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FloPro-Sampler™

Global FIA automated sampling system for small reactors

Flexible, versatile automation

The Global FIA FloPro-Sampler is a versatile Zone Fluidics sampling interface that automates the sampling of laboratory reactors. This fluid handling device allows unattended sampling and dilution of a host of different sample matrices and media. The resultant sample can be:

- archived to a fraction collector for subsequent assay,
- loaded into the injection valve of a chromatograph, or
- pumped through the flow cell of an in-line detector

The device comprises of two main parts: a sample probe and a fluidics engine.

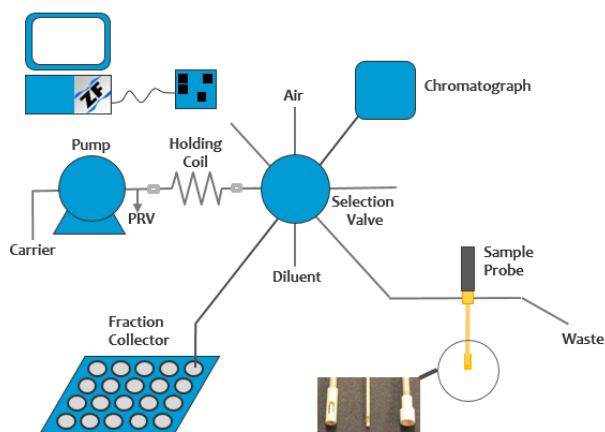
Sample probe

The sample probe comprises of an adjustable probe which can be mounted in a 24/40 tapered socket. The probe can be equipped with a filter tip for sampling supernatant solutions or an open port tip for sampling heterogeneous samples. A three-way valve allows selection of the sample probe or waste. The 24/40 adapter can also be equipped with a port for headspace sampling. Up to four sample probes

can be coupled to a single sampler allowing sampling from multiple process reactors. Various filter tips have been designed to allow specialized filtering options. Probe length is user specified. A metal version with metal stator can be used to match the temperature of the sample in the probe to the process temperature.

Fluidics Engine

The fluidics engine employs Zone Fluidics to automate sample and dilution sequences. This part of the FloPro-Sampler is equipped with a milliGAT® high precision, bi-directional, positive displacement pump, a multi-position selection valves, and a holding coil. The holding coil, is positioned between the 18-port selection valve and the pump. The selection valve also provides the connection to the chromatograph if direct measurement of the diluted sample is required. A pressure relief valve protects the pump against unintended over pressurization which can lead to leakage into the pump internals. Reservoirs for up to four diluents are mounted on the device. A multi-vial fraction collector is connected to the selection valve to provide an archive of diluted samples.



FloPro-Sampler Fluidics Manifold

Sequences have been developed to carry out two serial dilutions. The first dilution is carried out in close proximity to the sample probe to improve the robustness of the system for handling samples with a high dissolved solids load close to or above the saturation point of the solution. The second dilution is a larger dilution and is intended to bring the concentration of the sample down to the operational range of the preferred detection technology.

Sequence

Two bubble-separated zones of about 100 μ L of diluent 1 each are aspirated and dispensed towards the sample probe. A portion of the diluent zone distant from the pump is dispensed past the probe towards the waste vial. The three way valve is switched to the sample probe and the probe is primed by aspirating from the reactor. The two bubble-separated zones are then dispensed towards the waste vial such that the zone with primed sample is dispensed into the waste vial. The remaining diluent zone is positioned such that half of diluent is located in the tube between the sample probe valve and the other is in the tube to the waste vial. This portion will be the trailing zone when the zone stack is returned to the holding coil. Diluent is not allowed to enter the reactor. A fresh sample is aspirated and is drawn back towards the holding coil. Once the desired volume (typ. 20-50 μ L) of sample has been aspirated, the three way valve is switched and the parked zone follows the sample as it is pumped towards the 18 port valve. *En route*, the sample is dispersed into the diluent zones thereby lowering the concentration of the sample. A portion (possibly the entire portion) of this sample is then bracketed

between two large zones of diluent 2. The zone stack is bracketed between two small bubbles. In this dilution step, dilution ratios of between 1:10 and 1:100 are easily accommodated. The bracketing bubbles prevent the zone stack from being dispersed into the carrier. They also give rise to Taylor flow which mixes the sample with the diluent zones. This mixing occurs by pumping this bubble bracketed zone stack through the 3mL mixing coil or by dispensing it into a vial in the fraction collector.

When the diluted zone is to be measured directly in an in-line flow cell or loaded into the injector of a chromatograph, a portion of the dispersed zone is sampled and transported to the flow cell or injector between two bubbles. In the case of the fraction collector the entire diluted zone (typically 500 μ L) is dispensed to the fraction collector vial.

