OPERATING INSTRUCTIONS FOR

MODEL 6650B

UV-Photo-X Fluorescence Analyzer

P/N M6650
04/11/2014

DANGER
Toxic gases and or flammable liquids may be present in this monitoring system. Personal protective equipment may be required when servicing this instrument. Hazardous voltages exist on certain components internally which may persist for a time even after the power is turned off and disconnected. Only authorized personnel should conduct maintenance and/or servicing. Before conducting any maintenance or servicing, consult with authorized supervisor/manager.

Warranty

This equipment is sold subject to the mutual agreement that it is warranted by us free from defects of material and of construction, and that our liability shall be limited to replacing or repairing at our factory (without charge, except for transportation), or at customer plant at our option, any material or construction in which defects become apparent within one year from the date of shipment, except in cases where quotations or acknowledgements provide for a shorter period. Components manufactured by others bear the warranty of their manufacturer. This warranty does not cover defects caused by wear, accident, misuse, neglect or repairs other than those performed by Teledyne or an authorized service center. We assume no liability for direct or indirect damages of any kind and the purchaser by the acceptance of the equipment will assume all liability for any damage which may result from its use or misuse.

We reserve the right to employ any suitable material in the manufacture of our apparatus, and to make any alterations in the dimensions, shape or weight of any parts, in so far as such alterations do not adversely affect our warranty.

Important Notice

This instrument provides measurement readings to its user, and serves as a tool by which valuable data can be gathered. The information provided by the instrument may assist the user in eliminating potential hazards caused by his process; however, it is essential that all personnel involved in the use of the instrument or its interface, with the process being measured, be properly trained in the process itself, as well as all instrumentation related to it.

The safety of personnel is ultimately the responsibility of those who control process conditions. While this instrument may be able to provide early warning of imminent danger, it has no control over process conditions, and it can be misused. In particular, any alarm or control systems installed must be tested and understood, both as to how they operate and as to how they can be defeated. Any safeguards required such as locks, labels, or redundancy, must be provided by the user or specifically requested of Teledyne at the time the order is placed.

Therefore, the purchaser must be aware of the hazardous process conditions. The purchaser is responsible for the training of personnel, for providing hazard warning methods and instrumentation per the appropriate standards, and for ensuring that hazard warning devices and instrumentation are maintained and operated properly.

Teledyne Analytical Instruments, the manufacturer of this instrument, cannot accept responsibility for conditions beyond its knowledge and control. No statement expressed or implied by this document or any information disseminated by the manufacturer or its agents, is to be construed as a warranty of adequate safety control under the user's process conditions.

Safety Messages

Your safety and the safety of others is very important. We have provided many important safety messages in this manual. Please read these messages carefully.

A safety message alerts you to potential hazards that could hurt you or others. Each safety message is associated with a safety alert symbol. These symbols are found in the manual and inside the instrument. The definition of these symbols is described below:

GENERAL WARNING/CAUTION: Refer to the instructions for details on the specific danger. These cautions warn of specific procedures which if not followed could cause bodily Injury and/or damage the instrument.

CAUTION: HOT SURFACE WARNING: This warning is specific to heated components within the instrument. Failure to heed the warning could result in serious burns to skin and underlying tissue.

WARNING: ELECTRICAL SHOCK HAZARD: Dangerous voltages appear within this instrument. This warning is specific to an electrical hazard existing at or nearby the component or procedure under discussion. Failure to heed this warning could result in injury and/or death from electrocution.

Technician Symbol: All operations marked with this symbol are to be performed by qualified maintenance personnel only.

NOTE: Additional information and comments regarding a specific component or procedure are highlighted in the form of a note.

CAUTION: THE ANALYZER SHOULD ONLY BE USED FOR THE PURPOSE AND IN THE MANNER DESCRIBED IN THIS MANUAL.

IF YOU USE THE ANALYZER IN A MANNER OTHER THAN THAT FOR WHICH IT WAS INTENDED, UNPREDICTABLE BEHAVIOR COULD RESULT POSSIBLY ACCOMPANIED WITH HAZARDOUS CONSEQUENCES.

This manual provides information designed to guide you through the installation, calibration and operation of your new analyzer. Please read this manual and keep it available.

Occasionally, some instruments are customized for a particular application or features and/or options added per customer requests. Please check the front of this manual for any additional information in the form of an Addendum which discusses specific information, procedures, cautions and warnings that may be peculiar to your instrument.

Manuals do get lost. Additional manuals can be obtained from Teledyne at the address given in the Appendix. Some of our manuals are available in electronic form via the internet. Please visit our website at: www.teledyne-ai.com.

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DANGER
COMBUSTIBLE GAS USAGE WARNING

This is a general purpose instrument designed for use in a nonhazardous area. It is the customer's responsibility to ensure safety especially when combustible gases are being analyzed since the potential of gas leaks always exist.

The customer should ensure that the principles of operating of this equipment are well understood by the user. Misuse of this product in any manner, tampering with its components, or unauthorized substitution of any component may adversely affect the safety of this instrument.

Since the use of this instrument is beyond the control of Teledyne, no responsibility by Teledyne, its affiliates, and agents for damage or injury from misuse or neglect of this equipment is implied or assumed.

Introduction

1.1 Theory of Operation

The ability to monitor the concentration of an analyte in a process stream is critical for accurate and reliable process control. There are many techniques used to determine the analyte concentration of interest. One of the most sensitive sensing techniques is molecular fluorescence. Fluorescence occurs when a molecule absorbs light energy, either ultraviolet or visible, and rapidly emits light, at some longer wavelength. Fluorescence of this type is referred to as Stokes fluorescence. Fluorimetry characterizes the excitation and emission properties of the molecular species. Figure 1-1 shows an example of the excitation and emission spectrum from a hypothetical fluorophore.

Fluorimetry is concerned with two types of information: 1) The (spectral) wavelength distribution, which is characteristic of the electronic properties of the molecule, and 2) The intensity of the fluorescence, which is typically correlated to the concentration of the fluorescent molecule in the solution.

