A Computer Controlled Multi-Analysis Titration System

by
Dr Alexander C. Le Dain*, and Mr Tony White
*Manager of Programming
ICON Technologies Pty Ltd

Category:
Test and Quality
OR Production and Manufacturing

Products Used:
LabVIEW software
PC-DIO-96 board
PC-TIO-10 board
AT-232/4 board

The Challenge:
To develop a fully-automated titration system that would allow continuous, “round-the-clock” operation of two dual-channel, serially controlled Titrators, with intelligent scheduling of the input sample queue, and the capacity to vary the titration method on a “per sample” basis.

The Solution:
Use National Instruments’ LabVIEW software to build the application. The multi-threaded LabVIEW environment is ideally suited to building a complex application of this type that requires parallel streams of instrument I/O, with simultaneous data analysis, data distribution, and sample queue management. A range of “off-the-shelf” National Instruments’ hardware provides the multi-port serial interface, the timer functions to accurately control reagent additions, and the digital I/O for monitoring flow switches and driving switch valve relays.

Abstract
Alumina refinery process liquors are monitored “round-the-clock” via titrimetric analysis. A typical manual titration takes about 15 minutes per sample, and throughput of samples is a major bottleneck in optimising the production process. ICON Technologies developed a new automated titrimetric analyser for ALCOA World Alumina to replace a previously developed in-house system. The previous system used a custom “C” software interface to control an industry standard Gilson Autosampler, and Mettler Model DL70 Titrator. One major drawback with the existing system was the inability of the software interface to handle new generation Titrators that allow the simultaneous processing of two samples. The new analyser uses LabVIEW software to intelligently schedule samples for submission to two concurrently operating dual-channel Titrators. Average continuous throughput of the new analyser is expected to be around 20 samples/hour.

Background to the Application
The alumina refining industry uses caustic solution to extract alumina from bauxite ore. The caustic and alumina concentrations are critical parameters in this large scale, continuous extraction process, and must be monitored around-the-clock at multiple locations in the plant. Manually acquired samples are analysed via a labour-intensive titration process. Each titration takes approximately 15 minutes, with 2-5 minutes clean up between samples. A good analyst may achieve up to three titrations per hour for a single method on one set of equipment, but sustained throughput would typically average 18-24 titrations per analyst in a normal 8-hour working day.

ALCOA had previously developed a PC controlled titration system that could queue and schedule samples for submission to a Gilson Autosampler and Mettler Model DL70 Titrator at *ca*. 5 samples/hour “round-the-clock” sustained throughput.

Requirements of the New System
ICON Technologies was asked to develop a new automated analyser that would provide a general enhancement to functionality, allow migration to Windows NT, and improve throughput by taking advantage of new generation Titrators that could process two samples simultaneously and by utilising spare capacity on the Autosampler to run two Titrators in parallel. The aim for the new analyser was that it should run unattended “round-the-clock” at an average continuous throughput of 20 samples/hour (480 samples/day). By utilising spare capacity on the Autosampler this fourfold increase in throughput is achieved at around half the scale up cost of the previous system.

Other requirements for the new system were: 1) that it implement multiple titration methods and allow for the future addition of new methods; 2) that it allow for the substitution of different makes and models of Titrator to accommodate differences in existing equipment between refineries; 3) that it run control samples as required by pre-determined statistical control methods, independent of user input; 4) that it self-diagnose and self-correct errors as far as possible; 5) that it preserve backwards compatibility with a scripting language developed internally by ALCOA for its previous system; and 6) that it allow for future expansion in capacity up to four dual-channel Titrators.

The Working System

The new system is in the final stages of beta testing and has so far met or exceeded all its original design specifications. The system consists of a modular LabVIEW software application running under Windows NT, on a controlling PC connected via multi-port RS-232 to a Gilson Autosampler and Syringe Diluter, and two Mettler Model DL77 Titrators (see Fig. 1). Each Titrator services two custom-built beakers, each with lines for reagent addition, rinse water, and drain to waste. Up to five different reagents can be added to each beaker, with only one beaker able to access the reagent supply at any one time. Reagent is delivered in typical quantities of 10-50 ml via an FMI piston pump with an accuracy of better than 0.4 percent. All lines to the beaker are monitored with digital flow indicators to confirm operation, and the beaker includes an overflow indicator. All pumps, valves, relays and indicators are driven from a PC-TIO-10 counter-timer card and a PC-DIO-96 digital I/O card, via a custom-built switch box.

