FLOPRO-TRACKER

ZONE FLUIDICS ON-BOARD ANALYZER FOR CHEMICAL OCEANOGRAPHERS

USER MANUAL

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SYSTEM DESCRIPTION

The FloPro-Tracker was developed by Global FIA and is depicted in Figure 1 to meet needs expressed by Chemical Oceanographers. It is intended to be a portable system for carrying out tried and tested wet chemical assays. It makes use of Zone Fluidics to carry out sample preparation steps followed by wet chemical analysis. Zone Fluidics is an approach to flow-based automation that has been pioneered by Global FIA (Marshall, Wolcott, & Olson, 2003). In Zone Fluidics one or more unit operations are positioned around the central fluidics engine. A Zone Fluidics sequence comprises of a series of fluid manipulation steps whereby small zones of fluids are sequentially presented to the various unit operations to process the sample or and in the case of an analyzer transform it into a detectable species, measure an analytical property, convert the analytical signal to concentration using calibration curves, and prepare the device for subsequent measurements. Zone Fluidics is increasingly being used to automate sample preparation steps.

The FloPro-Tracker comprises of:

a. One or more FloPro Zone Fluidics analyzers mounted in
b. A rugged portable 19” rack enclosure together with
c. A high-brightness LCD display and integrated keyboard and mouse and coupled to
d. An automated self-cleaning filter sampler and controlled by
e. FloZF device control and data acquisition software installed on
f. Intel NUC computers which are mounted on the FloPro Analyzers.
g. GPS receivers installed on each FloPro provide the geographical location of the device during sampling and measurement

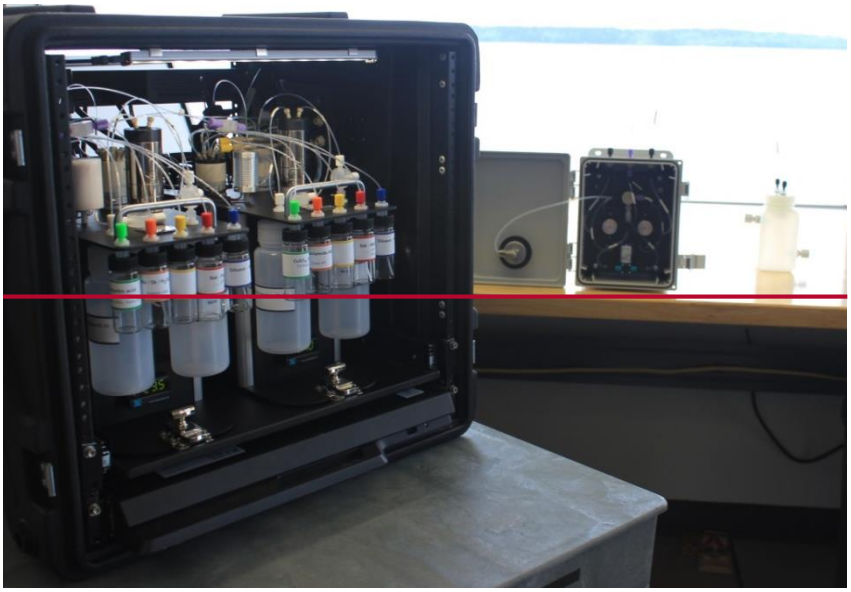

Figure 1: FloPro-Tracker with Sampling System

Two FloPro analyzers can conveniently be installed on a sliding shelf in the FloPro-Tracker rack. These analyzers are easily removed from the rack and can be used independently outside of the rack by connecting them to a standard LCD screen, keyboard and mouse.

Figure 2: FloPro Analyzers installed in the FloPro-Tracker 19" rack

The basic FloPro Analyzer is equipped with a milliGAT pump and two multiposition selection valves. In most instances, detection is spectrophotometric in which case the FloPro is equipped with an Ocean Optics spectrometer and bubble-tolerant flow cell. Other detection techniques such as fluorescence spectrometry, chemiluminescence, and certain electrochemical detection techniques have also been coupled to FloPro analyzers. Some chemistries benefit from heating steps and in these cases a heated reactor is included in the FloPro manifolds. Other unit operations such as cadmium columns for nitrate determination or a uv digester to digest organically bound analytes can also be coupled to the Zone Fluidics manifold. Where needed, additional unit operations can be added or developed to meet the requirements of a particular assay.

Figure 3: FloPro equipped with milliGAT pump, multi position selection valve, heated reactor, cadmium reactor (for NO_3 determination), spectometer and flow cell, GPS and miniaturized NUC computer

The 19" rack is equipped with a rack-mounted high-intensity LCD screen to allow for outdoor use. A keyboard and pointing device is integrated into the screen's 1U shelf. A KVM (keyboard, video, and mouse) switch allows switching of the display between the NUC computers that drive the two FloPro-Analyzers. All devices in the rack are powered from a single 100-250VAC power strip.

Figure 4: FloPro-Tracker with pull-out screen, keyboard and pointing device

The sampling system is a patented self-cleaning filter system where the sample probe is equipped with two filter elements. A pump circulates sample from the one filter element through a set of valves and back out through the second filter element. In the process, the second element is back-flushed. Periodically a timer switches the valves and the role of the two filter elements is swapped so that the element previously filtering is back-flushed and the back flush element begins filtration. A de-bubbler provides a bubble-free sub stream for measurement. The flow rate of the pump is adjustable as is the frequency with which the valves are switched. Several filter probes have been developed to meet a range of application areas. These are described in more detail below.

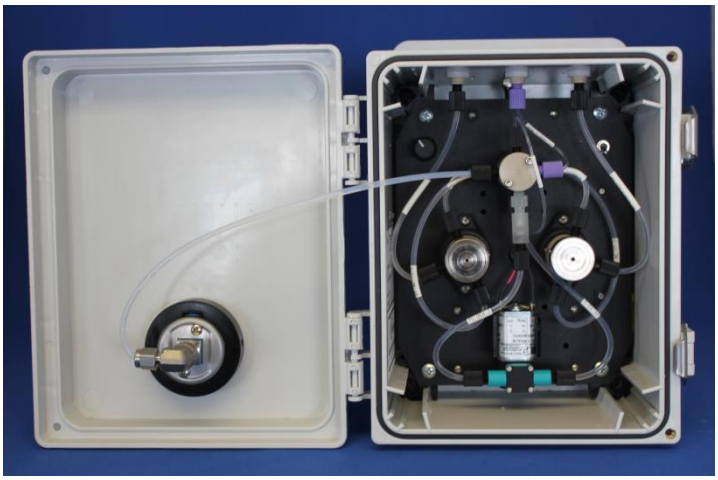

Figure 5: Sampling system control module

The FloZF software is a full-feature device control and data acquisition and manipulation package with support for a range of unit operations, detectors, and sampling systems. Devices are controlled in scripted sequences. Data are acquired from detectors and sensors and are presented in both tabular and graphic form. Quantification of the analyte is by means of calibration and is handled automatically by FloZF. Data can be exported to CSV files or an Excel spreadsheet. Individual results are time stamped and when the system is equipped with a GPS, they can also be plotted on a Google Earth map.