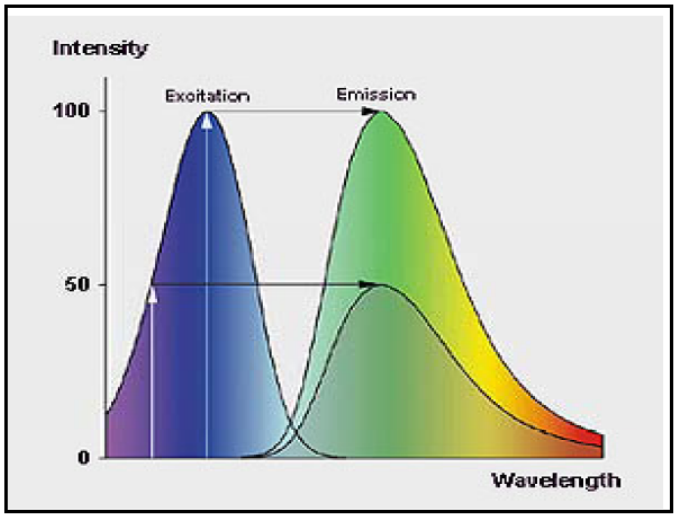

Figure 1-1: Molecular Fluorescence Example

1.2 Filter Based Flurometer Description

The Fluorometer is a filter-based analyzer. The Fluorometer measures the ability of the analyte of interest to absorb light in a narrow spectral region and emit light at a longer wavelength. A filter-based Fluorometer is a good choice when quantitative measurements are desired for a specific analyte in process. Additionally, the Fluorometer provides a relative measurement and can be calibrated with a known concentration standard(s) or correlated to measurements using standard laboratory methods resulting in a quantitative fluorescence.

A filter-based Fluorometer uses optical filters to provide specific excitation or emission wavelengths for molecular fluorescence. In the Fluorometer the filters are located internal to the transmitter and are specific to the application. Therefore, the Fluorometer is a dedicated instrument for monitoring only one specific analyte of interest. The filter sets used to configure the instrument are specific to the analyte of interest. In order to monitor a new analyte of interest, the user must return the Fluorometer to TAI for factory reconfiguration. The Fluorometer can be used for both quantitative measurements (determination of analyte concentration) and control measurements (switching of valves once a fluorescence level is attained, for example).

In brief, the Fluorometer works as follows: The light source launches excitation light into a fiber optic cable. The fiber optic cable transfers the excitation light to the Fluorescence probe mounted in the process. The fluorescence probe launches light into the process sample, and collects the molecular fluorescence (emission) from the sample. The emission light is then transferred from the probe through fiber optic cable to the analyzer. The emission light passes through an emission filter in order to remove any residual excitation energy collected by the fluorescence probe. The emission light then impinges on a detector and the fluorescence intensity is displayed on the analyzer.

Unlike many fluorescence units on the market, the Fluorometer utilizes a xenon flash lamp to provide excitation energy. This lamp allows the Fluorometer to be easily configured to meet any excitation wavelength requirement with the appropriate filter selection across the entire spectrum. The xenon flash lamp also has an extended lifetime compared to other common UV sources (deuterium, mercury vapor, etc.), which reduces the cost of ownership of the analyzer.

The remainder of this manual provides the user with the necessary information to operate the Fluorometer. In addition to standard

operation methods and procedures, a section detailing some application development objectives (Appendix 1) has been provided to aid the user in defining the parameters required to realize optimal process monitoring.

Definition of Terms

2.1 Excitation Filter

The excitation filter is used to select the range of wavelengths, the pass band, used to cause or excite the molecular fluorescence. Wavelengths not in the pass band are rejected and ideally never reach the sample. Removal of wavelengths outside the pass band minimizes the possibly of false fluorescence readings due to detection of light from the lamp which mimics the fluorescence signal.

2.2 Emission Filter

The emission filter is used to select the range of wavelengths, the pass band, to be passed to the measure detector. Wavelengths not in the pass band are rejected and ideally never reach the sample. It is critical that the excitation wavelengths never reach the detector, since it will respond to excitation light. Excitation light impinging the measure detector results in increased residual background levels, which reduces the dynamic range, the signal-to-noise ratio and the signal-to-blank ratio.

2.3 Measure Detector

The light detector is most often a photomultiplier tube, though photodiodes are increasingly being used. The light passing through the emission filter is detected by the photomultiplier or photodiode. The light intensity, which is proportional to the analyte concentration, is registered as a digital readout.

2.4 Span Filter

The span filter is used to check instrument operation. When a fluorescence filter is employed the span filter fluorescence value is dependent on the initialization method utilized and the standards used during calibration. The span filter is used to verify instrument operation and should not be used to calibrate the instrument.

2.5 Background Fluorescence

This refers to the fluorescence signal due to the probe/analyzer optical configuration, stray light, and fluorescence from the background material.

2.6 Sensitivity

The sensitivity of the instrument refers to the ability of the analyzer to detect a given level of analyte based on the molecular fluorescence from the analyte. The actual limits of detection depend on the properties of the analyte measured and the process conditions. Parameters such as pH, temperature, oxygen content, and background solvent, to name but a few may dramatically alter the fluorescence intensity measured. Typically, detection of parts-per-million (ppm) and parts-per-billion (ppb) analyte levels can be detected. In general, fluorescent measurements are 1,000 to 500,000 times more sensitive than absorbance based photometric measurements. Practically, sensitivity means the minimum analyte concentration that can be measured above background fluorescence in the process.

2.7 Specificity

The specificity is the ability of the analyzer to monitor one specific analyte in a mixture of background materials without interference from the background materials. In absorbance based photometric measurements, interference problems are common since many materials absorb light, making it difficult to isolate the targeted analyte in a complex mixture. However, Fluorometers are highly specific and less susceptible to interferences because fewer materials exhibit molecular fluorescence. Furthermore, if background materials do absorb and emit light, it is rare that they will emit the same wavelength of light as the analyte of interest.

2.8 Signal-to-Noise Ratio

Signal refers to the emission collected by the fluorescence probe and monitored by the analyzer using the internal span filter. Noise refers to the output from the instrument's electronics, which is present whether or not sample is being read plus any collection of errant wavelengths not removed by the optical filters. Noise is measured by

placing the fluorescence probe in air and in complete darkness (no stray light). For process monitoring, the signal-to-noise ratio is not as important as the signal-to background ratio.

