COMPUTER DATA ACQUISITION
AND ANALYSIS TOOLS
FOR THE CHEMICAL SCIENCES

FEATURING THE FS-522 WITH *FASTSpec*™
LAB INTERFACE, ASSOCIATED SENSORS
AND WINDOWS BASED SOFTWARE

E-Newsletter

January 2011

Volume II, Number 1,

In This Issue

Summer MicroLab Workshop at Montana State University: "Inquiry and Visualization in General Chemistry" July 18-20, 2011.

See Us at These Upcoming Meetings

191st 2YC3 Conference, Mt. San Antonio College, Walnut CA
March 25-26, 2011

ACS National Meeting Anaheim CA March 27-31, 2011

Northwest ACS Regional Meeting, Portland, OR
June 26-29, 2011

MicroLab can help you with all your titration needs in education and research. Use your imagination! With MicroLab, you have quite a toolbox!

Photometric Titrations Go Way Back...

See the article **"Photometric Titrations,"** Arthur L. Underwood**,** *J. Chem. Educ.*, **1954**, *31* (8), p 394-397

This is an excellent review of the nature of photometric titrations: background concepts, accuracy and precision compared to volumetric methods, and a number of specific applications that might be of interest to the reader. There have been some significant advances in spectrometers since the article was published that make the photometric titration approach even more desirable and far less cumbersome than with older spectrometer designs.

Quick Links…

Past Newsletters
Website
Catalog
MicroLab support
MicroLab Experiments

Ideas? Suggestions? *We want to hear from you!*

If you have a question about the capabilities of the FS-522, please ask us. You may be surprised at its potential in your situation! Email us (info@microlabinfo.com) or click the MicroLab Support in the Quick Links box (above) for other contact information.

If you have an interesting application of the MicroLab system in your lab, we would love to hear from you! Send us an email - just click on the link above.
If you want to contribute a featured lab application to the E-Newsletter, please contact the editor!
mjcollins@viterbo.edu

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What some of our colleagues using the FS522 say:

"MicroLab has made our labs much more economical. Experiments use smaller samples

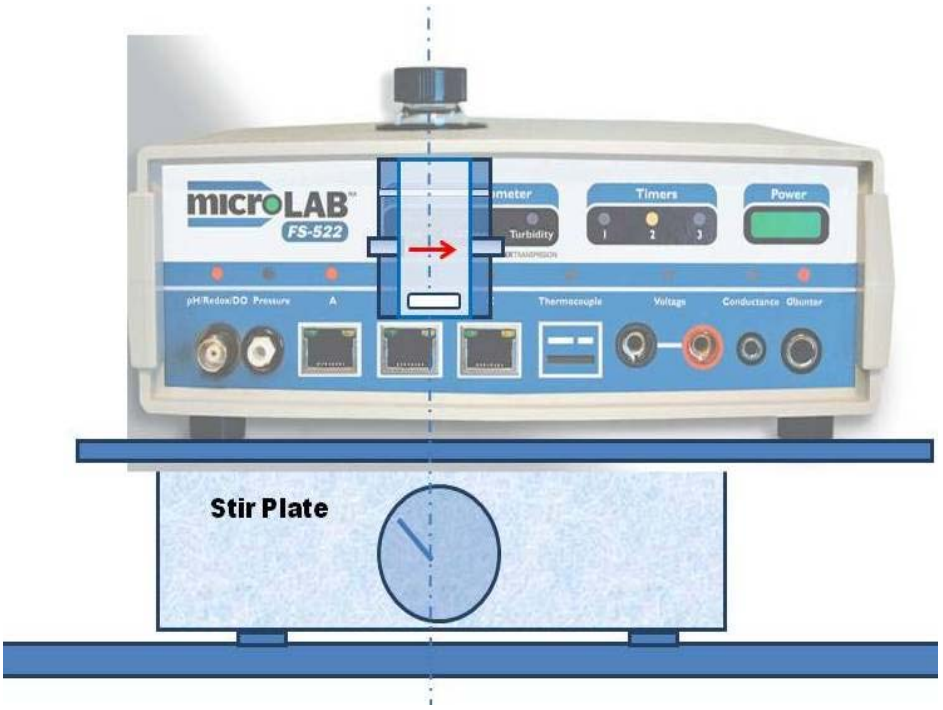

Figure 1. Representation of the FS522 with stirred vial in the *FASTSpec* compartment during a photometric titration. The vial's scew cap can be replaced with a cloth cover during the titration to minimize external stray light. The red arrow indicates light from an LED to a photodiode.

