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A SOFTWARE APPROACH TO ACCELERATING PROCESS ANALYZER DEPLOYMENT

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ABSTRACT

This paper describes a software approach designed to accelerate process analytical development and deployment by standardizing the interactions with diverse instruments, sampling systems, chemometric packages, and enterprise data systems and by integrating all required functionality at the analyzer level. In addition to the required control and data analysis functions, the system provides local displays in various formats, including statistical information and real-time trend plots, as well as database storage, alarms, and remote communications. It can be implemented on a standard PC or integrated into a dedicated process analytical "appliance" for locked-down deployment.

INTRODUCTION

The field of process analytical chemistry (PAC) – known in the pharmaceutical world as process analytical technology (PAT) – has developed rapidly over the past 25 to 30 years. In its early period, it was primarily concerned with the use of laboratory instruments such as chromatographs and spectrometers to analyze samples extracted from a process and transported to an analytical laboratory. The need for increased analytical speed and accuracy eventually led to the introduction of more robust instruments capable of being deployed in a factory environment. In addition, sample conditioning systems were developed to automatically extract samples from the process and transport them to the instrument while maintaining their condition (e.g. temperature, pressure, and composition) as nearly as possible to that representative of the process. Such sample conditioning systems are still used for the majority of process analytical installations – especially those employing techniques which inherently require an extracted sample.

The past twenty years have seen a steady increase in the number of process analytical installations that do not require sample conditioning systems. Many of these installations employ optical probes (typically near-infrared or Raman) which are inserted into a process pipe or vessel and connected to a spectrometer by means of optical fibers. Others employ gas or liquid flow cells rather than immersion probes. In such cases, the cell is usually in series with a process line or in a recirculating "fast" loop with sufficient flow velocity so that sample conditioning is not necessary. Still other installations, often involving blending or

drying, involve noncontact analysis. An installation of this type typically employs a spectrometer which views the process through a window on the process vessel. All of these installations have the advantage of eliminating the reliability issues often associated with sample conditioning systems and of insuring that a measurement is always made under actual process conditions. In addition, the use of fiber-optics allows an instrument to be located in a benign environment a substantial distance from the process.

The above hardware developments have been paralleled by the development of chemometric (multivariate analysis) techniques to extract process variable information from the raw data. These techniques have proven to be invaluable in helping to realize the potential of process analysis – especially in the near-infrared, a region in which strongly overlapping spectral bands render discrete band analysis impractical. The availability of chemometrics, along with fiber-optic communications, robust sampling devices, and process spectrometers has made wide-spread process spectroscopy a practical reality.

INDUSTRY INITIATIVES

With the proliferation of diverse sampling techniques, instrument types, instrument software, and chemometric packages in the late 1990's, it became apparent to major users that there was need for standardization and streamlining of process analytical hardware and software. This led to the establishment of three industry initiatives. (1), (2), (3)

NeSSI:

The New Sampling/Sensor Initiative (NeSSI™) is sponsored by the Center for Process Analytical Technology (CPAC) at the University of Washington. Its primary objectives are to foster standardization and miniaturization of sample conditioning systems, to provide the infrastructure needed to accelerate the use of micro-analytical sensors, and to lay the groundwork for the adoption of an open communication standard for process analysis. (4) An overall goal is to move the analytical systems out of the analyzer houses and to place them as close as possible to the major process equipment.

COPA:

The Chemometrics for On-line Process Analytics (COPA) initiative got its start at an Analect FTIR users meeting in 2000 and has been pursued at various CPAC and IFPAC meetings since then. (5), (6), (7) Initially, the goal was to create an interchange standard for chemometric models. However, this morphed into the broader goal of simplifying the routine analysis in a process environment using chemometrics. Open format models (e.g. ASCII), imbedded prediction dlls, and vendor-agnostic software systems have been the result. (8) One of the goals of our program has been to further the cause of the COPA initiative by providing a standardize set of interfaces and a format for implementing and integrating predictions from all of the commonly used chemometrics programs.

ConnI:

The Process Analyzer Connectivity Initiative (ConnI) got its start at about the same time as NeSSI and COPA. Its goal at the outset was "to develop a Connectivity Model based on a functional understanding and description of the overall Process Analyzer System. (9) Initially sponsored by CPAC, it has more recently been pursued on a less formal basis. In part, its objectives have been subsumed into those of the OPC foundation (see below). The considerations of ConnI and related activities have highlighted the complexity of the task of integrating individual analyzers into a plant wide process analysis and DCS

system. (10) This complexity is one of the reasons for our emphasis on integration at the analyzer (or process) level. Only by insuring that each analyzer system is self contained and semi-autonomous can we instill order in the overall scheme.