The software is installed on an Intel NUC computer which is mounted on the side of the FloPro. This compact computer is equipped with a 4th Gen Intel i5 processor and Windows 7 operating system. It's built in Wi-Fi means that the computer is accessible wherever Internet connectivity is available.

Figure 6: Intel NUC computer mounted on FloPro

The Globalsat USB GPS receiver streams data in standard WGS-84 format which is easily converted to longitude and latitude and can be plotted on a Google Earth map (requires internet connection to acquire initial map space). If an Internet connection is not available, the data are plotted on a x-y grid scaled to the far extents of the location space of the chosen data set.

Figure 7: BU-353 GPS receiver

ZONE FLUIDICS

Zone Fluidics (ZF) is the precisely controlled physical, chemical, and fluid-dynamic manipulation of zones of miscible and immiscible fluids and suspended solids in narrow bore conduits to accomplish sample conditioning and chemical analysis.

A *zone* is a volume region within a flow conduit containing at least one unique characteristic.

From an operational point of view, ZF is an approach to sample handling where a zone or zones are shuttled between and within an assembly of one or more unit operations where different sample processing steps are performed. Fluid handling is accomplished in a fluidics manifold like the one depicted in the following schematic.

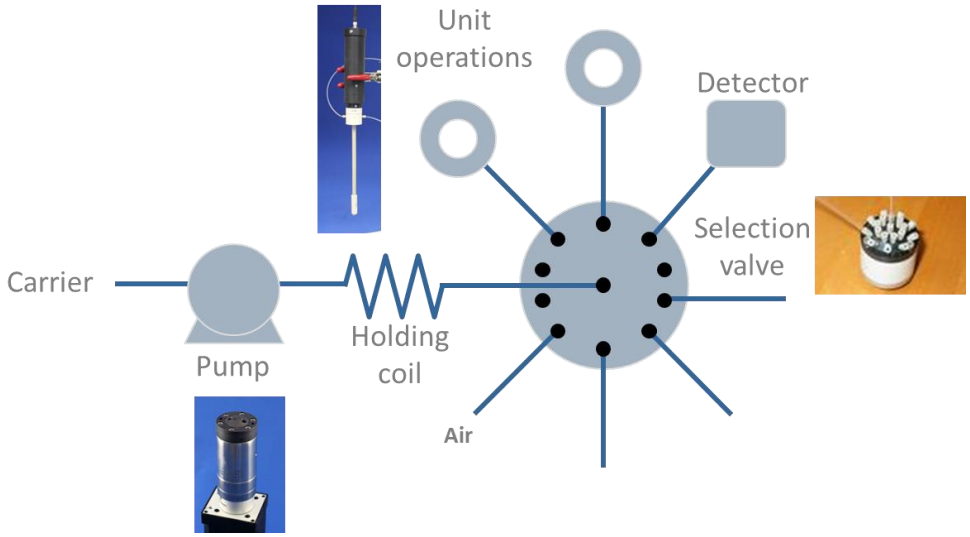

Figure 8: Zone fluidics as a versatile sampling interface

The pump and selection valve are key components in a ZF manifold. Zones of sample are aspirated into the holding coil. The selection valve port is then switched, allowing these zones to be transported via the pump to one or more unit operations.

Unit operations are hardware devices that transform the sample in one way or another and may include sample probes, heaters, separators, reactors, mixers, fraction collectors and detectors to name just a few. Unit operations may be employed for pre-analysis sample preparation or manipulation of the sample to form a detectable compound. The operation of a unit operation in a particular measurement sequence is controlled from the FloZF device control and data acquisition software.

Frequently, one port is left open to air so that air zones can also be included in the zone stack. These air zones can be used to prevent dispersion of one zone into another. Taylor flow, which results in thorough mixing, occurs when zones bracketed between two bubbles are pumped through tubing. Bubbles are also used to empty a tube of its contents.

FLOPRO-TRACKER FLUIDICS MANIFOLD

The specific fluidics manifold for the FloPro-Tracker is given in the respective method files for the chemistries deployed on the FloPro analyzers. Figure 9**Error! Reference source not found.** provides an example manifold and is used to describe typical operation. The pump moves fluids that are selected and controlled by the two multi-position selection valves (SV1 and SV2). The pump is protected from being over pressurized by the pressure relief valve. Chemical reactions take place as the zones of reagents drawn from vials connected to one of the selection valves are shuttled in and out of the holding coil and to and from unit operations such as the cadmium reactors depicted in this manifold. In some cases a heated reactor is included as a unit operation and sometimes the

holding coil is heated to accelerate reaction rates. Once a detectable species has been formed, the zone stack is flowed through or positioned in the flow cell of the detector where analytical parameters are measured. Waste solutions are pumped to the waste reservoir. Calibration standards are prepared *in-situ* by appropriate dilution of a stock calibration standard. Sample is drawn in via a sampling probe or from a direct coupling to an automated sampling system.

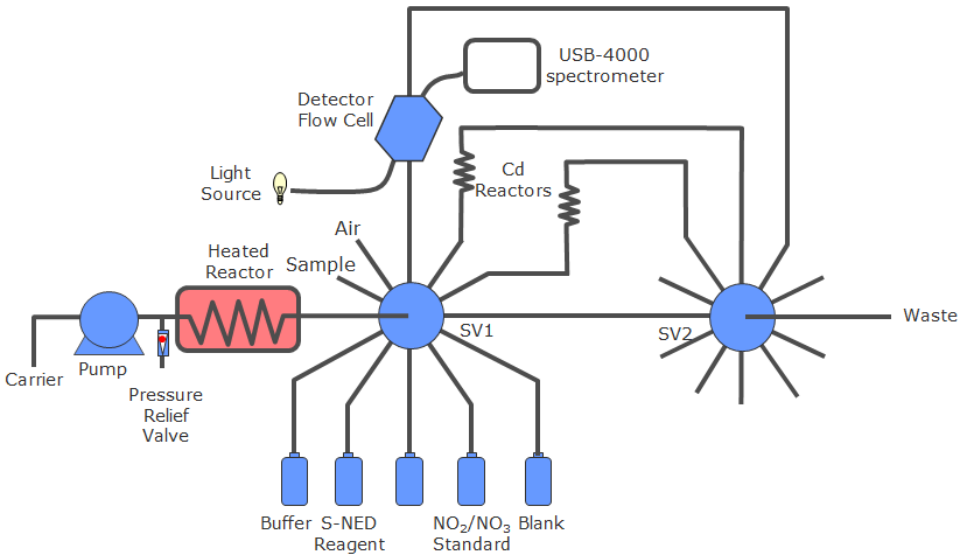

Figure 9: FloPro ZF manifold deployed in FloPro-Tracker

FLOPRO ANALYZERS

MILLIGAT PUMP

The microprocessor-controlled milliGAT™ pump (Wolcott & Marshall, 1996) is a positive displacement linear flow pump that consists of the pump head / motor / gear assembly, and an integrated micro-electric controller.

The milliGAT features that are of particular importance for the FloPro-Sampler include:

- Bi-directional smooth flow
- Flow range from 0.1 nL/sec – 167 µL/sec (6 nL/min to 10 mL/min)
- Meters precise volumes from nL – mL
- Excellent precision (± 12nL for 250 nL dispenses)
- No pulse-dampeners
- No check valves
- No syringes to re-fill or exchange
- Chemically inert
- Self-priming

To obtain good precision, the following flow rate guidelines should be followed.