2.9 Signal-to-Background Ratio

Signal refers to the emission collected from a sample with known analyte concentration by the fluorescence probe and monitored by the analyzer. Background refers to the process liquid containing no analyte of interest and any stray light present in the system. The signal-to-background ratio should be calculated during the application engineering phase of the project. Knowing this ratio will help determine when the stray light level changes and/or the background material fluorescence properties change. *Refer to Appendix 1 for additional details.*

2.10 Dynamic Range

Dynamic range refers to the range of concentrations an instrument can read, from the minimum to the maximum detectable. The minimum detectable concentration is determined by signal-to-noise and signal-to-background ratios. The maximum detectable concentration is determined by the compound's chemistry and by factors such as instrument sensitivity ranges, fluorescence (quantum) efficiency, specificity of optical filters, etc.

2.11 Linearity

Fluorescence intensity is typically directly proportional (linear) to concentration. There are, however, factors that affect this linear relationship. For example, variations in temperature, pH, dissolved oxygen content, stray light, turbidity, variation in the chemical composition of the background, etc. can dramatically affect the linearity of the fluorescence response. Practically, the linearity of the measurement is determined during the application engineering phase of the project. *Refer to Appendix 1 for details.*

2.12 Process Background

The liquid solution used to transport or sustain the analyte of interest in the process is referred to as the process background. This

solution has all the chemical constituents found in the process except the analyte of interest.

2.13 Mode 1 Initialization

Mode 1 initialization is a method for setting up the Fluorometer for process monitoring. **Mode 1 initialization is used to setup the Fluorometer when the fluorescence intensity versus analyte concentration and the background fluorescence of the process have been characterized.** Mode 1 initialization must be performed while the fluorescence probe is immersed in the calibration (reference) sample.

In Mode 1, fluorescence calibration is based on a process sample with a known analyte concentration. The background material for this sample must be the process background material. The fluorescence from the calibration (reference) sample is then used to set the midpoint of the fluorescence scale. During Mode 1 initialization, the zero fluorescence level is set electronically to 0 counts (display value), the midpoint range is set to 1000 counts (display reading after initialization), and the upper limit of the range is set to twice the midpoint value (1999 display value). Section 5 provides details the procedure for Mode 1 initialization.

Note: If the Fluorescence of the process has not been characterized, Mode 2 initialization must be performed first followed by an application engineering study, refer to Appendix 1 for guidance.

2.14 Mode 2 Initialization

Mode 2 initialization is a method for setting up the Fluorometer for process monitoring and/or application engineering studies. Mode 2 initialization is used to setup the Fluorometer when the fluorescence intensity versus analyte concentration and the background fluorescence of the process are **unknown (but the excitation/emission matrix has been fully characterized by our application engineering department)**. Characterization of the fluorescence response as a function of analyte concentration can be performed onsite, refer to Appendix 1 for details, or contact TAI Customer Service at:

Teledyne Analytical Instruments
16830 Chestnut Street
City of Industry, California 91748-1020, USA

Tel: 626-961-9221 or 626-934-1500
Fax: 626-961-2538 or 626-934-1651
Toll free: 888-789-8168
Email: ask_tai@teledyne.com

In Mode 2, fluorescence calibration is set electronically. Initialization may be performed while the fluorescence probe is immersed in any process sample, although for simplicity TAI recommends using the process background material without any analyte of interest. This sample is commonly called the zero or blank sample. During Mode 2 initialization, the zero fluorescence level is set electronically to 0 counts (display value) and the upper limit of the range is set to the 1999 display value. Section 5 provides details the procedure for Mode 2 initialization.

Diagnostics & Controls

3.1 Front Panel Controls

The front panel controls are used to adjust the display, Zero, initialization of the transmitter, and the 4-20mA output. Figure 3-1, below, identifies the position of each of these controls.

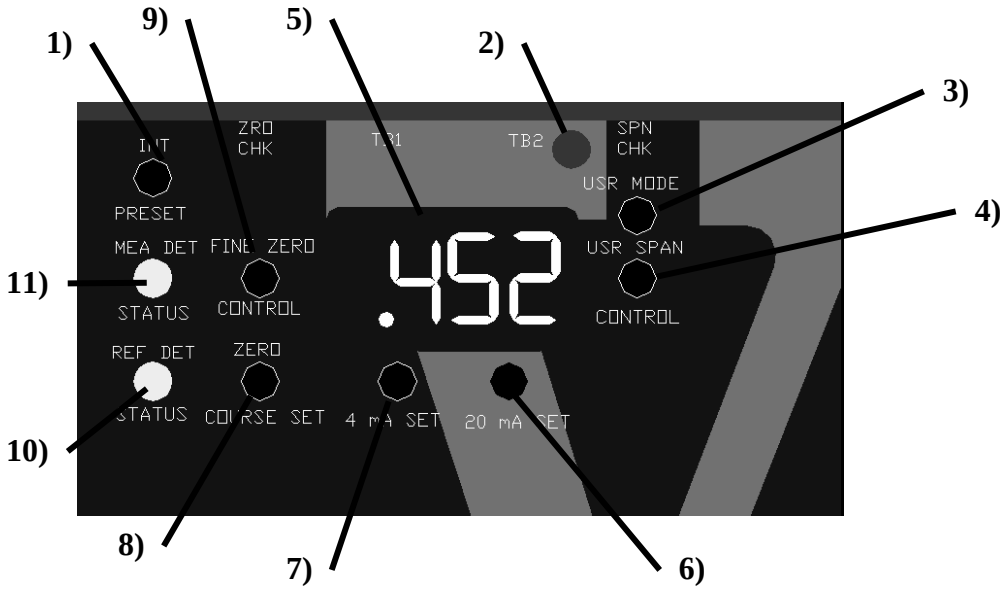

Figure 3-1: Photo-X Front Panel Controls

1) Initialization Preset Switch
2) Span check LED indicator
3) User mode indicator LED
4) User mode span adjustment
5) Digital display
6) 20mA adjustment
7) 4mA adjustment
8) Course zero adjustment
9) Fine zero adjustment
10) Reference detector switch
11) Measure detector status

Hardware Installation

In addition to the physical, optical, and electrical installation requirements of the Fluorometer it is recommended that the transmitter be placed in a clean, dry area of the facility. If the local area of where the electronics are mounted exceeds our upper temperature limit of 115°F (46°C), it is recommended that the transmitter be moved or purged with plant air to cool the enclosure.

