelengths.

Work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract W-7405-Eng-48.

A continually expanding line of state-of-the-art instrumentation, supported by the industry and technological specialists in Near Infrared Reflectance Analysis.



4. The resulting calibrations (or set thereof, if stacked operation is desired) are checked for internal consistency by checking the standard error outputs of the program. If acceptable, these are then rechecked with a verification set. At this stage, it is possible to go back to the wavelength selection and calibration coefficient search by deleting outliers, refining wavelength choices, or using indicator variables, to get further improvement. The goal of the exercise — a routine procedure for NIRA calibrations requiring a minimum of experience — seems to be in sight.

Near Infrared Spectral Search

James Rothrock, Milliken Research Corporation, Spartanburg, SC

Mr. Rothrock, a Research Chemist in the Analytical Chemistry Laboratory, has been with Milliken for 17 years. He is presently involved in thermal and infrared analytical work and problem-solving.

A method of using Near Infrared for qualitative determination, and also of using search routines as an additional aid to identification, was reported.

Search routine is primarily used for liquids, which are scanned through the near infrared region using a transmission instrument. The wavelength in nanometers and the percent transmission of each peak are recorded. The near infrared spectral data is then converted to a format generally used for mid infrared spectra. The data can then be entered into a data station having search capabilities. After manually entering data, the search routine can be run in the same manner as any mid infrared data. A library of liquids processed in the same manner is searched to locate the best match.

(Biographical information on Dr. Wetzel is included with Abstract of paper in FOOD & DAIRY SESSION)

Enhanced Data Entry for Routine Analysis with The InfraAlyzer 400 System

David Wetzel, Kansas State University, Manhattan, KS

The use of the InfraAlyzer 400 system for the analysis of high volumes of wheat samples during the peak production season has resulted in a need for a more rapid method of sample coding and sample number entry during analysis. Through the adaption of peripheral devices to the HP-85 microcomputer, a bar code sample-numbering system has been implemented which allows the operator of the Technicon InfraAlyzer system to "read" the bar code, run the sample on the InfraAlyzer system and store the sample data on magnetic tape for later output.

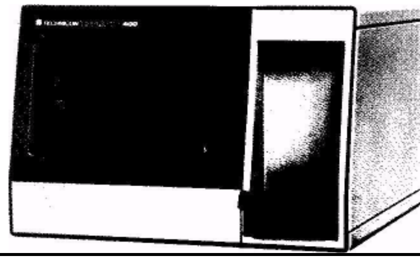
FTIR Transforms

W. F. McClure, North Carolina State University, Raleigh, NC

Dr. McClure, a Professor in the Department of Biology and Agricultural Engineering, has worked with NIRA since 1971. He is presently concerned with the development of instrumentation and techniques for measuring quality and composition of agricultural products.

As few as 11 Fourier coefficients computed from NIR spectra recorded with dispersion-type instrumentation can be used to estimate chemical composition of tobacco, forages and other products. Standard errors of estimate from an 11-term, step-wise calibration in the Fourier domain, are equivalent to standard errors of estimate using a 7-term log 1/R model in the wavelength domain. Since only 12 numbers are required per spectrum, computation time for calibration can be reduced by *96%* and storage requirements can be reduced by 98%. Removing the mean term from consideration during a Fourier calibration, corrects for variations in particle size.

[redacted]



Technicon InfraAlyzer 400 system

Derivative Treatments vs. Log 1/R

(Biographical information on Mr..Honigs is included with abstract of paper in PLENARY SESSION II

David Honigs, Indiana University, Bloomington, IN

There has been much debate among NIRA users as to the proper way to use reflectance data. Some claim that it is best to use log 1/R directly, while others claim that the first or second derivative of log 1/R is better. In general, it is difficult to prove that one data treatment is better than another. There are cases where log 1/R has been shown superior, and cases where derivatives have worked best. This may be because the instrumental signal-to-noise ratio is so large that any data-processing technique will work.

Proponents of the derivative methods have made the claim that the derivatives eliminate the bias and slope effects of variable sample preparation. This is indeed true. However, from recent work using Fourier transforms of NIRA spectra, it appears that broad spectral features carry the most information about sample composition. This suggests that derivative may not be the optimum data-processing technique.

PHARMACEUTICAL AND INDUSTRIAL SESSION

S. Gabriel, presiding
Milliken Research Corporation

Detergents: Actives and Moisture

Mr. Pietrantonio holds AAS and BS degrees in Chemical Engineering and Chemistry from the State University of New York. He has been associated with the development of automated methods for 14 years, and is presently a Supervising Scientist in charge of near infrared methods development and applications for the pharmaceutical, chemical, textile, meat and dairy industries.

Anthony Pietrantonio, Technicon Industrial Systems, Tarrytown, NY

NIRA was applied to the analysis of moisture, active ingredients, softener and phosphate in powdered detergents, and the suitability of NIRA was discussed. Various aspects of the methods developed — sample handling, data acquisition and analysis — were discussed. Results for moisture and actives were presented, and the potential for this technique in the detergent industry highlighted.

