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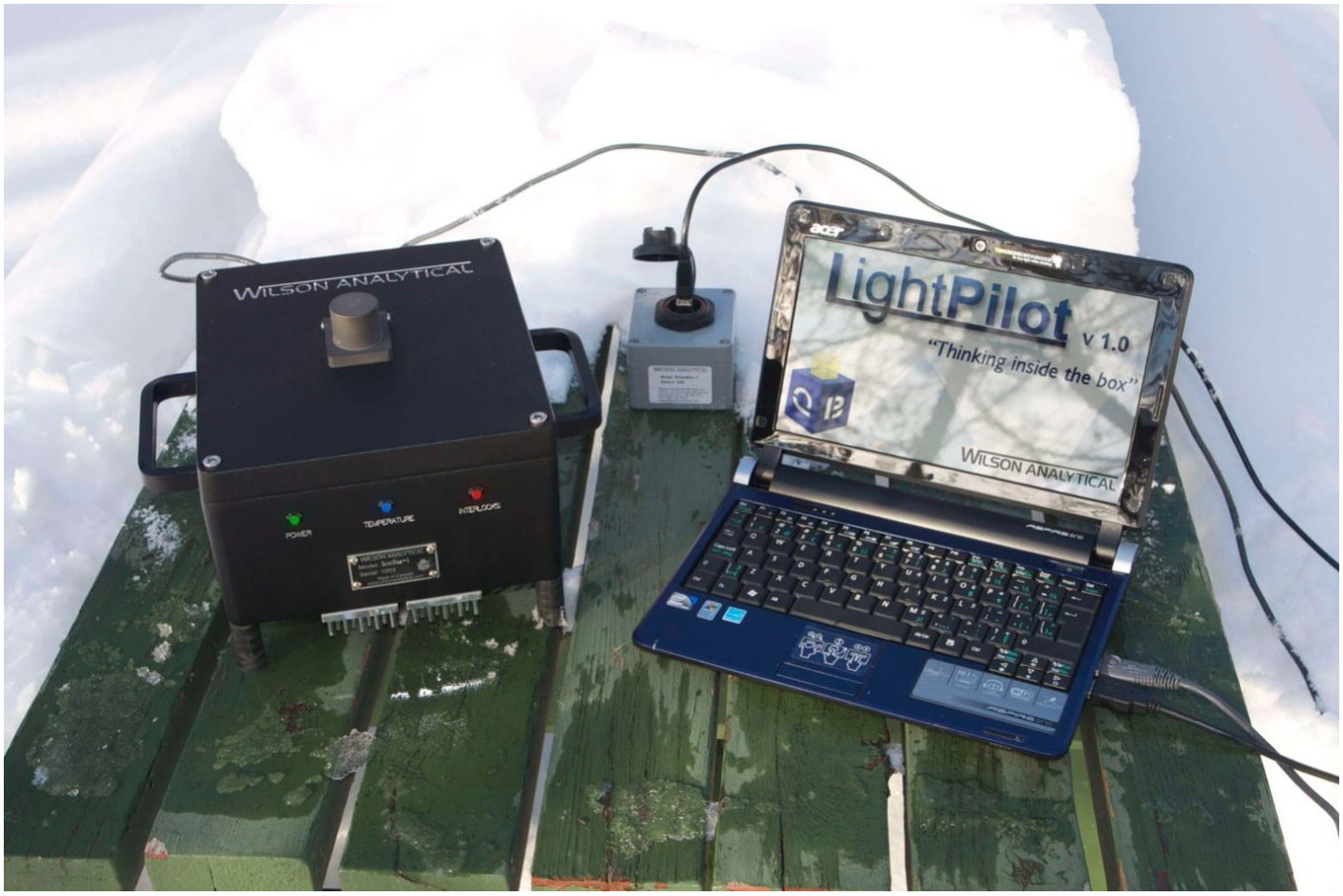
QuatBox™ by Wilson Analytical

Hardened Fluorescence Spectrometer

User's Manual



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Contact Information

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Introduction to the QuatBox™

Figure 1. Wilson Analytical's rugged fluorescence instrument, the QuatBox™.

The QuatBox™ is a ruggedized fluorescence spectrometer designed to provide field-based users with laboratory-quality analytical data. The instrument was designed to support field applications by making the measurement of chemical concentrations in water easy and reliable, even under adverse environmental conditions.

Several unique features make the QuatBox™ a valued and flexible addition to the corrosion specialist's toolkit:

- Runs from any 12V or 110V source
- Sample holder has easy to clean, highly scratch-resistant windows
- User friendly software that requires no specialized knowledge of chemistry or fluorescence
- Creates field-based calibration curves in a few easy steps
- Automatic temperature regulation of samples enhances data accuracy
- High sensitivity allows measurement of very dilute samples (low ppm)
- Built-in GPS unit automatically adds date, time and location stamps to all data files
- Integrated fuses and interlocks make the device inherently safe to use

Set-up & Quick Start

1. Take the QuatBox™ and set it up on a reasonably clean, flat surface.
2. Using the power cables provided, connect the device to any convenient 12V DC or 110V AC power source. The *Power* LED on the front of the QuatBox™ will turn green when the unit is energized.
3. Power up your computer which runs LightPilot Software.
4. Connect the 'WhereBox' [GPS receiver, see Figure 2] to your host computer using the long USB cable. (NB. The maximum length of this cable is 16 feet)

Figure 2. The WhereBox with the USB Cable

5. Place the WhereBox in a location that allows for the clear reception of GPS satellite radio signals.
6. Connect your computer to the USB port on the rear of the QuatBox™ using the short USB cable (The *Interlock* LED on the front of the QuatBox™ will turn from red to green.)
7. Double click on the blue W icon to start the LightPilot software.
8. Depending on the ambient temperature, the *Temperature* LED on the front of the QuatBox™ may be red (too hot), blue (too cold), or green (temperature within measurement range, factory set to 32 C +/- 5 C). If the *Temperature* LED is not green, simply wait a few minutes until the QuatBox™ has stabilized. Once all three indicator LED's are green, the QuatBox™ is fully operational, and ready to begin taking measurements.
9. If there is no calibration curve for the chemical you wish to measure, you must select *Control* from the mode menu in the LightPilot software and follow the steps on the screen to create one.
10. If a calibration curve for the chemical you wish to measure is already stored in the LightPilot software, select *Acquisition* from the mode menu, and follow the steps on the screen to measure the chemical concentration in a new sample.
11. For more information regarding LightPilot, refer to the LightPilot Software User's Manual.