Please follow the outlined sections below as a guideline to installing the Fluorometer in your plant environment.

4.1 Physical

1. Bolt the Fluorometer in place using mounting screws to a secure, rigid surface.
2. Run required electrical wiring for instrument power and 4-20 mA output signals.
3. If required, install dry and oil free purge air for the unit.
4. Run fiber optic cable in conduit or other appropriate protective measure as desired.
5. Install probe in sensing location, making sure there are no leaks and the probe has been fully tested for process conditions such as temperature and pressure.

Figure 4-1: Hardware

4.2 Optical

To remove or install the fiber optic cables from the probe, use the following procedure:

1. Unscrew the optic cables from the bottom of the light source and the control unit.
2. Unscrew the liquid tight connector's 1 ¾" nut which is connected to the conduit from the other part.
3. Remove the probe bracket from wherever it's attached to.
4. Unscrew the 1 $^{9}/_{16}$" nut from the probe. Do this by holding the probe by hand and using a wrench on the nut.
5. Once the nut has been removed from the probe, the two unions with the SMA fiber-optic connectors connected are exposed and the cable can be removed.
6. Remove the protective boots from the SMA 905 connectors on the fiber optic cable.
7. Clean the fiber ends using a lint free cotton swab dipped in either spectroscopic grade isopropyl alcohol (IPA, also referred to as 2-propanol) or methanol. Spectroscopic grade acetone can also be used, but may delaminate the cotton from the swab by dissolving the binding agent.
8. Connect the fiber optic cable
   One end of one cable to the probe,
   The distal end of the cable above to the source, and SIGNAL IN port.
   The second fiber gets connected to the outlet end of the probe and it's distal end gets connected to the SMA-905 fitting on the bottom of the transmitter labeled; "Measure Channel Input"
9. Finger tighten the SMA 905 connectors. *DO NOT use a wrench to tighten the nut*.

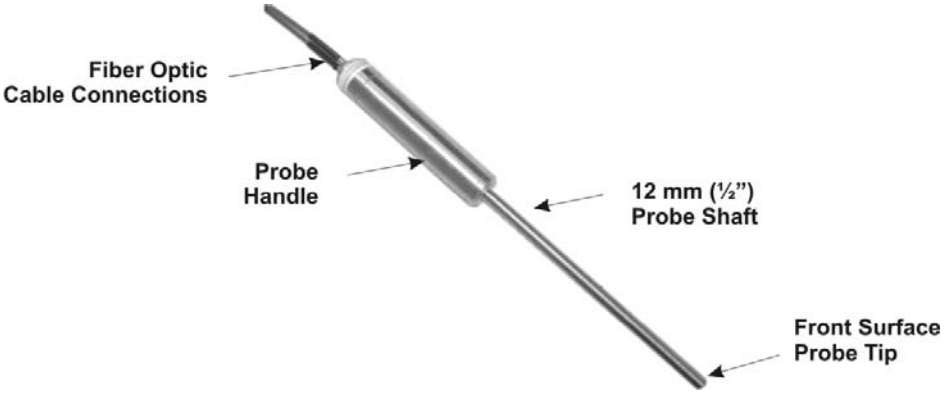

Figure 4-2: Fiber Optic Cable and Probe

4.3 Electrical

1. Connect the power and ground to the terminal strip mounted located below the Fluorometer.
2. Connect the 4-20 mA signal output line to the terminal strip located below the Fluorometer.
3. If required, connect the remote span filter insertion control line to the terminal strip located below the Fluorometer. The unit is supplied with a jumper on the terminal strip, which is the correct configuration for an instrument using span filter insertion.

Note: For remote operation of the span filter, the jumper can be replaced with a switch.

4. It is best if the unit is not initially powered up until after the initial optical setup is completed.

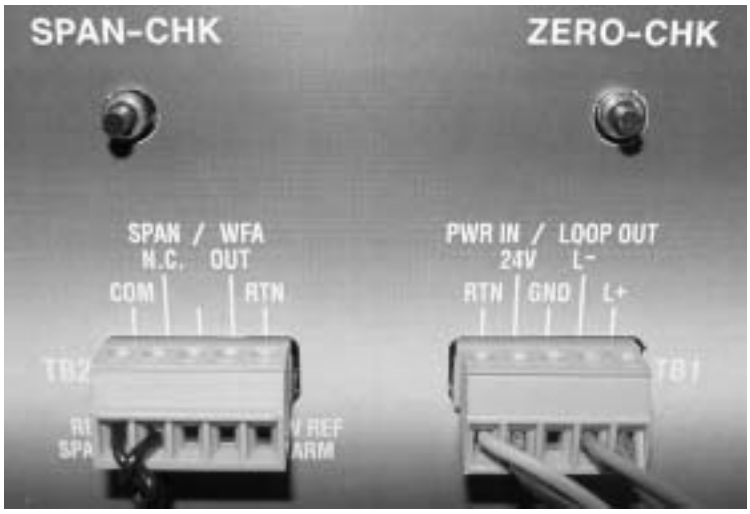

Figure 4-3: Top view of Flurometer

Setup & Operation

The Fluorescence transmitter is designed to monitor the fluorescence signal from a laboratory or process stream. Under certain conditions the fluorescence range of the sample is well defined and can be verified with standard reference materials such as zero and span solutions of know concentrations. However, this is not always the case when measuring fluorescence and is addressed in the design of the product via the mode selection.

There are two modes which can be employed for calibration/set up of the transmitter:

- Mode 1 — known range, known fluorescence
- Mode 2 — unknown range or fluorescence of sample

Typically when the user does not know what the expected range or the fluorescent value of the samples use mode 2 initialization. Mode 2 initialization requires the initialization preset switch (refer to Figure 3-1) be held for >8 seconds. In the mode 2 initialization, the ratio of the fluorescent value to the excitation value needs to be spanned properly in the display since this value is unknown the transmitter arbitrarily set to 20 times the original ratio. This is done with or without the probe in a sample. Once a real sample is available, the transmitter range may be determined. Once the transmitter range is known, a calibrated range, using mode 1 operation, may be set.