Specifically,
the patented design of the built-in *FASTSpec* spectrometer on the 16 bit MicroLab FS522, with its range of 360-940nm and circular array of detectors that supports cylindrical sample vials, greatly facilitates the addition of titrant directly into the spectrometer vial while it remains in the sample compartment. This results in very high photometric reproducibility. And the cylindrical vial gives very efficient stirring to create a homogeneous solution quickly.

and run more quickly, and students use their time more effectively."
-Virginia Wairegi, Rice University

"Y'all are doing wonderful work...MicroLab is a quantum leap in teaching the fundamentals of chemistry."
Sam Stevenson Marion Military Institute

"It used to be that students would spend a three-hour lab gathering data. Now, students can focus on what the data means; this enables them to decide quickly whether or not they need to do the experiment over. The discovery process - how the numbers relate to a concept - takes place in the lab, not when the students are writing their lab reports."
Dr. Carolyn Mottley Luther College

"I have been using the MicroLab FS-522 in our general and physical chemistry laboratories. I am impressed with the

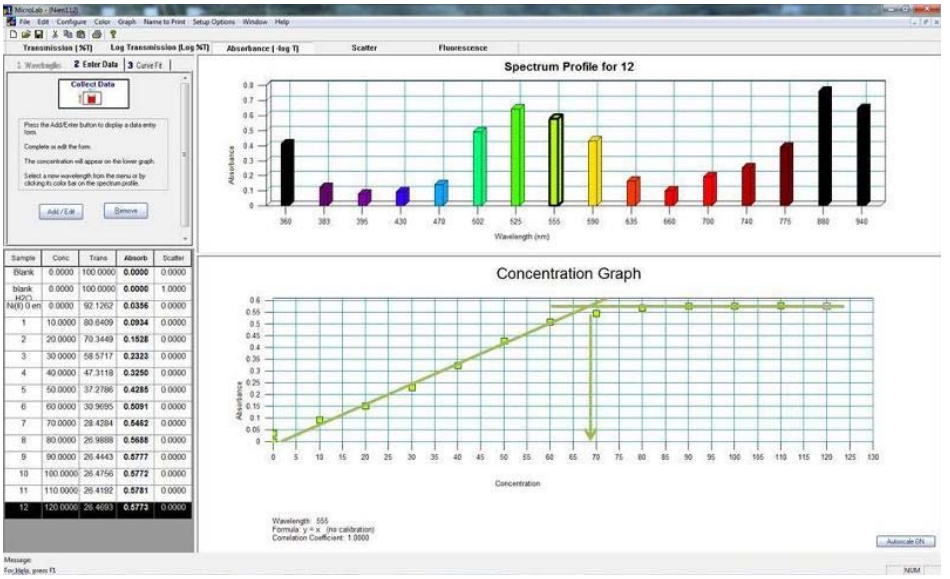

Figure 2. An example of a micromolar photometric titration using the FS522 built in FASTSpec spectrometer along with a prototype of the new Photometric Titration software package. The titration was carried out with replicate additions from a 10 uL auto-pipet. The vial was stirred throughout the experiment. Data can be exported easily to a spreadsheet where the pre-endpoint and post-endpoint data can be regressed to establish their intersection. The interpolated endpoint was 68.3 uL.

Some examples of photometric titrations that are suitable for use with the FS522 are illustrated below:

Titrations involving $KMnO_4$: Permanganate is suitable for a wide range of redox titrations under acidic conditions: $C_2O_4^{2-}$, Fe^{2+}, As^{3+}, Sb^{3+}, V^{4+}, H_2O_2, SO_3^{2-}, and NO_2^- can all be determined. End points are easily seen by the appearance of pink at the first drop of excess MnO_4^-. The intense color of the permanganate ion is an advantage in photometric titrations in that it allows high accuracy of end point determination on very small quantities of analyte and titrant because there is an abrupt change in slope of the absorbance vs. volume $KMnO_4$ at the end point. Extrapolations of the plot up to the end point and beyond the end point allow precise determination of the end point graphically, with <1% error on titrations involving only a few mL of $KMnO_4$ solution.

