

Application Bulletin 409

Analysis of chemicals using near-infrared spectroscopy

Branch

Chemicals

Keywords

Near-infrared spectroscopy, chemicals, process monitoring, chemical reaction, moisture determination, hydrocarbon, acid, resin, solvent.

Summary

This Application Bulletin shows the examples of NIR applications and the feasibility studies using NIRSystems in the chemical industry. It includes qualitative and quantitative analysis of various samples and parameters. Each application briefly describes the measuring systems used in the studies as well as the recommended instruments and the test results.

Introduction

NIR is extremely sensitive to the O–H absorbance; moisture determination is therefore a common application. Additionally, NIR is sensitive to N–H, C–H, and S–H bond absorbance, for what reaction monitoring and endpoint determination are good applications for organic chemistry reactions and processes. Many applications that have historically been measured with physical property testing, for example viscosity, may be measured with NIR if it is dependent on an intrinsic chemical characteristic, such as chain length or cross linking. NIR spectroscopy is well accepted as a rapid and accurate analysis method. Metrohm NIRSystems offer NIR instruments suitable to use in all working environments: laboratory, atline, inline and online measurement.

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No. 1: Monitoring surfactants in a water/isopropyl alcohol mixture

Summary

This application shows that NIR spectroscopy can be used to monitor surfactants in a water/isopropyl alcohol mixture. Thirty samples were provided for analysis ranging in total surfactant concentration from 22.02–30.56.

Instrument

Model 5000, transmission detector module, fiber optic bundle setup module, interactance fiber and immersion probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS SmartProbe Analyzer 2m Fiber	2.921.1610
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Sampling

Samples were scanned using an interactance immersion fiber optic bundle probe and 1 mm pathlength cuvette.

Spectra are recorded in the 1100–2500 nm wavelength range. A least-squares regression was performed at 1722 nm and the standard error of calibration (SEC) is 0.6 for monitoring total surfactant concentration.

Results

The results indicate that NIR can be used to develop a correlation between total surfactant concentration and peak intensity at 1722 nm.

No. 2: Monitoring the presence of a surfactant in a water solvent

Summary

This application note presents the test result of using NIR to monitor the presence of a surfactant in a water solvent. The samples had surfactant concentration ranging from 0.28–1.05%.

Instrument

Model 5000, transmission detector module, fiber optic bundle setup module, interactance fiber and immersion probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS SmartProbe Analyzer 2m Fiber	2.921.1610
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Sampling

All spectra were collected in the 400–1100 nm range using a fiber optic interactance immersion probe attachment. Only the spectral region from 400–1100 nm was used for this study due to the strong absorbance of water in the NIR region. An effective pathlength of 2 mm was used for this study. A least-squares regression for the surfactant was performed at 1050 nm (SEC of 0.04%).

Results

The results indicate that NIR can be used to monitor surfactant in a process reaction.

No. 3: Monitoring monoethylene glycol (MEG) in water

Summary

This application shows the use of NIR technique to monitor monoethylene glycol (MEG) in water. Thirteen samples with MEG concentration ranging from 0.05–1.08% were used for calibration. Three additional samples (MEG concentration 0.21, 0.57 and 0.59%) were used to validate the calibration model.

Instrument

Model 5000, transmission detector module, fiber optic bundle setup module, interactance fiber and reflectance probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS SmartProbe Analyzer 2m Fiber	2.921.1610
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Sampling

The samples were analyzed in the 1100–2500 nm region using a fiber optic interactance immersion probe. A pathlength of 2 mm was used for analysis. Due to the low concentration of MEG in water, each calibration sample was analyzed three times, repositioning the probe each time. Three spectral bands could be used to monitor MEG with minimal interference from the water: 1676, 1588, and 1710 nm. A regression model was developed at 1676 nm (SEC of 0.04%).

Results

The results indicate that NIR can be used to develop a correlation between total surfactant concentration and peak intensity at 1722 nm.

No. 4: Monitoring the level of hydrogen peroxide in water

Summary

This application briefly shows how NIR analysis is used to monitor the level of hydrogen peroxide in water. The samples range in peroxide concentration from 0–4% (by volume).

Instrument

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410
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Sampling

The samples were analyzed in transmission mode in the 1100–2500 nm region. The 2100 nm region shows an absorption for peroxide with no interference from the water. A calibration was developed at 2110 nm (SEC of 0.2%).

Results

The results indicate that NIR can be used to determine peroxide levels in water easily at the 0-4% level.

No. 5: Qualitatively differentiating aqueous solutions of aluminum chlorhydroxide and AIC136H20, and solids aluminum zirconium tetrach\glyp and glycine

Summary

This feasibility study presents the result of using NIR analysis to qualitatively differentiate aqueous solutions of aluminum chlorhydroxide and AIC136H20, and solids aluminum zirconium tetrach\Glyp and glycine.

Instrument

Model 5000, transmission detector module, fiber optic bundle setup module, interactance fiber and immersion probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS SmartProbe Analyzer 2m Fiber	2.921.1610
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Sampling

The samples were analyzed using a fiber optic immersion probe at 1 mm for the liquids. The solids were measured with the sample probe, but using the reflectance tip instead. The samples were analyzed in the 1100–2500 nm region. Differences could be seen throughout the spectra, therefore samples should be distinguishable.

Results

The results indicate that NIR can be used to monitor the level of dye in water based dye solutions.

No. 6: Monitoring water, methyl acetate, and methanol

Summary

This application proves that NIR spectroscopy can be used to monitor several parameters simultaneously like water, methyl acetate, and methanol in a mixture made of the three constituents.

Instrument

Model 5000, transmission detector module, fiber optic bundle setup module, interactance fiber and immersion probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS Process Analyzer MicroBundle SinglePoint	2.928.0110
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Sampling

Twenty samples were provided with water concentration ranging from 0–5%, methyl acetate concentration from 0–49%, and methanol from 49.7–99.7%. Acetic acid concentration remained constant at 0.3% throughout the samples. The samples were analyzed using a process analytics instrument in the 1100–2500 nm region. An interactance immersion fiber optic probe set to a 1 mm pathlength was utilized. Water had a unique absorption at 1920 nm. A calibration was developed at this wavelength (SEC of 0.2%). Methyl acetate was monitored using a single wavelength calibration at 1674 nm (SEC of 0.8%). Methanol was monitored at 2086 nm (SEC of 1%).

Results

The results indicate that NIR can be used to monitor water, methyl acetate, and methanol in these samples.

No. 7: Monitoring moisture in ethylene glycol samples

Summary

This application shows the use of NIR spectrometer to monitor moisture content in ethylene glycol samples. Six samples were provided with moisture ranging from 0–5%.

Instrument

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410
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Sampling

The samples were analyzed in transmission mode using a 2 mm pathlength cuvette. The 1100 to 2500 nm range was scanned. A calibration for moisture was developed at 1960 nm (SEC 0.1%).

Results

The results indicate that NIR can be used to determine moisture levels in ethylene glycol to better than +/- 0.2%.

No. 8: Monitoring various 1,4-butanediol chemical systems

Summary

The objective of this feasibility study was to determine if NIR could be used for certain chemical systems. The samples provided were a) 1,4-butanediol, poly(tetramethyleneglycol) and polyurethane mixture, b) 1,4-butanediol and c) poly(tetramethyleneglycol).

Instrument

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410
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Sampling

All spectra were collected from 1100–2500 nm in transmission mode. All samples were scanned in a 1 mm quartz cuvette. Unique absorptions for poly(tetramethyleneglycol) were found at 1225, 1825 and 1940 nm. For 1,4-butanediol, unique absorptions were found at 1490, 1800, 2075, and 2375 nm.

Results

The results indicate that NIR can be used to determine unique absorptions for the above stated materials.

No. 9: Monitoring water in monochlorobenzene

Summary

This feasibility study was aimed to prove that NIR can be used to monitor water in monochlorobenzene at the hundreds of parts-per million level.

Instrument

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument



NIRS XDS RapidLiquid Analyzer 2.921.1410

Sampling

The samples were analyzed in transmittance mode in the 1100–2500 nm region. Analysis was done in a 1 mm pathlength cuvette.

