

(19) World Intellectual Property Organization  
International Bureau



(43) International Publication Date  
31 March 2005 (31.03.2005)

PCT

(10) International Publication Number  
WO 2005/029541 A2

- (51) International Patent Classification<sup>7</sup>: H01L
- (21) International Application Number: PCT/US2004/025502
- (22) International Filing Date: 6 August 2004 (06.08.2004)
- (25) Filing Language: English
- (26) Publication Language: English
- (30) Priority Data:  
60/494,977 14 August 2003 (14.08.2003) US

(81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW.

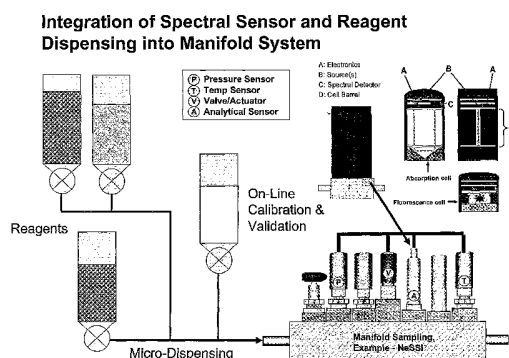
(84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

- (71) Applicant: MICROSPECTRAL SENSING, LLC [US/US]; 18 Ocean Street, South Portland, ME 04106 (US).
- (72) Inventors: COATES, John; 12 North Branch Road, Newton, CT 06470 (US). RATHGEB, Fernando; 31 Fales Road, Sharon, MA 02067 (US). STOWELL, Rand; West Side Road, Weld, ME 04285 (US).
- (74) Agent: CASEIRO, Chris, A.; Verrill & Dana, LLP, One Portland Square, Portland, ME 04101 (US).

Published:  
— without international search report and to be republished upon receipt of that report

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: SYSTEM AND METHOD FOR INTEGRATED SENSING AND CONTROL OF INDUSTRIAL PROCESSES



(57) Abstract: An integrated spectral sensing engine is based on a combination of energy sources (illumination) and detectors housed within an integrated package that includes the sample interfacing optics and acquisition and processing electronics. The focus is on a miniaturized sensor system that can be optimized for specific measurements and can be integrated into a manifold-based sample handling system. Design and fabrication components are selected to support high volume manufacturing of the sensors. Spectral selectivity is provided by either continuous variable optical filters or fabricated filter matrix components. The spectral response of the primary sensors covers the range from the visible (400 nm) to the nearinfrared (1100 nm), as defined by the availability of suitable low-cost solid-state detector devices. Provision is made to extend the range into longer wavelengths, and to shorter wavelengths for filter-matrix devices. A broad selection of measurement modes is defined and these include transmittance/absorbance, turbidity (light scattering) and fluorescence. On board data processing not only provides the primary data acquisition, as well as data massaging and the display and output of computed results. The targeted application of the spectral sensing devices are for water, pulp and paper, chemical and petroleum based industries. Alternative packaging regimes and the production of lower cost sensing devices can lead to the use of the spectral sensing devices in the medical, clinical, forensic and consumer based areas of application.

WO 2005/029541 A2

**System and Method for Integrated Sensing and Control of Industrial Processes****Background of the Invention**

## 1. Field of the Invention

[001] The present invention relates to integrated systems and methods for sensing parameters associated with industrial processes and controlling such processes based on the sensed information. More particularly, the present invention relates to a miniaturized spectral sensing system, with integrated sensed signal conditioning, signal exchange, and integration of the sensed information with appropriate process control for improved industrial processes.

## 2. Description of the Prior Art

[002] The processes employed to make products vary widely from industry to industry. Those processes may involve the use of complex machinery, interconnected machinery and equipment, and electrical and chemical exchanges. It is important to ensure to the greatest extent possible that the features and components of an industrial process interoperate as effectively as possible. Achieving that goal results in good products and good productivity. Additionally, it can minimize the impact of the process on the surrounding environment. Two related aspects of industrial process improvement include the need to understand the process itself, and control of the process based upon that understanding. In particular, it is important to be able to determine the characteristics or parameters at each stage of the process and for the final output of a process, such as the chemical composition, temperature, and/or pressure of a gas mixture used to make a chemical compound, for example. Based upon that information, it is important to be able to adjust the process, such as increasing process temperature, or changing the ratio of reactants, for example, if the output information deviates from an established set of parameters. The inability to monitor the process usually means that there is a risk of low quality product, which results in the need to make corrections after the product has been made. This is both inefficient, and leads to a high level of wastage, which can often lead to an environmental issue.

[003] There are two established approaches to the monitoring of a process for chemical composition or physical properties. They are the extraction of grab samples followed by remote analysis at a suitable control laboratory, and the use of on-line instrumentation. The first option is inefficient and is not effective for control purposes. The second option is usually expensive, and as a result, it is normal to implement a single analyzer at the end of the process. This has limited value for good process control because it

is too late in the process to make meaningful adjustments. For a complex process, the ideal situation is to have a multiplicity of measurement points and to monitor the process from the raw material through to the final product. This has to be cost effective to make the implementation of multiple sensing points worthwhile. One solution with optical instrumentation is to use a single analyzer but to multiplex the stream or the optical output. While this is an option, it has risks because it lacks redundancy--one instrument controlling an entire process. It is also limited in terms of its response, dependent on the number of points measured (measurements are made sequentially, not in parallel). The present invention uses multiple low cost sensing devices, a major advancement, and overcomes issues related to a lack of redundancy. In fact, one may use a redundancy of sensing devices to ensure maximum efficiency in the event of the failure of a single sensing device.

[004] Many industry standard methods for process control still rely on Proportional-Integral-Derivative (PID) controllers, a technology dating back 60 years. Once tuned, the system is only able to control the process with which it started. Should process behavior change after start-up, the controller cannot counteract disturbances and the closed-loop system may become unstable. The traditional fix for time-varying process dynamics is to start over and manually retune the loop whenever its performance degrades. While that may not be particularly difficult, repeated tuning can be tedious and time consuming, especially if the process takes hours to respond to a tuning test. Manual tuning may not even be possible should process behavior change too frequently, too rapidly, or too much. In the case of model based controllers, process characteristics such as gain, time constants, and dead time need to be estimated, often by trial and error, to attempt defining the shape of the process response curve. It is a long and complex process that requires significant investment of time and money. The present invention includes the option of a firmware-based control, which is also miniaturized, and is reduced in size to a small electronic board, which overcomes these established problems associated with both standard control systems and traditional model based systems.

[005] When measuring devices are integrated into a process it is normal to employ a sampling system. The sampling system is typically a collection of valves and sample conditioning devices (filters, mixing chambers, temperature control loops, etc.) that extract the sample from the main stream, and present the sample in an ideal format to the measurement system. Traditionally, this collection of valves and components takes up a rather large space, and can sometimes be as expensive as the measurement device to implement. In the miniaturization of the sensing devices, it makes little sense to use such a

system, in terms of efficiency and cost. Significant benefits are gained if the sensing device and the sampling system can be integrated where the sample volumes are matched to the sensing device itself. Recent developments in industrial process improvement initiatives have been centered on the mechanism for integrating sensing devices into sampling systems. A good example is the New Sensors/Sampling Initiative (NeSSI) sponsored by the Center for Process Analytical Chemistry (CPAC) at the University of Washington, an effort by an industrial consortium to standardize sensors and the sensing platform used for process monitoring. Initially, traditional parameters such as temperature, flow and pressure, which can be important indicators of process characteristics, have been addressed. The goal of the NeSSI is to make measurement techniques uniform across industries with an interest in participating in the initiative. The platform is a miniaturized version of traditional sample gathering and handling methodologies and, pursuant to the Instrumentation, Systems, and Automation Society (ISAS), standard SP76, establishes the interface of the sample gathering components with sensing devices used to assess the characteristics of the extracted sample.

[006] The benefits of the NeSSI system are its size, the ability to add components as standard modules, and the ability to integrate the sensing system to form a single stand-alone unit for sample extraction, conditioning and measurement. The objective is to develop sensing devices to be compatible with the goals of NeSSI. What is also needed is such a system that enables sampling and sensing at intermediate sites along the way of the process, thereby permitting process corrections earlier and minimizing defective product output. Note that NeSSI has been used here as a discussion point, and is not necessarily the only platform for consideration. There is a movement in a wide variety of fields that involve the handling of liquids, gases and vapors, including medical and clinical applications, where miniaturized valves and sample handling/conditioning are involved. The approach to integration of sensing within these platforms is also given consideration.

#### Summary of the Invention

[007] It is an object of the present invention to provide an integrated spectral sensor and an optional control system for controlling industrial processes. The system includes a sampling component, a spectral engine including a sensing component and a signal conditioner, a signal exchange system, and a controller. The sampling component can include, where required a multiport manifold on a miniaturized sample handling platform (such as NeSSI) for gathering one or more fluid samples (including reagents) related to the industrial process to be controlled. The sampling component interfaces with the spectral

engine that includes an optical sensing system for nonintrusive detection of features of the sampled fluid. The spectral engine further includes a light or energy source, spectral sensing component for measuring the characteristic chemical features of the fluid, a sample cell or chamber that is dimensionally optimized for the light source and sensing component, and a microprocessor for conditioning the signals output from the spectral sensor. The signal exchange system may be a wired or a wireless signal transfer device coupled locally or remotely to the sensor. The optional controller includes the signal processor and one or more embedded computer programs to evaluate the received signal information and make decisions on any process modifications to be made. The controller is a predictive controller that maximizes the ability to adjust process conditions as quickly as possible in response to the sensed process information. The controller interfaces with one or more process modification devices, including control valves, chillers and heaters, for modifying, pressure, flow rates and volumes, and temperatures of the fluid or fluids forming part of the industrial process.