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Introduction to the WhereBox

The WhereBox is a robust, weatherproof GPS unit designed for use with the QuatBox™. It connects to the laptop by way of a 16-foot USB cable (included with the WhereBox) and will automatically supply the precise coordinates of where you are sampling.

Figure 3. The WhereBox

You will be prompted in the LightPilot software to enter information on the customer and sample location by the dialogue box seen below, each time you take a sample.

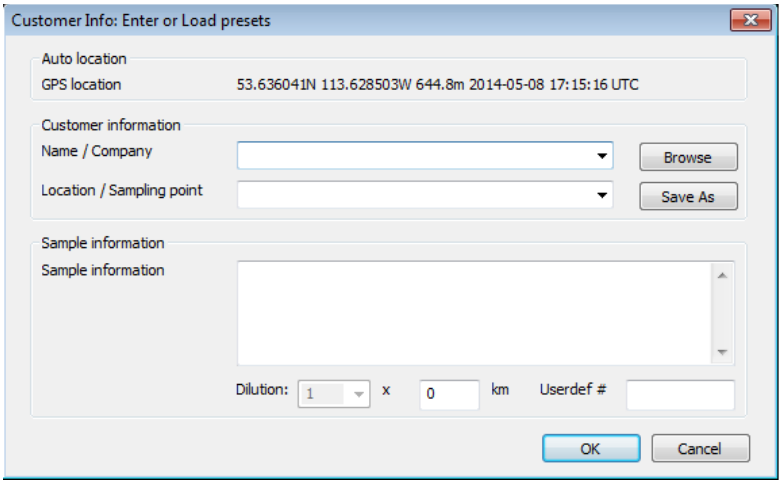

Figure 4. Dialogue box in the LightPilot software where you can write in sample information and location.

Note that beside the ‘Name/Company’ and ‘Location/Sampling point’ boxes, there are arrows. You can click these arrows to open a dropdown menu of previously entered sample locations and company names. Selecting one of these will save you the need to enter this information again if you are sampling at the same location multiple times.

After filling in the ‘Name/Company’ and ‘Location/Sampling point’ boxes, you can click the ‘Save As’ button to the right of them to save this information to the GPS coordinates. In future visits, if all the information remains the same, you can simply click ‘Browse’ (located above the ‘Save As’ box) and select the location in order to fill in the company and location information.

For the WhereBox to work, it must be in a location from which a GPS satellite radio signal can be reached. As such, it is best to place it out in the open; if indoors, it is best placed as near to the door or window as possible, especially if you are below ground. The 16 foot USB connection cable allows you to place the WhereBox anywhere within a 16-foot radius from where the laptop is set up. The bottom of the WhereBox is also magnetic, allowing it to be attached to any magnetic surface, such as the roof of a truck or wall of a metal shed. This improves the range of places the WhereBox can be set up, allowing for the best GPS satellite radio signal possible.

With LightPilot you have the option to use the WhereBox unit, or not. If you choose to use the WhereBox, the GPS coordinates, the date and the time will be displayed at the top of the screen in the Customer Information dialogue box as seen in Figure 4. If you choose not to use the WhereBox the GPS field will just contain default letters and question marks that can be safely ignored. For more information on how to install LightPilot, create a calibration curve and run an unknown sample on LightPilot, refer to the LightPilot User’s Manual that can be found on the Wilson Analytical website. www.wilsonanalytical.com

Operating The QuatBox™ : Power Up and LED Indicators

The QuatBox™ is designed to run on either 12V DC (using the supplied 12V power cord, see Figure 6) or 110V AC power (using the supplied power brick, see Figure 7). Either source can be plugged directly into the rear of the unit (see Figure 5 and 9), using the power cables provided, without the user having to flick a switch or change any settings. Connecting the cord turns on the unit, while disconnecting the cord turns it off, as there is no “power” switch on the QuatBox™.

Figure 5. Connections on the rear of the QuatBox. Note the cap in place over the power socket.

Figure 6. QuatBox 12V DC power cord with 12V vehicle plug and DIN plug.

Figure 7. QuatBox AC power supply with 120V cord and plug, and DIN plug.

Figure 8. DIN power plug on the QuatBox end of the power cables. This DIN plug makes a waterproof seal with the DIN power socket on the back of the QuatBox and has a rotating locking collar to securely hold the cable in place.

Figure 9. Connecting and locking the DIN power plug to the rear QuatBox power socket.

Suitable sources of power include a regular 110V AC supply outlet, a 12V DC ‘cigarette lighter’ outlet as found in trucks and other vehicles, or a stand-alone 12V DC battery pack. To connect up the power source, the waterproof cap must first be removed (by twisting counter-clockwise) from the QuatBox™ power socket on the rear of the unit (see Figures 5). Next gently insert the DIN power cord plug (see Figure 8) into the power socket on the back of the QuatBox™ (Figure 9). Properly seat the plug by slowly rotating it in the socket until it is correctly aligned and can be fully inserted into the power socket. At this point, if the plug is correctly positioned in the socket, the cable can be locked into place by the rotating locking collar clockwise until it stops. Successful power-up of the QuatBox™ is indicated by a green “*Power*” LED on the front face of the unit.

The QuatBox™ electronics are protected by several thermal and electrical fuses. In the event that any of these fuses break, the *Power* LED on the front panel will turn red. At this point, the QuatBox™ is inoperable and must be returned to Wilson Analytical for repair.

The QuatBox™ uses an intense ultra-violet (*uv)* light source to stimulate fluorescence in the sample. Without a cover for the sample chamber, it would theoretically be possible for this *uv* light to escape from the QuatBox™ via the cuvette and enter the eyes of the user. As a safety precaution therefore, the QuatBox™ is equipped with an interlock, such that the unit will not operate (the *uv* light source will not come on) unless the hinged sample cover is closed. When the hinged sample cover is open the *Interlock* LED on the front of the QuatBox™ is orange, indicating the instrument is not ready to be run.