Results

The results indicate that NIR can be used to monitor water at the hundreds of parts-per-million level in monochlorobenzene. While a calibration was not performed due to the difficulty in preparing standards, NIR calibrations are possible, using laboratory data on production samples. The determination can be done either in the 1900 nm region using cuvettes, or in the 1400 nm region, using a fiber optic probe.

No. 10: Detection of water in methane sulfonyl chloride, methane sulfonic acid and distinguishing between these substances

Summary

This study shows that NIR analysis can be used to detect water in methane sulfonyl chloride (MSC) and methane sulfonic acid (MSA) in the very low concentration range. For the MSC study, the water concentration ranged from 250–1000 ppm, while for the MSA, the concentration was 0–1%.

Instrument

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument



NIRS XDS RapidLiquid Analyzer 2.921.1410

Sampling

All spectra were collected in transmittance mode in the 1100–2500 nm region. Moisture was monitored in the 1940 nm region. A calibration for moisture was developed at 1962 nm with a SEC of 0.05%. For distinguishing between MSC and MSA, the 2360 nm region was used to develop a calibration (SEC of 0.07%).

Results

The results indicate that NIR can be used to monitor water in both MSC and MSA. It is also possible to distinguish between the MSC and MSA. Several spectral regions were identified.

No. 11: Monitoring the chemical reaction of nonene with diphenylamine

Summary

This NIR application is used to monitor the chemical reaction of nonene with diphenylamine. Ten partially reacted samples were provided for calibration purposes. The samples ranged from 1.1–1.8% nonene.

Instrument

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410
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Sampling

All spectra were collected in transmittance mode in the 1100–2500 nm region. Moisture was monitored in the 1940 nm region. A calibration for moisture was developed at 1962 nm with a SEC of 0.05%. For distinguishing between MSC and MSA, the 2360 nm region was used to develop a calibration (SEC of 0.07%).

Results

The results indicate that NIR can be used to monitor water in both MSC and MSA. It is also possible to distinguish between the MSC and MSA. Several spectral regions were identified.

No. 12: Monitoring levels of styrene-butadiene resin dissolved in methylene chloride

Summary

This feasibility study reports the result of monitoring the levels of styrene-butadiene resin dissolved in methylene chloride using NIR technique. The samples range from 0–11.55% resin.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410
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Sampling

The spectra were collected in transmission mode in the 1100–2500 nm region. A 1 mm quartz cuvette was used for the liquid polymers. The pure resin was analyzed in reflectance mode. A calibration equation was developed at 2164 nm (SEC of 0.04%). Another equation was developed at 1210 nm (SEC of 0.05%). This region could be enhanced by increasing the pathlength, which would make sample handling for lab and online measurements easier and more reliable.

Results

The results indicate that NIR can be used to monitor the level of styrene-butadiene resin, dissolved in methylene chloride.

No. 13: Determination of low level water in methyl isocyanate

Summary

This feasibility study was aimed to evaluate NIR's ability to monitor levels of water below 550 ppm in Methyl Isocyanate.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410
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Sampling

Six samples covering the range of interest were prepared and placed in 20 mm pathlength, screw-top, quartz cuvettes. The samples were scanned in transmission from 400–2500 nm with the region above 1600 nm being disregarded because of the expected high absorbance due to the large pathlength. Sample Selection was performed identifying all samples to be used for the Calibration Set. A second derivative math pre-treatment using a 10 nm segment and 0 gap was applied to the raw spectra and a Multiple Linear Regression performed.

Results

A single wavelength calibration equation was developed at 958 nm (a known water absorption region) producing a correlation (R^2) of 0.975 and a Standard Error of Calibration (SEC) of 36.5 ppm. The accuracy and precision of this equation can be improved through the use of more calibration samples.

No. 14: Monitoring the presence of ammonia in vinyl pyrrolidone

Summary

This NIR application is used to monitor the presence of ammonia in vinyl pyrrolidone (VP) in the very low concentration). Eight VP samples were analyzed with ammonia content ranging from 0–100 ppm dissolved ammonia.

System

Model 6500, sample transport module, transmission detector module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410
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Sampling

The samples were analyzed in the 400–2500 nm region in transmission mode. A 10 mm quartz cuvette was utilized for analysis.

Three portions of the spectrum were identified and related to N-H absorptions: 1990 (N-H combination band), 1524 (N-H first overtone), and 1040 nm (N-H second overtone). Each of these bands showed increasing intensity with increasing ammonia content. A calibration for monitoring ammonia was developed using the 1524 nm band (SEC of 14.0 ppm). Calibration models were also developed at the two other bands, however, the presence of interferences required more rigorous calibration methods to be used than linear least-squares.

Results

The results indicate that NIR can be used to monitor dissolved ammonia in vinyl pyrrolidone.

No. 15: Monitoring a binary mixture of N-methylpyrrolidinone and polyethylene glycol

Summary

This NIR application is used to monitor a binary mixture of N-methylpyrrolidinone (NMP) and polyethylene glycol (PEG). Five samples were provided for calibration, and one unknown was provided for validation. The NMP concentration ranged from 51.26–56.96%.

System

Model 5000, transmission detector module, fiber optic bundle setup module, interactance fiber and immersion probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS SmartProbe Analyzer 2m Fiber	2.921.1610
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Sampling

All samples were analyzed in transmission mode in the 1100–2500 nm range using an interactance immersion probe. A pathlength of 2 mm was used for analysis. The 1700 nm band increases with increasing NMP concentration. Since this is a binary system, the components are inversely correlated, one band can be used to monitor both constituents. The calibration at 1704 nm for NMP and PEG yielded a SEC of 0.03%.

Results

The results indicate that NIR can be used to quantitatively monitor N-methylpyrrolidinone and polyethylene glycol in a binary mixture.

No. 16: Monitoring the levels of water and fluorosulfonic acid in a mixed hydrofluoric-sulfuric acid stream

Summary

This feasibility study presents the result of using NIR analysis to monitor the levels of total water and fluorosulfonic acid in a mixed hydrofluoric-sulfuric acid stream.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410
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Sampling

The samples of the different acid mixtures were placed in a 1 mm quartz cuvette and analyzed in transmittance mode. The spectral range was 1100–2500 nm. Water bands appear in the 1450 and 1940 nm regions.

Results

The results indicate that NIR can be used to monitor water in a mixed acid stream of HF and sulfuric acid. It does not appear feasible to measure HFSO_3 , however, since no spectral features unique to this acid could be found which were not affected by the other components in the stream.

No. 17: Monitoring a process stream of diethylaniline (DEA) and diethylphenyl azomethine (AZO)

Summary

This application shows the use of NIR analysis to monitor a process stream of Diethylaniline (DEA) and Diethylphenyl Azomethine (AZO). Three samples were provided, the two pure components and a sample of 30/70 mixture of DEA versus AZO. A series of calibration samples were made from the two pure component samples. Ten samples were made with DEA concentration from 5–80%.

System

Model 5500, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410
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Sampling

The samples were analyzed in transmission mode from 400–1100 nm. The 20 mm pathlength was used, and the samples were measured at ambient temperature. DEA has a different color absorbance at 420 nm, and a strong absorption was seen at 1022 nm. A calibration was developed for DEA at 1022 nm (SEC of 0.6%).

Results

The results indicate that NIR can be used to monitor the concentration of DEA in a two component process stream of DEA and AZO. A linear response appears from 5–80% DEA.

No. 18: Monitoring methoxypropyl-amine and N,N-diethylaminoethanol in a complex mixture

Summary

This feasibility study presents the use of NIR to monitor methoxypropylamine without interference from cyclohexylamine or monoethanolamine in water based mixtures and also to monitor N, N-diethylaminoethanol in the presence of a complex mixture.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410
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Sampling

Each sample was diluted in water to approx. 10%. The spectra were collected in the 1100–2500 nm region. The following components can possibly be monitored at the following wavelengths: methoxypropylamine at 2285 nm, N, N-diethylaminoethanol at 2390 nm, cyclohexylamine at 2380 and 2430 nm, morpholine at 2095 nm. No unique bands were found for monoethanolamine (at least 1 interference was present).