Table 1: Flow rate guidelines

Action	Suggested Flow Rate
Aspirate and dispensing through the holding coil and other reactors	<15µL/sec
Metering reagents and calibration standards	5µL/s – 15µL/sec
Dispensing analyte to and through the flow cell	<20µL/sec
Flushing the flow cell	75-100 µL/sec
Flushing Mixing Coil	50µL/s – 170µL/s

MULTI-POSITION SELECTION VALVE

The multi-position selection valve (see Figure 10) selects one of 10 or 18 streams arranged in a circle around a central common port, with the remainder of the lines dead-ended. These valves are coupled to the same motor with integrated controller used for the pump and an embedded algorithm controls port selection. This valve is able to withstand pressures up to 250 psi (~17 bar) and has a chemically inert wetted flow path. The rotor flow path contains no unswept volumes and is scaled to match channel dimensions. The red channel in Figure 11 (for an 8-port valve) shows the channel machined into the rotor of the valve.

Figure 10: 18-port selection valve (left behind pump) and milliGAT pump (front), and 10-port selection valve (right)

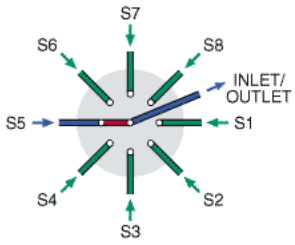

Figure 11: Flow path for the multi-position valve (only 8 ports are shown)

PRESSURE RELIEF VALVE

The FloPro-Tracker is equipped with a pressure relief valve immediately downstream of the pump. This valve provides protection for the pump in the event of a blockage or sequence programming error. If the pressure in the system exceeds 100 psi (~ 7 bars) this valve opens and the fluid is pumped to waste. To restore normal operation, the source of the blockage must be eliminated.

HEATED REACTOR AND HOLDING COIL

Several designs of heated reactor have been developed for the FloPro. In the case of the FloPro-Tracker the holding coil is made from an aluminum cylinder that is heated by means of a heater cartridge. The holding coil tubing is wound around the heated block. Where the chemistry is particularly heat sensitive an insulation layer is added and a cover holds the insulation in place. A thermocouple tracks the temperature of the reactor and a PID temperature controller maintains the temperature at the desired set point. Where long reaction times are needed, the flow is stopped in the holding coil for the required time. The temperature controller is located below and behind the carrier and waste reservoir (indicating a temperature of 23.4 °C in Figure 12).

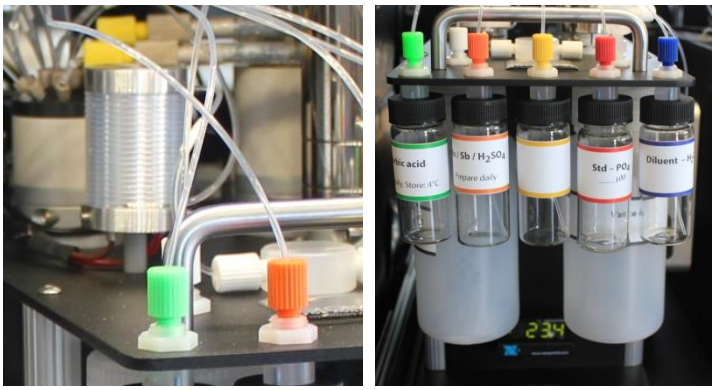

Figure 12: Heated holding coil (left) and temperature controller (right)

Some measurement chemistries such as phosphate are particularly sensitive to temperature. In these cases it is a good idea to auto tune the PID parameters at the desired set-point under the operational conditions. A sequence called **Temp AutoTune** is provided to automatically carry out this process. Parameters in this sequence can be adjusted. Note in order for auto tune to work, the present heater temperature must be at least 10 °C less than the set point. This sequence temporarily changes the set point to 20 °C and then waits for the temperature of the heater to cool to 10 °C below the desired set point before initiating the auto tune.

CADMIUM REACTOR

The cadmium reactor is constructed from a ¼ inch OD 0.190 inch (4.8mm) ID PFA plastic column with PEEK end fittings. The end fittings have a 0.030 inch (0.8mm) bore hole and ¼-28 female threads. The column is packed with

cadmium granules (Sigma P/N: 00623-50G) with a particle size of 0.3-1.6 mm. The packed portion has a length of 25 mm and a void volume of approximately 250 µL. The system is equipped with two reactors so that if one becomes contaminated it is easy to swap over to the other. In the main sequence the port of the selected reactor is assigned to the variable **CdReacPort** and can have a value of 9 or 10. It is important to keep the cadmium reactor filled with buffer solution when not in use for short periods of time (<2 weeks). If the system will be unused for a greater length of time then it is better to flush the column with DI water and then air dry it. When it is used again, it must first be reactivated before use.

If in the course of use, the reactor is exposed to air it may be necessary to re-activate the cadmium to obtain complete conversion of the NO_3 to NO_2. Activation of the column is handled in a semi- automatic manner by a sequence called **Activate Cd Reactor 1** or **Activate Cd Reactor 2**. Cd reactor 1 is plumbed to port 9 on the two valves and Cd reactor 2 is plumbed to port 10 on both valves. These sequences step through the steps needed to activate the columns. At each step the user is prompted to attach a vial containing the required regeneration solution. Four solutions are required: 6N HCl, DI H_2O, 2% $CuSO_4$, and NH_4Cl buffer. These solutions are aspirated and dispensed to the appropriate reactor. The preparation of the regeneration solutions is described in the nitrate / nitrite method. After activation, the cadmium granules will appear to be black in color.

If a brown precipitate begins to accumulate in the reactor, then the column should be removed and the precipitate should be flushed from the column using a syringe filled with DI water. This precipitate contains cadmium and should be treated as toxic waste. With time the cadmium will be consumed and when more than 20% of the column material has been lost then one end piece can be removed so that additional cadmium granules can be added. Heating the plastic column in boiling water will allow the end piece to be removed.

Figure 13: Cadmium reactor before activation

LED AND TUNGSTEN LIGHT SOURCE

FloPro analyzers are typically equipped with two light sources to cover the full spectral range of the spectrometer; a white LED powered by a constant current power supply, and a miniature tungsten – xenon lamp powered by a constant voltage power supply. The output from these two sources is blended using a bifurcated optical cable which is coupled to the inlet end of a bubble-tolerant flow cell. The path length of the flow cell is approximately 12 mm. The exiting light is channeled to the spectrometer in a single optical cable.

BUBBLE-TOLERANT FLOW CELL

The bubble-tolerant flow cell has been designed specifically to allow the efficient passage of bubbles through the flow cell. A common problem with photometric flow cells is that bubbles, which inevitably enter a fluidics manifold, are trapped in the flow cell and prevent the acquisition of analytically useful data. The user is left tapping the side of the cell in an attempt to knock the bubble loose.

Three principals were applied to promote passage of the bubbles through the cell:

1. Bubbles, by virtue of their buoyancy, have a tendency to move up
2. Flow rate is proportional to the cross section of the fluid channel; a small cross section means higher flows and a large cross section means slower flows. Higher flows imply greater shear forces which dislodge bubbles.
3. Bubbles tend to adhere to hydrophobic surfaces and conversely, hydrophilic surfaces readily shed bubbles.