In total there are three LED indicators on the front of the QuatBox™, which all serve a different purpose (see Figures 10 and 11). The *Power* LED indicates when the light source is ready to be ran, is running or if a fuse has blown. The *Interlocks* LED protects the user from harmful *uv* rays and helps remind the user to connect the unit to the computer. The QuatBox™ can only be used and controlled via the LightPilot software supplied with each unit. A computer must therefore be connected to the QuatBox™ via the USB interface at the rear of the unit before any measurements are possible. The *Temperature* LED helps indicate to the user the temperature of the sample, ensuring samples are tested at consistent temperatures.

Figure 10. LED indicators at the front of the QuatBox

Power LED: Green - unit energized and ready.
Red - internal fuse has blown. QuatBox™ will not run and must be returned for repair.
Blue - fluorescence excitation source is energized, and the QuatBox™ is acquiring data.

Interlocks LED: Green - both interlocks are closed; unit is ready.
Orange - the hinged cover for the sample holder is open
Red - no computer connected to the QuatBox™.

Temperature LED: Green - sample is within the set temperature range, between 22 and 32 C.
Red - sample is above the set temperature. (> 32 C)
Blue - sample is below the set temperature. (< 22 C)

Operating The QuatBox™ : Temperature Control

Temperature control is one of the four 'secrets' of successful fluorescence measurements. Accordingly, the QuatBox™ contains heating and cooling that keep the sample at close to constant temperature, regardless of the ambient conditions.

The sample temperature is monitored by a small sensor, placed in very close contact with the sample cuvette. It is the output of this sensor that controls the *Temperature* LED indicator on the front panel of the QuatBox™. The *Temperature* LED will only turn green (allowing the user to obtain measurements) when the sample temperature is within a pre-determined range (27 +/- 5 C). A digital display is provided on top of the QuatBox™, which constantly displays the temperature of the cuvette and sample in degrees Celsius.

The acceptable temperature range for measurements is factory set to 27 +/- 5 C (80.5 +/- 9 F), and so the *Temperature* LED will be blue if the sample temperature is lower than 22 C (71.5 F), and red if the sample temperature is higher than 32 C (89.5 F). If the *Temperature* LED is blue or red, no measurements are possible, but no action is necessary. The temperature control circuitry inside the QuatBox™ will bring the sample temperature into range, although this may take a few minutes, depending on the initial sample temperature. Once the sample has equilibrated to 27 C (+/- 5 C), the *Temperature* LED will turn green, and measurements can commence.

Note that heat is dissipated from the QuatBox™ via the aluminum case of the unit. It is a good idea, especially if the air temperature is greater than 27 C, to locate the QuatBox™ somewhere where there is some air movement around the case. This will enhance convective cooling and hence the efficiency and speed of temperature equilibration. The use of a fan may help.
If airflow alone proves insufficient for cooling at higher temperatures, the QuatBox™ can be run with an ice pack in direct contact with the aluminum bottom of the unit. The unit bottom is sealed and the QuatBox™ legs are plastic, so neither will be adversely affected by cold or moisture. If an ice pack is unavailable, the instrument may be operated while standing in a tray of water and/or ice. The liquid level in the tray must be high enough to touch the aluminum bottom of the unit, but not higher than the bottom of the Swagelok drain fitting on the back of the QuatBox™. See Figure 15 and 16 for a rear view of the unit and the drain.

Figure 11. The QuatBox™ with all three of the LED indicators showing green, indicating the instrument is ready for testing. Note the sample temperature display on the top of the unit.

Operating The QuatBox™ : Setting the Blank and Running Samples

To run a sample on the QuatBox™, you must use the LightPilot software, and select a calibration that is appropriate for the samples being run. If the software currently stores no suitable calibration, one must be created before use. For more information on how to create a calibration curve in LightPilot, please refer to the LightPilot User's Manual that can be found on the Wilson Analytical website at www.wilsonanalytical.com

Once the appropriate calibration for the samples is selected in the Acquisition Mode on LightPilot, it is only possible to acquire a spectrum if the QuatBox™ is in operational mode, as indicated by the three status LED's on the front of the instrument showing green.

First check that the *Power* LED is green, indicating that the QuatBox™ is energized and ready. Next insert the blank sample into the sample holder and close the cover. The *Interlock* LED should now also be green. If it is orange, the sample cover is not properly closed. Finally, check that that the *Temperature* LED is also green. If the sample just placed inside the QuatBox™ is colder than the designed running temperature of the QuatBox™ (less than 22 C or 71.5 F), the *Temperature* LED will turn blue, at which point the QuatBox™ will not operate. Conversely, if the sample temperature is above the designed running temperature of the QuatBox™ (greater than 32 C or 89.5 F), the *Temperature* LED will turn red, and again, the QuatBox™ will not operate. Should the *Temperature* LED indicate that the sample temperature is outside the operational window, simply wait until the QuatBox™ brings the sample temperature into the desired range and the LED turns green. The actual temperature of the sample is also continuously displayed on the top of the unit in degrees C as a reference, as seen in Figure 11.

Once there are three green LED's on the QuatBox™ front panel, the unit is ready to take measurements. Click 'Measure Blank Sample', and the software will prompt you to ensure that the blank sample has been placed into the cuvette holder before proceeding. If you have not already put the blank sample into the QuatBox, open the sample lid (the *Interlock* LED will change from green to orange), place the blank sample (zero concentration sample) into the cuvette holder inside the QuatBox™, and close the lid (the *Interlock* LED will change from orange to back to green). After clicking on ok, the *Power* LED will turn from green to blue, indicating that the measurement is in progress. Wait until the *Power* LED changes from blue back to green, at which point the data collection is complete.

The fluorescence spectrum of the blank sample will be displayed on the computer screen. It is likely to be relatively uninteresting as it is a control sample. If the spectrum does contain peaks or features that are more than a few hundred counts high, there is a good chance that either the wrong sample has been selected (i.e. the sample isn't really a blank), or if using a matrix-matched blank instead of pure water, that the sample matrix contains fluorescent material that could interfere with the measurement. It is also possible that the unit is dirty inside of the cuvette holder, and requires cleaning before proceeding with measurements. See the manual section on "Cleaning the Sapphire Windows" for the correct cleaning instructions.