OPC FOUNDATION:

The OPC initiative arose from the need to provide a common method in which to communicate between low-level IO devices and PLC/SCADA and DSC systems. During the 90s, automation companies were plagued with the need to continuously create and revise the drivers for a myriad of possible IO devices. With the advent of a common OPC IO standard known as OPC Data Access or OPC DA, the need to maintain this vast array of custom drivers became a thing of the past.

In more recent years, a follow-up initiative, driven through the OPC foundation, was created to provide a common interface for analytical instruments. This is known as the OPC Analytical Device Interface or OPC ADI. (11) The focus of this initiative is to make it easier for automation providers to communicate with a wide variety of analytical instruments.

APPROACHES TO PROCESS ANALYTICAL INTEGRATION

Chemical process analysis has been widely used in the petrochemical and related industries for at least fifty years. During most of this period, the emphasis was on the use of analytical instrumentation to gain insight into and to control individual processes. The ideal was for the analytical instrument (or analyzer) to fill a role analogous to a univariate sensor but with the capability to provide information about chemical composition as opposed to simple physical quantities such as temperature, pressure, and flow rate. One can think of this as a "measurement centric" or "process centric" view of process analysis.

The promulgation of the PAT initiative by the US FDA in 2004 led to a rapid increase in the attention being paid to process analysis within the pharmaceutical industry. As a result, many of the firms in this industry began to ramp up their analytical capabilities. At the same time – perhaps partly as a result of the highly regulated nature of this industry – there developed an increased emphasis on the integration of process analytical information into plant-wide and enterprise-wide information systems. This has led to a somewhat different view of chemical process analysis than that largely held in the petrochemical industries, a view which sees process analytical technology as a function to be controlled by and integrated into the supervisory layer of a company's infrastructure. We might call this the "supervisory" or "enterprise-down" view of chemical process analysis. This "Global" viewpoint naturally leads to the vision of the total integration of all operational information into a master intelligent system for process and enterprise optimization. Although this is an attractive goal, it should be pursued with caution. For example, in the case of process analytical data, integration should be done in stages. The raw analytical data should first be processed to provide predictions of process variables at a given measurement location. These can then be combined with other information at a higher level to model unit operation and eventually plant operation. This "organic" approach organizes data along functional lines – analogous to the human nervous system. In doing so, it limits the difficulty of data synchronization and minimizes the likelihood of over fitting and hence error.

One possible embodiment of the enterprise-down approach is illustrated in Figure 1. This embodiment takes advantage of the capabilities already present at the supervisory level to control the operation of the sampling system and to integrate sensor outputs and raw analyzer data into an overall chemometric model.

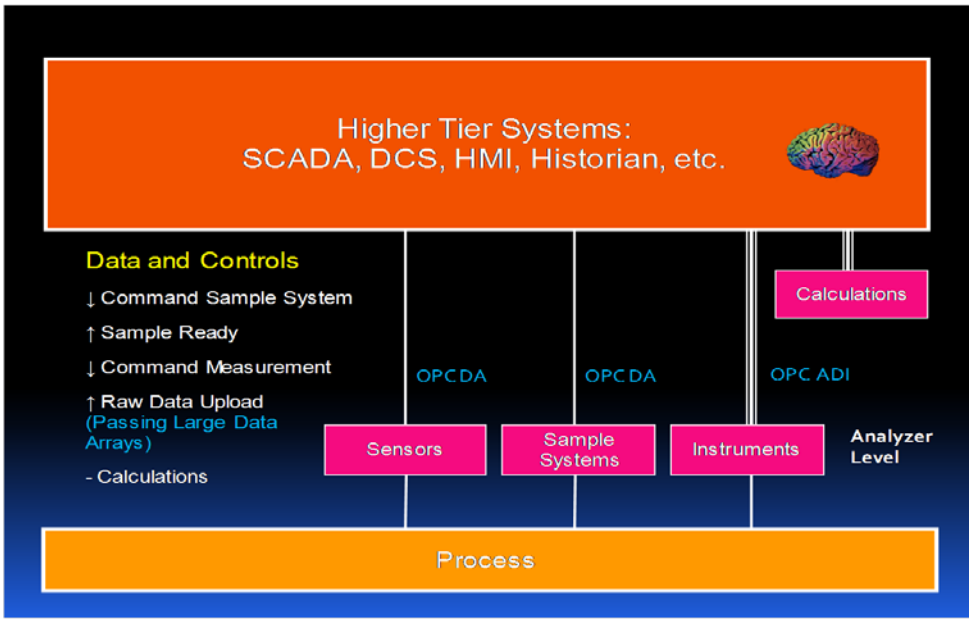

FIGURE 1. POSSIBLE EMBODIMENT OF THE ENTERPRISE-DOWN APPROACH TO ANALYTICAL INTEGRATION.