NIRA Applications in the Pharmaceutical Industry

Mr. Weedon, Senior Marketing Manager for TIS, received his BS from Saint Joseph's University in Philadelphia in 1976 Prior to joining Technicon in 1980, he was a Biochemist for SmithKline 8eckman Corporation in Philadelphia

Roy Weedon, Technicon Industrial Systems, Tarrytown, NY

NIRA has been successfully used in the Pharmaceutical Industry to perform rapid multi-component analysis and to identify raw materials and finished products. Practical applications relating to quantitative and qualitative analysis of pharmaceutical products are presented. The applications include multi-component analysis of parenteral drug solutions; intermediate compound analysis; bulk product analysis for purity, moisture and residual solvents; and identification of raw materials, finished products and packaging materials.

Technicon InfraAlyzer 500 system

Basics of Discriminant Analysis

Howard Mark, Technicon Industrial Systems, Tarrytown, NY

Dr. Mark, a Scientist with TIS, has been involved with NIRA for the past seven years. He is primarily concerned with investigating new approaches to handling data and determining ways to analyze data in order to optimize the use of Technicon NIRA equipment.

The application of the mathematical technique, Discriminant Analysis, to data collected by near infrared reflectance spectrometers is a promising approach toward using NIRA technology for qualitative as well as quantitative analysis. The technique is a mathematical way of determining how well spectra match, by looking at each spectrum as a point in multidimensional space. If a spectrum of an unknown material is "near" the spectrum of a known material when both are represented in this multidimensional space, then the unknown is probably the same as the known, because the spectra match.

The determination of whether spectra are "near" each other is made by measuring the distance between them using a non-euclidean distance measurement. The mathematical modeling of this distance measurement makes it possible to allow a computer to perform all the calculations and comparisons needed to classify an unknown sample, or to determine whether it does not fit any of the known categories.

AGRICULTURAL SESSION

W.F. McClure, presiding North Carolina State University

Rapid Analysis of Hops for Alpha and Beta Acids Using Near Infrared Reflectance Analysis

Ralph Pirritano, Technicon Industrial Systems, Tarrytown, NY

Mr. Pirritano has been an Applications Scientist with TIS for 16 years, and has specialized in NIRA for the past seven years. He holds a BA in Biological Sciences from Rutgers University and an MS in Environmental Health Sciences from New York University. He has presented several papers at the Pittsburgh Conference and is a member of the ACS.

The analysis of kiln-dried raw hops for alpha acids, and, to a lesser extent, beta acids, is the most important chemical measure of hops quality. A near infrared reflectance technique is described, which correlates well with the ASBC spectrophotometric method for alpha and beta acids. Multiple correlation coefficients of 0.99 and 0.97 were observed for alpha and beta acids, respectively.

Analysis time with the near infrared instrument is approximately 3 minutes, including sample preparation, compared to about 45 minutes for the spectrophotometric method. Regression data based on over 200 hops samples, and a sample handling procedure, are discussed.

Near Infrared Reflectance Spectroscopy to Analyze Forages

J. E. Winch, Crop Science Department, E. Valdez, Animal/Poultry Science Department and L.G. Young, Animal/Poultry Science Department, University of Guelph. Guelph, Ontario, Canada

Professor Winch has been in-charge of the Laboratory for Quality Analysis of forage since 1959. His main areas of concern are forage production research, with concentration on pasture production and physiology and forage quality. His work with NIRA instrumentation for the past five years includes installation of a forage filter

The study of near infrared reflectance has indicated that NIRA equipment can be used for analysis for pure species of grasses, for legumes and mixtures of grass and legumes, and for crude protein and *in-vitro* digestibility. The standard errors of prediction are within acceptable limits: 0.9 to 1.2% for crude protein and 2.2 to 3.1% for *in-vitro* digestibility.

The estimates of concentration of acid and neutral detergent fibers of forages were not as accurate as those of crude protein or *in-vitro* digestibility, but the standard error of prediction may be acceptable under some circumstances, such as where a simple ranking of breeding lines is required.

Calibrations are also being developed for haylage and corn silage. The parameters of concern are crude protein, *in-vitro* digestibility, acid and neutral detergent fiber and some mineral elements.

To insure continued use of calibrations, the range of moisture level in the samples used in the calibrations must be known, and the samples to be analyzed should be allowed to equilibrate to the moisture level for the calibration. Also, the particle size of the samples should be uniform and similar to that of the calibration samples.

Determination of Protein in Vegetative Alfalfa and Small Grains Using NIRA

Robert A. Isaac and William C. Johnson, University of Georgia, Athens, GA.

Dr. Isaac is Professor of Chemistry and Director of the soil testing & Plant Analysis Laboratory. Among other papers, he has presented at each of the previous NIRA Symposia, FACSS Conference and 1981 American Society of Agronomy Conference

The determination of protein nitrogen in vegetative alfalfa, wheat and corn appear to be quite feasible using the technique of NIRA. Correlation coefficients of 0.98 plus for wheat and corn, and 0.84 for alfalfa, were achieved, when compared to standard Kjeldahl analysis. (The alfalfa values can be improved significantly with better sample distribution; only six weeks' time had been devoted to the alfalfa at symposium time.)

The ability of the wheat and corn matrices to predict unknowns is similar. Approximately 99 percent of the samples tested agreed within 0.3% absolute with the Kjeldahl value. In addition, corn samples from the Midwest gave similar results on the Georgia matrix.