It is good practice to run the blank as a “sample” at this point to ensure that the instrument is functioning correctly. Leave the blank sample in the instrument, and click ‘Measure Data’. Then, click on the ‘Measure’ button to collect the sample data. Wait until the *Power* LED changes from blue back to green, and the fluorescence spectrum of the blank sample “run against itself” will be displayed on the computer screen. Since the blank has been compared to itself, the result should be a relatively straight line if the instrument is functioning correctly. The vertical “counts” axis autoscales when displaying spectra depending on peak height, so the “blank” florescence plot may look at bit jagged, but the data should not go up or down by more than a hundred counts across the spectrum. If there is an obvious “peak” in the spectrum, try repeating the blank background collection and then rerunning the blank as a “sample”. This should correct the problem, and give a nearly straight line as a spectrum. Once this is obtained, the unit is ready to collect sample data.

To run an unknown sample, insert the sample into the sample holder and close the cover. At this point the three status LED’s on the front panel should all show green. Click ‘Measure Data’ and insert any sample information as needed. The next step is to type in the dilution factor for that particular sample in the text box provided. For example, if the original sample was diluted by a factor of x100 prior to measurement, the user should type ‘100’ (without the quotes) into the ‘Dilution Factor’ text box. If no dilution was used, ensure that the dilution factor is set to ‘1’. Finally, click on the ‘Measure’ button to collect the sample data. Wait until the *Power* LED changes from blue back to green, and then remove the sample from the QuatBox™.

The fluorescence spectrum of the unknown sample will be displayed on the computer screen. The spectrum display will show a red vertical line at the wavelength selected during calibration for the concentration calculation. Typically, this will correspond to a major peak in the fluorescence spectrum of the test sample. Note that the calibration wavelength cannot be changed at this point; it is displayed for reference only. LightPilot will also calculate the concentration of the target chemical in the original sample, based on the dilution factor and the calibration curve that have been selected. The result is displayed at the top left-hand side of the screen and will be written into the data file when it is saved.

Further samples can be run at this point by changing the sample and then clicking ‘Measure Data’. There is no need to rerun the blank until the instrument is unused for several hours.

For more information on how to install LightPilot, create a calibration curve and run an samples on LightPilot, please refer to the LightPilot User’s Manual that can be found on the Wilson Analytical website at www.wilsonanalytical.com

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Cuvettes 101

Samples are normally introduced into the QuatBox™ in specially designed disposable plastic cuvettes. Some simple spectrometers use round cuvettes for quick measurements, but square cuvettes are required for accurate concentration determinations, and all Wilson Analytical systems are designed for square cuvettes. For best results we recommend using 10mm pathlength disposable PMMA fluorescence cuvettes for QuatBox™ measurements in water. These are available in boxes of 100 with caps from the Wilson Analytical website at www.wilsonanalytical.com, which also contains further information on cuvette types, wavelength ranges and solvent compatibilities.

Cuvettes are referred to by the distance between the INSIDE faces of the opposing cuvette walls, as this represents the pathlength, usually expressed in mm, that the spectrometer light travels through the liquid sample during the measurement. So, for example, a standard “macro” 10 mm cuvette has a distance of exactly 10 mm between the inside walls of the cuvette, giving a liquid sample pathlength of 10 mm, and a sample volume of approximately 3.5 mL. The outside dimensions of cuvettes can vary however, depending on the cell material and type of construction.

The QuatBox™ measures fluorescence, which is obtained by optically ‘exciting’ the sample solution at a 90-degree angle to the spectrometer detection system. This requires cuvettes with optically clear walls at 90 degrees to each other, and this is typically accomplished by making all four walls on the cuvette optically clear, with no ‘frosted’ sides for handling the cuvette (as is typical seen for “absorbance” cuvettes). These are known as “four sides clear” or “fluorescence” cuvettes.

Be aware that the instrument light passes through the lower third of the cuvette, and so when taking measurements, please ensure that cuvettes are always filled to at least the half-way mark (~2mL). It is important to ensure that the outer surfaces of cuvettes are dry before they are placed into the QuatBox™ sample holder. Drops or films of water or liquid will refract and scatter light, reducing the accuracy and repeatability of the measurements.

It is also a good idea to handle cuvettes only at the top, because grease, smears, dirt and fingerprints on the outside of the lower part of the cuvette will interfere with the accuracy of the measurements. We suggest writing sample information on the top 1/3 of the cuvette in order to distinguish between samples. Remember that all cuvettes look alike, and so should be labeled!

Disposable cuvettes (which do not require cleaning, and so are convenient for oilfield analyses) are available in various types of plastic, only some of which are suitable for use in the QuatBox™ with the 365 nm excitation required for corrosion inhibitor measurements. Polystyrene disposable cuvettes should not be used as they do not transmit light well in the *uv* range, while Poly(methyl methacrylate) or “PMMA” disposable cuvettes are preferred for their good transmission down to 300 nm in the *uv*.