In pursuing the enterprise-down approach to process analytical integration, automation providers have faced the need for standardization of instrument interfaces just as they previously had to deal with the standardization of I/O interfaces – leading to OPC-DA. The natural approach from this viewpoint was to extend the OPC specification to analytical instruments. This led to the OPC-ADI initiative.

The typical scenario, suggested by the automation industry, is for each instrument manufacturer to provide an OPC-ADI interface to connect to the supervisory level where the raw data is then processed. An issue with this approach is that large amounts of data are now being passed to higher-tier levels in real-time for processing. The processed data is then passed along for monitoring and control purposes. This imposes a significant bandwidth requirement on the network and also exposes the system to possible issues with respect to robustness and reliability.

Among the drawbacks of the enterprise-down approach are the following:

- It makes it impossible to develop, configure, and validate the measurement system detached from the supervisory level.
- It does not provide a migration path from laboratory development to on-line deployment.
- It necessitates the transfer of large arrays of raw data between the analyzer and the supervisory level in real time – thereby compromising system robustness.
- It invites the use of overly complex chemometric models, with the attendant likelihood of over fitting and hence error.
- It renders the measurement system inoperable in the case of the failure of any of the requisite communication channels.

INTEGRATION AT THE ANALYZER LEVEL

An alternative to the enterprise-down approach to analytical integration is to incorporate sufficient localized intelligence at the analyzer level to allow the instrument data to be processed near the lowest tier in the automation hierarchy – resulting in essentially a smart sensor. Only the resultant processed data, meta-data, outlier status, and performance validation results are routinely transmitted to the higher-tier levels – resulting in significantly reduced bandwidth requirements. Improved reliability is achieved not only by reducing the bandwidth but also by limiting the complexity of the information flowing between sub-systems. This results in a more lightly-coupled overall system with only the pertinent information being transmitted.

The focus of the work discussed below has been on the integration of the analytical instrument and all of its ancillary hardware and software components into a semi-autonomous integrated analyzer system, which outputs process variable predictions and related low data rate performance information and which can be fully configured and validated without recourse to the supervisory layer. The overall scheme is illustrated in Figure 2. Here the integrated analyzer system is imbued with sufficient intelligence to carry out all of the operations required to sample the chemical process, obtain the raw data, and perform the required chemometric data reduction. Resultant process variable predictions and outlier status are reported to the DCS and other supervisory level components via a robust, relatively low data rate communication channel employing OPC or Modbus connectivity. Raw data (e.g. spectra) can be stored locally and transmitted to the supervisory level as required. For many applications, it may be sufficient to transfer a representative sample of the raw data on a scheduled basis or when enabled by a triggering event.

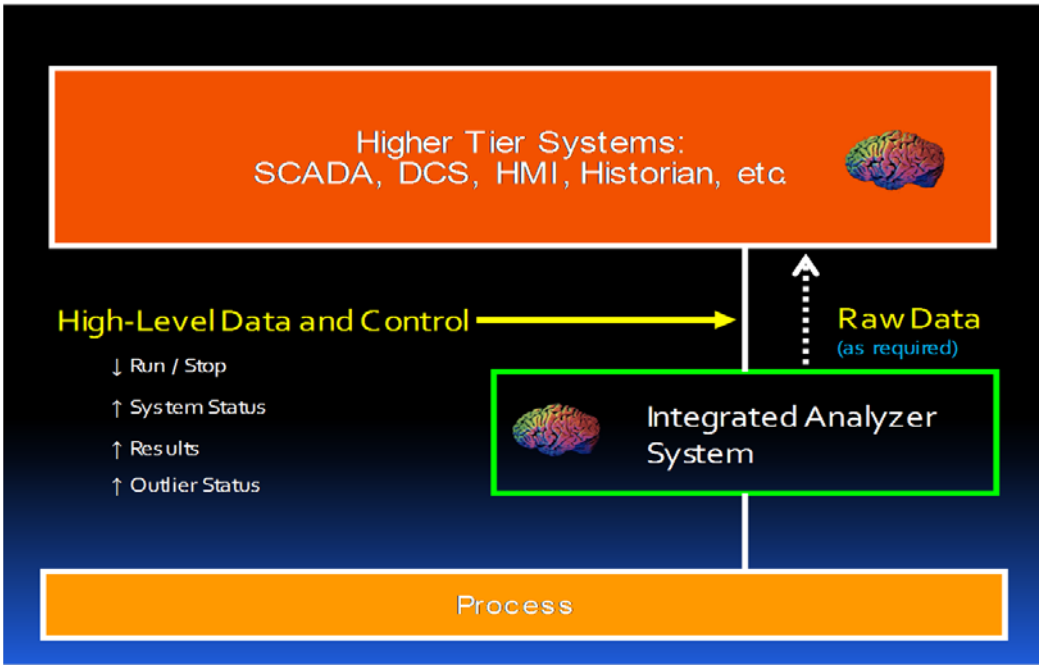

FIGURE 2. ANALYTICAL INTEGRATION AT THE PROCESS/ANALYZER LEVEL.