[008] Four example application areas have been identified that can benefit from this integrated sensor approach, and these include the water, pulp-and-paper, chemical, and petroleum industries. These applications require on-line, real-time sensors, sensors that are capable of operating in harsh environments, and can provide analytical and physical property measurements. The present invention addresses these needs. In the case of the chemical industry, a wide range of applications is envisaged in a wide range of industry sectors, from specialty chemicals, such as pharmaceutical products to commodity chemicals, such as petrochemicals and polymers. The applications go beyond just the manufacture of the raw material and basic chemicals, and can be extended to the blending and formulation of final products in key high energy consuming industries, including those linked to consumer products. It is to be understood that the present invention has broader applicability than the application areas cited.

[009] In conventional industrial process plants, process sensors and actuators are hardwired using copper wire or fiber optics networking. Because of the high costs associated with installation, maintenance, and constant reconfiguration of a process, there is a large opportunity for a major cost advantage for using a wireless communications path to interconnect the sensors and actuators. This is particularly important when a network of sensors is being employed. Also, when using an optimizing predictive adaptive control system, better communications between sensors improves the overall efficiency. For this reason, the present invention contemplates the option of employing wireless connectivity to establish data signal transfer.

[10] The integrated sample, sensing and control system of the present invention provides a more granular and immediate picture of relevant information associated with an industrial process. That picture is coupled with effective predictive process control to yield improved productivity with corresponding reduced impact on the process's surrounding environment. These and other advantages will become more apparent upon review of the following detailed description and the accompanying drawings.

#### Brief Description of the Drawings

[011] FIG. 1 is a simplified representation of the elements of an example spectral sensing engine: source, sample interface, light analyzer and detector.

[012] FIG. 2, comprising FIGS. 2A-2C, show example combinations of optical filters and detector arrays.

[013] FIG. 3 shows the electronic components for the integrated spectral sensor.

[014] FIG. 4 is a simplified representation of standard package configurations for on-line sensors: absorbance/transmission and fluorescence cells.

[015] FIG. 5, comprising FIGS. 5A-5D, illustrates alternative packaging configurations of on-line sensors: turbidity, NIR and immobilized reagent versions.

[016] FIG. 6 is an example integration of spectral sensor and reagent dispensing into manifold system.

[017] FIG. 7 is an example spectral sensor response in the visible region: colored dyes.

[018] FIG. 8 is an example spectral sensor response in the near infrared region: common chemicals.

[019] FIG. 9 is a simplified flow diagram of the steps associated with the function of the optional predictive-adaptive controller.

[020] FIG 10 is a schematic representation of an optional predictive-adaptive control system.

[021] FIG 11 is an example schematic representation of the predictive-adaptive control system in a multi-loop implementation.

[022] FIG 12 shows an example mesh-network for wireless communications with multiple sensors.

#### Detailed Description of the Preferred Embodiments of the Invention

[023] The present invention is an integrated system for sensing process characteristics and controlling the process based on the sensed information. The sensing aspect of the invention preferably includes one or more miniaturized optical spectral sensors located at multiple points within a process or an individual process unit. This provides a means to monitor a process from start to finish, with key intermediate points also covered, as opposed to the traditional approach of monitoring the product as it is produced at the end of the production line. An optional component of the system is a wireless communication interface, based on a proprietary adaptation of a standard wireless platform, associated with the multiple spectral sensors to allow them to interface with the control component of the system. The system further includes an optional control component as an adaptive-predictive control system that makes use of the feedback from the sensors and provides optimized process control.

[024] An important component of the spectral sensor technology can be broadly described as an optical spectrometer on a chip. While optical sensors have been available, the present invention integrates an optical filter assembly with a light or energy sensitive array. The optical filter technology used is either in the form of a continuous linear variable filter (LVF), or a filter array (patterned filter or mosaic). In the LVF form the resultant device, or spectral sensing component, is the most versatile and can be utilized for many applications. An example format of an LVF -based spectral sensor is shown in FIGS. 1, 2A and 2C. The spectral sensing component is preferably implemented as part of a photodiode or a Complementary Metal-Oxide Semiconductor (CMOS) array detector package. The sensing component of the present invention is based on existing optical sensing technology modified for the present purpose. An example component has been marketed as a commercial device by OCLI (a JDS Uniphase company), known as a MicroPac. The device was not produced in a form that was compatible with the proposed application, and was intended for lab-based experiments that demonstrated feasibility. As developed, the MicroPac was based on a silicon photodiode array offering spectral ranges of 400 nm to 700 nm (visible) and 600 nm to 1100 nm (short wave near Infrared (NIR)). The MicroPac had a complex construction featuring a gradient index (GRIN) lens as an optical interface between the filter and the photodiode assembly. This was required to preserve the spectral resolution of the filter because the detector used was a standard commercial package. In the current embodiment, the LVF is directly bonded to the detector array, and in this form does not require any form of resolution retaining optics. Sensors derived from these components based on the LVF can be used for absorption measurements in the visible and near infrared (NIR),

as well as fluorescence measurements in the visible. Examples of data have been acquired in all of these modes, and example spectral response curves for the visible and NIR ranges are provided in FIGS. 7 and 8. The short wave NIR provides good differentiation based on chemistry and composition based on vibrational overtones of the component molecules. However, in cases, such as the digestions in pulp and paper applications, where visible absorbing and fluorescence centers are also expected to be important, the visible version for the spectral sensor is used. For applications involving chemistry, where the species to be measured is not normally visible, the analysis may be performed with the addition of a reactive chemical reagent. The reagent may be added as an additional chemical to the process stream, or the process stream can interact directly with versions of the reagent that are immobilized on or in a solid substrate, which forms part of the optical path as shown in the examples in FIG 5.

[025] Those skilled in the art of optical sensing technology will recognize that the short-wave Near Infrared (700 to 1100 nm) works well for a wide range of liquid-based measurements. In this region, sample pathlengths from 1 to 10 cm are considered to be optimum, dependent on the material to be measured. Such pathlengths may be used for direct measurements made on petroleum and liquid chemical products, while shorter path lengths may be required for darker materials, such as digestion products and materials with high aromatic content. Although spectral changes in this region are subtle, they can be readily correlated with both composition and key chemical and/or physical properties. Tools such as multivariate modeling, sometimes known as chemometrics are common for such applications. These would be used as appropriate, and the calibration coefficients generated from the modeling are stored on flash RAM located on-board the sensor.

[026] It is also possible to consider indirect methods of measurement while measuring in this silicon spectral region. These are methods are as noted above, where a reagent interacts with the stream either as a liquid reagent or on a substrate that interacts with the process stream, and spectral changes are observed for the substrate. Examples are pH or chemical reactivity of the stream, where the stream reacts with a reagent that is immobilized in a porous solid matrix, such as a sol gel or a membrane (organic or inorganic). Systems featuring immobilized reagents can also be used for gas phase measurements where the gas or vapor is chemically reactive and is able to form a colored species once it interacts with the immobilized reagent. An example of such a reaction is with carbon monoxide and a derivative of hemoglobin. If the reaction is reversible, the sensor may be used for continuous monitoring. in cases where the resultant reaction is a color change or a change in a level of

fluorescence. The optical sensor system of the invention may be used to conduct direct measurements, as well as with immobilized reagents necessary for applications in the water industry, and in the pulp industry, for example, especially for the measurement of materials such as sulfides, both in the liquid and gas phases. For applications that feature liquid based reagents, the reagent or reagents are metered into a mixing chamber which exists as one of the components of the manifold system. Applications based on chemical reagents do not necessarily require long optical pathlengths, and pathlengths ranging from 0.25 cm to 2.00 cm are expected to be the norm.

[027] As defined, the spectral sensor can be constructed from either a continuously variable filter (defined as the LVF) or from a filter matrix or mosaic. This latter approach is usually optically more efficient and less expensive than the LVF approach. It is often more specific in application, but less versatile than the LVF system. An illustrated example of a matrix-based spectral sensor is provided in FIGS 2 and 3. The version shown is a 4-channel RGBW sensor, and is capable of handling a wide range of color-based applications.

[028] The sensor hardware for the present invention is not limited to silicon-based photo-sensing devices, and alternative detector arrays can be used, including InGaAs, PbS, PbSe and also Micro Electronic Mechanical System (MEMS)-based devices. Such devices would be considered for extensions into the longer wavelength NIR and for the mid-IR. The format of the proposed sensor platform may be extended into these other spectral regions. In the case of the mid-IR spectral region, these are expected to be mainly for gas and vapor phase applications. Note that for some applications, the silicon-based detectors may be used in conjunction with an immobilized reagent for certain gas-phase measurements. For example, vapor analysis for pulp digestions may be handled by this approach, especially for the detection of sulfides.