Fig. 1 – The working system, showing the control PC, Gilson Autosampler and Syringe Diluter, two Mettler Titrators and their associated beakers, the switch box, and piston pumps.

The LabVIEW software application includes modules for receiving and scheduling batches of samples, controlling the Gilson Autosampler and Syringe Diluter, controlling the Mettler Titrators, analysing the returned data, archiving data to the local disk and the network LIMS system, handling errors, and running automatic and manual maintenance routines.

Samples can be submitted in random order by the operator(s) at any time. The system queues samples for processing based on their priority and method of analysis (some methods require a dedicated beaker available on only one Titrator). The Sample Submission and Run Status screens give extensive visual feedback to the operator about the overall system state and the current queue status of any sample (see Figs. 2 & 3). Each submitted sample has an accompanying sample file that is modified as the sample moves through the preparation, titration, and analysis stages.

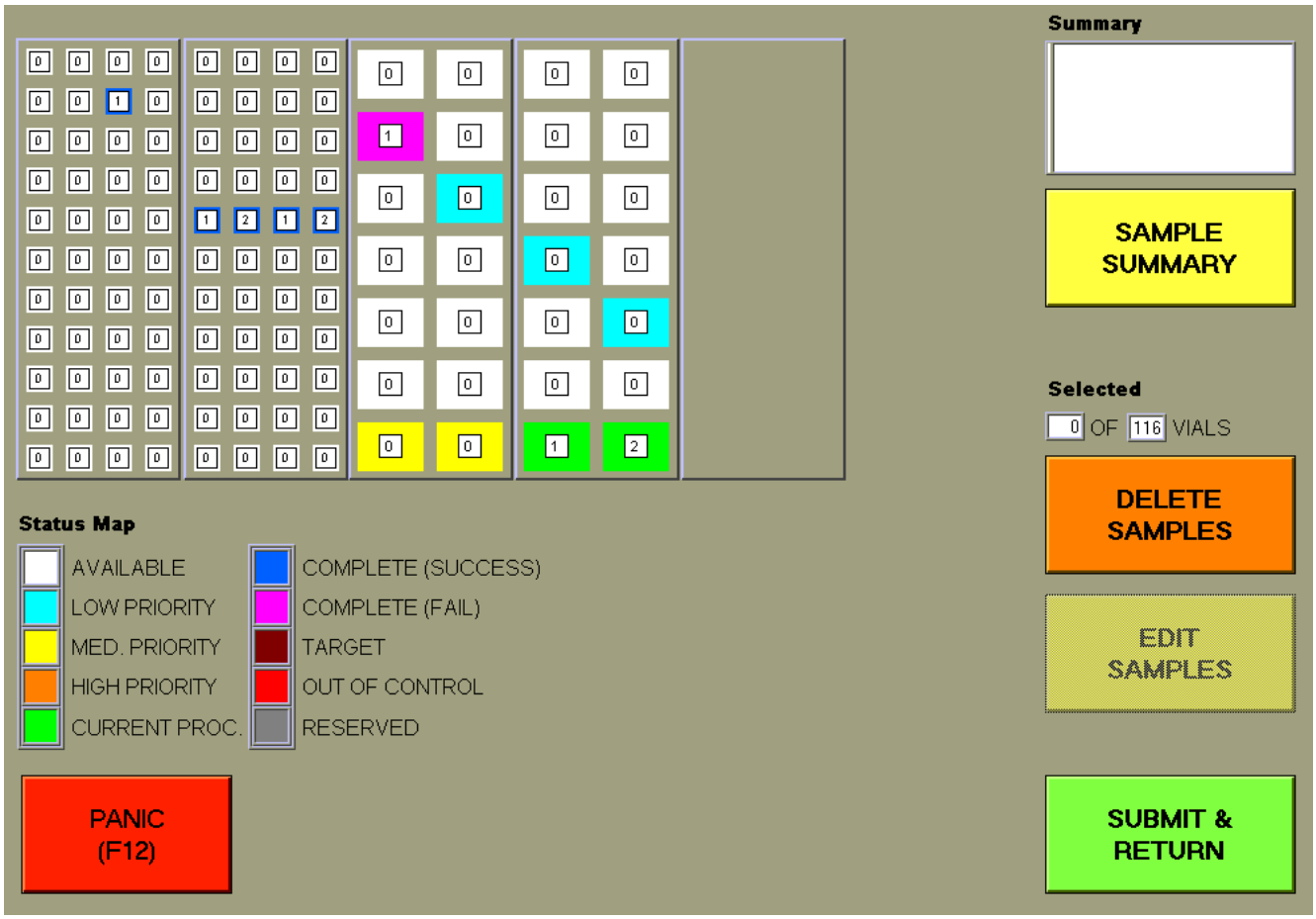

Fig. 2 – The Sample Submission screen. Samples are mapped to locations in the Gilson Autosampler, and coded with a colour according to priority. As samples move through the process their colour code is updated, and they are tagged with a number according to the beaker on which they are executing. This screen shows that two medium priority samples and three low priority samples are awaiting execution, two samples are currently executing on beakers 1 and 2, five samples have completed execution successfully, and one sample has failed to complete successfully.

To maximise flexibility samples are processed according to operator-selected scripts written using ALCOA's ESL scripting language. The use of ESL scripts means that the amount and order of each reagent addition can be varied, so that existing analysis methods can be "tweaked", and new methods can be added, without rewriting any underlying code. ESL scripts also control all the routine maintenance operations for the system. Scripts are translated into the unique commands required for the Autosampler and Titrator by a low-level driver VI, so the system can easily incorporate alternative Autosamplers and Titrators, provided their gross functionality is similar. All drivers were developed around a standard interface to allow for future extensions to the ESL scripting language.