Mode 1 is used to set the full scale range of the unit but it does so by splitting the range at the midpoint of the full scale. That is to say that if you had a sample of concentration of X the full scale would be 2X. The calibration sample would read 1000 counts and have a 12mA output. If on the other hand you wanted a full scale range of X you would have to dilute your sample to X/2 to get a full scale of X.

The display output is in arbitrary count units. To convert the arbitrary unit to relevant engineering units the 4-20mA output must be manipulated using an external device. The transmitter display cannot be set to relevant engineering units. Additionally, the user can adjust the output display over the full scale range once it has been established.

Note: The above readings are all based on defined excitation and emission wavelengths for the given sample or intended application.

5.1 First Power-Up Following Installation

1. The Fluorometer comes preconfigured from the factory using the supplied fiber optic cable and fluorescence probe. Preconfiguration at TAI involves application specific filter sets and optimization of detection capabilities. Preconfiguration should insure turnkey operation upon arrival.
2. Make sure that the probe is located in complete darkness. Stray light in the measurement area will result in an increased baseline response level and will limit the effective monitoring capabilities of the instrument.
3. Immerse the fluorescence probe in a process sample. **TAI recommends that the setup sample be the process background.**
4. Make sure that all fiber optic cables are properly connected
5. Make sure that all electrical connections to the transmitter have been made according to the wiring diagram.
6. Power up the unit.
7. Allow the lamp to flash for a minimum of 1 hour before completing the rest of the initialization sequence.
8. After lamp warm-up and stabilization, the REF DET LED should have a steady green output. The MSR DET LED may have a flashing yellow, flashing green, or a solid green output. The indicator LEDs are located to the left of the display along the outer edge of the Fluorometer housing.
9. Perform Mode 2 initialization
    a. Press the INT PRESET, located in the upper left hand corned of the Fluorometer, for at least five (5) seconds. Please see Section 2 for a description of Mode 2 operation.
    b. Both the REF and MSR DET LEDs exhibit solid green or flashing green output.

c. The value on the display is the fluorescence level in the process. If the sample is the background material, this is your background fluorescence level.

d. Insert the span filter using the SPN CHK button located on the upper end of the Fluorometer housing. When the filter is in place, the FLTR-IN LED will change from green to red.

e. Wait 2 minutes for the output to stabilize and the unit will read an upscale value.

f. Both the REF and MSR DET LEDs should have solid green or flashing green output.

g. If the indicator LED color does change color, refer to the Diagnostics section below.

h. Depress the SPN CHK button to remove the filter from the beam path. The FLTR-IN indicator will change from red to green.

i. Wait 2 minutes for the output to stabilize and the unit should display the background fluorescence level.

j. Both the REF and MSR DET LEDs should have solid green or flashing green output.

k. The unit is now ready for either application engineering studies or process monitoring.

10. During initial setup, there is no need to adjust the 4 mA, 20 mA or display settings. These adjustments should be made only during the setup for application engineering or process monitoring.

Note: The First Power-Up Following Installation sequence should be completed anytime the unit has been powered down for an extended time period (> 7 days)

5.2 Application Engineering

Refer to Appendix 1 for suggested Application Engineering suggested procedures. Unless this has been supplied by TAI you will have to establish a relationship between your samples and the response from the instrument. You will need to develop a compound specific

calibration curve and you will have to determine your limit of detect ability, and dynamic range.

5.3 Initialization for Process Monitoring: Mode 2 Operation

Mode 2 process monitoring is used whenever the user wants to forego the application engineering sequence or when the application engineering results indicate that Mode 2 operation is sufficient to meet the monitoring requirements of the user.

Use the outline below for initialization of the unit in this mode:

5.3.1 Case 1: Process Monitoring Following Hardware Setup Without Application Engineer

Setup was completed during the *First Power-Up Following Installation* sequence (see Section 5.1) and no additional setup is required, although adjustments to the display, 4 mA and 20 mA levels may be desired. Instructions for altering these levels are given below. **TAI strongly suggests that no changes to the 4 mA, 20 mA and display levels occur until after the user has collected data on the process fluorescence.**

5.3.2 Case 2: Process Monitoring Without Application Engineering

This case is valid when the user desires to monitor the process without performing application engineering studies, or the process has changed slightly and the user wants to monitor the changes without performing a complete application engineering study, or to determine if full application engineering studies are warranted.

The steps outlined below assumes that the hardware was initialized properly (see above) and that the Fluorometer has been functioning properly since installation.

1. The fluorescence probe must be immersed in the background material.
2. Perform Mode 2 initialization

a. Press the INT PRESET, located in the upper left hand corned of the Fluorometer (refer to Figure 3-1), for at least five (5) seconds. Please see Section 2 for a description of Mode 2 operation.

b. Both the REF and MSR DET LEDs should have solid green or flashing green output.

c. The value on the display is fluorescence level in the process. If the sample is the background material, this is your background fluorescence level.

d. Insert the span filter using the SPN CHK button located on the upper end of the Fluorometer housing. When the filter is in place, the FLTR-IN LED will change from green to red. Refer to Figure 3-1.

e. See Section 2 for a description of the span filter.

f. Both the REF and MSR DET LEDs should have solid green or flashing green output.

g. If the indicator LED color does change color, refer to the Diagnostics section below.

h. Depress the SPN CHK button to remove the filter from the beam path. The FLTR-IN indicator will change from red to green.

i. Wait 2 minutes for the output to stabilize and the unit should display the background fluorescence level.

j. Both the REF and MSR DET LEDs should have solid green or flashing green output.

k. The unit is now ready for process monitoring.

3. There is no need to adjust the 4mA and 20 mA levels for process monitoring, since the presence of analyte will move the fluorescence level upward from the background level.

4. Instructions for adjusting the 4mA, 20 mA and display levels are given below.

5.3.3 Case 3: Process Monitoring Following Application Engineering

This case is valid when the user has determined that Mode 2 monitoring is sufficient to meet the monitoring goals in process. This is

the simplest monitoring method since ***no calibration standard is required.***

The steps outlined below assume that the hardware was initialized properly and that the Fluorometer has been functioning properly since installation.