Figure 3 illustrates the typical components of a semi-autonomous integrated analyzer system. The example contains one or more analytical instruments, sample systems, a mathematical processing technique (e.g. chemometrics), database storage, and a communication mechanism to transmit the resultant processed data and the raw spectroscopic data. The typical sequence of operations would start by commanding the sample system to present a representative sample to the analytical instrument. This may employ local controls for temperature, pressure or flow. Next, an instrument data collection is initiated. The raw data is then processed via a mathematical technique such as PLS. Finally, the resulting processed data is sent to the PLC/DCS or SCADA system. Raw data can be stored locally and transmitted to the higher level for archiving and administrative analysis as required.

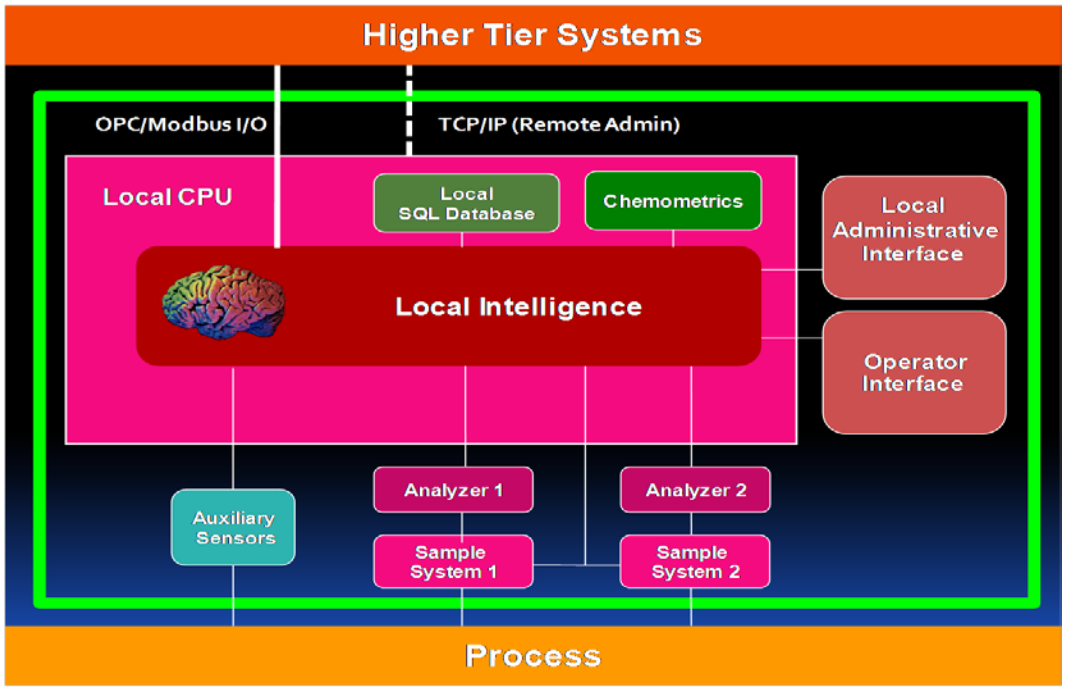

FIGURE 3. ELEMENTS OF A SEMI-AUTONOMOUS INTEGRATED ANALYZER SYSTEM.

The overall goals of the integrated analyzer system development program have been both to accelerate the deployment of process analytical systems and to insure the validity and accuracy of the data produced. A more specific goal has been to enable the analytical data system to be deployed in the form of a series of locked-down analytical appliances which can be prevalidated for use in specified applications, in effect providing a series of dedicated analytical sensors.

USER REQUIREMENTS

At the outset of the integrated analyzer system program, user requirement inputs were solicited from a number of key potential users. These are summarized below along with some comments.

1. A Common Platform for Feasibility Studies, Method Development, and On-Line Monitoring: During the early stages of feasibility analysis and modeling, the analytical requirements are likely to be substantially different than those encountered during on-line process monitoring. For example, it is often necessary to rapidly survey a number of different samples and analytical options. As a result, the emphasis is often on real-time visualization rather than on chemometric modeling. The instruments

employed at this stage are more likely to be broad-capability laboratory instruments rather than the more dedicated process instruments. Needless to say, it is very desirable for the same software tools to be available over the full range of development steps.

2. Drivers for all Required Instruments and Software Packages: While the OPC-ADI specification offers a standardized approach to instrument interfacing, it places the burden for interfacing on the individual instrument vendors. Our approach is to provide the flexibility of either using the OPC-ADI specification or of employing a pure TCP/IP based driver optimized for each instrument. The latter approach has the inherent advantages of intrinsic network compatibility, reliability, and connectivity thereby leveraging all of the prior developments of standard network interfacing. OPC, in contrast, requires additional software (i.e. "tunneling") in order to be network compliant. This introduces additional overhead and complexity.

