# Measurement & Control Engineering Center (MCEC)

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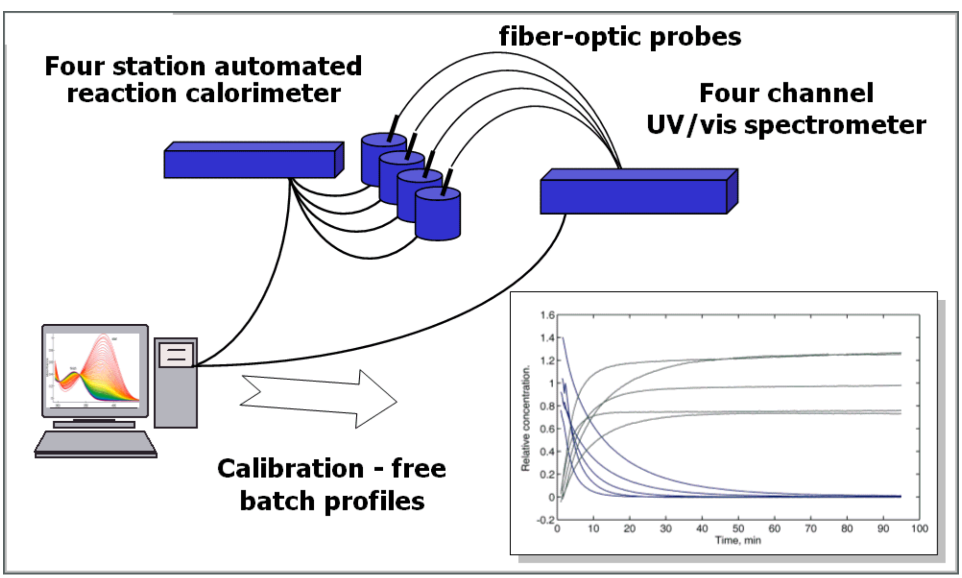
## Automated Reactor with Fiber-Optic Spectroscopic Monitoring

Industry scientists have gained a new tool to enhance the development of chemical manufacturing processes thanks to MCEC's chemical reactor coupled with in-situ optical monitoring and data analysis software. An MCEC project led by chemistry professor Paul J. Gemperline, East Carolina University, and small business partner, H&A Scientific, Inc., Greenville, N.C., resulted in a four-station, automated chemical reactor with fiber-optic spectroscopic monitoring. By comparing multiple lab-scale batch reactions, the new instrument allows development scientists in the pharmaceutical and chemical industries to quickly determine process conditions with better yields, faster reaction times, and lower material and energy costs. The technology is able to estimate composition profiles of starting materials, intermediates, and products using novel mathematical resolution techniques developed by Dr. Gemperline. The mathematical resolution technique uses non-negative alternating least squares, a type of self-modeling curve resolution (SMCR). Multi-batch SMCR analysis of consecutive batches permits standardless comparisons of consecutive batches to determine which produced more or less product and which batch proceeded faster or slower. The product was introduced at the 2001 Pittsburgh Conference, the country's largest chemical instrumentation exhibit. Software for the product has broader application and has been licensed to IntelliFORM. For more information, contact Paul Gemperline, 252-328-6767; e-mail: gemperlinep@mail.ecu.edu.



## Technology to Monitor and Enhance Fluidized Bed Operations

MCEC research led to the development of new measurement and data analysis techniques to monitor fluidized bed operations. The project was initiated in MCEC and supported by a sponsor; in addition, the sponsor funded a separate proprietary development project outside of MCEC to protect its intellectual property. The technique that has been developed applies nonlinear time series analysis tools based on chaos theory to analyze data from high-speed delta pressure measurements. Based on these results, the status of fluid bed operation can be inferred. This new technology may be used to monitor and improve the performance of fluidized bed operation, thus improving unit efficiency. For more information, contact Duane Bruns, 865-974-5317; e-mail: dbruns@utk.edu.