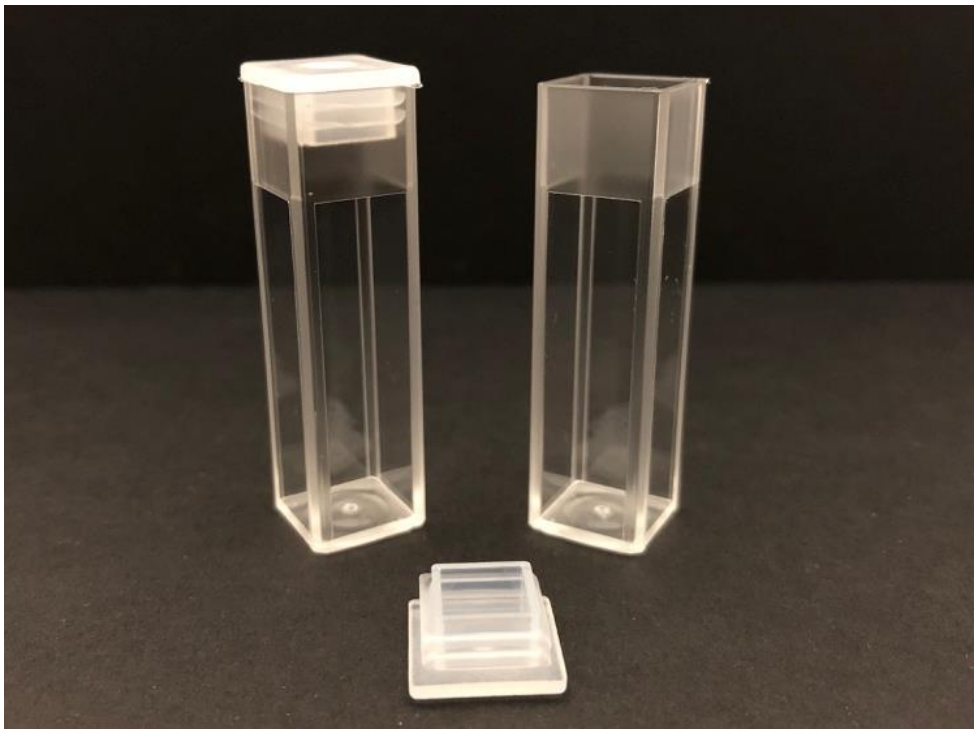

Figure 12. Disposable fluorescent cuvettes available online at www.wilsonanalytical.com.

Although the plastic cuvettes are mass-produced, there is typically a slight variation in their external dimensions. This is important to note, because the QuatBox™ sample holder has been manufactured to accommodate the cuvettes as a snug fit. This design is deliberate and ensures that the sample temperature sensor is able to respond rapidly and accurately to the various sample temperatures that are presented. Very occasionally we have encountered plastic cuvettes that are slightly oversize, such that they do not fit inside the QuatBox™ sample holder. If this occurs, please discard that particular cuvette and use another; **under no circumstances should the user try and force an oversize cuvette into the sample holder**.

Please note that disposable plastic sample cuvettes are often incompatible with organic solvents. If you are planning to use solvents in your work, we strongly recommend that you ensure beforehand that the particular disposable cuvettes to be used will stand-up to the chemicals in question by checking with the Wilson Analytical website for plastic/solvent compatibilities. If disposable cuvettes are unsuitable, it is possible to obtain glass or quartz fluorescence cuvettes that are far more chemically robust, but also much more expensive. The cost of these cuvettes ($25 to $200 each, depending on material and construction quality) means that they must be reused, and as such thoroughly cleaned between samples. Please consult with Wilson Analytical before using non-plastic cuvettes in the QuatBox™, as an adjustment to the temperature sensor position may be required to fit the cuvettes into the sample holder without damage.

Good quality fluorescence cuvettes (both disposable and reusable, suitable for all types of samples) are available from the Wilson Analytical website at www.wilsonanalytical.com, which also contains further information on cuvette types, wavelength ranges and solvent compatibilities.

Using the Solid-State Reference Sample

A solid-state reference (SSR) sample is supplied with each QuatBox™. The reference material is encased in an anodized aluminum holder that fits snugly inside a regular disposable 10mm x 10mm cuvette. The reference is obviously designed for re-use, and so the plastic cuvette acts as a protective housing. If and when the external cuvette becomes scratched, damaged, or unclean simply slip the reference out of the old cuvette and place it in a new one.

Figure 13. Wilson Analytical's Solid State Refence (SSR)

The Wilson SSR works as a 'relative' fluorescence reference sample, which is well suited to its purpose as a Quality Control (QC) check to ensure that the instrument is running correctly and calibrated properly prior to measuring samples. Since the concentration value obtained for the SSR is dependent on the calibration curve used to run it, providing a 'certified' ppm value for the SSR is not possible. Its 'value' is always determined experimentally for each calibration curve produced. See Figure 14 for a plot of the spectrum obtained on the Wilson SSR when run in a QuatBox™ with 365 nm excitation.

Once a calibration curve is completed for a particular corrosion inhibitor to be measured by the QuatBox, that curve should be used to determine the numerical value of the Wilson SSR for at least 30 measurements, preferably collected over the course of a week or so. The average value and two standard deviation of these measurements are then calculated and recorded as the QC value (the average) and range (+/- 2 std. dev.) for the SSR for that particular calibration curve.

At the beginning of each session, or whenever the user wishes to check that the QuatBox™ is operating normally, simply select the appropriate calibration file, run a blank determination, then insert the SSR standard and acquire a spectrum. If the QuatBox™ returns a value within two standard deviations of the accepted value for the SSR when using the specified calibration curve, then it is operating normally. Remember that a different QC value (the average) and range (+/- 2 std. dev.) will be obtained for each calibration curve. However, if the machine performance is all that is being verified

by the QC measurement (not each calibration curve individually), then the same calibration curve can safely be used each time to ensure that the instrument is functioning correctly.

If the reference check returns an out of range value for that curve, the most likely causes are a dirty or damaged SSR outer plastic cuvette, and/or dirty windows in the sample holder. If the cuvette holding the reference standard appears to be clean and undamaged, proceed with cleaning the windows in the sample chamber and then re-measure the reference. The QuatBox™ should then return a reading within the accepted value if operating normally. If the received signal is still not within the specified range, repeat the process. If this does not resolve the issue, contact Wilson Analytical at info@wilsonanalytical.com.