3. Sample System Configuration and Control: In most cases, the sample conditioning system (if employed) is integral to the operation of the analyzer system and needs to be synchronized with data collection and the other functions of the system. The control of the sample conditioning system locally makes it possible for the analyzer system to continue functioning even if there is a failure of communication with the supervisory level. In addition, it allows the operation of the sample system to be validated in conjunction with the rest of the analyzer system independently of the supervisory data system.

4. Flexible method development capability: The concept of the analyzer system as an autonomous entity capable of functioning independently of other data systems implies the existence of a comprehensive method development capability. This capability should include a simple yet powerful scripting environment – capable of handling any requirement – as well as the ability to fully test any method off-line.

For the program being discussed, a compact instruction set specifically tailored to the needs of chemical process analysis was created. This is based on TCL (Tool Command Language) which is widely used in the semiconductor industry and several of the national labs. TCL has inherent advantages in that it is easy to extend, embed, and customize to meet the needs of the most demanding process analytical applications. TCL typically requires much less programming than other languages to accomplish the same task. In addition, TCL utilizes an embedded interpreter so that the user can develop applications without the need to compile and execute outside of the development environment, resulting in rapid prototyping of functionality.

5. System diagnostics, real-time data display, and alarming: System diagnostics can include automated self verification of the instrument, periodic checks of software functionality, sample system tests, inclusion of a watchdog timer function, and passing of outlier status along with chemometric predictions to the DCS. Local real-time raw data, trend, and statistical displays are essential if the measurement configuration is to be developed, validated, and maintained independent of supervisory level data systems.

6. Archiving of data in a local or remote SQL database: The storage of raw and process data, metadata, system attributes, and operator attributes in an SQL database streamlines the organization of this information as well as its transfer to other data systems. In particular, it provides for searching and reporting based on any combination of attributes. Finally the use of the database provides the "store and forward" capability needed in the event of a communications failure.

7. Remote communications using common protocols: Typical I/O applications utilize OPC or Modbus protocols to transport digital and real-valued data both to and from a process analytical system and other higher-tier automation systems. In order to assure longevity as well as compatibility with networking standards, it is believed that any newly developed solution should include the use of the TCP/IP protocol.

EXAMPLES OF PROCESS ANALYTICAL INSTALLATIONS

The software developed to meet the above requirements has been discussed in detail elsewhere. (12) Here, we will simply give two examples illustrating the use of this software to provide semi-autonomous integrated analyzer systems. These examples illustrate two distinct types of requirements, one involving a complex sample conditioning system and the other involving the multiplexing of multiple in-line optical probes so as to create, in effect, a series of dedicated smart sensors.

EXAMPLE 1: SPARGING-INFRARED WASTEWATER ANALYSIS:

This is an example of an autonomous integrated analyzer system which employs a complex sampling system. Its purpose is to sample the wastewater flowing through the main drain of a large chemical manufacturing complex and to measure the concentrations of four specific pollutants several times an hour, twenty-four hours per day. It functions by sparging a stream of air through a volume of water so as to transfer the dissolved pollutants from the water to the air stream where their concentrations can be measured in an infrared gas cell coupled to an FTIR spectrometer. (13)

The major system components of the sparging-IR wastewater analyzer are illustrated in Figure 4. A sample of wastewater is periodically metered into a cylindrical sparging vessel. Plant air at a fixed flow rate is then introduced into the bottom of the vessel by means of a sparging nozzle. As the air flows through the liquid, the volatile pollutants are transferred to the air stream at a rate determined by their solubility, vapor pressure, and water temperature, in accordance with Henry's law. The air stream is transferred to a low volume infrared gas cell (Axiom Analytical, Inc. Model LFT-210) (14) which is coupled by means of metallic (Axiot™) light guides to a Bruker Matrix-M FTIR spectrometer (15) All aspects of system operation are controlled by the process analytical software under discussion. (13)