Results

The results indicate that NIR can be used to quantify four of the compounds in the presence of the others in a water based mixture. Only monomethanolamine did not have a unique absorption in the presence of the other components, and therefore the ability to quantitatively measure it is questionable.

No. 19: Monitoring low level hydrocarbons and moisture in carbon tetrachloride

Summary

This feasibility study was aimed to show that NIR can be used to detect low level hydrocarbons and moisture in carbon tetrachloride. Five samples were analyzed.

System

Model 5000, liquid sampling system and Model 5000, transmission detector module, single fiber optic setup module, interactance fiber and immersion probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410
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Sampling

Two approaches for sampling were used: cuvettes for laboratory simulation, and single fiber optics for process simulation. The samples were analyzed in the 1100–2500 nm region using a pathlength of 10 mm. Using cuvettes, moisture bands were observed at 1920 and 1440 nm. Also observed were hydrocarbon bands between 1600 and 1800 nm. Using single fiber optics, the same spectral features are evident.

Results

The results indicate that NIR can be used to detect low level hydrocarbons and moisture in carbon tetrachloride. The analysis can be performed using either cuvettes or a single fiber optic probe. The limitations of the single fiber are signal attenuation above 2300 nm, and sensitivity to bending in the fiber.

No. 20: Monitoring water, chloroform, carbon tetrachloride, dibromobenzene, chlorine, sulfate and oil in a bromine liquid

Summary

This application shows that NIR spectroscopy can be used to monitor several parameters simultaneously even in the very low concentration like water (13–55 ppm), chloroform (13–36 ppm), carbon tetrachloride, dibromobenzene, chlorine (20–148 ppm), sulfate and oil (0.1–0.23 ppm) in bromine liquid.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410
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Sampling

The samples were analyzed in transmission mode in the 1100–2500 nm region. A 20 mm pathlength cuvette was used for analysis. As with bromine, carbon tetrachloride, chlorine, and sulfate do not show NIR absorptions. Therefore, calibrations for these components were not performed. A calibration developed for moisture at 1920 nm yielded a SEC of 4 ppm. For chloroform, a calibration was developed at 2254 nm (SEC of 4 ppm). For oil, a calibration was developed at 2206 nm (SEC of 0.03 ppm).

Results

The results indicate that NIR can be used to monitor moisture, chloroform, and oil in bromine liquid. The remaining constituents do not have NIR absorptions, therefore, NIR cannot be used to monitor these constituents.

No. 21: Monitoring parts-per-million (PPM) levels of moisture in phenol

Summary

This feasibility study was aimed to prove the NIR's ability for monitoring parts-per-million (ppm) levels of moisture in phenol. A total of seven samples were analyzed with the concentration of moisture ranging from 170–10,000 ppm.

System

Model 5000, sample transport module, transmission detector module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410
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Sampling

The samples were analyzed in the 1100–2500 nm region in transmission mode. A 10 mm pathlength sealed cuvette was used for analysis. Each phenol sample was loaded into the cuvette under a nitrogen atmosphere, and then allowed to equilibrate for ten minutes to a temperature of 50 degree Celsius.

Spectral changes in the 1912 nm moisture band were easily identified. A least-squares regression performed at this wavelength yielded a SEC of 66 ppm. By removing an outlier sample which was 'estimated' to be 630 ppm moisture, the SEC was reduced to 36 ppm.

Results

The results indicate that NIR can be used to monitor moisture in phenol.

No. 22: The determination of water in emulsions

Summary

The objective of this study was to measure the concentration of water in emulsion samples with varying levels of turbidity.

System

NIRSystems Model 6500 monochromator with a Liquid Sample module at room temperature. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410
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Sampling

Eight samples with water concentrations from 23–44% were prepared. The samples were shaken vigorously to ensure sample homogeneity then scanned at room temperature in transmission from 400–2500 nm in 4 mm quartz cuvettes followed by measurement in 8 mm disposable cuvettes.

Results

Applying a second derivative math pretreatment to sample spectra collected in the 4 mm cuvettes and using a simple linear regression at wavelength 1466 nm (SEC of 1.37) was produced. Predicting 10 samples held (SEP of 1.7). Using the same math pretreatment and regression technique for the samples collected in the 8 mm disposable cuvettes produced an equation at wavelength 980 nm (SEC of 1.17).

It was shown that NIR spectroscopy can be successfully used to measure water in emulsion samples, even when they are held at room temperature and exhibit turbidity.

No. 23: Monitoring free acid, hydroxyl amine, and ammonium sulfate in the process

Summary

This application shows that NIR spectroscopy can be used to monitor free acid, hydroxyl amine, and ammonium sulfate in the process stream in a temperature range of 23–45 °C. 28 samples were analyzed with concentration of free acid from 0.2–3.0 N, hydroxyl amine from 50–130 g/l, and ammonium sulfate from 5.0–17.50 g/l.

System

Process Analytics Model 6500 with interactance fibers and immersion probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS Process Analyzer	2.928.0110
MicroBundle SinglePoint	



Sampling

The samples were analyzed in the 1100–2500 nm region using an interactance immersion probe. A pathlength of 6.5 mm was used in the study of both fatty acids. The acid value was monitored at 1432 nm (SEC of 2). The low correlation was due to difficulty in maintaining a constant sample temperature, therefore, a denominator term was added to the model (1432/1500 nm, yielding a SEC of 1). Iodine value was monitored at 2118 nm (SEC of 1). Again, due to varying temperatures, 1804 nm was added as a denominator term, reducing the SEC to 0.5).

Results

The results indicate that NIR can be used to quantitatively monitor the amount of free acid, hydroxyl amine, and ammonium sulfate in a process stream.

No. 24: Monitoring moisture in acetonitrile

Summary

This feasibility study was aimed to prove the NIR's ability for monitoring moisture content in acetonitrile samples within the range of 50–200 ppm. Anhydrous liquid acetonitrile was received for analysis.

System

Process Analytics Model 5500, interactance fiber and reflectance probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS Process Analyzer	2.928.0110
MicroBundle SinglePoint	



Sampling

All samples were analyzed using a Process Analytics instrument scanning from 400–1100 nm. An interactance immersion probe with a pathlength of 2 cm was used. One hundred grams of acetonitrile was weighed into a beaker, then water was added using a micropipet with a glass capillary delivery tip. Two sample scans were collected for each level of moisture. A regression was performed at 958 nm. A standard error of 9.40 was obtained; however, after a time lag at 73.5 ppm was taken into consideration, the standard error was reduced to 4.3 ppm.

Results

The results indicate that NIR can be used to determine moisture content in acetonitrile samples. The lower limit of detection may be limited by the obtained standard error.

No. 25: Relating the tear properties of the finished product to the modified silica samples

Summary

This feasibility study was aimed to prove that NIR can be used to relate the tear properties of the finished product to the modified silica samples. Of thirty samples supplied, twenty seven were used for calibration and three were used as unknown. The mean tear ranged from 51.3–92.9.

System

Model 5000, reflectance detector module, spinning sample module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer Solids 2.921.1120



Sampling

All samples were analyzed in reflectance mode in the 1100–2500 nm region. Each sample was analyzed three times, reloading and rotating. The spectra were then averaged to obtain one spectrum for each sample. A calibration for mean tear was developed at 2262 nm (SEC of 14). A calibration for median tear was also developed at this wavelength with the same SEC. For tear range, a calibration was also performed at 2262 nm (SEC of 13). These three constituents were highly intercorrelated.

Results

The results indicate that NIR can be used to sense the tear parameter of the silica samples. This NIR method shows extremely high correlation for what is an indirect determination. Therefore, it may be that tear is related to a chemical modification in the base silica.

No. 26: Monitoring the level of vitamin E oil on a silica substrate

Summary

This NIR application is used to monitor the level of vitamin E oil on a silica substrate. The samples provided were the vitamin E oil, uncoated silica, and four samples of silica coated with various levels of vitamin E in the 39.3–51.7% range.

System

Model 5000, reflectance detector module, spinning sample module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer Solids 2.921.1120



Sampling

The samples were analyzed in reflectance mode in the 1100–2500 nm region. Three different portions of each sample were loaded into the sample cell, and then the spectra were averaged. The region above 2000 nm shows bands for vitamin E without interference from silica. A calibration was developed at 2088 nm (SEC of 1%).

