

## REAL-TIME XYLENE ISOMER QUANTIFICATION USING CHEMOMETRIC RAMAN ANALYSIS

© 2000 Roper Scientific, Inc. All rights reserved.

Raman spectroscopy is a very powerful technique for identifying and quantifying materials based upon their molecular and crystal-lattice bond vibrations. Raman spectra are similar and complementary to infrared spectra. Raman, however, is unaffected by aqueous matrices. It is noninvasive and is able to make measurements through sealed vials or via fiberoptic probes in process environments.

Raman spectroscopy has been used more in recent years due to the convergence of several technologies: CCD detection systems with high sensitivity in the NIR, small powerful diode-based lasers, and fiberoptic probes with integrated laser-and-signal filtering. These products, along with high-aperture short-focal-length spectrographs, provide quality Raman spectra with low fluorescence interference in compact, easy-to-use packages.

While using Raman for identifying and verifying composition of materials is widely accepted, using Raman for quantification of components in a matrix has had only limited success. With the use of multivariate analysis, i.e., PLS and PCR, matrix-component quantification can now be routinely accomplished. Roper Scientific can supply complete packages that will allow real-time content quantification of multiple components in a mixture.

The isomeric concentrations of meta, ortho, and para xylene are important to quantify for the production of many polymer compounds. Raman spectroscopy presents the fastest and easiest method of making this determination. While there is one characteristic peak in para xylene that has no interferences from the other isomers, there is sufficient interference between all three to preclude a concentration calculation of them from only peak-height analysis. In this experiment, the three isomers of xylene were combined in various concentrations and the percent composition of each was predicted using PLS. A training set of 15 samples was used to produce a model with the Galactic Industries Corporation's PLSplus/IQ™ chemometrics package. The samples were mixed with a precision of approximately 1 percent. The resultant model was able to make predictions with a standard deviation in the same range

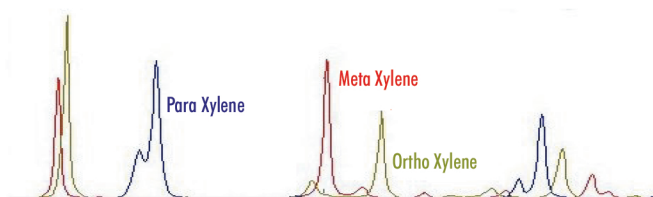


Figure 1. Overlay of pure isomers.

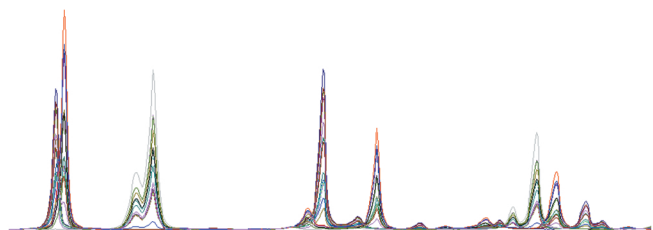
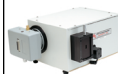


Figure 2. Overlay of 15 mixed samples used in training set.



# A P P L I C A T I O N N O T E

## R O P E R S C I E N T I F I C

### Experiment

All data were taken through 5-ml glass vials using a fiberoptic probe. An Acton Research SpectraPro® 300i with a 1200-g/mm grating and an Acton Research SpectruMM™ GS CCD detection system using a 1024 x 128 back-illuminated chip were employed for the detection. A Process Instruments 785-nm diode laser was used for excitation. The fiberoptic probe used a 90- $\mu$ m excitation fiber and delivered approximately 100 mW to the sample. Collection of the spectra was through a 200- $\mu$ m fiber. Spectra with sufficient signal-to-noise ratios were collected in the 1-second time domain, although spectra taken with a longer integration time were also used in this experiment. Data acquisition and processing was done using a Grams/32®-compatible version of Acton Research SpectraSense™ software. PLSplus/IQ software was employed to create the chemometric model. Once the model was created, on-the-fly calculations of the concentrations of the three isomers were made in real time and output to SpectraSense's unique process-monitoring interface. The data was then displayed in both pie-chart and spreadsheet formats.

### Conclusions

Real-time discrimination and quantification of xylene isomers could be made in the seconds time domain using Raman spectroscopy with virtually no sample preparation. The technique used in this demonstration can be utilized for a broad range of applications.

### Hardware used in this experiment:

SpectraPro 300i	model 306 with single-entrance slit and multichannel adaptor 1200-g/mm grating blazed at 750 nm giving 1000-cm <sup>-1</sup> coverage 600-g/mm grating blazed at 750 nm giving 2000-cm <sup>-1</sup> coverage 300-g/mm grating blazed at 750 nm giving 4000-cm <sup>-1</sup> coverage
SpectruMM GS CCD	with back-illuminated 1024 x 128 chip, 24.6 mm x 3.0 mm
Raman probe	with integrated laser-blocking filter used in signal fiber and laser bandpass filter used in excitation fiber
Laser	Process Instruments 300-mW diode laser

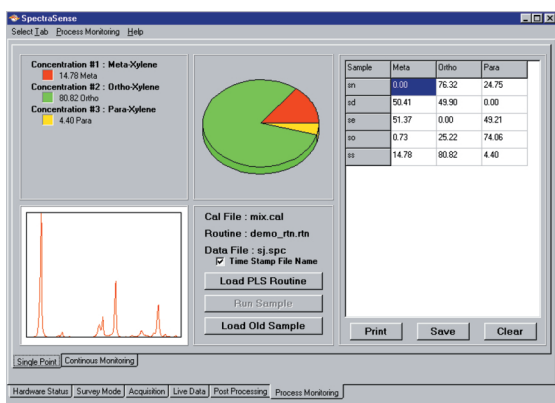


Figure 3. SpectraSense process-monitoring interface with graphical and tabular output of % composition in mixed samples.

Contact **Roper Scientific, Inc.** for more information:

530 Main Street Acton, MA 01720  
tel: 978.263.3584  
fax: 978.263.5086  
email: mail@acton-research.com  
web: www.roperscientific.com

3660 Quakerbridge Road Trenton, NJ 08619  
tel: 609.587.9797  
fax: 609.587.1970  
email: info@roperscientific.com  
web: www.roperscientific.com

3440 East Britannia Drive Tucson, AZ 85706  
tel: 520.889.9933  
fax: 520.573.1944  
email: info@roperscientific.com  
web: www.roperscientific.com

Rev A1