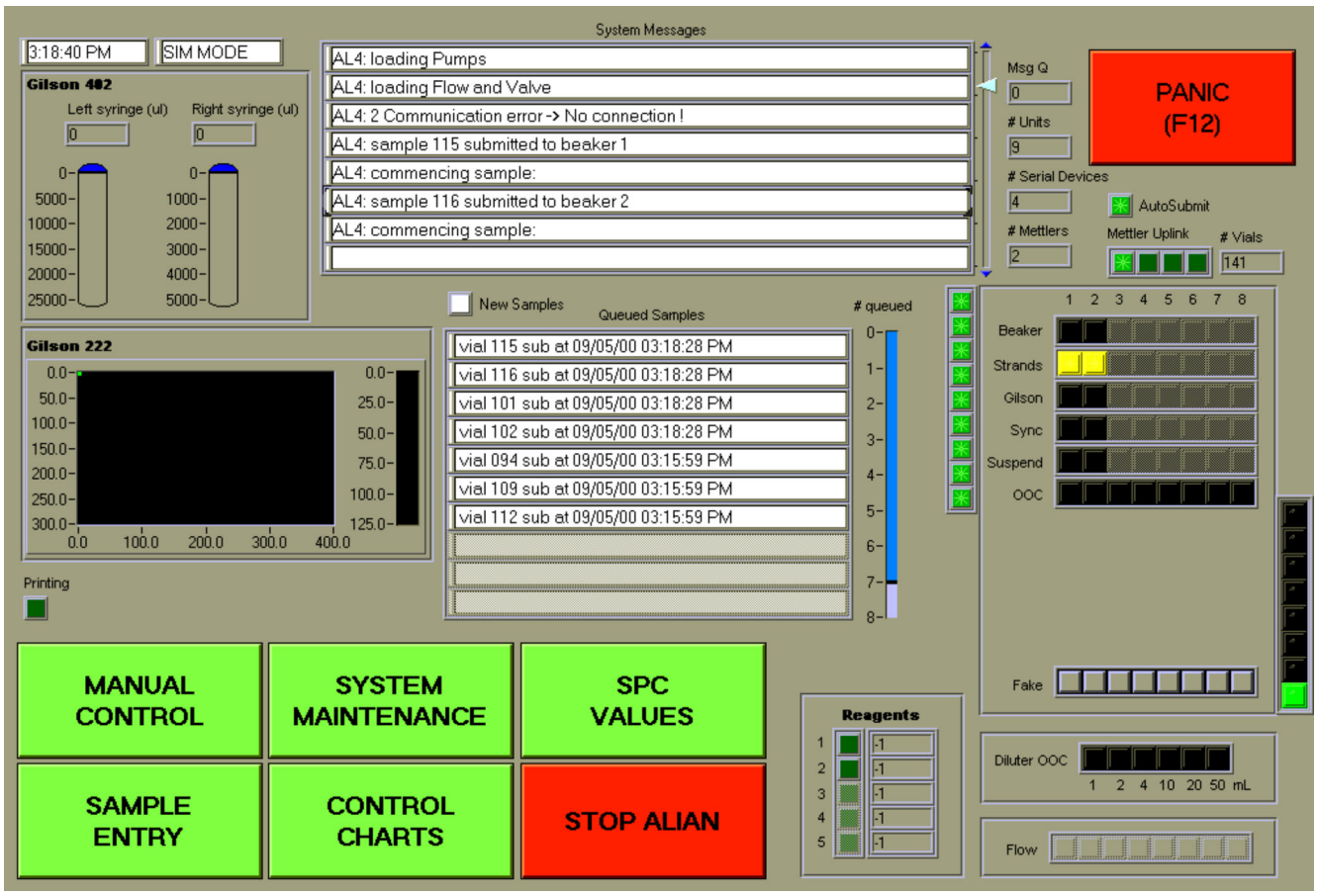

Fig. 3 – The Run Status screen is the normal display mode. It provides extensive feedback on the current state of the system including overall performance, a log of system messages, the current sample queue, the status of each beaker and its associated resources, and a real time animated display of the Autosampler head position and Diluter syringe. It also provides password protected access to manual control and system maintenance modes.

The Mettler Titrator drivers in particular were not trivial to develop. The Mettler DL77 series are very intelligent and can manage a large percentage of the titration process under local control. During this process they alternate between wanting to behave like a server (waiting for the PC to initiate events) and wanting to behave like a client (requesting services from the PC). At all times they must "see" a continuously active communications link from the PC, or they can put themselves irreversibly off-line. In these circumstances reliable messaging and error handling in the presence of major background processes were critical to the success of the system. Under LabVIEW control the new system communicates reliably to multiple Titrators at rates up to 9600 baud, compared with 1200 baud communications to a single Titrator in the previous system.

An Ideal LabVIEW Application

LabVIEW is ideally suited to building an application of this type. It includes all the base functionality required to build the operator interface, and to manage the DAQ and serial I/O. It is inherently suited to the parallel execution of the separate processes for sample submission, sample queuing, control of the Autosampler, management of the two Titrators, and data analysis and archiving. LabVIEW is also well suited to modular development, meeting the requirement for incorporation of new methods, alternative Autosamplers and Titrators, and expanded capacity with minimum disruption to the underlying code.

Equally critical to the timely implementation of the application were some of LabVIEW's more advanced features such as accessible ActiveX support, high level implementation of Queues and Semaphores, and simple re-entrant execution of sub-VIs. Each of the four beaker strands is handled by a re-entrant call to a multiple instance of the same sub-VI. The in-built Queue functions were used to handle sample submission, system messaging, and error handling. The in-built Semaphore functions were used to manage queued use of the single Autosampler and Diluter, key communications between the PC and the Titrators, and access to relay switching of the shared pumps.

ActiveX support became critical during the development of the LabVIEW interface to the Mettler DL77. The supplier released, for the first time, an upgraded version of the system level driver as an ActiveX control. The new control was fully integrated into the LabVIEW application, which was at an advanced stage of development at this point, within one working day, and performed flawlessly from day one.

At this point we have not had to invoke anything more sophisticated than the default two-thread execution system to achieve the desired performance. It is comforting to know that more sophisticated multi-threaded execution options are readily available if required.