1. The fluorescence probe can be immersed in any process sample. Since application engineering has been performed, the user has generated a calibration curve such that the fluorescence level can be immediately correlated to an analyte concentration.
2. Perform Mode 2 initialization.
    a. Press the INT PRESET, located in the upper left hand corned of the Fluorometer, for at least five (5) seconds. Please see Section 2 for a description of Mode 2 operation.
    b. Both the REF and MSR DET LEDs exhibit solid green or flashing green output.
    c. The value on the display is fluorescence level of the process.
    d. Use the span filter to verify unit operation. While this step is not actually required since the process fluorescence has been characterized, it is recommended so as to verify the Fluorometer operational viability.
        - Insert the span filter using the SPN CHK button located on the upper end of the Fluorometer housing or, if equipped, using the remote span filter switch. When the filter is in place, the FLTR-IN LED will change from green to red. Refer to Figure 3-1.
        - Wait 2 minutes for the output to stabilize and the unit should read an upscale value.
        - Both the REF and MSR DET LEDs should have solid green or flashing green output.
        - If the indicator LED color does not change color, refer to the Diagnostics section below.

- Depress the SPN CHK button to remove the filter from the beam path. The FLTR-IN indicator will change from red to green.
- Wait 2 minutes for the output to stabilize and the unit should display the background fluorescence level.
- Both the REF and MSR DET LEDs should have solid green or flashing green output.
- The unit is now ready for process monitoring.

3. There is no need to adjust the 4mA and 20 mA levels for process monitoring, unless desired by the user. Instructions for adjusting the 4mA, 20 mA and display levels are given below

5.4 Initialization for Process Monitoring: Mode 1 Operation

Mode 1 process monitoring is used once a calibration curve has been developed (see Appendix 1). This initialization sequence requires that the user generate a process sample with a **known analyte concentration**, which is used to calibrate the fluorescence scale.

Outlined below are the steps for initialization of the unit, assuming that hardware installation and start-up has occurred and that the Fluorometer is working properly:

1. The fluorescence probe must be immersed in a calibration sample.
2. Perform Mode 1 initialization.
    a. Press the INT PRESET, located in the upper left hand corned of the Fluorometer , for at least three (3) seconds, but no longer than five (5) seconds.
    b. Both the REF and MSR DET LEDs should have solid green or flashing green output.
    c. Wait 2 minutes for the reading to stabilize.
    d. The value on the display should be 1000 ± 20 counts and the 4-20 mA level should be 12.000 ± 0.164 mA.

e. Insert the span filter using the SPN CHK button located on the upper end of the Fluorometer housing. When the filter is in place, the FLTR-IN LED will change from green to red.

- Wait 2 minutes for the output to stabilize and the unit should read an upscale value.
- Both the REF and MSR DET LEDs should have solid green or flashing green output.
- If the MSR indicator LED color does change color, refer to the Diagnostics section below.
- Depress the SPN CHK button to remove the filter from the beam path. The FLTR-IN indicator will change from red to green.
- Wait 2 minutes for the output to stabilize and the unit should display the calibration sample level.
- Both the REF and MSR DET LEDs should have solid green or flashing green output.

f. The unit is now ready for process monitoring.

3. There is no need to adjust the 4mA and 20 mA levels for process monitoring, since the presence of analyte will move the fluorescence level along the calibration curve determined by the user during the application engineering studies.

Instructions for adjusting the 4mA, 20 mA, and display levels are given below if the user wants to adjust the levels.

5.5 Adjusting the Display

The display values can be adjusted for both Mode 1 and Mode 2 operation using the following instructions. For clarity, the directions describe changing the display following a Mode 1 initialization since the Fluorometer, will display a value easily correlated to a process variable. Figure 3-1 can be used to locate all the adjustment points described.

1. Immerse the probe in a process sample, preferably with a known analyte concentration and therefore fluorescence level.

2. The Fluorometer is always configured in USER MODE. The display is displaying the fluorescence intensity in arbitrary counts.
3. The display and 20 mA output levels are interrelated in USER MODE. Consequently adjusting the display value alters the 4-20 mA output signal level.
4. The display adjustment is to the right of the display. The adjustment is designated USR SPAN CONTROL
5. Display Adjustment (Mode 1 initialization):
    a. The calibration sample is being monitored
    b. If the display reading is close to 1000 counts (off by <100 counts) use the USR SPAN CONTROL to adjust the display to read 1000 counts
6. Adjusting the display level after Mode 2 initialization is not recommended

5.6 Adjusting the 4 mA Level

The zero level or 4 mA level can be adjusted at any time during the monitoring process; however, it is strongly recommended that the 4 mA level be adjusted when the fluorescence probe is immersed in the background sample. The Fluorometer/probe combination exhibits a fluorescence signal so even if the background material does not fluoresce and there is not stray light in the measurement area there will be a non-zero fluorescence measured. It is not critical that the background fluorescence be zero, only that the background fluorescence level is stable. Furthermore, the zero fluorescence level is set electronically in both Mode 1 and Mode 2, not by process samples.

The following procedure is followed for both Mode 1 and Mode 2 initialization. Refer to Figure 3-1 for the locations of the adjustments discussed.

1. Immerse the probe in a process sample, preferably the background material with no analyte of interest.
2. Monitor the 4-20 mA signal using an appropriate meter.
3. Note the display reading.

4. Depress and hold the ZRO CHK button. This disconnects the Fluorometer output from the detector circuitry of the Fluorometer, and allows the 4 mA level to be set.
5. The display should read approximately 0000 ± 20 counts and the 4-20 mA signal should be 4.000 ± 0.164 mA.
6. To adjust the display:
    a. Changing the display once ZRO CHK is depressed will alter the display level observed only after the ZRO CHK button is released.
    b. The display adjustments are located to the left of the display. While depressing the ZRO CHK button note the display. If the display reading is close to 0000 counts (<100 counts off) use the FINE ZERO CONTROL to adjust the display.
    c. If the display is >100 counts off use the COURSE ZERO CONTROL to bring the reading near 0000 and then fine tune the adjustment with the FINE ZERO CONTROL.
7. To adjust the 4 mA level:
    a. The 4 mA adjustment is located below the display.
    b. With the ZRO CHK button depressed, adjust the 4 mA level until the 4-20 mA signal is in the 4.000 ±0.164 mA range
    c. Release ZRO CHK button.
    d. Note the display reading.
    e. If the displayed reading changed by more than 20 counts, reinitialization of the Fluorometer may be required for accurate process monitoring. This is especially true for Mode 1 operation where the calibration sample is set to a specific fluorescence level.
8. It is not recommended that the zero level be altered for Mode 2 initialization.