The use of a non-ionic surfactant such as Capstone FS-31 (Du Pont) is crucial to ensure that all surfaces are wetted and rendered hydrophilic. A trace (0.01%) of Capstone FS-31 should be part of the carrier stream composition whenever this is possible. When the chemistry or other fluidic components preclude the inclusion of a surfactant in the carrier, the bubble-tolerant flow cell should periodically be conditioned overnight with a wash solution containing 0.01% of Capstone FS-31 in DI water.

USB 4000 SPECTROMETER

The USB 4000 features a 3648-element Toshiba linear CCD array and 16 bit A2D converter for increased signal-to-noise and enhanced electronics for acquiring spectra and photometric data. FloZF supports rapid data acquisition, plotting of spectra as well as absorbance verses time profiles. Multiple wavelengths can be followed and simple arithmetic operations can be carried out on response data. The specific optical bench typically employed in FloPro has the following characteristics:

- VIS region windows,
- no entrance slit,
- no long pass absorbing filter,
- standard mirrors,
- #3 grating (350-850nm)
- L4 - detector collector lens

REAGENT VIALS AND RESERVOIRS

The carrier and waste bottle are Nalgene 8 oz. (250 mL) LDPE bottle with no cap liner (P/N: 2103-0008[1]). The reagent and calibration standard vials are 6 dram (25 mL) glass vial with phenolic caps and PTFE cap liner (P/N: CT242767-25[2]). The caps are fitted with CTFE universal vented vial holders to facilitate connection to tubing and bulkhead mounting. A set of color coded vinyl labels is provided for the reagent vials that match the color of the tubing fittings.

Figure 14: Reagent vials and reservoirs

GPS

The Globalsat USB GPS receiver (PN: BU-353[3]) includes the latest SiRF Star III GPS chipset and an active patch antenna to ensure a high degree of GPS accuracy. The receiver has a fast time to first fix and a flashing red LED on the underside of the receiver indicates that a fix has been made. It supports NMEA 0183 data protocol. Data are streamed in standard WGS-84 format which is easily converted to longitude and latitude and can be plotted on a Google Earth map (requires internet connection to acquire initial map space). If an Internet connection is not available, the data are plotted on an x-y grid scaled to the far extents of the location space of the chosen data set. FloZF supports data acquisition from the GPS receiver and allows results to be tagged with the GPS location as well as a time stamp.

[1] http://www.thermoscientific.com/en/product/nalgene-wide-mouth-ldpe-bottles-closure.html

[2] http://www.discountvials.com/6-dram-glass-vial-w-cap-pkg-of-25/

[3] http://www.globalsat.com.tw/products-page.php?menu=4&gs_en_product_id=2&gs_en_product_cnt_id=28

POWER

The FloPro makes use of a switch-mode power supply to provide 24VDCand 5VDC power for the various devices. This means that it can be powered by AC line voltage in the range 100-250VAC. The power socket is equipped with 0.5Amp slow-blow fuses.

USB PORTS

Communication with the FloPro-Sampler is via a USB 2.0 port. The FloPro is also equipped with a USB hub. The hub is used to provide additional USB ports for peripheral devices such as the USB 4000 and GPS receiver. Devices on the FloPro make use of virtual COM ports on the USB hub. The driver for these ports can be downloaded from FTDI[4].

NUC COMPUTER

The NUC computer from Intel is mini-PC equipped with a 4th generation i5 Intel processor and an Intel HD 5000 graphics interface. Connection to a LCD screen is via a mini-HDMI connector. Two 4 GB DDR3 RAM chips have been installed. Furthermore, the NUC has been fitted with an Intel Network 7260.HMWG WiFi Wireless-AC 7260 H/T Dual Band Wi-Fi card and a 120GB mSATA internal solid state drive. Windows 7 home premium 32 bit operating system has been installed. Its small 117mm x 112 mm x 50 mm dimensions allow for mounting this mini-PC on the side of the FloPro.

Figure 15: NUC computer and power supply mounted on the side of the FloPro analyzer

[4] http://www.ftdichip.com/FTDrivers.htm

FLOPRO-TRACKER ENCLOSURE

LCD SCREEN, KEYBOARD AND POINTING DEVICE

The keyboard, LCD screen and pointing device are housed in a 1U pull out shelf in the FloPro-Tracker 19" rack enclosure. To lift the screen slide out the drawer. Then clasp the screen on the right side and lift it up.

Figure 16: Viewing the screen

The power switch for the screen is on the LCD membrane just below the LCD screen – refer Figure 17. Touching the membrane will light up the backlight for the membrane switches.

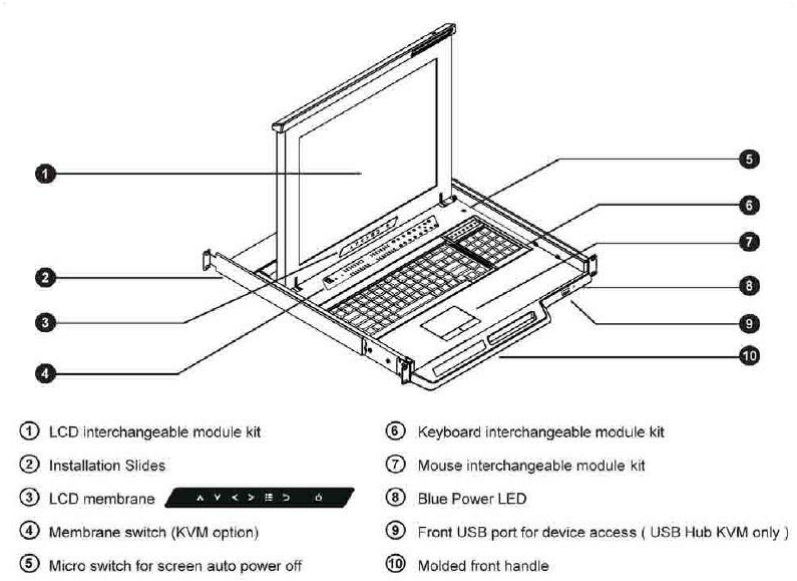

Figure 17: Screen drawer diagram

The video input to the screen comes from the console screen connector on the KVM switch by means of an HDMI cable. The HDMI cable is equipped with an HDMI to DVI adapter on the KVM switch end. **Under normal conditions it should not be necessary to unplug the HDMI cable. It is a good practice to power down the system before unplugging the HDMI cable.**

The keyboard and pointing device cable connects to an all-in-one 15-pin connector on the 1U shelf. This cable plugs into the console USB connector designated with a keyboard icon on the KVM switch.

To slide the drawer back into the 19" rack, there are two latches on each side of the screen drawer (see Figure 18) that must be slid forward as the drawer is slid back.