Results

The results indicate that NIR can be used to monitor vitamin E added to a silica substrate. The vitamin E is not evenly distributed in the samples provided, so that the samples used for the primary analysis and the NIR measurements may not have had the same levels of vitamin E. This inhomogeneity was averaged out by measuring the NIR spectrum of three portions of each sample.

No. 27: Quantitating phenylalanine and water in L-phenylalanine cake samples

Summary

This study shows that NIR analysis can be used for quantitative analysis of phenylalanine and water in 'wet' L-Phenylalanine cake samples. Values for phenylalanine and water ranged from 65.84–74.9% and 15.4–25.1% respectively.

System

Model 5000, reflectance detector module, spinning sample module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer	2.921.1110
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Sampling

The cake samples were measured in reflectance in the 1100–2500 nm. Each sample was scanned four times while rotating and reloading the sample between scans. For the wet L-phenylalanine cake, a calibration was developed at 2082 nm (SEC of 2%). For water, a calibration was developed at 1454 nm (SEC of 1%).

Results

The results indicate that NIR can be used to quantitatively monitor phenylalanine and water in L-Phe wet cakes.

No. 28: Monitoring moisture content in concentrated iron ore samples

Summary

This feasibility study presents the result of using NIR analysis to monitor moisture in Type I and Type II iron ore samples. Volumetric additions of 1 ml water provided approximate 1% increments in moisture for the 100 g samples, with moisture range of 6–12%.

System

Model 5000, transmission detector module, fiber optic bundle setup module, interactance fibers and reflectance probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120
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Sampling

The samples were analyzed using a fiber optic reflectance probe in the 1100–2500 nm region. Due to mixing problems with the Type I samples, we did not proceed with Type II. A standard regression for moisture was performed at 1928 nm, yielding a standard error of 2%. However, after eliminating the higher concentrations of moisture (due to mixing problems), a better SEC was obtained, 0.5%.

Results

The results indicate that NIR can be used to monitor moisture in iron ore samples. However, sample homogeneity is a concern. Online analysis with additional averaging can provide results which approach the accuracy of the primary method of analysis.

No. 29: Detection and differentiation of coatings on aluminum

Summary

This feasibility study was aimed to prove the NIR's ability to detect and differentiate coatings on aluminum. The samples provided for this analysis were bare aluminum, and two samples of aluminum coated with a thin layer of different materials.

System

Model 5000, reflectance detector module, spinning sample module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120
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Sampling

The scan range was 1100–2500 nm. The analyses were done in reflectance mode. Aluminum can samples were cut into 1.5 inch wide strips, and placed into a coarse sample cell, and backed with foam to hold the sample flat against the cell window. The aluminum spectrum is much higher in absorbance than the two coated samples since the bare aluminum is not an efficient diffuse reflector, rather it is a very efficient specular (mirror-like) reflector. Differences between bare aluminum, and the two coatings are seen in the 1700–2100 nm region.

Results

The results indicate that NIR can be used to differentiate the two different coatings on aluminum from each other at the coating levels provided. Also, the bare aluminum can be differentiated from the two coatings.

No. 30: Monitoring volatiles in solimide powder

Summary

This NIR application is used to monitor volatiles in solimide powder. The volatile components in solimide are mainly methanol, either free or bound as a methyl ester, but could also contain water, as one other possible volatile compound. The moisture range was 0.42–0.60%. The samples provided had a range of volatiles from 20–23% by weight, of which approximately 0.5% was due to water.

System

Model 5000, spinning sample module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer	2.921.1110
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Sampling

The spectra were collected in reflectance mode in the 1100–2500 nm region. The samples were loaded into a standard sample cup. A calibration was developed at 2264 nm for volatiles (SEC of 0.3%). This wavelength is due to methanol, the major volatile component. Addition of other wavelengths for the other volatile components did not improve the calibration. A moisture determination was also made (1920 nm, SEC 0.05%).

Results

The results indicate that NIR can be used to monitor volatiles and moisture in solimide powders. The spectral contributions of water and methanol were determined so that the wavelengths used in the calibrations were assignable to the chemical constituents being analyzed.

No. 31: Detecting the thickness of a silicone layer on solar cells

Summary

This study shows that NIR analysis can be used to detect a silicone layer on silicon solar cells. Five samples were analyzed: dimethyl silicone, quartz glass, silicon wafer, and two silicon solar cells.

System

Model 5000, transmission detector module, fiber optic bundle setup module, interactance fibers and reflectance probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS SmartProbe Analyzer 2m Fiber	2.921.1610
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Sampling

The samples were analyzed using an interactance reflectance probe in the 1100–2500 nm spectral region of the raw materials, only the dimethyl silicone contained any strong NIR absorptions. Absorptions were found in the 1700 nm region as well as the 2300 nm spectral region. After comparing a single layer to a double layer and a quadruple layer, the 1696 nm band was identified as a region which could be used to monitor the thickness of the silicone layer.

Results

The results indicate that NIR can be used to monitor the thickness of the silicone layer.

No. 32: Monitoring layers of a heat seal coating on foil

Summary

This application briefly presents how NIR analysis can be used to determine layers of a heat seal coating on foil. Three samples of foil were provided: one set of raw foil with no coating, one set of foil with one layer of heat seal coating, and one set of foil with two layers of heat seal coating.

System

Model 5000, remote reflectance probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS Process Analyzer DirectLight/NonContact	2.928.0310
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Sampling

The spectral range used in this study was the 1100–2500 nm region. A remote reflectance module was used for sample analysis. Each sample was scanned ten times for calibration purposes. A calibration was developed at 2302 nm (SEC of 3).

Results

The results indicate that NIR can be used to monitor heat seal coating thickness.

No. 33: Monitoring the concentration of sodium azide in an airbag formulation

Summary

The NIR application is used to monitor the concentration of sodium azide in airbag formulation. Of secondary concern was monitoring sodium nitrate and Bentonite clay in the same formulation. Fifteen samples were provided with sodium azide concentration ranging from 66.23–74.27% (w/w). Bentonite range was 0.5–5.2%, while nitrate concentration was 2.9–6.4%.

System

Model 5000, reflectance detector module, spinning sample module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120
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Sampling

The samples were analyzed in the 1100–2500 nm region. Two sampling approaches were used: the fiber optic probe, and the coarse sample cell. Sodium azide has 3 major bands: 1346, 1634, and 2112 nm. The sodium nitrate has 2 bands: 2386 and 2424 nm. Bentonite has a band at 2196 nm. Calibrations for azide were developed at 2112 nm (SEC of 2%) and 1346 nm (SEC of 1%). A calibration for Nitrate was developed at 2424 nm (SEC of 0.5%). A calibration for Bentonite was developed at 2196 nm (SEC 0.7%)

Results

The results indicate that NIR can be used to monitor azide, sodium nitrate, and bentonite clay in airbag formulations. The sampling methodology is the greatest contributor to the error in the accuracy and precision.

No. 34: Quantifying ferric oxide in clay

Summary

This feasibility study was aimed to prove the NIR's ability to monitor ferric oxide in Clay. Pure constituents representing Ferric oxide, Silica, Titania and Alumina were analyzed for this study.

System

Model 5500, transmission detector module, fiber optic bundle setup module, interactance fiber and reflectance probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS SmartProbe Analyzer 2m Fiber	2.921.1610
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Sampling

The samples were analyzed in the 400–1100 nm region using a powder probe. Spectra were collected for each sample by resting the probe above and in direct contact with the sample. Spectral differences are evident between the ferric oxide and the other three constituents. Ferric oxide displays a region of unique absorption in the visible wavelength region (474–700 nm).

Results

The results indicate that NIR can be used to monitor ferric oxide. Calibration development would require a substantial number of samples, representing the entire range of concentration for each constituent. Methodology is the greatest contributor to the error in the accuracy and precision.

No. 35: Monitoring moisture, clay and starch in foundry sands

Summary

This feasibility study presents the result of using NIR analysis to monitor moisture, clay and starch in foundry sands.