5.7 Adjusting the 20 mA Level

The 20 mA level can be adjusted for both Mode 1 and Mode 2 operation using the following instructions. Although the display and 20

mA levels are interrelated in USER MODE, adjustment of the 20 mA level will not alter the display value. For clarity the directions describe changing the 20 mA level following a Mode 1 initialization since the Fluorometer, will display a value easily correlated to a process variable Figure 3-1 can be used to locate all the adjustment points described.

1. Immerse the probe in a process sample, preferably with a known analyte concentration and therefore fluorescence level.
2. Monitor the 4-20 mA signal using an appropriate meter.
3. The Fluorometer is configured in USER MODE. The display is displaying the fluorescence intensity in arbitrary counts.
4. Note that any adjustment of the display levels will adjust the 20 mA level; therefore only adjust the 20 mA level after setting the display.
5. The 20 mA adjustment is located below the display.
6. With the display reading 1000 counts the 20 mA signal should be 12.000 mA
7. Adjust the 20 mA level until the 4-20 mA output is 12.000 mA.
8. Adjusting the 20 mA level after Mode 2 initialization is not recommended.

5.8 Diagnostics

5.8.1 Front Panel Lamp Diagnostics

The MSR DET STATUS and REF DET STATUS lamps show the relative signal energy present. When the LED's change their colors or go from constant to flashing this is an indication that the energy levels on the appropriate detector has changed. Reasons for changes in the LED output could be sample changes, filter attenuation, flow cell/probe fouling or broken fibers.

The signal levels on the measure and reference channels are shown by the MSR DET STATUS and REF DET STATUS lamps. (Refer to Figure 3-1) .

Causes and solution keys:

1. If both lamps show steady or flashing green, the energy levels are sufficient and no change is needed.

2. If either lamp shows red, the photomultiplier of that channel is overloaded.
3. If either lamp shows steady orange, the energy level of that channel is at the low limit for the high measurement range. Cables, filters, and cell should be checked and the energy level should be improved, if possible, to give a steady green indication.
4. If either lamp shows flashing orange, the energy level of that channel is below the reliable measurement range. The optical system must be revised to bring the lamp indication back to steady green, if possible.
5. Flashing Yellow Detector LED: there is not enough light striking the detector.
6. Red detector LED: there is to much light intensity on the detector.

5.8.2 Rectifying the Problem: Flashing Yellow Indicators

1. Verify that the lamp is emitting light.
    a. Remove the fiber optic cable from the lamp assembly.
    b. Place a white piece of paper or business card in front of the lamp assembly. The card will exhibit either a bluish purple or reddish flashing spot when operating properly.
    c. If no flashing is occurring contact TAI for additional instructions
    d. If flashing is observed, reconnect the fiber optic cable to the lamp assembly
2. Verify that the fiber optic cable is securely attached to lamp, probe and analyzer.
3. If steps 1 and 2 do not solve the problem, then the probe must be removed from the measurement location and checked for cleanliness
    a. Remove the fiber optic cables from the probe.
    b. Remove the probe from the measurement area.

c. Visually examine the probe for deposits. NOTE: a visual inspection will only detect gross deposits.

d. Clean the probe tip using a solvent known to remove the process background material and the analyte of interest. Gently rub the optical surface with a damp lint free cloth or cotton swab to remove deposits.

e. Rinse the probe tip using spectroscopic grade isopropyl alcohol (IPA or 2-propanol) or methanol to remove residual cleaning solvent.

f. Dampen a cotton swab or lint free cloth with the rinse solvent and gently rub the optical surface.

g. Air-dry the probe for a few minutes.

h. Reinsert into the process and follow the required initialization sequence (see above).

1) If steps 1-3 are not sufficient to return the Fluorometer to operation, contact TAI for assistance.

5.8.3 Rectifying the Problem: Red Indicators

If there has been a major change in the probe installation or process piping, very likely, there will be a stray light leak into the system. Verify that the monitoring location is in complete darkness. If stray light is not present at the monitoring location, contact TAI for assistance.

Appendix

A-1 Application Engineering Assistance

TAI's customers and partners working to validate our hardware for specific applications where the application engineering and development work is undertaken at customer locations, may use this document as a guide. This document is a suggested procedure only and is based on an understanding of the hardware operational requirements, not detailed knowledge of the specific application parameters and variables. These suggested experiments are the first sequence only, additional experiments will probably be required to fully characterize the process fluorescence.

A-1.1 Series 1 - Initializing the Unit and Determination of the Probe/Analyzer Signal Level

The following initialization sequence assumes that application engineering is being performed in a laboratory setting. The hardware installation is assumed to be minimal (unit is placed on a bench).

1. Mount the fluorescence probe on a lab stand. The probe comes from TAI with a black PVC cap over the sensing tip. Leave this cap in place. Initialization of the unit will occur with the cap in place. The initialization sequence below will result in determination of the probe/analyzer residual fluorescence level.
2. Connect the electrical, 4-20 mA output and fiber optic cables as described in Section 4.
3. Power-up the Fluorometer.
4. Allow the system to operate a minimum of one hour before completing the rest of the initialization instructions.
5. After lamp warm-up and stabilization, the REF DET LED should have a steady green output. The MSR DET LED should have either a solid green or flashing green output. The indicator LEDs are located to the left of the display along the outer edge of the Fluorometer housing. Refer to Figure 3-1. If the indicators are

non-green, refer to the Diagnostics section in Section 5. Preconfiguration at the TAI factory should eliminate the occurrence of non-green indicators.