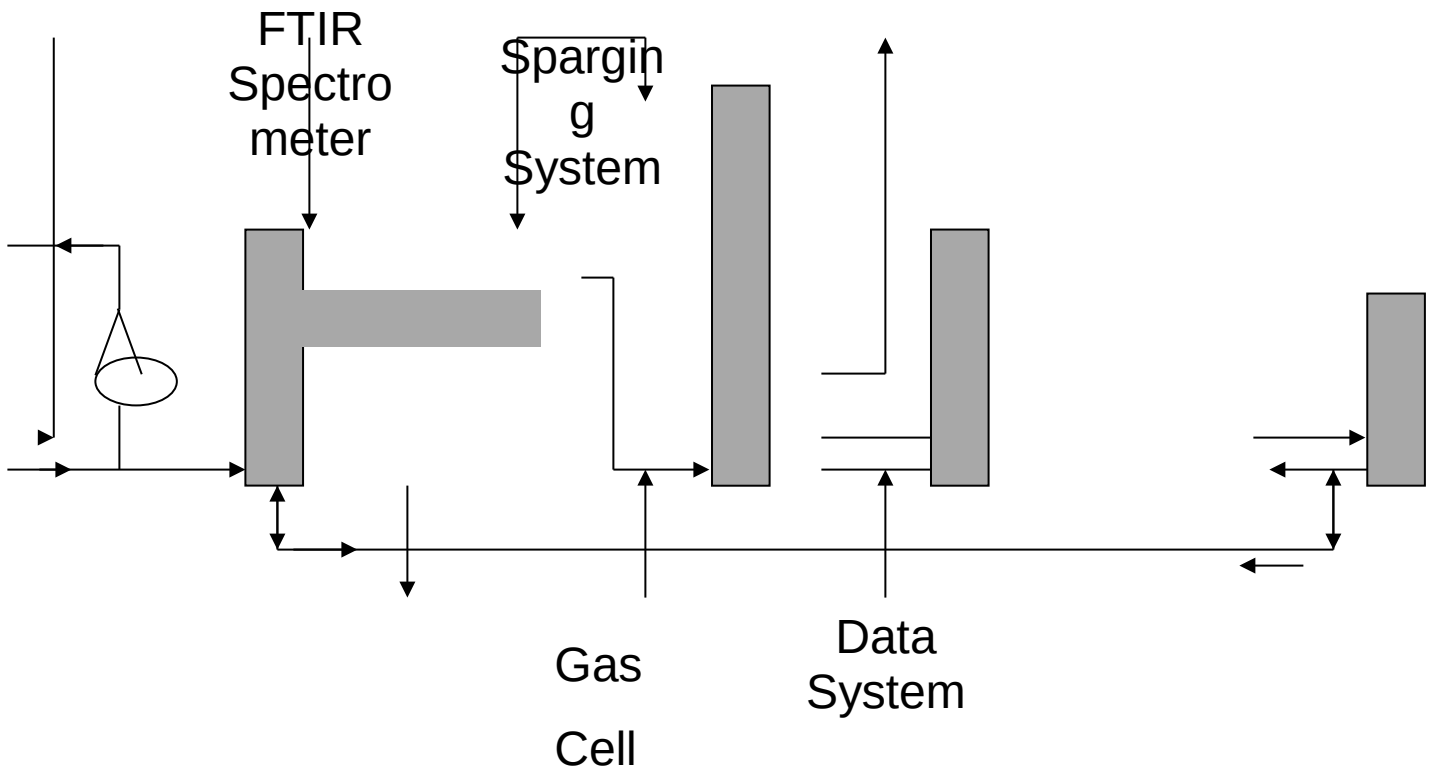

FIGURE 4. WASTEWATER ANALYZER SYSTEM COMPONENTS

In detail, the wastewater analyzer sampling system is somewhat more complex than Figure 4 alone would suggest. For example, the system switches between two sample streams, as well as between sparging and back-flush air flow, provides for flushing the sparging vessel with clean water, controls the gas cell temperature, and meters in an antifoam agent, when required. In addition it monitors water temperature, air flow rate, and the pH of both streams. In all, the software needs to process signals from eight different sensors and issue instructions to nine different control points.

The scripts used to control the operation of both the sampling system and the spectrometer and to perform the chemometric predictions are resident in the system's "Set-up" screen. Below is an example of the particular script used to control the spectrometer, predict concentrations, and plot the results. In this example, the FTIR spectrometer is commanded to take eight co-added absorbance scans using the parameters from the XPM file C:/MyXMP.xpm, as illustrated by the first statement below. The data and meta-data from the scan are passed into the local structure variable x1. This variable is then used in the second statement to run the PLSPlus/IQ embedded run-time engine against the data and the PLS calibration C:/MyCal.cal. The prediction and other metric information are passed into the local variable x2 for 0^{th} constituent. The final statement plots the prediction to the "Run-time" window.

```
set x1 [ ControlInstrument Bruker Absorbance 8  {xpm = C:/MyXMP.xpm} ]

set x2  [ RunPrediction PLSIQ {C:/MyCal.cal} $x1 0 ]

PlotRTTag structure $x2 Concentration Name Units Color
```

Scripts of this type can be developed using an integral script composing environment. Individual lines of code are written by filling in the blanks in a series of pop-up windows and then pressing "insert". These can be tested off-line by using the script composer's plotting tool. Once approved, a script can be pasted into the main set-up window and saved as a new measurement configuration.