System

Model 5000, reflectance detector module, spinning sample module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer Solids 2.921.1120



Sampling

The samples were analyzed in the 1100–2500 nm region in reflectance mode. A standard powder cup was used for analysis. Unique features for the clay (near 2200 nm) and the starches were located. The dextrin and cereal binders have very similar spectra and probably cannot be measured independently, but possibly can be measured together. Two distinct regions, 1450 and 1940 nm (calibration at 1944 nm, SEC of 0.6%), were found for monitoring moisture. The sands also contain either magnetite (black) or hematite (red). If magnetite is present, quantitation of the other components is virtually impossible. However, quantitation with hematite is possible.

Results

The results indicate that NIR can be used to monitor moisture in foundry sands using NIR spectra. The analysis of other constituents such as clay also appears feasible, as demonstrated using the dried foundry sands. It should also be possible to monitor clay in the wet sands, but this would probably require a separate calibration equation. Dextrin and cereal binders could be monitored together.

No. 36: Monitoring the primary amine present in a treated clay

Summary

This feasibility study was aimed to prove that NIR can be used to determine the concentration of primary amine present in treated clay. Five clay samples were provided which varied in amine treatment from 0.7–2.3%.

System

Model 5000, reflectance detector module, spinning sample module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer Solids 2.921.1120



Sampling

All NIR absorbance spectra were recorded from 1100–2500 nm in reflectance mode. Samples were placed into a standard sample cup and analyzed. A calibration for amine was developed at 2024 nm (SEC of 0.1%). Another calibration was also developed using the 1st overtone of NH at 1536 nm (SEC of 0.1%).

Results

The results indicate that NIR can be used to monitor primary amine concentration. Tertiary amine concentration should also be examined.

No. 37: Monitoring primary and tertiary amines in clay samples

Summary

This NIR application is used to monitor primary and tertiary amines in clay samples. Five clay samples treated with tertiary amine (0.2–1.0%) and five samples treated with both primary (1.2%) and tertiary amine (0.2–1.0%) were provided, along with five samples treated with just primary amine (0.7–2.3%).

System

Model 5000, reflectance detector module, spinning sample module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer Solids 2.921.1120



Sampling

All NIR spectra were collected from 1100–2500 nm. A calibration for primary amine was monitored at 2024 nm (SEC of 0.1%). For tertiary amine, a calibration was developed at 2054 nm (SEC of 0.02%). For tertiary amine in the presence of primary amine, a calibration was developed at 2270 nm (SEC of 0.04%).

Results

The results indicate that NIR can be used to determine of primary and tertiary amine in the presence of primary amine treated clays. Since the concentration of primary amine was held constant in the presence of tertiary amine, linear regression could not be performed upon this sample set. With much unique absorption bands existing for both primary and tertiary amine and regression analysis provided above, it is expected that the measurement of primary amine in the presence of tertiary amine is possible.

No. 38: Monitoring quaternary amine in clay samples

Summary

This application shows the use of NIR spectroscopy to monitor quaternary amine in clay samples. The sample set included uncalcined base clay, four concentrations of amine treated base clay with quaternary amine in the 0–8% range, and one amine treated base clay containing additives at their normal manufacturing concentrations.

System

Model 5000, reflectance detector module, spinning sample module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer Solids 2.921.1120



Sampling

Sample spectra were collected from 1100–2500 nm in reflectance mode using a micro-sample cup. The correlation is affected at most wavelengths by the presence of the additives; however, the 1628 and 1660 nm bands are not severely affected. A calibration for quaternary amine was developed at 1628 nm (SEC of 0.06%). Adding a second wavelength to the equation (1628 + 1262 nm) reduces the SEC to 0.02%. This was done to minimize the effect of the additives in this region.

Results

The results indicate that NIR can be used to monitor quaternary amines in clay samples.

No. 39: Monitoring water present in clay soil, sharp soil and dark sand

Summary

This NIR application is used to monitor water present in clay soil, sharp soil, and dark sand. Nine samples, three of each type with varying water levels, were analyzed.

System

Model 5000, transmission detector module, fiber optic bundle setup module, interactance fiber and reflectance probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS SmartProbe Analyzer 2m Fiber	2.921.1610
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Sampling

The samples were recorded in reflectance mode using a fiber optic interactance probe. The scan range was 1100–2500 nm. Each sample was thoroughly mixed, and the spectra recorded by placing the interactance probe directly onto the soil.

Results

The results indicate that NIR can be used to show a linear increase in absorbance to the amount of water added.

No. 40: Monitoring propellant mixtures for sodium azide, sodium nitrate, clay (bentonite), iron oxide, and moisture

Summary

This application shows the use of NIR technique to monitor sodium azide, sodium nitrate, clay (bentonite), iron (III) oxide and moisture in propellant mixtures. Three prepared samples were provided. Sodium azide ranged from 51.95–61.92%, sodium nitrate from 1.05–5.01%, clay from 4.02–4.07% and iron from 29–42.98%. A single production sample was also provided.

System

Model 5000, reflectance detector module, sample transport module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120
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Sampling

The samples were analyzed using a remote reflectance detector module in the 400–2500 nm region. A calibration for sodium azide was developed at 1632 nm (SEC of 0.5%, an isosbestic point for the other constituents). For sodium nitrate, a calibration was developed at 1804 nm (SEC of 0.3%). This is a region where the other constituents are relatively non-absorbing. A calibration for iron oxide was developed at 774 nm (SEC of 0.8%). Moisture is monitored at 1916 nm (SEC of 0.09%).

Results

The results indicate that NIR can be used to monitor sodium azide, sodium nitrate, and moisture in the propellant samples. Further studies appear necessary to assess the feasibility of quantitating iron oxide and clay.

No. 41: Quantitatively determining the amount of polyamine in an adhesive coating

Summary

This study shows that NIR analysis can be used to determine the amount of polyamine in an adhesive coating. Five samples were received: two raw materials of the adhesive coating (an epoxy polymer and a polyamine paste), and three samples from three different rolls of the final product containing 0.2, 1.7, and 2.7% polyamine paste.

System

Model 5000, remote reflectance probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer	2.921.1110
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Sampling

The samples were analyzed in reflectance mode using a remote reflectance probe attachment. The samples were analyzed in the 1100–2500 nm region. A calibration was developed for the polyamine at 1980 nm with a SEC of 0.04%.

Results

The results indicate that NIR can be used to quantitatively detect the presence of a polyamine in adhesive.

No. 42: Determining differences between good and bad samples of adhesive

Summary

This feasibility study was aimed to prove the NIR's ability to differentiate between good and bad samples of an adhesive. The difference between good and bad was not determined by any analytical technique, rather by performance of the adhesive in the product. This study was intended to see if any differences in the NIR spectra might be used to provide a test for adhesive quality.

System

Model 5000, transmission detector module, cuvette module, cuvette adapter module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410
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Sampling

The samples were analyzed in the 1100–2500 nm region in transmission mode using a 1 mm pathlength cuvette. Spectral differences could be seen at 1920, 2000, and 2150 nm. These regions all indicate differences in those samples denoted as bad.

Results

The results indicate that NIR can be used to distinguish between good and bad adhesive samples.

No. 43: Monitoring styrene, butadiene, and total solids in a latex reactor

Summary

The NIR application is used to monitor styrene, butadiene, and total solids in a latex reactor line. Total solids concentration ranged from 4.25–17.84%, while styrene concentration ranged from 4.89–8.26%. Butadiene concentration ranged from 12.39–28.09%.

System

Process Analytics Model 6500 with remote reflectance probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS Analyzer PRO – DirectLight/NonContact	2.928.1130
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Sampling

The samples were analyzed using a Process Analytics instrument with a remote reflectance probe.

A calibration for percent solids was developed at 764 nm (SEC of 0.8%). For styrene, a calibration was developed at 1630/1740 nm. The divisor wavelength is used to correct for multiplicative scatter in the samples caused by the change in total solids and particle size. An SEC of 0.09% was obtained. For butadiene, a calibration was developed at 1704/1776 nm (SEC of 1%). Again, the divisor term was included to correct for scatter differences caused by the variation in total solids content.

Results

The results indicate that NIR can be used to monitor percent solids, styrene, and butadiene content in a latex reactor.