6. Perform Mode 2 initialization
    a. Press the INT PRESET, located in the upper left hand corned of the Fluorometer (refer to Figure 3-1), for at least five (5) seconds.
    b. Both the REF and MSR DET LEDs exhibit solid green or flashing green output.
    c. The value on the display is the fluorescence level due to the probe/analyzer configuration. Record this value for later analysis. Table 1 below provides an example of how to organize measurement data.
    d. Insert the span filter using the SPN CHK button located on the upper end of the Fluorometer housing. When the filter is in place, the FLTR-IN LED will change from green to red.
    e. Wait 2 minutes for the output to stabilize and the unit should read an upscale value.
    f. Both the REF and MSR DET LEDs should have solid green or flashing green output.
    g. If the indicator LED color does change color, refer to the Diagnostics section.
    h. Depress the SPN CHK button to remove the filter from the beam path. The FLTR-IN indicator will change from red to green.
    i. Wait 2 minutes for the output to stabilize and the unit should display residual fluorescence level of the probe/analyzer configuration.
    j. Both the REF and MSR DET LEDs should have solid green or flashing green output.
    k. The unit is now ready for either application engineering studies or process monitoring.
7. During the application engineering process there is no need to adjust the 4 mA, 20 mA or display settings.

A-1.2 Determination of the Probe/Transmitter Fluorescence in a Non-Fluorescing Liquid:

1. Prepare the measurement area to be used for liquid testing.
    a. For lab analysis a wide mouth *amber bottle*, which has been wrapped with tape works as a measurement vessel. The cap of the bottle needs to be equipped with a feed through that will hold the probe. (Use P/N 53FCAL calibration bottle/ fitting).
    b. In the pilot system, mount the fluorescence probe in the monitoring location using the appropriate connections.
    c. For both installations it is imperative that there is no stray light in the system and the probe is in complete darkness.
2. If the Fluorometer is on, turn off the unit. Remove the protective cap from the probe tip and mount the probe in the testing location, which has been filled with a non-fluorescing material with solution characteristics as close to the process background as possible. It is critical that the solution is *completely non-fluorescing*, so that only the refractive index difference between air and liquid sample may alter the fluorescence level. Ultra pure water, RO water or DI water are probably good choices for the test material.
3. Power-up the unit and wait about 5 minutes. The power down does not affect the initialization settings.
4. After lamp stabilization, the REF DET LED should have a steady green output. The MSR DET LED should have either a solid green or flashing green output. If the indicators are non-green, refer to the Diagnostics section in Section 5.
5. Record the fluorescence level on the display
6. Insert the span filter.
    a. Wait 2 minutes for the output to stabilize
    b. Record the fluorescence level due to span filter insertion.
    c. Remove the span filter.
    d. Wait 2 minutes for the output to stabilize and the unit should display residual fluorescence level of the probe/analyzer configuration.

1) Calculate the difference between the air and liquid fluorescence levels. The difference is due to the change in refractive index between air and the liquid sample.

A-1.3 Determination of the Process Fluorescence:

This experimental sequence is the first step in characterizing the process fluorescence. The Fluorometer will use the Mode 2 initialization used for the previous experimental measurements.

For laboratory analysis using a small vessel (recommend, P/N 53FCAL), the analyst will need to collect pull samples from the process which covers the entire range of analyte concentrations. It is recommended that at least six process samples be analyzed. The first sample will be the process background material and should not contain analyte of interest.

For pilot process testing, it is critical that the initial process liquid be background material with no analyte of interest. The process can then be run normally and monitored.

The steps outlined below are based on discrete sample analysis, but can be easily modified for continuous process monitoring.

1. If the unit is not on, power-up the unit and allow warming up for at least 1 hour. If the unit has been running, just begin the sequence.
2. Place the background material in the sample vessel, wait 2 minutes and record the fluorescence level. This reading is your background fluorescence and is a combination of the fluorescence properties of the background material, stray light and the probe/ analyzer fluorescence.
3. The dynamic range of the instrument, for the application engineering sequence can be determined at this time using the following equation:

   Range Multiplier = 1999/Display Value

4. Gently wipe the probe tip with a lint free cotton swab or cloth dampened with the process background material and gently rub the optical surface. This insures that each sample is tested without fouling of the optical surface. This step is not possible in the pilot system.

5. In sequence, measure the fluorescence from each of the process samples. Make sure to wait 2 minutes after placing the probe in contact with the solution before recording the fluorescence reading. Make sure to clean the probe tip after each sample.
6. If none of the samples go off-scale (a 1 on the display in the fourth digit), then using the data obtained, plot the fluorescence intensity versus analyte concentration for the data. Determine the linearity of the output response and a possible calibration sample.
7. If one or more samples caused the unit to go off-scale (a 1 on the display in the fourth digit), then additional application engineering sequences are required. Most likely a Mode 1 initialization study, with the highest on-scale sample used as the calibration sample. The initialization sequence for Mode 1 is described in Section 5.
8. Once the process fluorescence has been characterized and the calibration procedure determined, repeat the experimental series using the desired initialization mode to verify that the correct monitoring approach has been selected.

Table A1: Data Organization

Series 1: Air and Liquid Probe/Analyzer Fluorescence Level

Material (*Air, Liquid, Solid*)	Probe/Transmitter Fluorescence	Span Filter Fluorescence

Series 2: Process Sample Analysis

Sample Number	Analyte Concentration	Measured Fluorescence
0		
1		
2		
3		
4		
5		
6		

Series 3: Monitoring verification using selected calibration (initialization) procedures.

Sample Number	Analyte Concentration	Measured Fluorescence
0		
1		
2		
3		
4		
5		
6		

A-2 Notes

1. Convert 4-20mA output to counts;
   Where X=mA output
   (X-4/16)1999 = Counts

2. Convert 4-20mA output to engineering units;
   (X-4/16) max value = Max Engineering Unit

   The maximum engineering unit value should be 2x the calibration sample value (Mode 2 operation) or the sample with a Mode 1 fluorescence reading of 1999 counts.

3. TAI utilizes two types of span filters. Metallic film ND filters are used when fluorescent filters are not available for the application specific excitations and emission wavelengths. The neutral density filter reduces the measured signal by a fixed percentage whenever employed. For example, a ND filter may have a 70% pass so if the process reading is 980 counts, inserting the span filter will result in a reading of 686 counts (nominal).

4. Generally, the reference detector LED should always be solid green, although flashing green is acceptable. Generally, in zero fluorescencent material, the measure detector LED should be yellow, or flashing green. Generally, in process or calibration solution the measure detector LED should be flashing green or solid green.

   A solid red detector indicates to much signal intensity. If this appears, the unit must be returned to factory for evaluation.
   A flashing yellow LED on either detector is a low signal level and the unit must be returned to factory for evaluation.