[029] The approach offered is described as being based on a spectral engine. The spectral engine includes the spectral sensing device (described above), and the energy source, which can be either a broadband or narrowband source, dependent on the mode of measurement (broadband sources are used for NIR and visible absorption, narrowband sources are used for turbidity and fluorescence). White LEDs and tungsten bulbs are used as broadband sources, and individual LEDs are used as narrowband sources. Another component of the spectral engine is the sample interface, which is typically a cell or chamber. One of the key benefits offered by the system are that the cell can be optimized in size based on the source and spectral sensing system. The sizes of the detection devices are 1 x 8 mm and approximately 3 x 3 mm (matrix sensor). Scaling the sample cell to these dimensions

results in a net cell volume of a few microliters up to around 80 microliters. The advantage gained here is that a minimum sample size is required, which effectively eliminates any sample temperature effects, and significantly reduces the amount of reagents that have to be dispensed for reagent-based applications. The volume reduction on reagents can be up to 1000 times, which reduces reagent vessel capacities, dramatically reduces consumption, and cuts operating costs. The final critical set of components of the spectral engine are the electronic components. An example of the functional electronics is provided in FIG 3. Up to two microprocessors, and possibly more, may handle the initial data handling (processor #1) and then the data massaging (processor #2). A final processor may be employed and feature onboard memory to store methods, calibrations and results, and to handle communications to displays (if required), external devices via serial connections and also wireless communications if the option is used.

[030] It is known that measurements from industrial processes require a degree of sample conditioning. Normally this necessitates the use of a sampling system in conjunction with a process analyzer. Such an approach negates the cost and size benefits of the proposed sensor technology. For this reason, the present invention combines the spectral sensing device with a miniaturized sample handling platform. The NeSSI generation II platform is used as a practical example of a miniaturized modular approach to sample handling, based on a manifold, with the provision for modular sensor integration. The spectral sensors of the present invention can be designed to be compliant with the ISAS SP76 standard which defines the interface and format of the NeSSI platform. The sensors can also be designed to interface with any other form of miniaturized sample handling.

[031] The spectral sensor implementation based on NeSSI may be implemented in several ways. FIG 6 provides a view of the sensing device integrated into a modular base compliant with NeSSI. FIGS. 4 and 5 show example configurations for the spectral sensor that meet these requirements. This includes a basic transreflectance (absorption/transmission) arrangement wherein the source (B) radiation is directed into the sample chamber, and the beam is reflected from the base of the module up into the sensing area of the spectral sensor(C), which is powered and controlled by onboard electronics (A). These electronics can be linked to an external network, which may include wired transmission or wireless telemetry. An alternative arrangement provides a geometry that can be used for fluorescence measurements, for light scattering measurements, or for dark samples. FIG. 5 shows alternative arrangements that can be used for turbidity, near-infrared and also for applications involving immobilized agents.

[032] The optional control system component of the present invention is an advanced predictive and adaptive model-based controller suitable for critical and/or inherently unstable processes. Unlike prior art controllers, it achieves peak performance in a matter of hours, resulting in major savings of time, money and energy. The predictive controller does not require a complex predetermined or theoretical mathematical model of the process. Instead the system "learns" the process dynamics while in use and this is achieved regardless of the complexity of the underlying process behavior. High order and non-minimum phase transient response characteristics are captured by the system and are included in the model. A comprehensive model is developed based on actual process dynamics, not a theoretical model. The controller then uses this model to make accurate forecasts of process response. A PC-based controller platform suitable for integration into the sensor platform is the predictive software made available by Universal Dynamics Technologies, Inc. of Richmond, British Columbia, Canada and offered under the name Brainwave™.

[033] The control system includes a form of the Brainwave™ platform adapted for the control component of this invention in the form of firmware, incorporated into hardware electronics. The control function is in the form of software, firmware, hardware, or microcode as part of the overall miniaturized form of the invention. While the control function may be employed as a control means for the entirety of an industrial process, it is contemplated that the controller provides local optimization of control for small segments of processes, small stand-alone processes and individual process units. The control system has two main parts: the adaptive model building component (Process Model Identification), and the predictive controller. Process Model Identification is achieved quickly by using a function series approximation technique called Dynamic Modeling Technology (DMT) that is based on Laguerre polynomials. The controller monitors process response to changes in the controller output and other feedforward input variables. The transient response of the process is modeled using a Laguerre function series. The coefficients of this series are automatically calculated so that an accurate model of the process transfer function is obtained.

[034] In process control, process transfer functions are transient and the Laguerre functions are well suited to modeling these types of transient signals because they have a similar behavior to the process being modeled. The Laguerre modeling method produces a set of weights for the Laguerre functions in the series so that when summed, a high fidelity model of the original transient signal is obtained. The set of "weights/coefficients" is called the Laguerre spectrum for the signal. In short, changes in the transient behavior of the process will trigger the controller to adjust the model to the new characteristics by automatically

generating a new Laguerre spectrum. The effects of measured process disturbances are also modeled to incorporate adaptive feed-forward compensation into the control strategy resulting in further performance improvements. As illustrated in FIGS. 9 and 10, for each model, there is an internal representation of the 'state' of the system with respect to the recent changes in each input to the process (i.e., Control output, FF1, FF2, and FF3). This representation is used together with the model to make predictions about future changes that may occur in the process. Each model contributes its component of the expected change in the process. All of these predicted changes are summed together to reveal the 'net' effect on the process in the future. The process models, the system state, and the net predicted change in the process are then used to determine the necessary control action. This problem is solved by calculating a control change that (if all conditions remain unchanged) returns the process to the set point as rapidly as possible. This technique is known as *horizon predictive control* whereby the process model is used to compare the predicted value of the process to the desired target value. Any difference becomes the amount that the process has to change and the process models are used to solve the control action required.

[035] In an embodiment of the invention requiring control of multiple functions, a multi-loop approach is used for the control of large coupled processes as represented in FIG. 11. Advanced controls can be applied to most complex applications such as distillation columns, pulp and paper processing, multi-unit batch reactors, and other complex processes where manipulation of one process variable disturbs many others or where parallel process are attempting to achieve multiple objectives simultaneously. In the proposed control system, these problems become opportunities for improvement that translate into lower cost, higher quality, increased production, and a more efficient use of energy.

[036] The optional control system of the invention is designed to improve the accuracy and efficiency of an existing industrial process control arrangement. This is enhanced by the use of the spectral sensing devices, which are ideally distributed at key locations throughout the process. There is minimum hardware required and it readily becomes the technology of choice where PID controllers cannot deliver increased levels of process control performance. It may be interfaced to existing Distributed Control Systems (DCS) using an OLE for Process Control (OPC) server. The system may be configured to synchronize the predictive-adaptive controller with the DCS so that safety logic automatically switches back to traditional PID control in the event of a malfunction.

[037] In order to make full use of a multiple sensor-based control system in a manufacturing plant it is necessary to develop an economical method for interfacing the

sensors to the control system. Conventionally, one would hardwire the sensors in place, but in a system where a multiplicity of sensors is used, the efficiency and cost benefits can be rapidly diminished based on the cost of installation. The present invention provides the optional feature of wireless signal exchange. It is known that traditional approaches to the implementation of wireless in a plant environment are fraught with difficulties because of environmental interference. However, the present invention minimizes the negative impact associated with such a signal exchange method by using redundant wireless connections. As shown in FIG.12, a mesh based wireless communications system (*Comsys*) is designed to provide a reliable fully connected path between the sensors and the control system.

[038] The optional *Comsys* system has two major elements: 1) Wireless Network Access Points (NAPs) and 2) Wireless Sensors (S). In a preferred embodiment, the NAPs are fixed modules in a ring that loops around the plant or the process unit, and are powered by AC mains line power with battery backup. The distance between each NAP is such that they have a reliable wireless path between each NAP. Even if there are no sensors within range of an NAP (as shown in right side of FIG. 12), there is an NAP provisioned to ensure the ability to relay communications traffic from point-to-point. Reliability is further enhanced in such a mesh network by allowing for the NAPs to be arranged in a ring that loops into the network operations center, so that the failure of any one NAP will not cause a loss of connectivity and control. The sensors shown in FIG. 12 may be line or battery powered with the necessary power control intelligence to conserve battery power and report battery health and battery condition as required. Each sensor point communicates data to the nearest NAP, which in turn will manage contention on the network and relay the sensor traffic back to the network operations center and the control system. To further enhance the system operation and reduce the costs of installation and reconfiguration, an application layer protocol resides on top of IEEE 802.11b standard designed to manage wireless traffic, and to provide for self discovery and configuration of the system. The application layer optionally provides functions for self-discovery, geolocation, and test of the network.

[039] The fundamental aspects of the present invention lead not only to increased productivity, but also to an energy saving and process efficiency capability in at least the four target industries: water, paper, chemical, and petroleum industries. In the case of water, it also leads to increased public safety. It is expected to provide similar advantages in consumer-oriented markets, including food and dairy processing, beverages, and household

products (cleaners, etc.). The technology of the current invention will be particularly useful in the four noted fields of application:

**Water application:** continuous monitoring of public water supplies for residual chlorine content, and similar parameters that are important to public safety. Residual chlorine (disinfectant) is an important marker in drinking water supplies for the unexpected introduction of toxic and biohazard materials. The latter can range from the accidental introduction of bacteria via a breakage in a feeder pipe, to the deliberate introduction of dangerous materials. The system can also be configured to measure heavy metals (lead, chromium, mercury, etc.) and also environmental contaminants, such as phosphate, nitrate and arsenic. While the systems as described can be configured for public water supplies, stripped-down versions can also be configured for public buildings, office buildings, hospitals, and even residential water supplies.

**Petroleum applications:** monitoring of process streams for raw materials, intermediates and final products. In an average refinery there are many process units, and many of these units have several critical points where measurements can be made. Examples include the reformer, cat crackers and the blenders. The convenience and cost potential of the invention may enable many more points to be monitored, thereby permitting a higher level of overall predictive control. Many of these processes are energy intensive, and so significant savings in terms of improvements in efficiency and reductions in environmental emissions are anticipated with the implementation of this technology.