Titrations involving complex formation: Cu(II)-EDTA, Ni(II)-EDTA, mixtures of Fe(III) and Cu(II) with EDTA, mixtures of Bi(III) and Cu(II) with EDTA. Again, abrupt changes in slope in plots of absorbance vs. volume titrant allow for precise graphical determination of the end points on small samples.

Turbidometric titrations: some precipitation reactions lend

versatility and the low cost of this interface, it opens new possibilities for experiments."
Dr. David Saiki California State University Bakersfield

"MicroLab's software is an enormous aid for non-major students to visualize data collection in real time, and leads them to clearly understand the concept of the lab."
Dr. Angie Sower Montana State University

"I'm continually amazed at the research quality data we get from MicroLab. We can do things in teaching and in under-graduate research at a small institution that we never dreamed possible."
Dr. Tom Kuntzleman, Spring Arbor University

"You have an exceptional product.
Money is very tight, and I wouldn't be spending this much of it if I didn't think that the MicroLab

themselves to being followed on the MicroLab FS522 unit, which has the ability to measure scattered light. Underwood suggests that Ni(II) with dimethylogyoxime; Pb(II) with citrate; and Cr(III), Al(III), and Fe(III) with phthalate can all be titrated turbiditmetrically with good accuracy and precision.

The titration data are obtained in a manner analogous to that used to develop the Beer's Law standard curve (see Newsletter Volume 1, Number 4), but the analyst enters volumes of titrant (or moles) from the keyboard instead of concentrations of standards. The titration curves that develop can be examined at any wavelength in real time with a click of the mouse.

Conventional acid-base titrations: following an indicator color change to determine the end point; following a colored acid with a strong base, eliminating the need for indicators; investigating indicator response against pH during a titration. See for example **"Acid-Base Indicators: A New Look at an Old Topic,"**Ara S. Kooser , Judith L. Jenkins and Lawrence E. Welch , *J. Chem. Educ.*, 2001, *78* (11), p 1504.

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Determination of an Equilibrium Constant: "Photometric titrations" can also be used to study equilibrium constants for a reaction of the type **D + X = DX** in which DX absorbs light. An excellent description of a conceptually simple method that is suitable for reactions that do not go to completion is given in the article "**Equilibrium constants from spectrophotometric data: Principles, practice and programming,"** Richard Ramette, *J. Chem. Educ.*, **1967**, *44* (11), p 647.

The Ramette method kind of got lost since he described not just the algebra but an outdated BASIC program to solve the equations. However, the math involved lends itself nicely to spreadsheet solutions, so it is probably time to give it a new look. The raw [*concentration, absorbance*] MicroLab data must be exported to a spreadsheet to determine the Keq, a trivial operation with the MicroLab software (File, Export as, Comma Separated Variable (CSV) for MS Excel etc).

units were the best such devices on the market. I think that they will transform and reinvigorate the way we teach chemistry at Oglethorpe."

Dr. Keith Aufderheide
Oglethorpe University

"MicroLab has given us a great step forward in the Physical Chemistry lab."

Dr. Clemens Heske
The University of Nevada Las Vegas

"We used the built-in spectrophotometer to study the absorption/ transmission properties of different food dyes. The students really took to the graphs produced for transmittance and absorbance ... they all said it made the ideas we were talking about really clear to see the two graphs."

Dr. "Skip " Wiley
Middlesex Community College

$$Fe^{3+} + SCN^{-} = FeSCN^{2+}$$

C_{SCN}	C_{Fe}	A (450nm)
0.001	0.0003	0.1617
0.002	0.0003	0.2845
0.003	0.0003	0.394
0.003	0.0003	0.386
0.005	0.0003	0.5529
0.008	0.0003	0.725
0.008	0.0003	0.7075

Concentration/absorbance data (data taken from Ramette reference above). The MicroLab *FASTSpec* spectrometer could be used to do this experiment using the 470 nm LED.