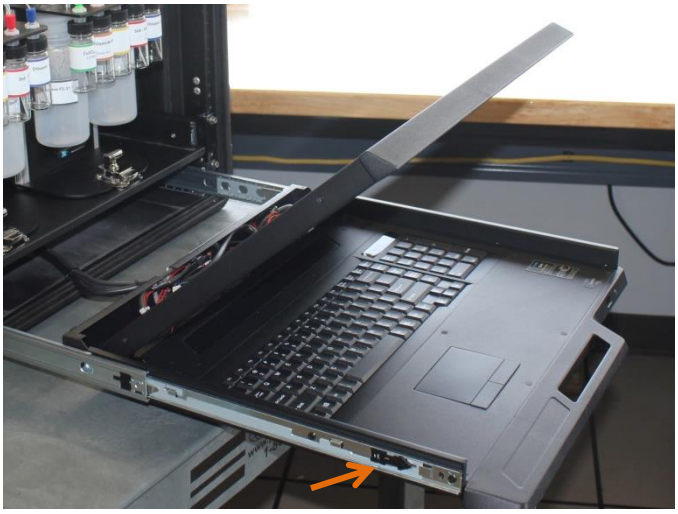

Figure 18: Drawer release latches for LCD screen drawer

To clean the screen, gently wipe the surface using a dry micro-fiber cloth. Use as little pressure as possible.

Take care not to spill liquids on this device as it contains no protection against liquid spills. If there is a danger of splash or rain, then this device is vulnerable.

ANALYZER SHELF

The two FloPro analyzers are mounted on a pull out shelf that incorporates a drip tray. The analyzers have two tabs at the back which slide in under a receiving bracket at the back of the shelf. First slide the analyzer in as far as it will go. The latch should line up with its receiving catch on the front side of the analyzer. By firmly pressing down on the front of the analyzer base plate in the vicinity of the latching mechanism, the analyzer is fastened to the shelf. Twisting the fastening mechanism knob releases the latch and the analyzer can slide forward and after disconnecting all of the wires it can be lifted out of the 19" rack enclosure.

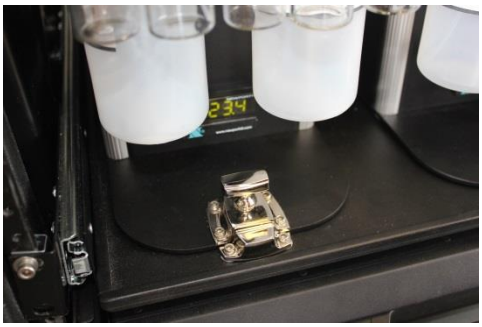

Figure 19: Analyzer latch

The shelf is held in a closed position by a shelf latch on the back right side. Flipping this latch over allows the shelf to slide out.

Figure 20: Analyzer shelf latch

KVM SWITCH

The KVM switch allows the two NUC computers to connect to a single keyboard, video and mouse. Switching between computers is achieved by pressing the Scroll Lock key twice and then the Enter key.

POWER ENTRY AND DISTRIBUTION

The NUC computers have their own switch mode power supplies as does the KVM switch and LCD screen. The FloPro analyzers also have their own internal power supplies. All are plugged into a 1U power strip accessible on the back side of the analyzer by removing the back cover. The power strip is wired to the power and sample entry module which is mounted in the side of the FloPro-Tracker enclosure (Figure 21). This module is covered with a cover plate when in transit.

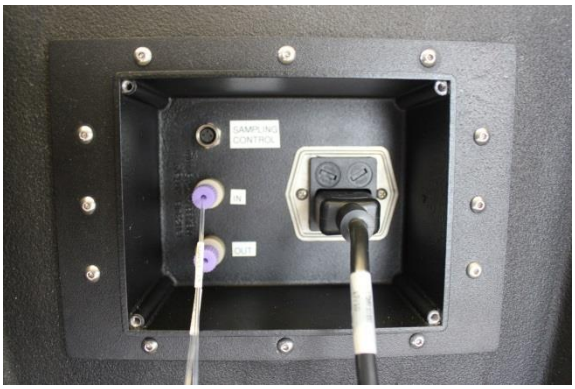

Figure 21: Power and sample entry module

SAMPLE ENTRY

The sample in and out bulkhead fittings are located in the power entry module. Tubing is not terminated in these bulkhead connectors but passes through them to eliminate the possibility of leaks at this point. In transit the sample in and out tubes are rolled up and stowed in the power entry and sample entry module.

SAMPLING SYSTEM WITH SELF-CLEANING FILTER

A common approach to automated sampling employs a fast loop that brings unprocessed sample solution to the analyzer where a series of filtering, de-bubbling, and other sample conditioning steps are carried out. This approach frequently is prone to clogging and a requirement for frequent scheduled maintenance on the filter element.

The Global FIA sampling system eliminates these problems. Filtration is carried out at the sampling point so that filtered sample solution is transported in the sampling manifold. Also, by using two filter elements, one filter element is being back-flushed while the other is filtering. Each filter therefore has a 50% duty cycle. The other 50% of the time, it is being back-flushed. Periodically the respective role of the two elements is reversed. Fresh filtered sample is carried close to the analyzer thus reducing lag times. An in-line de-bubbler provides bubble free sample solution to the analyzer. The FloPro analyzer draws sample from this de-bubbled sample point.

A compact sample control system manages the stream switching and pumping of the sample stream. This control system can be controlled by the analyzer but can also work independently of the analyzer. Several filter geometries have been developed to meet a range of application requirements. When not in use the sampling system should be pumped dry and the filters inspected for damage. Where necessary, repair, replacement, or cleaning should take place. To preserve pump life-time, the pump should be stopped when the analyzer is not operational.

PRINCIPLE OF OPERATION

During sampling, one of the elements of the dual filter (FIL) is the inlet to the sampling system while the other is the outlet. Two solenoid valves (V1 and V2) provide a means of switching the flow periodically so that the roles of the two elements are exchanged. The timing of this switch is dependent on the particle load and nature of solid component in the sample stream and is user-selectable. The following diagram depicts these two situations.

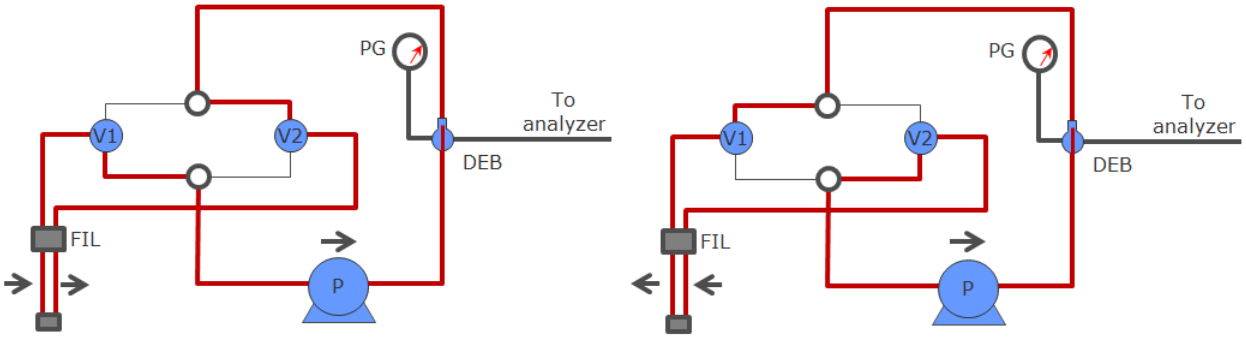

Figure 22: Flow through the self-cleaning filter and its control manifold

The diagram on the left of Figure 22 shows the fluid circulation path with the valves in one position. In this configuration, process liquid enters one filter element through the first valve (V1) under combined process pressure and pump (P) suction, then the filtered liquid passes through a gravity bubble separator (DEB), and back through the second valve (V2) to the second filter element, through which it returns to the process stream. Thus, while one filter element is filtering the sampled stream, the other is being back-flushed by the filtered solution returning to the sample source. A fraction of the filtered stream is removed at the bubble separator to supply the analyzer.