**Chemical applications:** there are numerous potential applications for the invention in the chemical and petrochemical industries. These can range from the production of raw materials to various processes used in the pharmaceutical and biotechnology industries. It is anticipated that it is well suited for the petrochemical related industry, where a significant amount of energy is involved, and where good monitoring and control can provide better overall efficiency and product quality. A practical example is the production of carbon black, where the composition of the input streams is used to provide additional information to the control system.

**Pulp and paper products:** there are several potential areas of application for the present invention in the pulp and paper industries. The most important, from the point of view of control, are probably in the digestion, pulping, and bleaching areas. The use of near infrared for the measurement of parameters such as Kappa has already been demonstrated, and the use of visible methods are feasible for determining lignin-related information. Another important area is bleaching. Both direct methods involving NIR, and indirect methods, involving

immobilized agents are expected to work for this application. Control of both the digestion and the bleaching are important for the overall process, and good control parameters for digestion are expected to provide important energy savings.

[040] While the present invention has been described with particular reference to certain specifically described components and methods, it is to be understood that it includes all reasonable equivalents thereof.

## CLAIMS

Having thus described the invention, what is claimed is:

1. An optical spectral sensing device, for determining properties of a sample, said device comprising:

an integrated energy source and an integrated spectral sensing detector package,

an optimized sample chamber or cell dimensionally designed to match the active area of the spectral detection device without the aid of additional interfacing optics;

integrated electronics for providing energy for said source and for receiving a signal generated by said spectral detector in response to energy coupled to said detector by said sample chamber or cell, said integrated electronics providing direct output of sample properties of said sample;

on-board computer processing means including memory storage for data, calibration coefficients, methods and results;

on-board data communications including the output to a visual display and communications of results to a process monitoring computer; and

a manifold-based sampling system to provide the primary interface to said spectral sensing system, and to provide necessary support for the analytical system in terms of sample conditioning and reagent interfacing.

2. The device of claim 1, wherein said optical structure is formed in configurations that allow for at least one of transfectance, transmittance/absorption, fluorescence, or light scattering measurements.

3. The device of claim 1, wherein said optical structure is fabricated from a material that can be machined, polished or molded to provide a sample interface with a cross-section that has optimal coupling between the energy source and spectral detection system.

4. The device of claim 1, further comprising an integrated spectral measurement system either based on the use of a continuous linear variable filter (LVF) or discrete optical components that comprise a matrix or mosaic-based filter-detector measurement system.
5. The device of claim 4, wherein when a plurality of faceted surface pairs exists, the angles of the facets for the individual pairs may be different to facilitate angle tuning of the optical structure.
6. The device of claim 3, further comprising a cylindrical based package, wherein the cylindrical base facilitates sealing and a compatible interface to the NeSSI Series II manifold-based sampling system.
7. The device of claim 1, wherein said optical structure of the sample interface is optimized to minimize the volume of sample required and/or reagents required for a specific measurement.
8. The device of claim 7, wherein in the optical transmission/absorption mode where energy passes into the sample, energy traverses the sample into a retro-reflective optical structure and re-enters the sample interface and is imaged on to the spectral sensing device.
9. The device of claim 7, wherein, in an optional optical scattering mode, where energy passes into the sample, interacts with the sample, and scattering radiation that results from this interaction is observed by a pick-off mirror system in the optical structure, said energy propagating at  $90^\circ$  with respect to normal passage of energy through the sample.
10. The device of claim 1, wherein said source and detector are located at matched positions to provide an optimum passage of energy through said optical structure utilizing at least one of internal reflection, light transmission and light scattering.
11. The device of claim 9, wherein in the case of scattering or fluorescence, the detector is mounted in secondary positions, thereby ensuring optimum energy collection.

12. The device of claim 9 wherein, the integration of the source and detector constitutes the formation of an integrated internal sensing module to complement the optical structure.
13. The device of claim 1, having an integrated internal sensing module with an energy source, wherein said source comprises a solid-state device primarily designed to emit a near continuum of light or energy with an optimized spectral range of 400 nm to 700 nm.
14. The device of claim 1, having an integrated internal sensing module with an energy source, wherein said source comprises a broadband tungsten source, primarily designed to emit a near continuum of light or energy with an optimized spectral range of 500 nm to 1100 nm.
15. The device of claim 1, wherein said optical structure, and said package are housed within an enclosure that protects internal components from the operating environment and the sample.
16. The device of claim 15, wherein said enclosure is fabricated from machined metal parts.
17. The device of claim 16, wherein said device is renewable and disposable.
18. The device of claim 16, wherein said enclosure is constructed of a thermally insulating material to reduce the internal heat within the sensor, or said enclosure is structured externally to include cooling fins or structures to dissipate heat from the device.
19. An integrated sensing module having a detector comprising a solid-state device either matched directly in spectral response to a source, or capable of responding to wavelengths over a broad spectral range from one or more sources, sensitive in spectral regions of UV-visible wavelengths (350 nm to 700 nm) near infrared (600 nm to 2500 nm) and mid-infrared (2.5  $\mu\text{m}$  to 25  $\mu\text{m}$ , 2500 nm to 25000 nm).
20. The sensing module of claim 19, wherein said detector is located in either a primary location for the detection of source radiation from transreflectance or transmission absorption measurements, or a secondary location at the center or close to the center of the module, for

the detection of radiation from fluorescence or from energy scattered from a fluid under study, or both locations.

21. The sensing module of claim 19, further comprising one or more on-board processors, wherein reference spectral data recorded in the absence of analyte material can be stored in flash RAM, and wherein said reference spectral data can be used to provide a ratio of energy detected by a sample measurement and energy obtained from the reference data, thereby providing a direct measure of the energy absorbed by the sample and system optics.

22. The sensing module of claim 21, wherein the spectral data recorded can represent the optical interaction of multiple analytes thereby enabling more than one analysis to be performed, or where a more complex analysis can be performed in the event that a condition being monitored provides different independent responses at different wavelengths, said sensor module providing an output for either multiple components or the measurement of a complex condition by use of modeling techniques.

23. The sensing module of claim 19, further comprising a processor for processing the signal produced from the detector or detectors, and an on-board memory provision to enable calibration coefficient data to be stored for simple and complex calibrations, including simple linear response calibrations, second order and higher response calibrations, and multivariate models for complex calibrations and/or multicomponent analyses, and wherein said on-board memory supports storage of methods to link calibration data.

24. The sensing module of claim 19, further comprising a processor for processing a signal produced by the one or more detectors, and an on-board memory provision to enable multiple calibrations to be stored, and multiple methods for using said calibrations.

25. The sensing module of claim 23 further comprising built-in or downloaded calibration coefficients for said processor.

26. The sensing module of claim 21, wherein said processor further comprises one or more of:

a computation means for performing computations including concentration calculations;

processing of intelligent sensor outputs;

support for standard data formats, standard protocols and standard forms of communications, including serial and bus-oriented communications formats; and

support of hardware-based and wireless-based communications protocols.

27. The sensing module of claim 22, wherein the sensing module is integrated into a modular manifold-based sampling system, and where the mechanical interface is optimized to mate with hardware that is defined by industry standards.

28. The sensing module of claim 27, wherein the sensing module includes sampling modules that can be used to dispense and mix reagents with the sample stream. The resultant reacted solution is fed into a sample interface that is optimized in internal dimensions to minimize the volume of chemical reagents required for the measurement.

29. The sensing module of claim 25, wherein output therefrom is received by a process control system, wherein said process control system includes one or more built-in predictive-adaptive control algorithms to monitor the process and to provide instructions for the optimization of the control of the process being monitored.

# Elements of an Example Spectral Sensing Engine: Source, Sample Interface, Light Analyzer and Detector

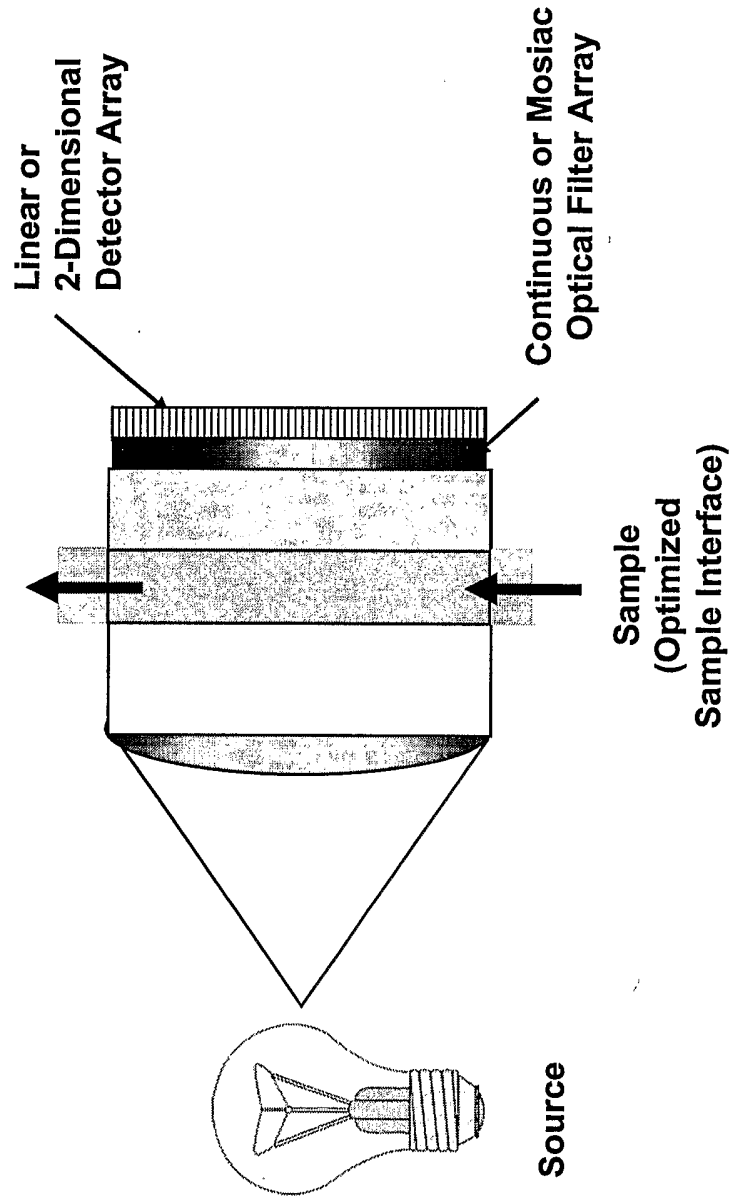
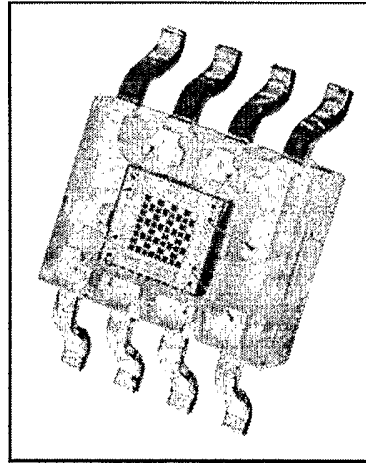