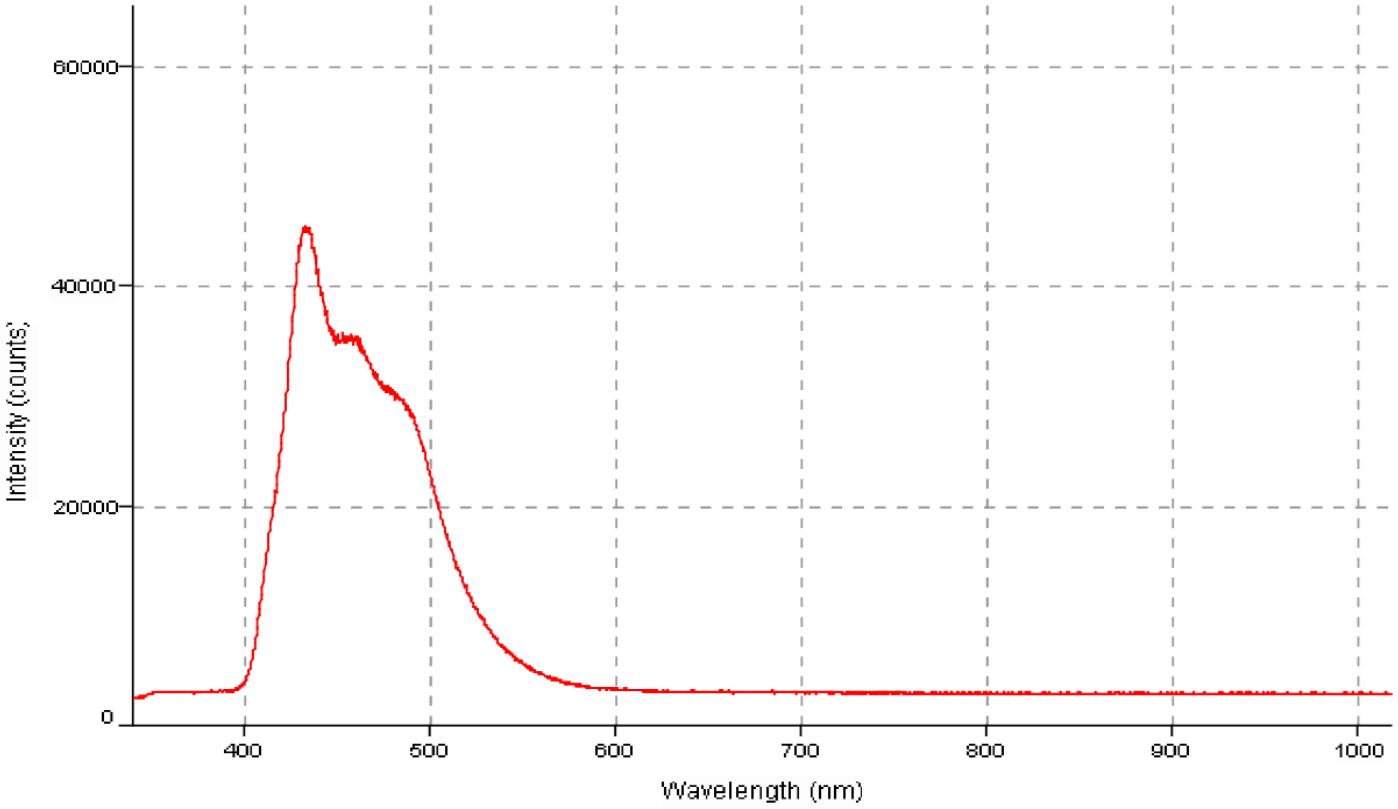

Figure 14. Fluorescence spectrum obtained with 365 nm excitation of the Wilson SSR QC Test Sample

Cleaning the Sapphire Windows

The optical windows in the QuatBox™ sample holder (cuvette holder) are manufactured from single-crystal sapphire (Al_2O_3), making them extremely resistant to scratches and chemicals attack. Nevertheless, any damage or staining to these windows will adversely affect the results from the QuatBox™, and so care should be exercised during the cleaning process. Never insert sharp objects into the cuvette holder.

The sapphire windows, which are located near the bottom of the anodized aluminum cuvette holder, are waterproof and are gasketed in place. This makes the entire cuvette holder assembly internally watertight and resistant to cleaning solutions. In addition, there is a drain at the bottom to remove liquids, the outlet of which is a stainless-steel Swagelok fitting at the lower rear of the instrument (see Figure 15). Before starting to clean the windows simply attach the supplied Swagelok hose-barb fitting (with a suitable length of plastic hose) to the drain fitting on the back of the unit, so that the wash fluid can flow into an appropriate sink or container (see Figure 16). This design allows the windows to be cleaned while still inside the fully assembled QuatBox™, by simply opening the QuatBox™ hat (sample lid) and using an appropriate length brush to clean them.

Figure 15. The stainless-steel drain fitting on the rear of the QuatBox™, circled in yellow.