A series of trend plots obtained during a test run of the sparging wastewater system is shown in Figure 5. The lower region of this screen (the "Run-time" window) provides statistical information and indicates alarm levels and display ranges. Concentration predictions can be determined from peak trend values, in conjunction with measured temperature, or by integrating the areas under the plots. The integrated area approach enables calibrations that are independent of both water temperature and sparging efficiency. However, the peak measurement aproach is somewhat easier to implement and has been found to provide measurement accuracies that are consistent with the customer's requirements. (Note that the Run-time window can be configured to display any desired combination of raw data, predictions, and statistical information.)

The sparging-IR wastewater analyzer operates 24/7. It has proven very successful, saving many times its cost each year by eliminating the need for costly and highly inaccurate manual sampling and by rapidly detecting contaminates so that remedial action can be taken before any harm is done. The system runs autonomously, routinely reporting only status, alarms, and exceptional information to the plant-wide network. All raw data is saved locally on a rolling basis for one week unless an alarm occurs, in which case the data for the previous week are transmitted to the plant network for further analysis and archiving.

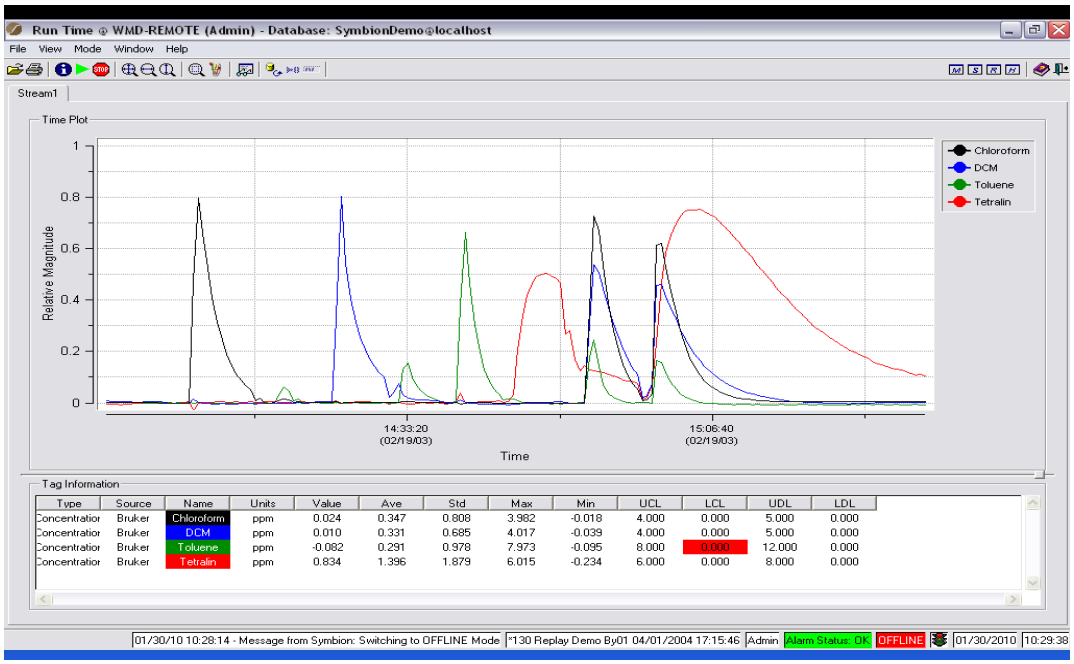

FIGURE 5. WASTEWATER ANALYZER TREND PLOTS DURING A TEST RUN.

EXAMPLE 2: A FULLY INTEGRATED CHEMICAL PRODUCTION FACILITY:

The second example is a system that does not employ sample conditioning or stream switching. Instead, it uses fiber-optic multiplexing to switch between a series of near-infrared probes (recently increased to a

total of 13) which are permanently installed in various processes in a chemical manufacturing plant. The overall system configuration is illustrated in Figure 6. The hardware includes of a single FT-NIR spectrometer and two fiber-optic multiplexers, one integral to the spectrometer and the other located in a building several hundred meters from the analyzer. The probes consist of eleven transmission probes and two diffuse reflectance probes.

The process analytical software program controls all aspects of the multiplexed analytical system operation. In particular, it sequences the operation of the thirteen sampling probes and provides a unique measurement configuration for each probe, including a background reference spectrum, a set of collection parameters, math preprocessing, a chemometrics calibration file, and output data specifications.

Each of the thirteen collection channels is currently configured to provide up to eight output trends as well as outlier information for each of these. Thus, the system could, in principle, provide up to 13 x 16 = 208 independent output signals. In practice, fewer than half of these are currently employed.