Figure 1 of 12

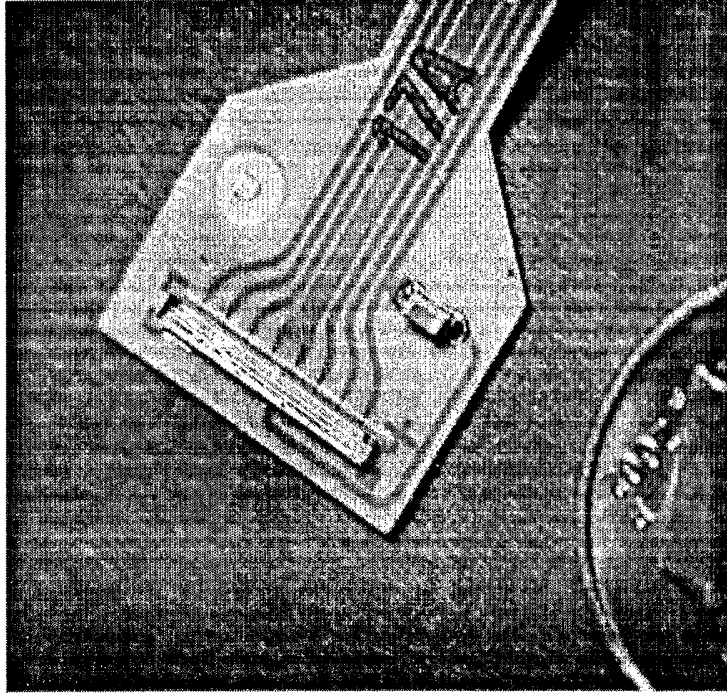
# Example Combinations of Optical Filters and Detector Arrays