No. 44: Qualitatively distinguishing between latex emulsions and between mineral spirit samples

Summary

This feasibility study was aimed to prove that NIR technique can be used to distinguish between samples of latex emulsions which differ in the nature of polymer and also to distinguish between mineral spirits differing in the amount of aromatics present.

System

Model 5000, transmission detector module, fiber optic bundle setup module, interactance fibers and reflectance probe and immersion probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS SmartProbe Analyzer 2m Fiber	2.921.1610
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Sampling

The samples were analyzed using a fiber optic probe sampling system. An interactance reflectance probe was used for the latex emulsions, while an interactance immersion probe was used for the mineral spirits.

Spectral differences were easily identified in the 1630–1820 nm region for the latex emulsions. A library was developed to distinguish between these products. For the mineral spirits, an intensity difference in the aromatic band at 1690 nm was used to qualitatively distinguish between these samples.

Results

The results indicate that NIR can be used to distinguish between latex emulsions differing in the nature of the polymer as well as to distinguish between mineral spirit samples differing in aromatic content.

No. 45: Quantifying the amount of antidusting/anticaking agent sprayed onto potash

Summary

This NIR application is used to quantify the amount of antidusting/anticaking agent sprayed onto potash. The antidusting and anticaking agent is a mixture of an amine (5%) and a wax (95%). Fourteen samples were supplied. Nine samples, treated with levels of coating varying from 0 to 4.0 lbs/ton, were used for calibration development. Three unknown samples were used for prediction. A sample of the wax and the amine were also supplied.

System

Model 5000, reflectance detector module, spinning sample module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer	2.921.1110
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Sampling

Reflectance spectra were collected from 1100–2500 nm. A calibration for the agent was developed at 1728 nm (SEC of 0.16 lbs/ton).

Results

The results indicate that NIR can be used to monitor the coating levels of an antidusting/anticaking agent on potash.

No. 46: Monitoring the alkylation process

Summary

The application shows the use of NIR analysis to monitor the alkylation process. Provided was a mixture of the starting reactants, a sample taken at the intermediate stage in reaction, and the final alkylated material.

System

Model 5000, transmission detector module, fiber optic bundle setup module, interactance fiber and immersion probe was used for this application.

The equivalent and recommended instrument

NIRS XDS Process Analyzer	2.928.0110
MicroBundle SinglePoint	



Sampling

The samples were analyzed using the probe set to a 4 mm gap. The 1700 nm region is seen to increase from the reaction hold stage (intermediate) to the final reaction stage, and no evidence of this band is present in the starting materials. It also appears that the band near 1680 nm, which is due to one of the starting materials, appears to be present in a small amount in the sample taken at the reaction hold stage, but is less intense in the final reaction sample.

Results

The results indicate that NIR can be used to monitor an alkylation reaction directly. The suspended solids in the samples provide a spectral interference, which could lead to problems with the analytical precision. By using a probe designed to minimize the effects of the suspended solids, and by compensating for variations in scattering, acceptable analytical precision is expected.

No. 47: Quantifying a light amine in a mixture of heavy amines

Summary

The NIR application is used to quantify the presence of a light amine in a mixture of heavy amines. Light amine content ranged from 5–70%.

System

Process Analytics Model 5500 with interactance fibers and immersion probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS Process Analyzer	2.928.0110
MicroBundle SinglePoint	



Sampling

The samples were analyzed using a Process Analytics instrument with an interactance immersion probe set to a 10mm pathlength. The samples were analyzed in the 400–1100 nm range. A PLS calibration was developed using three factors. A SEC of 0.4% was obtained.

Results

The results indicate that NIR can be used to monitor a light amine in a mixture of heavy amines.

No. 48: Determining the sensitivity of measuring protein in a saline solution

Summary

This study shows that NIR analysis can be used to determine the sensitivity of measuring protein in a saline solution. The samples provided were the saline solvent, and two solutions of the protein.

System

Model 6500, transmission detector module, fiber optic bundle setup module, interactance fibers and immersion probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS SmartProbe Analyzer	2.921.1610
2m Fiber	



Sampling

The spectra were collected in transmittance mode in both the 400–1100 nm region and the 1100–2500 nm region. The pathlengths utilized were 10 and 1 mm, respectively. The absorbance bands near 2100 and 1550 nm are assignable to N-H groups in the added solute. A shoulder on the side of the 970 nm water band is present for the two samples containing the added protein.

Results

The results indicate that NIR can identify protein absorptions in many regions of the spectrum. These regions could be used to monitor protein in saline-based fluids, however, no calibrations were developed.

No. 49: Monitoring saponification value in various oil products

Summary

This study shows the NIR application used to monitor saponification (SAP) value in various oil products. The goal was to develop one calibration equation incorporating all products. Sixteen oil products were provided for analysis, with SAP value ranging from 14.5–197.4.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer 2.921.1410



Sampling

The samples were analyzed in transmission mode in the 1100–2500 nm region. A 2 mm pathlength cuvette was used for analysis. The samples were heated to 50 degree Celsius in an oven, and then maintained at 50 degree Celsius using the temperature controlled module. A calibration was developed at 2126 nm for SAP value for all oils (SEC of 10). A PLS calibration was also developed in the 2050–2200 nm region yielding a SEC of 10 using 2 factors.

Results

The results indicate that NIR can be used to develop one calibration for all of the oils. However, better results would be obtained if calibrations were developed for groups of similar products.

No. 50: Determining acid value and iodine value in a fatty acid

Summary

The NIR application is used for determination of acid value and iodine value in a fatty acid. Twenty samples of the fatty acid were provided.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer 2.921.1410



Sampling

The samples were analyzed in the 1100–2500 nm region using an interactance immersion probe. A pathlength of 6.5 mm was used in the study of both fatty acids. The acid value was monitored at 1432 nm (SEC of 2). The low correlation was due to difficulty in maintaining a constant sample temperature, therefore, a denominator term was added to the model (1432/1500 nm, yielding a SEC of 1). Iodine value was monitored at 2118 nm (SEC of 1). Again, due to varying temperatures, 1804 nm was added as a denominator term, reducing the SEC to 0.5).

Results

The results indicate that NIR can be used for the determination of acid value in and iodine value in a fatty acid. This report has also demonstrated that calibration equations may be developed which help to compensate for spectral variations which occur due to changing temperatures.

No. 51: Monitoring the pour point of lube oils

Summary

This study shows that NIR analysis can be used to determine the pour point of lube oils. The lab reported pour point ranged from 55–70.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410
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Sampling

The samples were analyzed in the 1100–2500 nm range in transmission mode. The oils were loaded into a 1 mm quartz cuvette, and held in the liquid sample cell holder at ambient temperature. The region of largest variation occurs between 2200 and 2400 nm. The increase in intensity of the 2300 nm band with pour point is evident. For this indirect measurement, more than a single wavelength calibration model is probably warranted. The 2428 nm band was used as a denominator term. For the calibration model 2300/2428 nm, a SEC of 4 was obtained.

Results

The results indicate that NIR can be used to monitor the pour point in lube oils. For this application, a single term equation will probably not predict pour point as well as a two term equation, since pour point is affected by more than one component.

No. 52: Monitoring the level of rolling oil in a rolling oil emulsion

Summary

This feasibility study reports the result of monitoring the level of rolling oil in a rolling oil emulsion. Rolling oil, water, and contaminant oil were provided for analysis.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410
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Sampling

The samples were analyzed in the 400–2500 nm region in transmission mode. At small pathlengths, the 2000–2200 nm region shows unique absorptions from the rolling oil, while the other materials are relatively featureless. Since a 4 mm pathlength will be used, only the 700–1300 nm region is of use. In this region, many overlaps occur between the rolling oil and the other materials. A PLS calibration would probably be necessary in order to monitor the rolling oil content in the emulsion.

Results

These results indicate that NIR can be used to monitor rolling oil concentration in a rolling oil emulsion.