The basic idea is that one guesses the molar absorptivity of the absorbing product (which is unknown since it is never formed quantitatively) to compute an equilibrium constant value for each data point. The molar absorptivity guess is iterated to minimize the standard deviation of the range of K_{eq} values computed for each data point. See Figure 3. The built in Excel analysis tool "Solver" can also be used to obtain the best fit. *Feel free to contact the editor for a sample spreadsheet that illustrates this using the data shown and other data.*

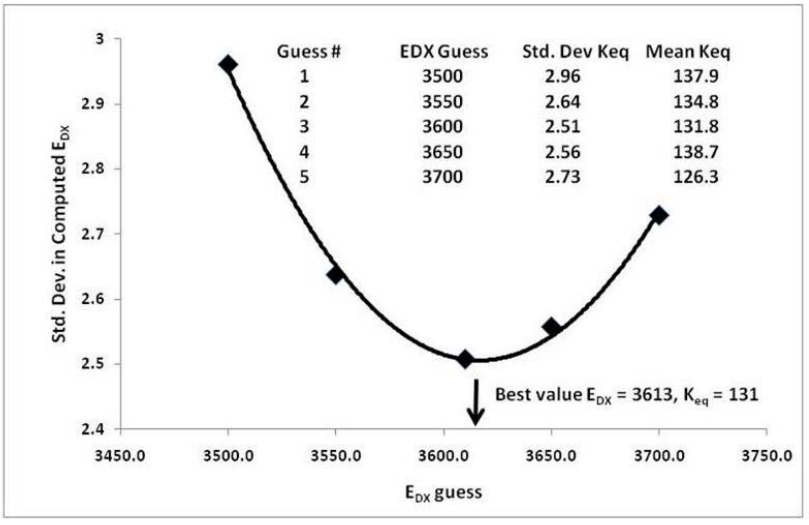

Figure 3. Spreadsheet results after data treatment.

Use MicroLab's sensors to carry out lots of other kinds of titrations.

Redox titrations:

Ce(IV)-As(III) titrations can be carried out on very small amounts of As(III) with dilute Ce(IV) to get part per thousand results on less that a milligram of As(III). The potential of the Ce(IV)/Ce(III) half cell can be monitored with a Pt or Au wire indicating electrode against any reference electrode, such as Ag/AgCl or calomel using the MicroLab FS522 **Voltage input sensor jack.** The volumes can be entered from the keyboard.

Broken pH electrodes make great redox indicating elecrodes.

Save your broken pH glass combination electrodes - each one has an internal reference electrode and a length of Pt wire - just smooth out the broken glass edges, plug it in to the MicroLab BNC mV/pH/ISE jack, and you are ready to measure potentials to +/-2500 mV.

Mixtures of halide ions can be titrated with $AgNO_3$ using a silver wire indicating electrode against any reference electrode. To avoid errors from leakage of halide from the reference electrode to the sample, a solution of KNO_3 in a tube with a porous frit is used as a salt bridge to isolate the analyte from the reference electrode solution. The cell potential is measured with the MicroLab Voltage input.

The MicroLab FS522 unit, with its pH/ISE BNC connector, can also be used with an ion-selective combination electrode (ISE) to follow the course of a titration, analogous to a pH potentiometric titration. Have you ever thought of using a Ca electrode to do a Ca-EDTA titration? Why not give it a try?

Conductimetric titrations: "Conductimetric Titrations: A Predict-Observe-Explain Activity for General Chemistry," K. Christopher Smith, Etinosa Edionwe, and Bayyinah Michel, *J. Chem. Educ.*, **2010**, *87* (11), pp 1217-1221

This paper describes the titrations of strong and weak acids against a strong base. The weak acid titration is carried out initially at 0.5 mL increments of NaOH and then again at 0.2 mL increments. This last titration allows the students to see an initial decrease in conductivity

as those few H^+ ions are replaced by Na^+ ions at low concentration. This would be a cinch with the FS522 and the Model 160 Conductance probe.