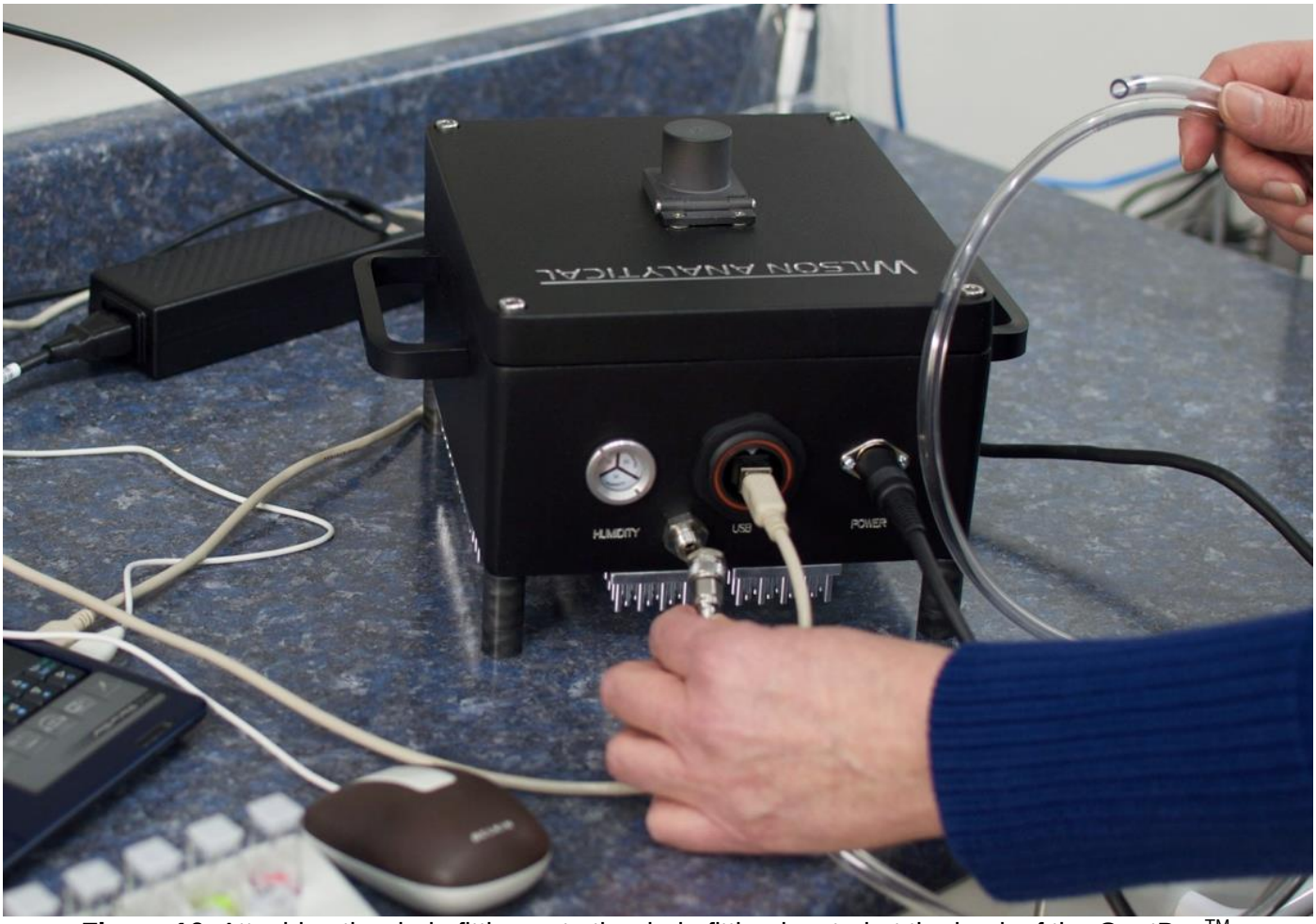

Figure 16. Attaching the drain fitting onto the drain fitting located at the back of the QuatBox™

To clean the windows, we recommended soapy water as the cleaning fluid, which should be applied to each of the windows in turn with a cotton swab or test-tube brush. Applying pressure on the surface of the windows during detergent and water washing is quite acceptable but avoid cleaning them by rubbing with abrasive materials (see Figure 17).

Figure 17. Gently washing the sapphire windows in the QuatBox™ with a cotton swab and soapy water

Once the windows have been cleaned, it is very important to remove any soap film, smears or residue that could impede or weaken the transmission of light. This is best achieved through the application of clean, lukewarm water and clean swabs. After a thorough final rinse with water (preferably distilled or deionized), a rinse with good quality, clean methanol can be used to speed drying, if desired. Drying of the windows can be finished with a soft cloth or lens tissue wrapped around the end of cotton swabs, making as sure as possible that the windows are left free of water, streaks, films and lint. A flashlight is usually helpful to examine the cuvette holder windows, which are mounted in the sidewalls of the lower part of the sample chamber. If no clean drying materials are handy, then the windows can be air-dried using a hair dryer to blow hot air through the open sample chamber, or failing all else by leaving the unit overnight with the sample chamber cover left open.

After cleaning and drying the windows, select the appropriate calibration file and re-run the Wilson SSR. The QuatBox™ should now return a reading within +/- 2 std. dev. of the accepted range of the calibration curve to be operating normally. If the received signal is still not within the specified range, repeat the process. If this does not work, contact Wilson Analytical at info@wilsonanalytical.com.

Note that it is not possible for the user to remove the sample holder from the QuatBox™, or remove the windows from the sample holder. Both operations require the top of the QuatBox™ to be removed, an operation that will void the warranty if attempted by the user.

Desiccant Indicator

The QuatBox™ contains a high-capacity desiccant that is designed to keep the dewpoint inside the instrument extremely low to avoid moisture condensation if the unit is cooled excessively at any point. Apart from potentially damaging the electronics and sensors inside the QuatBox™, moisture or ice condensation on the inside of the windows or optics might interfere with light transmission, and hence reduce the accuracy and repeatability of the measurements.

A humidity indicator is attached to the rear panel of the QuatBox™ so the user can see that the desiccant is not spent (see Figure 18). If the 30 % panel in the humidity sensor turns lavender in colour, the desiccant needs to be replaced. This operation can only be done by Wilson Analytical, as it requires the top of the QuatBox™ to be removed, an operation that will void the warranty if attempted by the user.

Figure 18. Humidity indicator located at the back of the QuatBox™

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Exterior Maintenance and Technical Support

The user should take every precaution not to spill chemicals into the QuatBox™ sample holder. Only clean, dry cuvettes should be placed inside the QuatBox™ itself. However, the sample holder has been designed with robustness in mind, and it will tolerate a wide variety of chemical spills and contamination, provided it is cleaned rapidly and thoroughly. See 'Cleaning the Sapphire Windows' procedure above for how to clean the cuvette holder and windows.

The most damaging chemicals to the sample holder, and indeed to the QuatBox™ itself, will be strong acids or alkalis, because the outer case and sample holder are manufactured from aluminum. Although all the accessible QuatBox™ components have been anodized or coated to provide chemical and corrosion resistance, the lifetime and accuracy of the unit will be enhanced if its surfaces are kept clean and chemical-free.

If you are experiencing problems with your QuatBox™, please refer to the relevant section of this user's manual for advice and troubleshooting suggestions. For example, low intensity may be due to dirty optical windows, while problems regulating the sample temperature may be due to insufficient airflow around the instrument when in a hot environment.

If referral to the manual and the remedial action suggested has not solved the problem, please send an e-mail with details of the issue you are facing to the following address: info@wilsonanalytical.com. Please remember to include your e-mail address and phone number in any correspondence.

A representative of Wilson Analytical will contact you as soon as possible to discuss the problem. If the situation cannot be remedied remotely, a Return Authorization Number (RAN) will be issued. At that point, the unit must be returned to us for repair. Unless the instrument is under warranty, it should be shipped pre-paid, and the enclosed documentation should include the RAN that was issued to you, your contact details and shipping address, and a summary of the problems you have encountered with the unit.

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Warranty

Wilson Analytical guarantees that the QuatBox™ product will remain free of defects due to materials or workmanship for a period of one year from the date of purchase.

This warranty is void if:

1. The QuatBox™ has been subjected to unusual or preventable chemical exposure, or physical excess beyond that expected from normal 'wear and tear'.
2. The top cover of the QuatBox™ has been removed.
3. Attempts have been made to modify the instrument or use it other than in accordance with the instructions and recommendations contained in this manual.

If it is necessary to repair or service the QuatBox™ during the warranty period, please send an e-mail with details of the issue you are facing to the following address: ken@wilsonanalytical.com.