No. 53: Monitoring water, acetic acid, beta-picoline and dimethylacetamide (DMAC) in a solvent stream

Summary

This study shows that NIR analysis can be used to monitor the levels of water, acetic acid, beta-picoline and dimethylacetamide (DMAC) in a solvent stream. Sixteen samples from two different sample streams were provided for analysis along with the four neat components. Water ranged from 6.18–14.81%, acetic acid ranged from 8.97–12.34%, DMAC from 62.7–71.6%, and B-picoline from 4.77–8.37%.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410
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Sampling

The samples were analyzed in transmission mode in the 400–2500 nm region. Quartz cuvettes were used for analysis. Regions unique to each component were identified. Water can be analyzed at both 1450 and 1940 nm. A regression was performed at 1442 nm (SEC of 0.3%). A regression for acetic acid was performed at 1676 nm (SEC of 0.2%). A regression for DMAC was performed at 1198 nm (SEC of 0.6%). A regression for B-picoline was performed at 1138 nm (SEC of 0.08%).

Results

The results indicate that NIR can be used to monitor water, acetic acid, DMAC and beta-picoline in a solvent stream.

No. 54: Qualitatively monitoring a mixture of hexamine, silicon dioxide, water, benzoic acid, calcium stearate, and DIDP oil

Summary

This feasibility study was aimed to see the NIR's ability to monitor the concentration of hexamine, silicon dioxide, water, benzoic acid, calcium stearate, and DIDP oil in a complex matrix.

System

Model 5000, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410
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Sampling

All samples were analyzed in the spectral region from 1100–2500 nm. The water and DIDP oil were analyzed in transmission mode using a 0.5 and 2 mm pathlength quartz cuvette. Powder samples were analyzed in reflectance mode using a standard sample cup. A water absorption (free from the other constituents) can be seen between 1430 and 1460 nm. A change in hexamine intensity is found at 1688 nm. For silicon dioxide, a region at 1404 nm could be used. The DIDP oil shows strong absorptions at 1720 and 2265 nm. No unique absorption bands of the remaining two constituents, a more complex algorithm such as PLS may be necessary for calibration development.

Results

The results indicate that NIR can be used to determine hexamine, water, silicon dioxide, and DIDP oil. In order to develop calibrations for all of the constituents in this matrix, more samples are needed.

No. 55: Monitoring methyl ethyl ketone (MEK) in ethanol

Summary

The NIR application is used to monitor the presence of MEK contaminant in ethanol. Four samples of MEK in ethanol were provided, with the concentration of MEK ranging from 0.05–1%. Also, to monitor methanol in ethanol with methanol concentrations ranging from 0.5–5%.

System

Model 5000, transmission detector module, fiber optic bundle setup module, interconnect fibers and immersion probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS Process Analyzer	2.928.0210
SingleFiber SinglePoint	



Sampling

The spectral region for this study was 1100–2500 nm. The spectral analysis was performed in transmission mode using a single-fiber optic immersion probe, with the pathlength set to 4 mm. A calibration equation was developed at 1678 nm (SEC of 0.1%) to monitor MEK in ethanol. The 2029 nm (SEC of 0.3%) wavelength was utilized to develop a calibration model for monitoring methanol in ethanol.

Results

The results indicate that NIR can be used to detect the contaminant MEK in ethanol, as well as the presence of methanol in ethanol. The study demonstrated that the use of single-fiber optic bundles would increase the precision of the measurement; however, it probably would not greatly affect the sensitivity.

No. 56: Determination of moisture and free fatty acid (FFA) in fatty amide and moisture and polymerization in hydrogen cyanide (HCN)

Summary

This application briefly shows how NIR analysis is used to determine moisture and FFA in fatty amide and moisture and extent of polymerization in HCN. Moisture ranged from 0.2–0.88%, FFA ranged from 4.43–8.29% in fatty amide. Moisture ranged from 0.2–0.68% for HCN.

System

Model 6500, transmission detector module, immersion probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS Process Analyzer	2.928.0110
MicroBundle SinglePoint	



Sampling

Samples were analyzed in the 400–2500 nm region using a fiber optic immersion probe. A pathlength of 10 mm was used for both fatty amides and HCN samples. A calibration was developed at 1416 nm (SEC of 0.02%) for moisture. A calibration for FFA was performed at 1578 nm (SEC of 0.8%). For water in HCN, a calibration was developed at 1146 nm (SEC of 0.002%). The polymerization reaction proceeds from yellow to colorless, therefore, the 470 nm band was used to monitor extent of polymerization.

Results

The results indicate that NIR can be used to monitor moisture in fatty amides. A better group of samples is needed for calibration development for simultaneously monitor FFA. Using an instrument that covers the visible region allows determination of the extent of polymerization of HCN.

No. 57: Analysis of alginates and xanthans

Summary

This feasibility study was aimed to show the NIR's ability for analyzing alginates and xanthans. Four samples of alginates and four samples of xanthans were provided.

System

Model 5000, transmission detector module, fiber optic bundle setup module, interactance fibers and immersion probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer	2.921.1110
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Sampling

The samples were analyzed in reflectance mode in the 1100–2500 nm region. The samples were analyzed in standard sample cups. A PLS regression using six factors was performed in the 1125–2475 nm region. A SEC of 0.004 was obtained.

Results

The results indicate that NIR can be used to analyze both alginates and xanthans. When validating, better results were obtained using the PLS equation rather than the MLR; however, the calibration set is very limited. When a larger set is used, it may be discovered that the MLR model is preferred.

No. 58: Monitoring peroxide in a reaction stream

Summary

This study shows that NIR analysis can be used to monitor peroxide content in a reaction stream. Ten samples were analyzed with peroxide content ranging from 0–1.061 g/kg.

System

Process Analytics Model 5000 with transmission fibers and probes was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS Process Analyzer MicroBundle 4 Channels	2.928.0120
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Sampling

The samples were analyzed in transmission mode using a fiber optic transmission pair set with a 2 mm pathlength. The scan range was 1100–2500 nm. An absorption due to peroxide was found at approximately 1900 nm. However, due to the complexity of the reaction stream, a more complex regression algorithm, PLS, was utilized. The three factor calibration model yielded a SEC of 0.06 g/kg.

Results

The results indicate that NIR can be used to monitor peroxide content in a reaction stream.

No. 59: Monitoring hydroquinol production in a reaction stream

Summary

This study shows that NIR spectroscopy can be applied to monitor hydroquinol production in a reaction stream. Samples ranging from 20–150 g/kg (HQL) were analyzed.

System

Process Analytics Model 5000 with transmission fibers and probes was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS Process Analyzer	2.928.0120
MicroBundle 4 Channels	



Sampling

The samples were analyzed in transmission mode in the 1100–2500 nm region. A fiber optic transmission pair, set to a 2 mm pathlength, was used for analysis. The 2090 nm band is associated with a hydroxyl absorption (HQL). A more complex regression algorithm, PLS, was utilized due to the widely varying temperature of the reaction stream. Four factors yielded a SEC of 1.2 g/kg.

Results

The results indicate that NIR can be used to monitor hydroquinol production in a reaction stream.

No. 60: The quantitation of toluene, methanol, and water in effluent streams

Summary

This feasibility study was aimed to evaluate NIR's ability for quantitative analysis of toluene, methanol, and water in effluent streams.

System

Samples were scanned in transmission from 1100–2500 nm using a NIRSystems Model 6500 Monochromator and OptiProbe module equipped with an Interactance Immersion probe set to a 2 mm pathlength (1 mm gap). This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS Interactance OptiProbe Analyzer	2.921.1510
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Sampling

Samples were collected in jars from the effluent streams. The sample jars were shaken prior to scanning to disperse insolubles present in the liquid.

Results

A second derivative math pre-treatment with a 20 nm segment and 0 gap, was applied to all spectra. A Partial Least Squares regression was applied to the data to generate a calibration equation for each constituent. A calibration for toluene was developed in the range of 15–30%, a Standard Error of Calibration (SEC) of 0.08–0.09 and a Standard Error of Validation (SEV) of 0.06. For methanol, a calibration was developed ranging from 60–80%, an R^2 of 0.99, an SEC of 0.04–0.2, and an SEV of 0.04–0.12. Under these same conditions, a calibration of water was developed in the range of 4–5%, SEC of 0.02–0.04, and an SEV of 0.02%. For each model, additional samples are required to produce a more robust model.