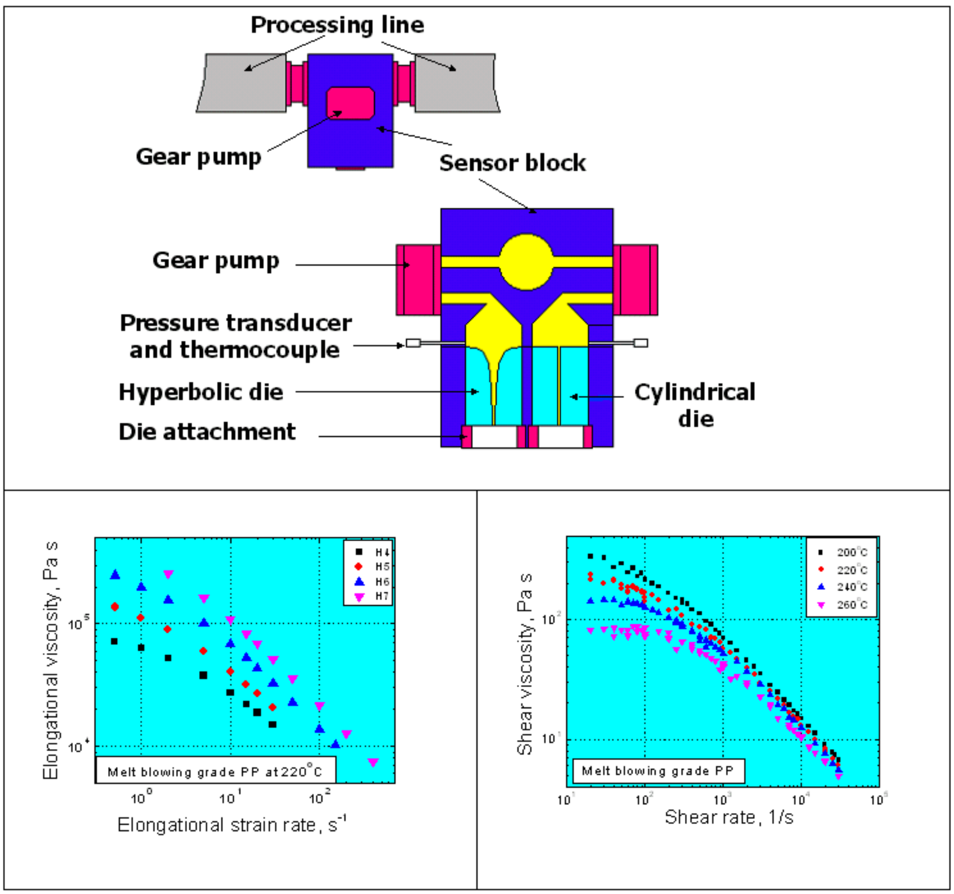
## Raman Spectroscopy for On-line Measurement of Chemical Composition in Manufacturing Processes

Initial MCEC research led to a major research grant from the Department of Energy Office of Industrial Technology to develop Raman spectroscopy for on-line chemical measurements. In collaboration with Eastman Chemical, MCEC demonstrated the practical application of Raman spectroscopy to measure the composition of a chemical mixture in a distillation column. Research at MCEC is widely recognized in the early proof of concept. Numerous vendors now manufacture devices that are applied to on-line Raman composition measurement in many manufacturing situations. For more information, contact Charlie Moore, 865-974-5339; e-mail: cfmoore@utk.edu.

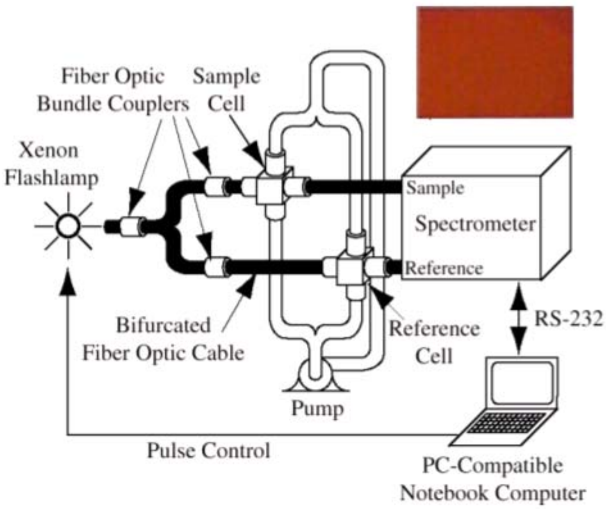

*Right*: The UT's department of chemical engineering unit operations lab. This device is similar to the industrial unit but much, much smaller. The industrial column is the size of a Saturn rocket but we could not provide pictures of the experimental setup because of proprietary concerns.

# Rheology Measurement Technology

John Collier, an MCEC PI at the University of Tennessee, has invented a hyperbolic die and developed the hyperbolically converging flow technique for the measurement of elongational viscosity of polymer melts and solutions over much higher (of industrial interest) ranges of Hencky strains and elongational strain rates, as compared with other techniques. Rheometric Scientific has marketed this die as an accessory for the ACER capillary rheometer. Also, based on the same new technique, an on-line rheological sensor is being developed for the simultaneous measurement of both elongational and shear viscosity. For more information, contact John Collier, 865-974-2421; e-mail: collier@utk.edu.



# On-line Optical Sensors for High Acidity and Basicity




Novel optical sensors for high acidity and basicity measurements have been developed by MCEC researchers at the University of Tennessee. These sensors are based on thin sol-gel films encapsulated with selected acid-base indicators, and are designed for the highly acidic and alkaline systems (such as 1-11 M HCl or 1-10 M NaOH) encountered in many industrial processes. Methods have been developed for correction of interferences from metal ions (acid probe) and organic co-solvents (base probe). The sensors have response times less than 5 sec, and are durable for repeated measurements. The probes provide reliable alternatives to the manual sampling and titration approaches often used for acid and base determination in corrosive environments, enabling online acidity and basicity monitoring and control. The PI is working with vendor companies to study commercialization of the technology. For more information, contact Ben Xue, 865-974-3443; e-mail: xue@utk.edu.