As in the case of our first example, the multiplexed system operates semi-autonomously, simply providing process variable, outlier information, and a watchdog timing signal to the DCS on a routine basis. Additional information, such as raw data, are provided to the plant network as needed. This system constitutes a fairly extensive example of an integrated analyzer system. At the same time, the net result of this integration is to provide, in effect, a set of thirteen smart sensors the operations of which are sequenced so as to allow the use of a single spectrometer and software program.

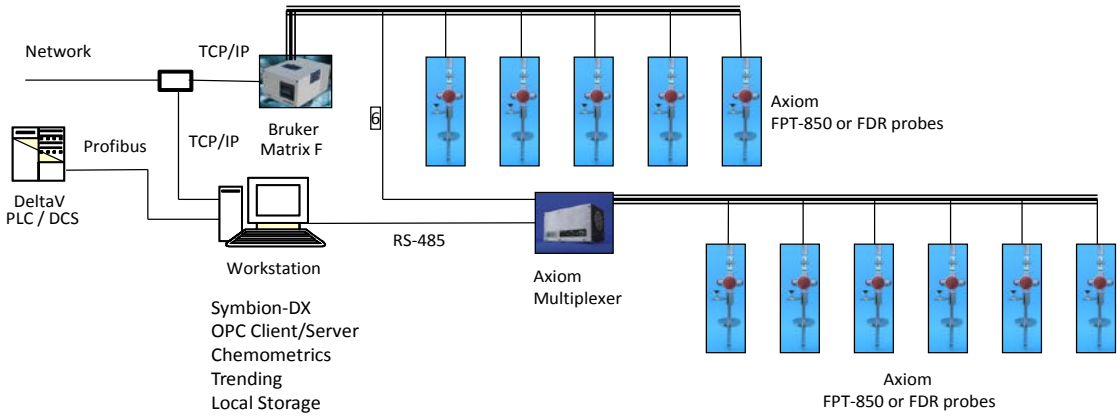

FIGURE 6. INTEGRATED CHEMICAL PRODUCTION FACILITY ANALYZER SYSTEM CONFIGURATION.

THE ANALYTICAL APPLIANCE

As noted above, one of the goals of the program outlined here has been the provision of analyzer systems which function semi-autonomously and which provide functionality analogous to that of a series of traditional sensors. This goal can be best achieved by packaging the intelligence of the system in the form of a dedicated analytical appliance. Among the requirements for such an appliance are the following:

- Robust tamper-proof hardware – e.g. no moving parts
- Embedded operating system
- Embedded process analytical software
- Prevalidated (locked-down) measurement configurations
- Strict revision control
- Store and forward capability
- Regulatory compliance

Such a system has been implemented using a ruggedized small footprint industrial computer. Other form factors that have been identified include a Pico-ITX (100 x 72 mm). This would allow an instrument provider to embed the appliance in its own system so as to incorporate all of the functionalities of the standardized process analytical software package within their product. A water-proof form factor has also been identified. This would allow the appliance to be deployed in areas that required NEMA-style compliance. Finally, a rack-mount form factor would allow the appliance to be incorporated into a standard 19'' industrial chassis.

CONCLUSIONS

Analytical Integration at the analyzer/process level provides:

- **Standardization** in the form of a common platform for feasibility, development, and process monitoring as well as common software for all instruments and analytical methods.

- **Extensibility**, i.e. the ability to handle the most complex analytical and control tasks and to integrate these into unified measurement configurations.

- **Accessibility** in the form of local control, display, database storage, and reporting as required.

- **Semi-autonomous Operation** allowing the system to operate independently of other systems, thereby providing intrinsic robustness – including the ability to continue functioning in the event of a communication failure.

- **Data Reduction at the Measurement Point** with only process data being passed to higher level systems in real time thereby minimizing required bandwidth and enhancing overall network reliability.

- **Hardened Deployment**, i.e. the potential for deployment using a dedicated analytical appliance.

In addition to these operational benefits, local integration provides an analyzer system which can be developed, configured, tested, and validated without requiring interaction with the supervisory level or other data systems. Furthermore, it organizes information along functional lines, providing a clear picture of the condition at each measurement point and unit operation before passing this information to a higher tier for further integration. By clearly distinguishing between different logical levels of integration, analytical integration at the process level also makes optimum use of the skill sets of clearly identifiable specialists such as analytical scientists and analyzer engineers.

An autonomous analyzer system can be deployed in the form of a dedicated "analytical appliance". As such, it can be both standardized and configurable. Standardization is provided by employing a fixed, hardened industrial computer, the "Windows Embedded™" operating system, and a fixed embedded version of the process analytical software program. Configurability is provided by employing a series of measurement methods or "Configurations" which can be developed and validated on a standard PC and then uploaded to the appliance through a secure link. As a result, the analytical instrument, combined with its ancillary hardware and software becomes, in effect, a series of smart sensors outputting predictions of specified process variables measured at identified locations within a chemical process.

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