The figure on the right of Figure 22 shows the fluid circulation path with the valves switched to their second positions. In this configuration, sample solution enters the second filter element (which had been back-washed during the first cycle by the returning clean sample solution) and is pumped through the valve (V2), pump (P) and gravity bubble separator (DEB), and is returned to the process via the first valve (V1) and through the first filter element — back-washing it of any filter cake formed in the first cycle.

Flow through the pump and gravity bubble separator is thus uni-directional, while flow through the two filter elements is bi-directional, with each filter element spending 50% of the total duty cycle being back-washed. The frequency of switching of the valve is configurable between 0.1 second (5 minutes would be a realistic minimum) and 10 hours. It is common to switch the valves every 15 minutes.

SAMPLING SYSTEM CONTROL UNIT

The sampling system control unit comprises of a diaphragm pump (P), two solenoid valves (V1 and V2), a timer and a PWM power supply to regulate the pump speed. A pressure gauge provides an indication of how well the sampling system is working. The sampling system operates independently of the analyzer but ensures that there is always a fresh sample at the sampling point when the analyzer needs it.

A toggle switch provides an on-off switch for the control unit. A speed dial allows control of the pump flow rate. When the system is powered, it takes a while to flush all bubbles from the system. If bubbles persist then all fittings should be tightened to eliminate leaks. It may also be necessary to replace or repair the filter element if the bubbles originate at the filter element. Occasional bubbles should not have an adverse effect as the de-bubbler will pass bubbles and keep them away from the sampling point.

The timer is located behind the fluid mounting plate in the control unit. To change the timing of the valve switching:

- Disconnect the power to the control unit.
- Disconnect the tubing to the sampling point and two filter elements inside the control box.
- Remove the four mounting plate screws.
- Carefully lift up the mounting plate being careful not to put strain on the wires supplying the control unit.
- Adjust the pulse (P) and range (R) settings on the timer board which is located at the bottom of the plate (see Figure 23). The switch times (T1 and T2) for the two valves are set by range jumpers (R) that select hours (h) , minutes (m) or seconds(s)) and the pulse length dials (P) which select a time between 1 and 60.
- Setting shown below if for approx. 15 minutes.

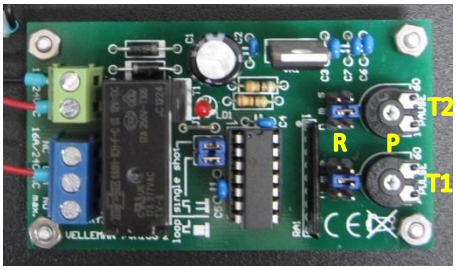

Figure 23: Timer control board

Some back pressure is required to force the sample through the loop to the analyzers. A pressure of 5 to 10 psi gives a steady flow through the analyzer loop. Some adjustment can be made to the back pressure by employing 0.030 inch (0.8 mm) ID tubing on the outlet from the de-bubbler instead of the 0.062 inch (1.6 mm) ID $^{1}/_{8}$ inch (3 mm) OD tubing used in the rest of the sampling manifold.

The pump speed is set so that the pressure indicated on the pressure gauge is between 5 and 10 psi. You can extend pump life by reducing the flow rate through the pump.

If there is a big difference is pressure readings (>5 psi) for the two switch states then examine the tubing for blockages or the filter elements for damage or fouling.

Monitor the flow of sample out of the sample waste from the analyzer enclosure to ensure that the sampling system is delivering fresh sample to the analyzers.

SAMPLE PROBES

To obtain the maximum possible flow through the filter elements, a conditioning period is usually necessary (especially for filters with porous Teflon filter elements). The initial conditioning can be accomplished by simply immersing the filter probe active portion in clean solvent chemically similar to the material to be sampled (water for aqueous streams), and simply running the system for a period of time (a couple of hours has been sufficient in most cases). For permanent installations, installation in the process and simply running the system overnight will probably be sufficient. As the filter elements become fully "wetted out", the backpressure indicated on the pressure gauge will drop and the pump flow rate can be reduced. You can help wet out the filter by storing it in a surfactant solution when not in use. Just pumping sample through the system for several hours will eventually wet out the filter elements.

Several filter geometries have been developed and are shown in Figure 24. Probe A has two 8 inch (200 mm) porous tubular filter with a pore size of 25 ± 10 µm. Probes B will accept any circular 1 inch (25 mm) filter disk. The two semi circles visible are separated internally by a barrier and provide the two filter elements in a single filter disk. Probe C comprises of two filter elements and will accept 0.5 inch (12 mm) filter disks or porous UHMWPE frits (15-45 µm pore size). In the figure one filter is shown without filter element installed (L) and the other (R) has the frit installed.

Figure 24: Filter probes

SCHEDULED MAINTENANCE

Maintenance schedules can only be determined with experience gained from use with the particular sample stream. The typical manual maintenance may involve removing the probe from its mounting, and inserting the filter elements into a vessel containing a suitable solvent for the major filtrate constituents, then circulating the solvent under action of the sample loop pump through the entire sample loop until the indicated pressure drop returns to a satisfactory level. Depending on the sample, this step should only be necessary between once a month and once a year.

For certain applications it may be necessary to remove bio-fouling or precipitated salts in the filter element by soaking the filter in bleach or acid. For filter probes with replaceable filters, the filter element can simply be changed out if it begins to foul.

When not in use the sampling system should be flushed of old sample to avoid clogging with precipitated salts.

SYSTEM SETUP

UNPACKING

1. Unpack the shipping containers and check for shipping damage. If there is any shipping damage, do not discard the packaging and contact Global FIA. Compare the contents with packing list and notify Global FIA immediately of any shortages.
2. A video detailing assembly instructions is available from Global FIA.

FLOZF SOFTWARE

FloZF is a device control, data acquisition, and data processing package designed for the development and operation of fluidic systems such as the FloPro-Tracker. It includes functionality to carry out automated calibration and continuous measurement of sample from a sampling point. Results are stored and include peak characteristics, concentration, spectra and certain diagnostic information. Method projects are provided for the assays deployed on the FloPro analyzers. **Projects** consist of hardware **Device** configuration information, the **Sequences** to control the devices, and data acquired from any detector devices in the Project. Full details of the software are available in the FloZF User Manual available from Global FIA.

RUNNING METHODS

Before the method is executed it is necessary to:

- Fill the Carrier reservoir with carrier solution (refer to the method description for the appropriate carrier solution.
- Ensure that the waste reservoir is empty

- Load the vials with reagents, standard, and blank solutions.
- Start up the sampling system and ensure that sample is being delivered to the analyzer by observing the pressure on the pressure gauge of the sample control unit and by looking at the outflow at the sample out tube.
- Execute the **STARTUP** sequence.
- Evaluate the reference spectrum to ensure that the detection system is working correctly.
- Execute the assay method sequence and select calibrate.
- Execute the assay method sequence and select sample. Provide a sampling run name and number of measurements you will carry out.
- Execute the **SHUTDOWN** sequence.
- Remove reagents and standards and empty the waste reservoir.