A representative of Wilson Analytical will contact you as soon as possible to discuss the problem and if necessary, issue a Return Authorization Number (RAN). The unit must then be shipped to us for repair at the following address:

Wilson Analytical Services Inc.,
#2, 215 Carnegie Drive, St. Albert,
Alberta, Canada. T8N 5B1.
Telephone: (780) 702-0610

The instrument will be repaired or replaced free of charge, but please enclose documentation that includes the RAN that was issued to you, your contact details and return shipping address, and a summary of the problems you have encountered with the unit.

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Appendix – Wilson Analytical QuatBox

Best Practices for Measuring Corrosion Inhibitor Residuals with the QuatBox

1. Keep the Sample Temperature Constant

It is important to realize that the amount of light emitted by any given sample (the fluorescence intensity) will depend on its temperature. The higher the temperature, the less light is emitted, even if the sample concentration remains the same. This temperature dependence means that it is only valid to compare concentration measurements obtained by fluorescence if they were obtained at close to the same temperature. This means that in practice, it is best to ensure that the samples are run within plus or minus 5 degrees C of the temperature at which the calibration curve was obtained at.

This is easy to do with a QuatBox™, as it contains heating and cooling that keep the sample at close to constant temperature, regardless of the ambient conditions. In order to keep an eye on the sample temperature, there is a digital Celsius *Temperature* display on the top panel of the QuatBox™. This display reads from an internal temperature sensor placed very close to the sample cuvette. In addition, the *Temperature* LED indicator on the front of the unit also monitors the temperature inside of the QuatBox™. It will only turn green (allowing the user to obtain measurements by enabling the light source) when the sample temperature is within a pre-determined range, which is 27 C +/- 5 C.

2. Keep the Sample Concentration in the Linear Range

The way to get the most accurate results from any analytical technique is to make sure there is a “linear relationship” between the concentration of the chemical that is being measured, and the output of the measuring device. In other words, if a concentration of 50ppm gives a signal, or response, of 1000 counts, a sample with 100ppm of the same chemical should give a response of 2000 counts. When this is true for a given system, we say we are working in the “linear range”.

Things are no different with fluorescence, and for most fluorescent chemicals, there is a natural range where a doubling of the concentration gives a doubling of the detector response. The exact range will depend on the chemical being used, but typically, concentrations below about 100ppm will give a linear response.

Above this natural “linear range”, what happens is that an increase in sample concentration gives less than the expected increase in detector response, and great care must be taken not to underestimate the amount of chemical truly present. This phenomenon is called “quenching” or “self-quenching” of the fluorescence.

As 100ppm is less than the required dose for many field chemicals, the phenomenon of quenching means that samples obtained directly from tanks, flowlines or other process equipment must usually be diluted before being measured. For example, if the expected concentration of chemical in a process

stream is 500ppm, a dilution of at least 10x, and perhaps 100x is necessary to ensure that measurements are being taken in the “linear range”.

The QuatBox™ uses an intense light source to make accurate fluorescence measurements possible down to 1ppm, or in some cases even lower. This means that sample concentrations of 100ppm (already on the edge of the linear range) can be further diluted by a factor of 100x if necessary and still measured accurately. This level of dilution has the additional great advantage of reducing matrix effects, such as extreme salinity, to insignificant levels.

3. Measure the Sample Concentration as Quickly as Possible

Chemicals that can be measured by fluorescence are detectable because they absorb light. Sometimes this light is in the visible range, but often it is in the ultra-violet, invisible to the naked eye, and of much higher energy. (It is over-exposure to ultra-violet radiation that causes sunburns.) The fluorescence process is very fast indeed, with the QuatBox™ collecting data in less than a minute, and this speed of measurement is an advantage over other techniques, such as dye extraction or chromatography, that typically take 30 minutes or more to complete.

However, the absorption of light, as well as making fluorescence possible, can destabilize the molecule being detected, and even cause it to chemically change and degrade. This is a much slower process than fluorescence (hours to days), but this photosensitivity means that samples that are stored in glass or clear plastic containers will exhibit lower and lower fluorescence signals over time. If the amount of light being received by the sample is uncontrolled (which is usually the case when samples are collected over time and then sent to a central laboratory), then the concentrations measured in those samples may be lower than the true (original) values.

Because the QuatBox™ allows the user to determine analytical concentrations to laboratory standards within minutes of obtaining a sample, any errors due to photosensitivity or light exposure are avoided completely.

4. Be Aware of any Interferences Present and Remove Them

Fluorescence is an optical analysis technique and is greatly affected by solids and oil droplets in the water samples being tested. Water samples must be thoroughly filtered prior to analysis by the QuatBox™. Coffee filters work well in the field, but the sample must be filtered repeatedly until no visible oil or solids are present, and the sample is completely clear. Use a fresh filter for each filtration for increased filtration efficiency, and to avoid sample contamination. Unless the original sample was full of oil, two filtrations are usually enough. A final filtration with a 0.25 or 0.45 micron syringe filter may be required as well if very fine solids or liquids are suspended in the original water sample.

Figure 19. Filtrating a Sample using a #2 Coffee filter, before testing

Many organic chemicals will absorb light and exhibit fluorescence, and sometimes there are naturally occurring substances in water samples that can mask or interfere with the fluorescence signal from the chemical of interest. This can happen for example when the water sample comes from an oil or gas producing formation, in which case the produced water can contain water soluble organic substances such as naphthenic acids, or polyaromatic hydrocarbons that have strong fluorescent responses.

The QuatBox™ is a full fluorescence spectrometer that shows the user the complete fluorescence spectrum of each sample. Even without being trained in fluorescence, it will be obvious to most users that the fluorescence spectrum from, for example, a corrosion inhibitor of interest, is the same time and time again; only the intensity of the spectrum varies with the concentration. However, if a fluorescent interference such as a naphthenic acid is present in the sample, the spectrum will look completely different, alerting the user to the contamination in the sample.

Once interferences such as naphthenic acids or polyaromatic hydrocarbons have been encountered, they must be removed from the samples before meaningful data can be obtained. The easiest way of doing this is to include an absorbent filter in the dilution step, such that the desired volume of sample passes through a solid phase extraction (SPE) cartridge, chosen to remove that specific interference.

Figure 20. Overview of the QuatBox™.

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