**Figure 2A: Direct Coupling of Optical Filters and the Detector Array (LVF Version Illustrated)**



**Figure 2B: Example Integrated Detector Array Component With Filter Matrix**



**Figure 2C: Example Integrated Detector Array Component Mounted on Flex Connector**

Figure 2 of 12

# Electronic Components for the Integrated Spectral Sensor

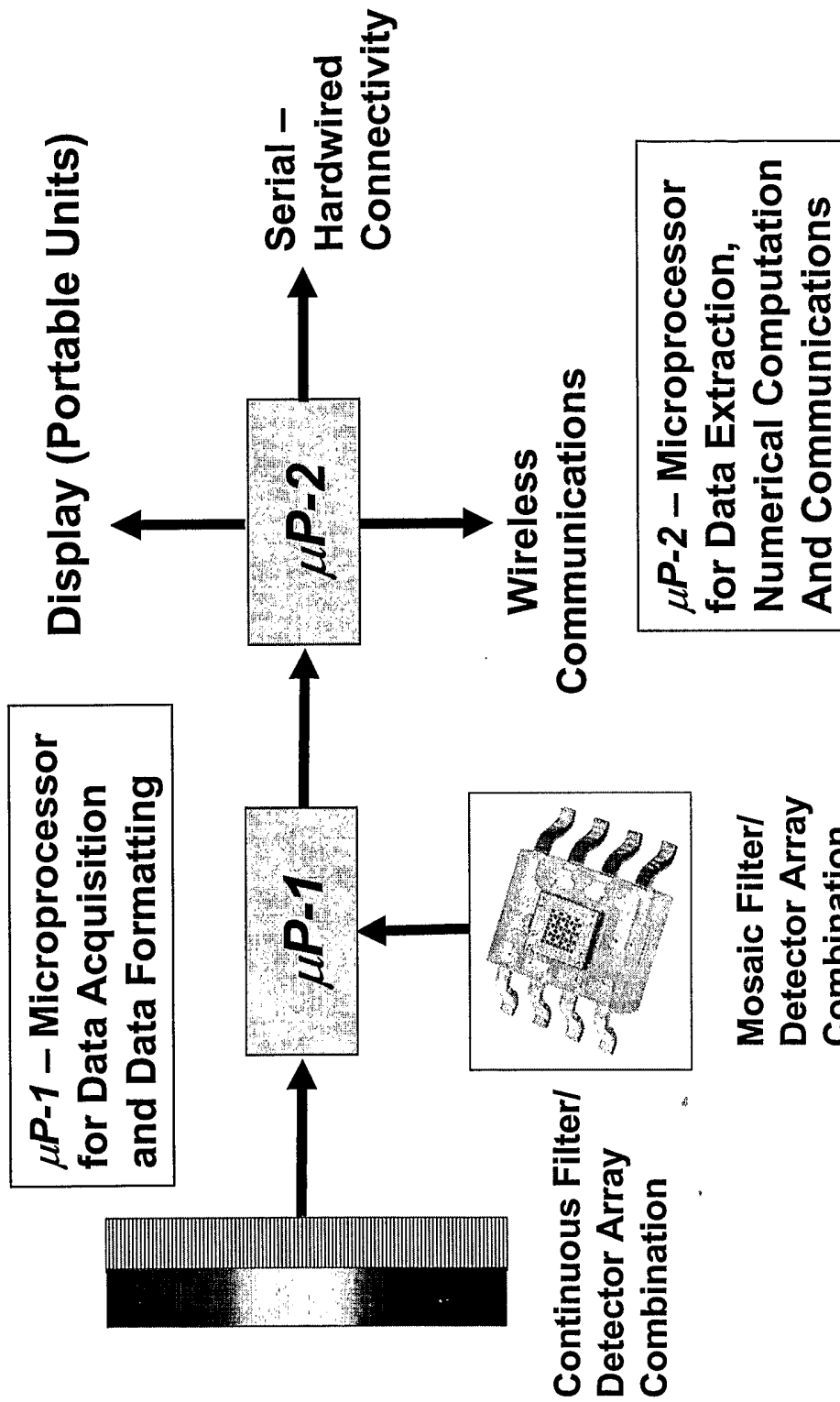


Figure 3 of 12

# Standard Package Configurations for On-Line Sensors: Absorbance/Transmission and Fluorescence Cells

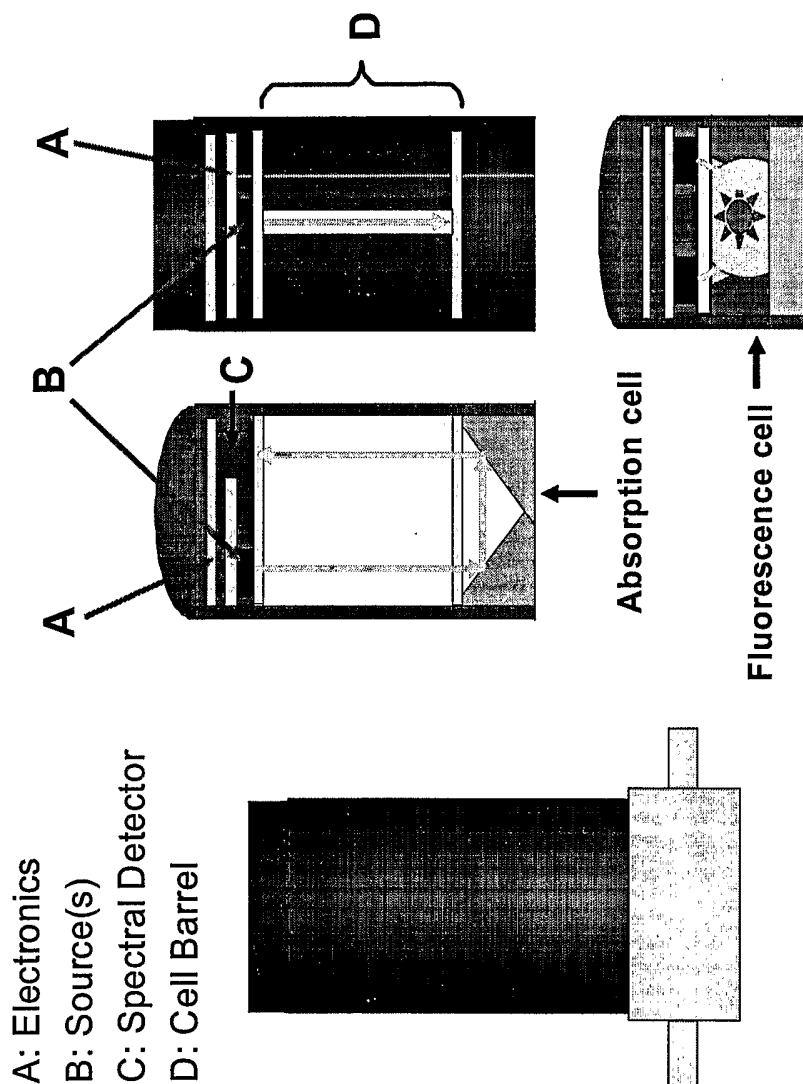


Figure 4 of 12

# Example Alternative Packaging Configurations of On-Line Sensors: Turbidity, NIR and Immobilized Reagent Versions

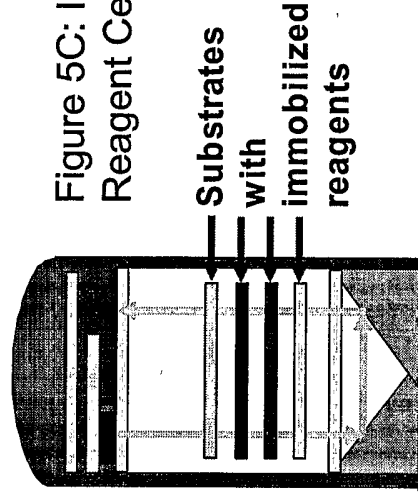
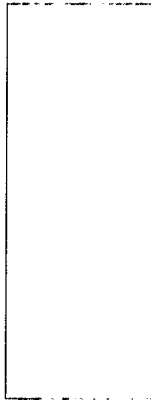