METHOD SEQUENCE

Once a project has been loaded, the method sequence is executed by dragging it from the Resource Sequences panel on the Develop tab to the Sequence / Step Name field and then pressing the Run button .

FloZF steps through the sequence controlling devices and acquiring and processing the data.

HOUSE-KEEPING SEQUENCES

In addition to the method sequence, there is also a **STARTUP** and Shutdown sequence. As their names imply, these sequences are used when starting up and shutting down the instrument.

The **STARTUP** carries out a variety of steps in order to prepare the system so that samples can be measured. Steps to prime reagent lines, flush the holding coil and reactors, set the heater temperature and prepare the detector for measurement are contained within this sequence.

The **SHUTDOWN** sequence carries out a series of steps to wash out any remaining reagents or sample from the tubing lines and fill these lines with clean carrier. It also sets the set point in the heater to normal ambient temperature and switches off the spectrometer light sources.

AUTOMATED MEASUREMENT

While it is possible to run a method from the **Develop** page, it is preferable to run from the **Measure** page (refer to Figure 25).

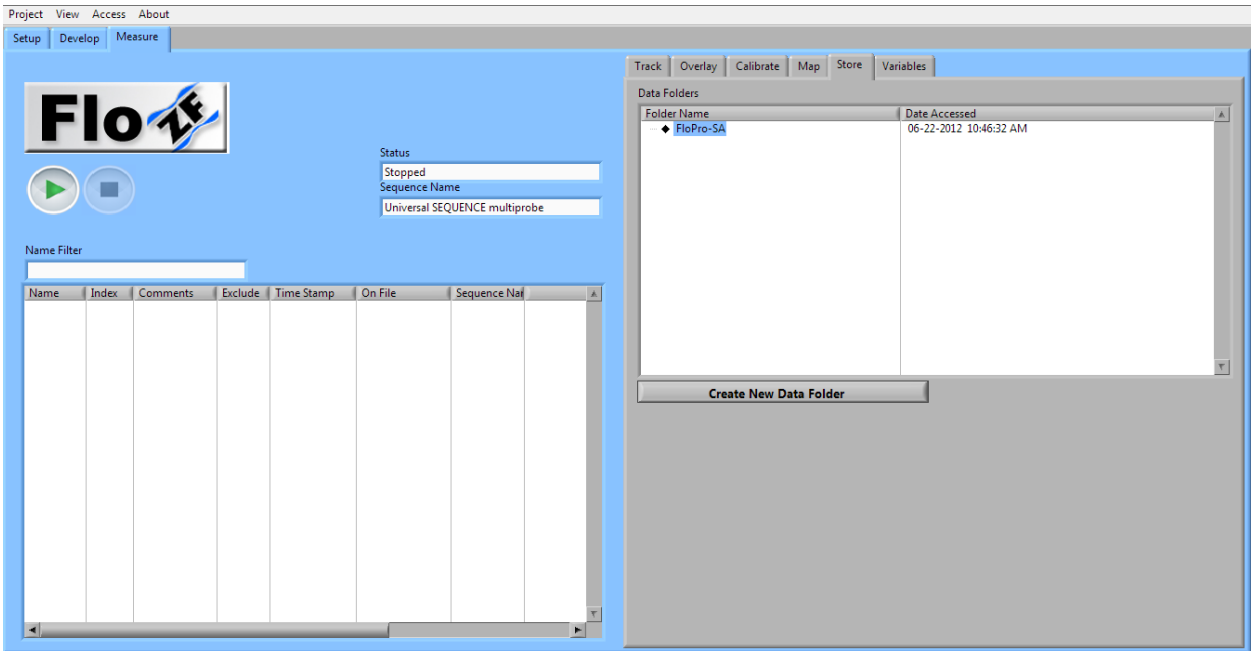

Figure 25: FloZF Measure page

The **Sequence Name** field specifies the sequence that will be executed when the Run button is pressed. You can add sequences from the **Resource Sequences** List by right clicking the Sequence name field to pop up a **Modify List** menu option

The **Name Filter** field allows you to just show samples in the data file whose sample name match the **Name Filter** mask**.**

The **Status** field shows the run status of the selected sequence.

A **Run** and **Stop** button are used to execute or stop a sequence. The **Run** button transforms into a **Pause** button while the sequence is running.

Figure 26: RUN STOP and PAUSE buttons

The **Create New Data Folder** button in the **Store** tab of the **Measure** page provides a means of creating new data folders. It is a good idea to create a new data folder for each sampling campaign.

The data folders for the current project are listed in the **Data** Folders field where it is possible to swap to a different data folder, sort data folders alphabetically or by date, or delete whole data folders by selecting a Data folder and pressing **Ctrl-Delete**. **Note –** you cannot recover a data folder once it has been deleted.

ACCESSING DATA

RESULTS TABLE

MEAUREMENT PROFILES AND SPECTRA

CALIBRATION DATA

EXPORTING TO EXCEL

SYSTEM SHUTDOWN AND TRANSPORT

APPENDIX A: TROUBLESHOOTING TIPS

CLEARING BLOCKAGES

Occasionally blockages occur within the system. If there is a blockage in the system, carrier fluid will drip from the pressure relief valve (open to the bench top). After identifying the location of the blockage, disconnect the fitting to this port as well as the tubing routed into the selection valve common port. Manually select the blocked port number from the instrument setup tab in FloZF. Attach a short line of tubing (equipped with the correct fitting) to a syringe and connect the syringe to the common port of the selection valve via this line. The syringe should be filled with distilled water. Pushing distilled water through the blocked port via the common port should effectively clear the blockage.

BUBBLES FRAGMENTING

Bubbles are frequently used to separate a zone stack from the carrier solution and prevent dispersion. For instance, bracketing a zone stack with bubbles for detection purposes with a spectrometer enables a higher degree of precision to be achieved. However, bubble fragmenting can negatively affect one's data and are therefore undesirable. Fragmented bubbles within the tubing can be explained by a few different possibilities, which are typically quite easily corrected. When flow rates are too high, bubbles tend to fragment. If bubble fragmenting is observed while running at a high flow rate, simply decrease the flow rate for the particular step that is resulting in bubble fragmenting and observe whether the problem persists. Bubble fragmenting also occurs when the tubing is not properly wetted. Washing the tubing line with IPA followed by Micro-90 will properly wet out the line. Ensure that the line is clean by washing several times with carrier following the IPA/Micro-90 treatment. Increasing the surfactant concentration of the carrier solution is also beneficial in reducing the frequency of bubble fragmenting.

NO COMMUNICATION WITH DEVICES

If communication is lost with the devices, exit FloZF, power down the MobiChem. Unplug the USB cable. Wait 30 seconds. Plug it in again and power up MobiChem. Start up FloZF.