Fraction collector

The fraction collector is built into the device and employs state of the art servo motors and controllers with high precision motion control mechanical elements. This ensures fast axis motion, fool-proof positioning, and quiet operation. The collector vials conform to a 96-well plate standard footprint and can be configured with vials to suit the end user's needs. This means, for example, that individual septum sealed vials or a vial holder that can be directly transferred to the system used to analyze the samples can be accommodated. A wash station minimizes carryover from one sample to the next.

Interfacing to a chromatograph

Direct injection into a chromatograph is best achieved by using the chromatograph's injector which is plumbed to the selection valve of the FloPro-Sampler. A digital trigger from the FloPro-Sampler is sent to the chromatograph to signal the injection and start of data acquisition. We have successfully coupled the FloPro-Sampler to Agilent's ChemStation software on an Agilent 1200 chromatograph.

Software

The device control and data acquisition software is called FloZF and provides a convenient drag-and-drop means of setting up device control sequences or altering key parameters in an Excel spreadsheet to change the dilution ratio, preferred diluent, or

specific sample handling protocol. Startup, shutdown, and dilution sequences are included.

Configurable and scalable

The standard configuration is depicted above. Additional unit operations can be plumbed into the 18 port valve to allow further processing of the sample in the FloPro-Sampler. Up to four sample probes can be attached to the FloPro-Sampler. The sample probe filter tip and open port tip are interchangeable. The sample probe can be locked in place once the optimum sampling height has been established.

Sample types

The FloPro-Sampler has been tested with homogeneous and heterogeneous samples with and without filtering. Samples have been obtained from reactors with reaction temperatures between ambient and 70 °C. Samples with concentrations above the saturation point of the reaction solvent at ambient temperature are easily handled by carrying out the first dilution in close proximity to the sampling point using a diluent with good solubility characteristics.

Figures of merit

COV of serial dilution sequence: 1-2%

First dilution: 1:1 to 1:10

Second dilution: 1:10 to 1:100

Sample extracted from the reactor 20-50 µL

Cycle time: 5-10 minutes

Diluent usage first dilution: 100-200 µL

Diluent usage second dilution: 100-1500 µL

Wetted materials

The standard sampling probe is made from PPS with a Teflon lined sampling tube – a PEEK version is available. The standard filter tip is porous PTFE and is held in place with a ceramic screw. Seals are made from Teflon. The 3-way valve body is made from PPS. Selection valve wetted materials are stator – PPS, rotor – UHMWPE. Tubing is made from PFA. More exotic valve components are optional.

The needle of the fraction collector is made from stainless steel.

The wetted components in the pump includes Teflon, ceramics and similar plastics to those used in the selection valves.

Size

FloPro-Sampler: 230 mm x 300 mm x 150mm

Probe length: 1cm to 45cm (adjustable)

Fraction collector vials: 54 x 1.5mL (typical)

Power and communication

Power: 110-250VAC

Communication: USB to RS-485

Software compatible with Windows XP, Vista, Windows 7 and Windows 8

Interfacing with external devices

Standard handshaking via TTL or contact closure digital IO

Technical Point of Contact

David Holdych

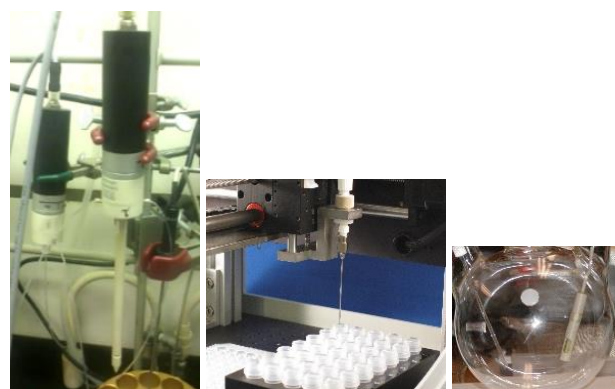
David@GlobalFIA.com

Pricing

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| 1. FloPro-Sampler including sample probe | \$28,875 |
| 2. Additional sampling probe (PEEK) | \$2,495 |
| 3. Additional sampling probe (PPS) | \$2,495 |
| 4. Chromatograph trigger cable | \$55 |

Accessories and spares

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| 1. ¼-28 fittings (5 pack) | \$11 |
| 2. 6-40 fittings (ea) | \$14 |
| 3. PFA tubing (3m) | \$13 |
| 4. Replacement tubing (ea) | \$25 |
| 5. Filter tip (ea) | \$45 |
| 6. Open port tip (ea) | \$25 |
| 7. Fraction collector vial holder (ea) | \$125 |
| 8. Fraction collector needle (ea) | \$38 |



Notes