Figure 5C: Immobilized Reagent Cell, Example 1

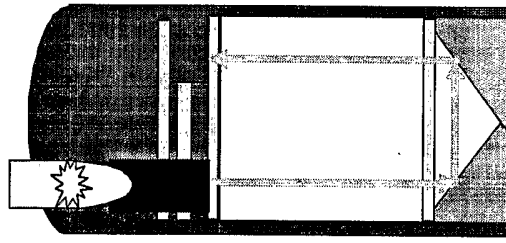


Figure 5B: NIR Absorption Cell With Remote Tungsten Lamp Source

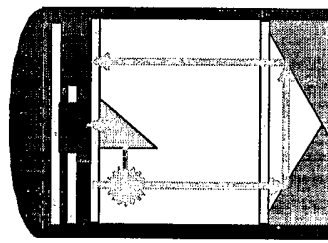


Figure 5A  
Turbidity Cell

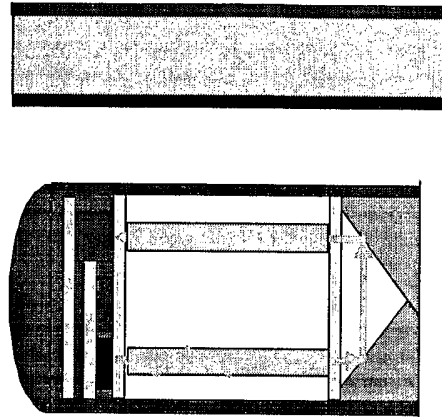


Figure 5D:  
Immobilized Reagent Cell,  
Example 2

Immobilized agents on light guides

# Integration of Spectral Sensor and Reagent Dispensing into Manifold System

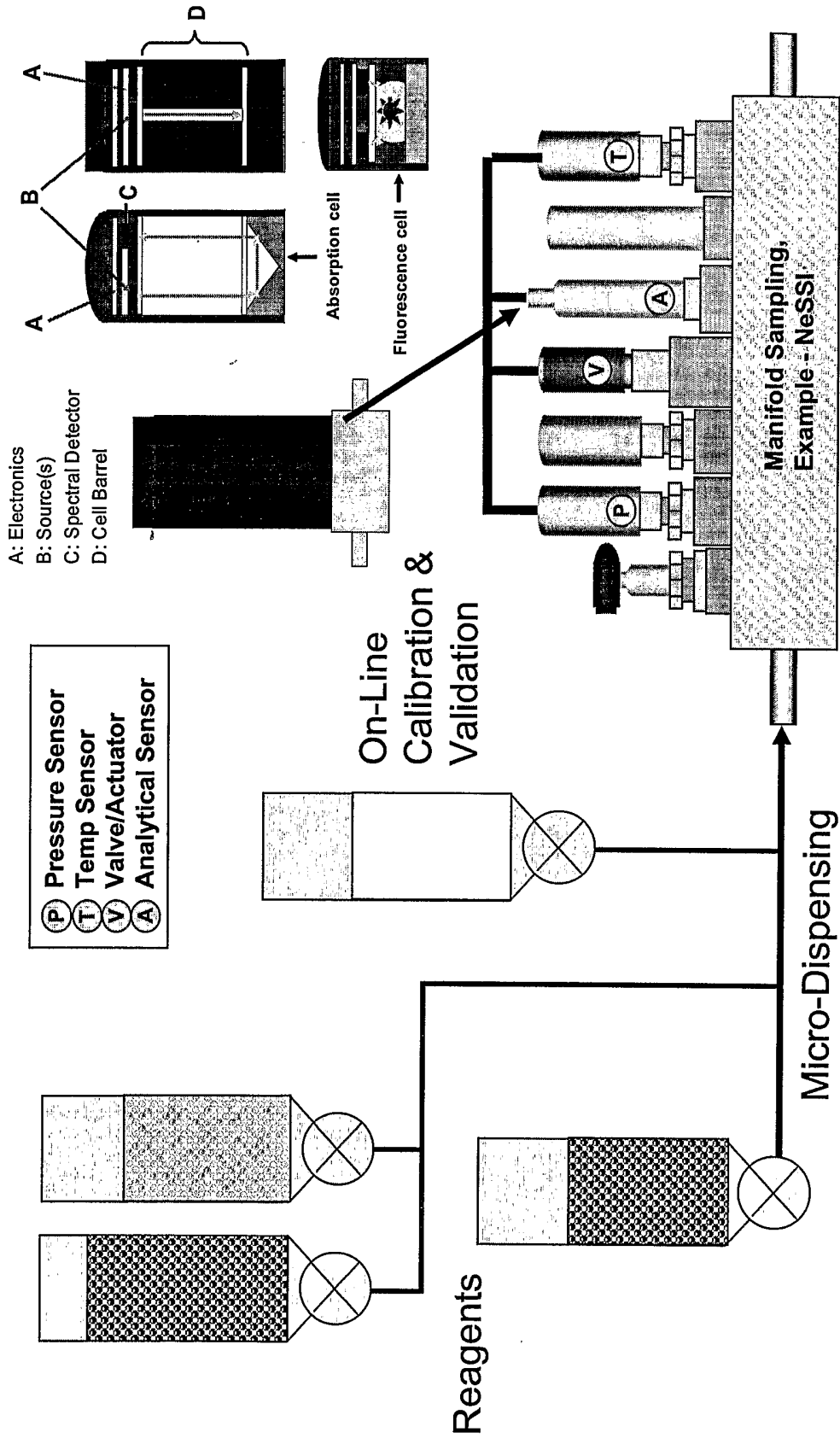


Figure 6 of 12

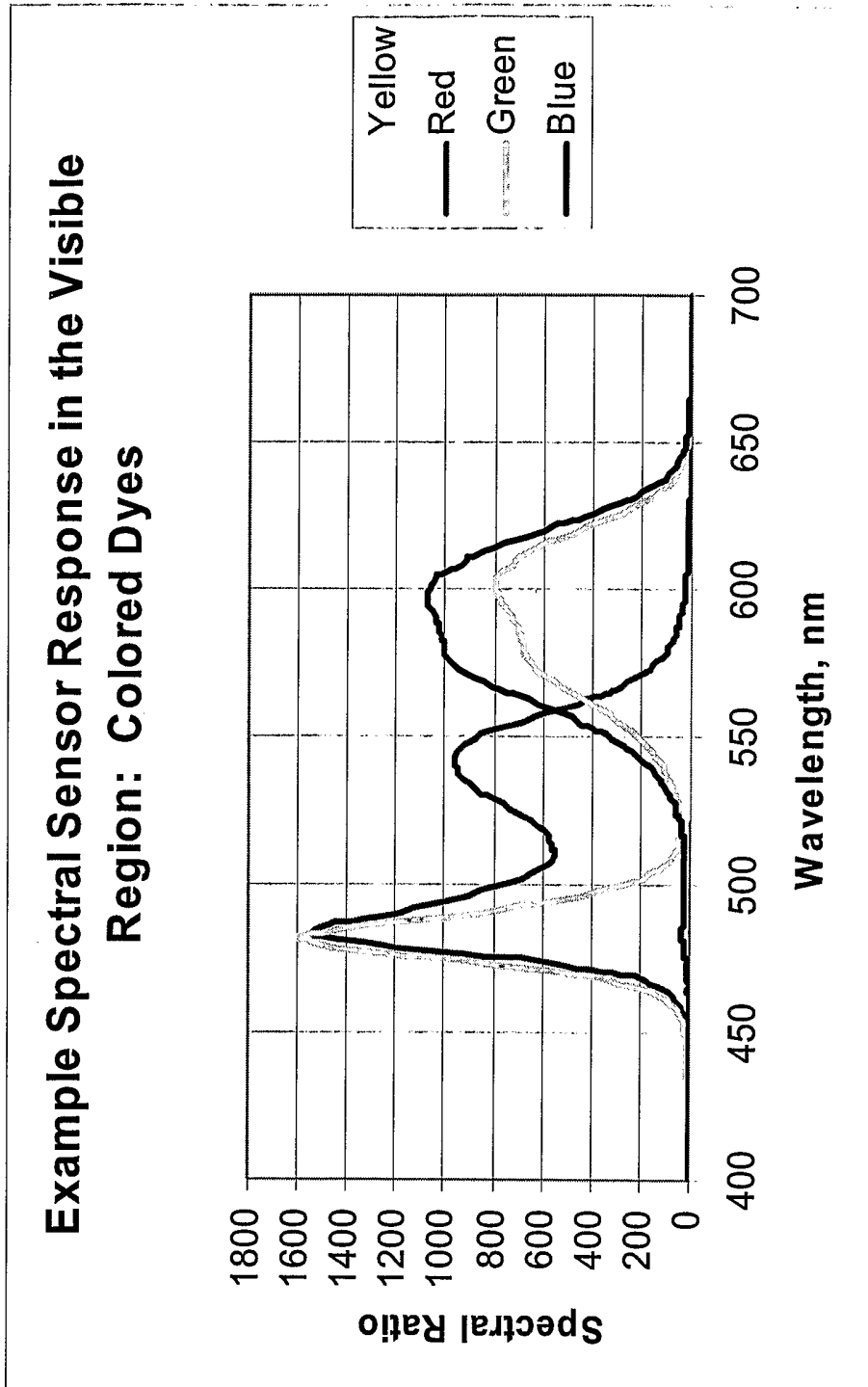


Figure 7 of 12

**Example Spectral Sensor Response in the  
Near Infrared Region: Common Chemicals**