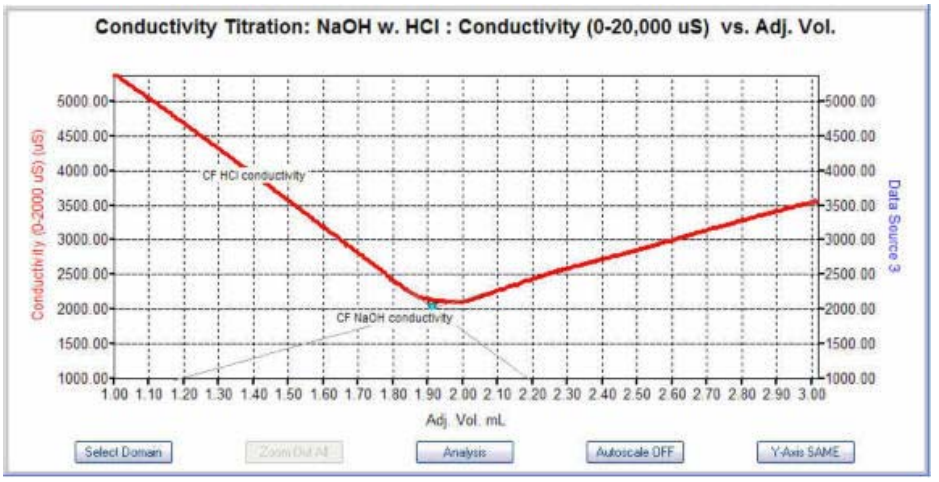

Figure 4. Strong acid-strong base conductimetric titration using the MicroLab FS522 and the Model 160 Conductance Probe in conjunction with the Model 154 Constant Volume Drop Dispenser and Model 226 Drop Counter. The conductivity decreases as H+ ions are replaced by Na+ ions up to the end point. Beyond the end point, the conductivity increases as excess OH- ions are added. Notice that the endpoint is only 1.91 mL NaOH.

Potentiometric and conductimetric titrations: "A Conductimetric-Potentiometric Titration for the Advanced Laboratory," Louis C. Rosenthal and Lawrence C. Nathan, *J.Chem. Educ.*, 1981, *58* (8), p.656-658.

Figure 5. The Model 254 is the automated version of the constant volume drop dispenser for controlling the addition of reagent via the MicroLab software. The drop rate is set with the thumbscrew, but the dispensing is solenoid controlled via keyboard, program loop, or another sensor. The drops are counted by the Model 226 drop counter below.

This article describes an approach to determining simultaneously the acid ionization constants of various amino acids and the ionic conductivities of the various ions of those acids. The MicroLab FS522 unit with pH electrode and Model 160 Conductivity Sensor would be a plug-and-play way to do this experiment easily. Using in the Model 154 Constant Volume Drop Dispenser (or the automated version, Model 254, shown above) and the Model 226 Drop Counter would add to the precision of the experiment.

Meet the Editor: Michael Collins

Michael Collins is Emeritus Professor of Chemistry at Viterbo University in La Crosse, WI, USA. He taught undergraduate chemistry for 38 years at virtually every level - from introductory chemistry for

liberal arts, nursing, pre-med, biology and chemistry majors to advanced courses for senior chemistry and biochemistry majors. He was the 1988 CASE Wisconsin Professor of the Year and has won awards at Viterbo for his scholarship, teaching, and service. He has been active in his local American Chemical Society section, and chaired the planning committee for the Great Lakes Regional Meeting that was held in La Crosse.

His interest in computer data acquisition began in the early 1980s, and he became convinced of its ability to enhance the lab experiences of his students as well as to prepare them to function in a modern lab setting. He has developed experiments across Viterbo's curriculum that use MicroLab for guided inquiry experiments as well as for more routine data logging and analysis. He has also given presentations on the role of computers in the laboratory to facilitate learning chemistry and in the assessment of lab outcomes. He has been using MicroLab products since they first arrived on the scene, and he continues to develop ideas for new applications of MicroLab in undergraduate teaching and research.

Please contact us at MicroLab for more information and to learn how simple it is to put these experiments and others just as exciting into your lab classes with the MicroLab FS522 and accessories.

Thanks for reading! We invite your feedback, ideas, and suggestions. As college educators ourselves, we on your MicroLab team value your feedback.

Sincerely,

Your MicroLab team
info@microlabinfo.com