DEBUGGING SEQUENCES

When a bug occurs within a complex sequence, it is often easiest to break up the sequence into its simplest component parts and run each portion separately. On the sequence page, expand the sequence by clicking once on the "+" sign to the left of the sequence in the Resource Sequences window. Drag the smaller sub-sequence to the Work Sequence panel and press the play button. Carefully observe the system as each sub-sequence is executed to identify the location of the bug. The sequence may simply need to be changed/re-written at that particular location. If the bug pertains to the software itself, contact David Holdych FloZF Software Manager, for software support (david@globalfia.com or #253-549-2226).

APPENDIX B: RECOMMENDED SPARES

Part No	Description	Qty	UOM
MG1-NM	milliGAT pump head	1	EA
C15-310M	Rotor for 10 port valve	1	EA
C25G-34R18	Rotor for 18 port valve	1	EA
SP-8	Sampling probe	1	EA
SP-0.5	Sampling probe, replaceable 0.5 in filter	2	EA
SP-1	Sampling probe, replaceable 1 in filter	1	EA

APPENDIX C: CONSUMABLES PARTS LIST

Part No	Description	Qty	UOM
CT242767-25	Reagent vials, 25mL	24	EA
2103-0008	Carrier reservoir, 8OZ (2003-0008)	2	EA
SS-1	Storage Satchel	1	EA
FS-31	Capstone FS-31 surfactant (formerly Zonyl FSN)	1	mL
BW	Box Wrench	1	EA
TC-2	Tubing cutter	1	EA
16T-030	Tubing, PFA 1/16 in OD, 0.030 ID	5	M
8T-062	Tubing, PFA 1/8 in OD, 0.062 ID	5	M
FF-16W	Flange-free fittings, for 1/16 in tubing, white, (5 pack)	3	PK
FF-8BL	Flange-free fittings, for 1/8 in tubing, black (5 pack)	3	PK
1032FN-16M	Zero dead volume fittings, 10-32, medium (10 pack)	1	PK
1032FF-16G	Ferrules for 10-32 fittings, grooved, PEEK (10 pack)	1	PK
640FF-16	6-40 Fitting for 1/16 in tubing	5	EA
P-1428N	Plugs for 1/4-28 ports, (5 pack)	1	EA
640W	Wrench for 6-40 fittings	1	EA
MUF	Make-up fitting for 10-32 and 1/4-28 fittings	1	EA
W-XE	Tungsten lamp, super bright	1	EA
FUSE-0.5	Fuse 250v IEC SLO 5x20mm 0.5A	2	EA
FUSE-6.4	Fuse 250v IEC SLO 5x20mm 6.4A	2	EA
CD-R	Cadmium reactor, 25 x 5 mm	1	EA
F-12-PE	Porous filter for filter probe, 12mm diam., UHMWPE	20	EA

APPENDIC D: WETTED MATERIALS

PTFE

The piston tips used in the pump are made from PTFE.

Polytetrafluoroethylene is the generic name for the class of materials such as Teflon®. It offers superior chemical resistance and lends itself to good sealing characteristics. Because it's so easy to handle, it is often used in low pressure devices such as the pump. Volatile compounds of low molecular weight can permeate PTFE. Fumes of strong acids such as hydrochloric acid can permeate the PTFE and should be constantly purged from the internals of the device

PAEK

The pump port end cap and some valve components and the sample probe make use of a PAEK-based composite. The green tubing between the reagent reservoirs and the selection valve is made from PEEK.

Polyaryletherketone is the generic name for the family of polyketone compounds. PAEK includes PEK, PEEK, PEKK, and PEKEKK, which differ in physical properties and, to a lesser degree, in inertness. This composite resists all common HPLC solvents and dilute acids and bases. However, concentrated or prolonged use of halogenated solvents may cause the polymer to swell. Avoid concentrated sulfuric or nitric acids (over 10%).

VALCON P

The pump rotor is made from Valcon P.

This composite, the majority of which is PTFE and carbon, has been used extensively in many analytical applications that use Valco valves. It is routinely used in other applications at 1000 psi, 75°C, and can also be used at temperatures approaching 200°C with decreased sealing tension.

VALCON-E3

The pump port end cap is made of a PAEK-based composite called Valcon-E3.

Polyaryletherketone is the generic name for the family of polyketone compounds. PAEK includes PEK, PEEK, PEKK, and PEKEKK, which differ in physical properties and, to a lesser degree, in inertness. This composite resists all common HPLC solvents and dilute acids and bases. However, concentrated or prolonged use of halogenated solvents may cause the polymer to swell. Avoid concentrated sulfuric or nitric acids (over 10%).

SAPPHIRE

The milliGAT® piston chambers are made of sapphire and are sealed in place with a Viton® o-ring in the low flow pump.

Sapphire has a unique set of properties including high strength, hardness, surface smoothness, and excellent chemical compatibility. It is commonly used in applications where a combination of exceptional mechanical and chemical properties is essential.

VITON®

Viton® O-rings are used to seal the piston chambers in the pump rotor in the low flow pump. Besides its excellent mechanical properties, Viton® provides the best proven fluid and chemical resistance of the commercial non-fluorinated elastomers.

Kalrez has been found to be superior in certain instances and a special pump can be constructed with Kalrez o-rings.

PFA

PFA Tubing is made from standard PFA resin (perfluoroalkoxy), Chemfluor® PFA Tubing is widely used in the semi-conductor, laboratory, environmental and pharmaceutical industries where ultrapure chemicals (including water) require precise quality control. Volatile compounds of low molecular weight can permeate PTFE.

QUARTZ GLASS

The optical conduits in contact with the fluid in the bubble tolerant flow cell are made from quartz glass.

BOROSILICATE GLASS

The reactor in the MD cell is made from borosilicate glass (also called Pyrex™). Some reservoir contains are made from borosilicate glass.

LDPE

Carrier and waste reservoirs are made from either borosilicate glass or low density polyethylene (LDPE). Chemically LDPE is unreactive at room temperature although it is slowly attacked by strong oxidizing agents and some solvents will cause softening or swelling. It may be used at temperatures up to 95° Celsius for short periods and at 80° Celsius continuously.

GUIDELINES

Specific reagent combinations should be tested to ensure compatibility with pump components. Since the least chemically inert component in these composites is PEEK, for optimum component life time, the following guidelines[5] given for PEEK should be followed:

Acids and bases	No stronger than 1 M
H_2SO_4	Resistant to 10%
HCl	Resistant to 20%
HF	Not Resistant to 70% (no data on lower conc.)
HNO_3	Resistant to 20%
dibutylamine in toluene	Not Rated
TBAH	Not Rated (Dionex uses PEEK as column material at 1 mM)
NaOH	Resistant
H_2S	Resistant
CH_3SH	Not Rated
acetone	Resistant
methyl ethyl ketone	Resistant at Room Temp.
methyl alcohol	Resistant
toluene	Resistant at Room Temp.
xylene	Resistant
chloroform	Resistant
chlorobenzene	Resistant at Room Temp.
iodine/iodide in water	Not Rated
sodium hypochlorite (household bleach)	Resistant at Room Temp.
permanganate	Resistant at Room Temp.
hexane	Resistant at Room Temp.

[5] Corrosion Resistance Tables, Schweitzer, Marcel Dekker, 1991

www.zeusinc.com/product_sheets/resins/peek/chemical_compatibility.html