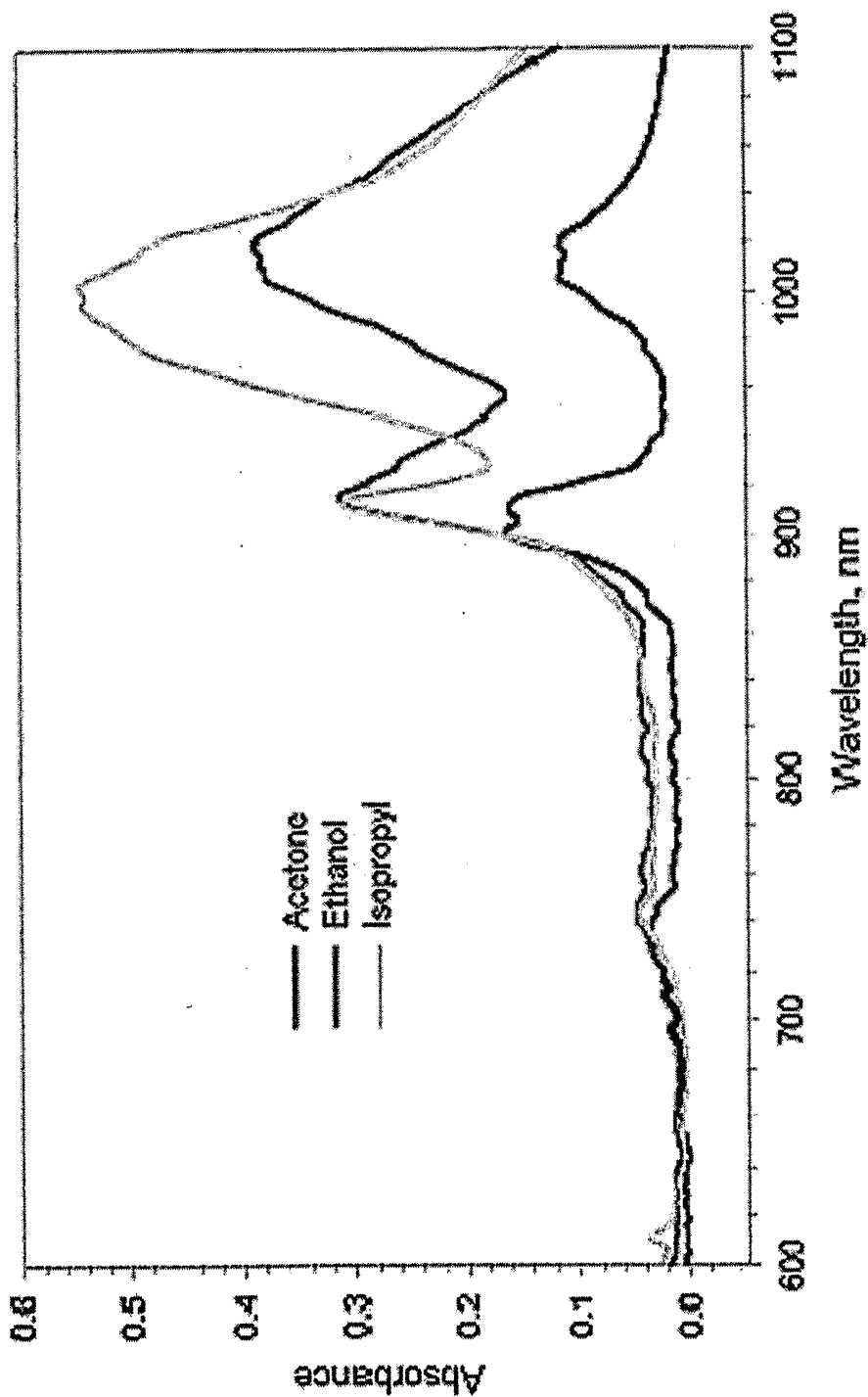


Figure 8 of 12

# The Function of the Optional Predictive-Adaptive Controller

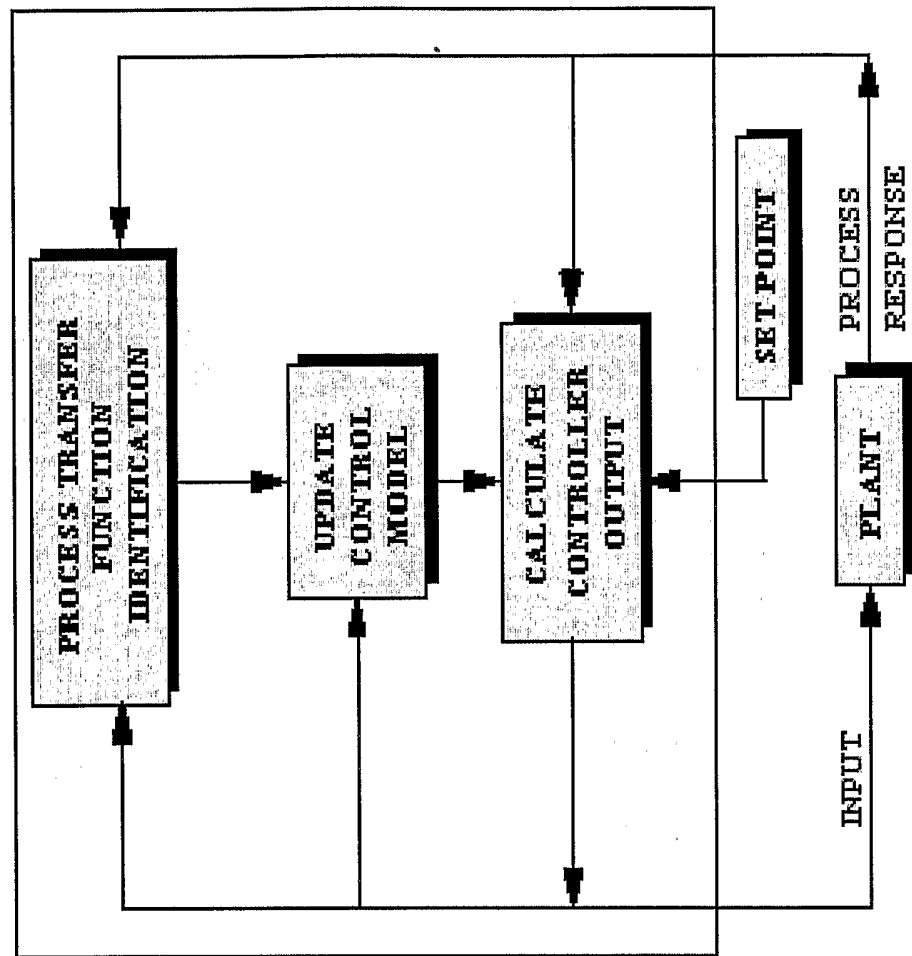


Figure 9 of 12

# Schematic of Optional Predictive Control System

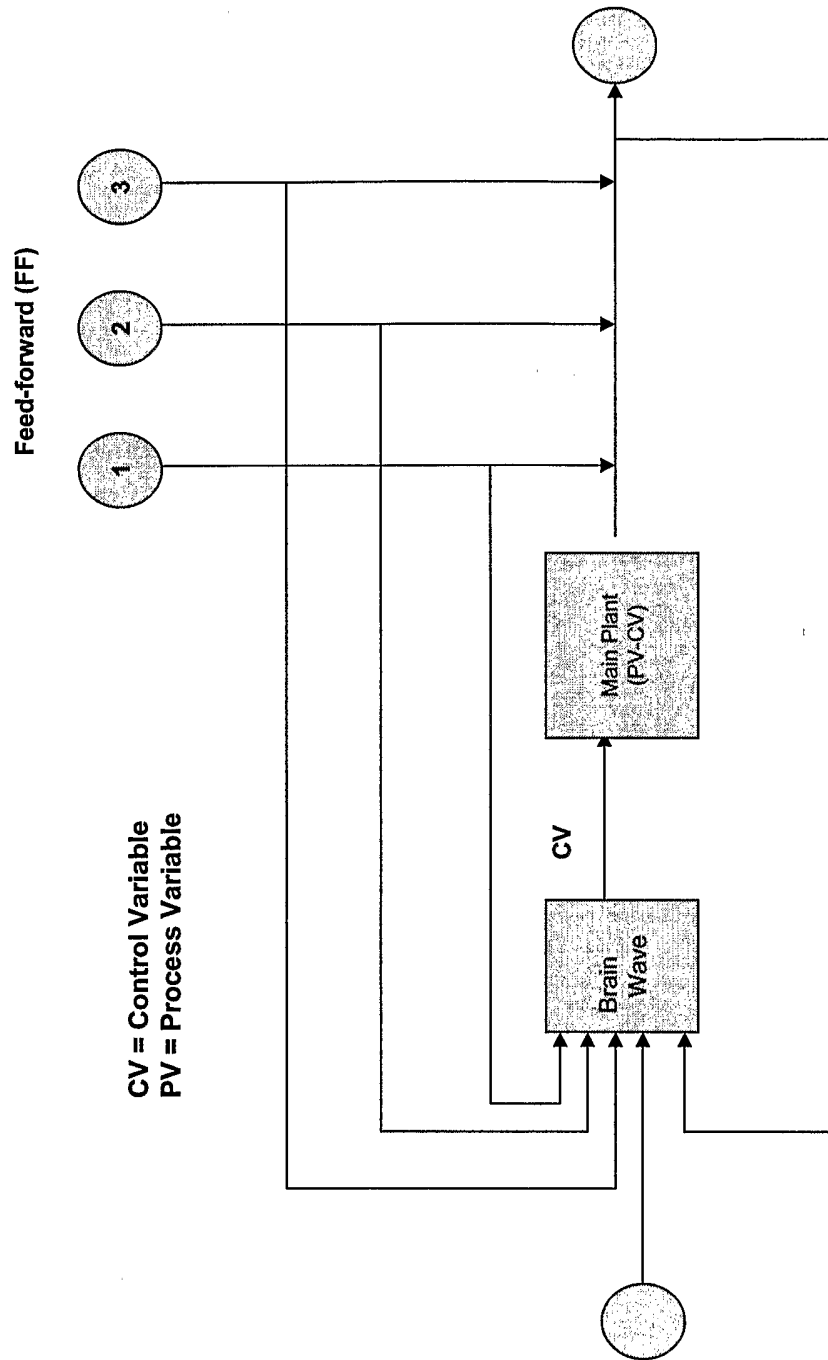


Figure 10 of 12

# Example Schematic of Predictive Control System in a Multi-Loop Implementation

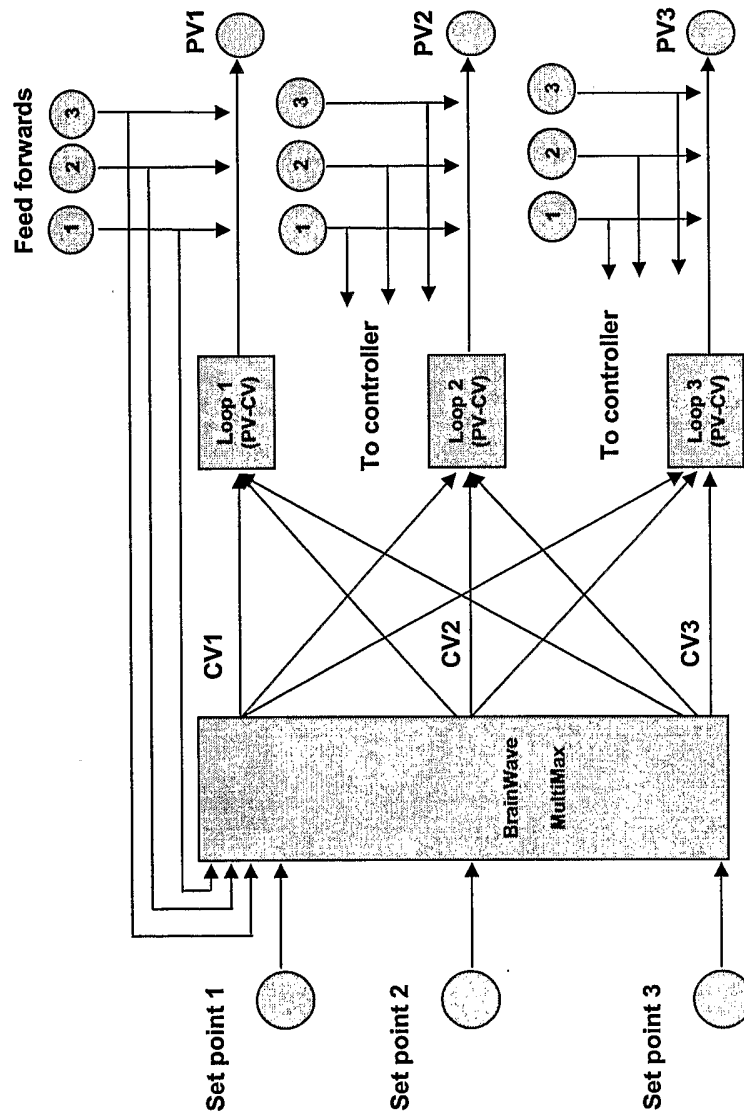


Figure 11 of 12

# Example Mesh-Network for Wireless Communications with Multiple Sensors

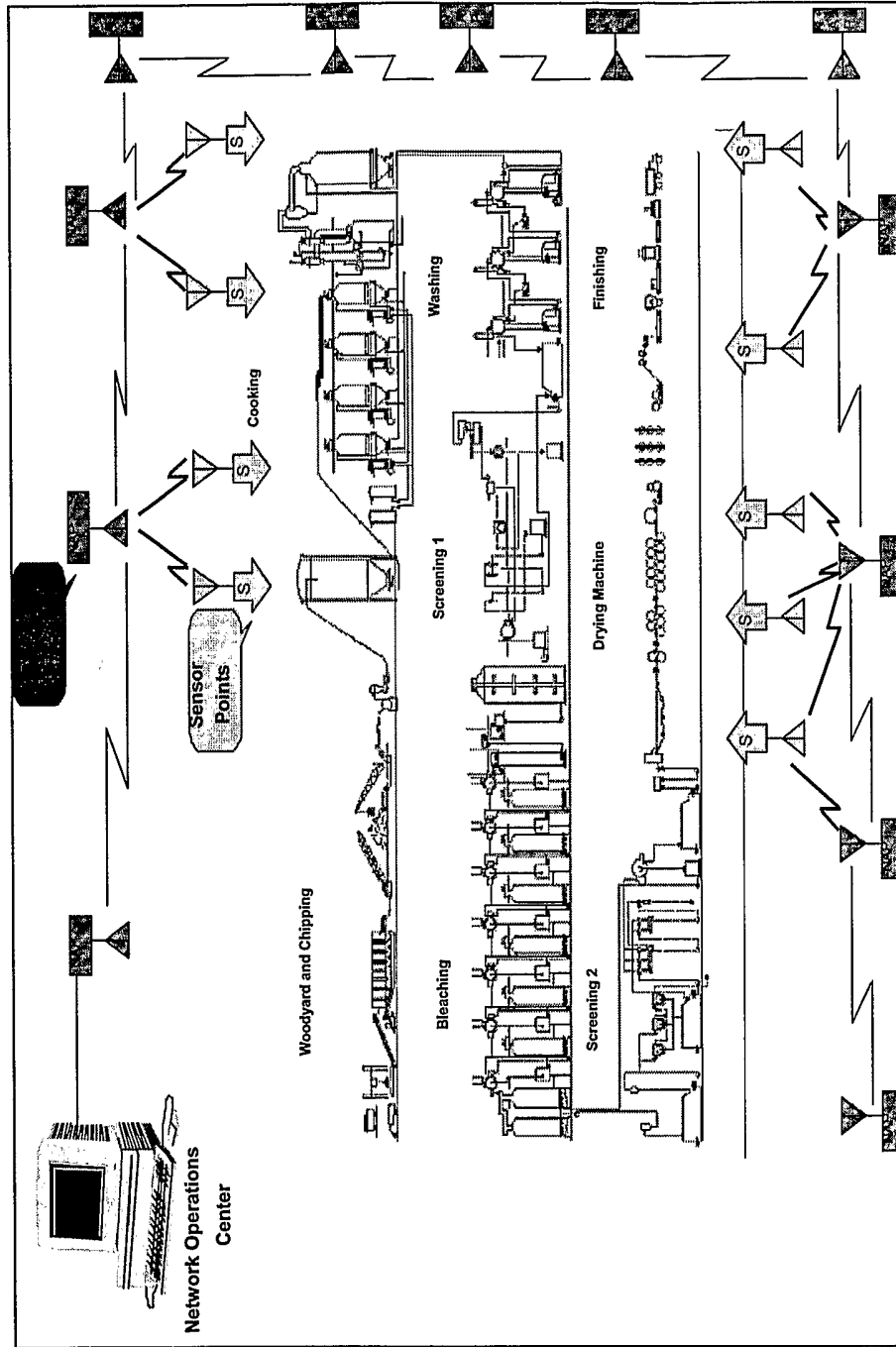


Figure 12 of 12