No.61: Monitoring impurities in a detergent builder process reaction

Summary

This feasibility study was aimed to evaluate the NIR's ability to determine impurities in a process reaction (detergent builders). There are five impurities of interest in this reaction: sodium malate (0.16–0.5%), sodium maleate (0.5–1%), sodium tartrate (0.6–1.5%), sodium fumarate (0.7–1.5%), and sodium formate (2.9–5.5%).

System

Model 5500, liquid sampling system was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidLiquid Analyzer	2.921.1410
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Sampling

The samples were analyzed in the 400–1100 nm range. Spectral data was collected in transmission mode using a 1 mm and 10 mm quartz cuvette. The variations for these impurities occur in the same area (700–800 nm), therefore it may be necessary to use PLS for developing calibrations.

Results

The results indicate that NIR can be used to perform quantitative modeling due to spectral differences apparent between the individual impurities. Calibration work with production samples should be performed to finalize feasibility.

No.62: Monitoring a reaction slurry for water, acetone, and methyl butynol

Summary

This application shows the result of NIR analysis for monitoring a reaction slurry for water, acetone and methyl butynol. The samples provided were solutions (~7.5%) of these constituents in diisopropyl ether, and a KOH/diisopropyl ether slurry to closely mimic the actual sample to be monitored.

System

Model 6500, transmission detector module, fiber optic bundle setup module, interactance fiber and immersion probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS Interactance OptiProbe Analyzer	2.921.1510
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Sampling

The NIR spectra were collected in the 400–2500 nm range in transmission mode. The NIR spectra of the slurried samples were measured using a fiber optic immersion probe, which is the sample presentation scheme that would be used for online measurement. Unique features for water, acetone, and methyl butynol are found in the 700–1600 nm region.

Results

The results indicate that NIR is sensitive to changing levels of water and acetone as demonstrated using spiked samples. Spectral variations were seen in the spiked samples where each constituent was expected to have an unique absorption.

No. 63: Measuring a coating of propionic acid on zinc oxide

Summary

This feasibility study was aimed to evaluate the NIR's ability to measure a coating of propionic acid on zinc oxide. The propionic acid coating ranged from 0–0.62%. Twelve samples were provided, eight of which were of similar surface area. The remaining four were either larger or smaller in size.

System

Model 5000, remote reflectance probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS Process Analyzer	2.928.0310
DirectLight/NonContact	



Sampling

Samples were analyzed in the 1100–2500 nm spectral region using the remote reflectance sampling attachment. Each sample was reloaded and scanned five times. A calibration was developed at 2240 nm. A correction term was added to the calibration equation to normalize the spectra for water content (2240/1930 nm, SEC of 0.02%).

Results

The results indicate that NIR can be used to monitor the propionic coating on zinc oxide.

No. 64: Monitoring the levels of stearic acid in magnesium stearate and monitoring the levels of toluene in an organic solvent

Summary

This application shows the use of NIR spectroscopy to monitor the level of stearic acid in magnesium stearate (measured as percent Mg Stearate in range of 90–99%) and to monitor the levels of toluene in an organic solvent (six samples in the 0.05–0.5% range).

System

Model 5000, sample transport module, reflectance detector module, transmission detector module, temperature control module was used for this application

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120
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NIRS XDS Interactance OptiProbe Analyzer	2.921.1510
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Sampling

The samples were measured in a standard sample cup. The liquids were scanned using an interactance/immersion fiber optic probe which was inserted into a beaker containing each sample. A calibration was developed at 2158 nm (SEC of 0.1%) for stearic acid in Mg stearate. A calibration for toluene in the solvent was developed at 1684 nm (SEC of 0.02%).

Results

The results indicate that NIR can be used to monitor the level of stearic acid in magnesium stearate and also to monitor the level of toluene in an organic solvent at less than one percent levels.

No. 65: Diphenylamine and ethyl centralite in propellants

Summary

This feasibility study was aimed to evaluate the NIR's ability to predict the concentration of the stabilizers diphenylamine and ethyl centralite in propellants.

System

The NIRSystems instrument used to collect the spectra was a model 6500 Monochromator with a Multi-Mode Analyzer sampling system. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120
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Sampling

Samples were dark colored pellets varying in size. The number of samples scanned, usually about 18, varied depending upon which propellant was being evaluated. Approximately 100 additional samples were scanned for use in model validation. Samples were scanned in reflectance from 1100–2500 nm using a coarse sample cell. A second derivative math pre-treatment using a 20 nm segment size and gap of 0, was applied to the raw spectra.

Results

For diphenylamine, a second derivative math pretreatment and a single wavelength regression using 1660 nm produced R^2 of 0.95 and SEC of 0.1 for values ranging between 0.2 and 1.8%. Second derivative pretreatment with a single wavelength regression was also used for ethyl centralite selecting wavelength 2216 nm. Expanded data sets and the evaluation of a Partial Least Squares regression technique is suggested for model robustness and stability.

No. 66: The quantitation of fragrance in wax

Summary

The objective of this study was to evaluate NIR's ability to quantitate a fragrance in wax samples.

System

Spectra were collected from 400–2500 nm using a NIRSystems Model 6500 monochromator equipped with the Vial Heater module set at 60 °C. Due to the attenuation of the signal when using fiber optics, spectral information above 2200 nm was not used for these measurements. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS Transmission OptiProbe Analyzer	2.921.1520
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Sampling

Wax samples were prepared gravimetrically and placed in disposable, 8 mm glass vials. The solidified samples were brought to a constant temperature which melted them and made them translucent prior to scanning.

Results

The pure fragrance was scanned first followed by wax samples containing 4.5–4.9% fragrance. The fragrance was found to have a strong NIR pattern. A second derivative math pre-treatment (segment 10, gap 0) was applied to the spectra and an MLR regression was applied. A correlation of 0.97 and a standard error of calibration of 0.03 was identified using wavelength 1726 nm for the fragrance.

No. 67: Monitoring hydroxyethyl cellulose, acetone, ethanol, and water in a mixture

Summary

This NIR application is used to monitor hydroxyethyl cellulose, acetone, ethanol and water in a mixture. The samples contained between 5 and 15% cellulose, 63 and 78% acetone, 3 and 19% ethanol, and between 2 and 11% water.

System

Process Analytics Model 5000 with interactance fibers and immersion probe was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS Process Analyzer	2.928.0110
MicroBundle SinglePoint	



Sampling

The samples were analyzed in the 1100–2500 nm region in transmission mode. A 3 mm pathlength was used for analysis. For monitoring ethanol, a calibration model was developed at 1698/1626 nm (SEC of 0.3%). The divisor term was included to correct for pathlength variations due to the light scattering effects of the cellulose. A calibration for water was developed at 1418/1342 nm, (SEC of 0.3%). A calibration for acetone was developed at 1676nm/1634 nm (SEC of 0.8%). For cellulose, a calibration was developed at 1874 nm (SEC of 0.8%).

Results

The results indicate that NIR can be used to monitor cellulose, acetone, water, and ethanol in a mixture. Transmission measurements yielded excellent results, however for mixtures containing greater than 25% cellulose, it is likely that a reflectance measurement will yield better results.

No. 68: Monitoring moisture concentration in a powdered pesticide

Summary

This feasibility study presents the result of using NIR technique to measure moisture in a powdered pesticide. Ten sealed samples with moisture varying from 6.2–9.5% were provided for this study.

System

Model 5000, reflectance detector module, spinning sample module was used for this application. This analyzer is no longer available.

The equivalent and recommended instrument

NIRS XDS RapidContent Analyzer Solids	2.921.1120
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Sampling

The samples were measured in the 1100–2500 nm range in reflectance mode. The samples were shaken in their sealed containers prior to the NIR measurement to break up the clumps that had formed in the samples, and to insure a uniform distribution of moisture throughout the sample. The samples were placed into a powder sample cell immediately after opening and scanned. Two aliquots of each sample were scanned in case the moisture was not uniformly distributed. Calibrations were developed at 1906 nm (SEC of 0.06%), and 1416 nm (SEC of 0.04%).

Results

The results indicate that NIR can be used to determine moisture in the powdered pesticide samples. The accuracy was good (+/- 0.065% or less) indicating that the sensitivity of this measurement is quite good. Expanded data sets and evaluation of a Partial Least Squares regression technique is suggested for model robustness and stability.