Flow-based Analysis for Bench-Top Analysis and On-Line Monitoring

What is Flow-based analysis?

Flow-based analysis is the generic term that describes a group of automated sample manipulation procedures that make use of unsegmented flowing streams in micro-conduits (0.030" or 0.7 mm id.). Most common among these techniques is Flow Injection Analysis (FIA). FIA is the process whereby a small segment of sample solution, typically 20 to 100 µl, is injected into a flowing carrier stream. This stream either contains a reagent, or the stream is merged with a reagent stream to produce a compound which can be determined in a flow-through detector. Sequential Injection analysis (SIA) is a variation of FIA where a selection valve is used to produce a stack of well defined zones of reagent and sample. These zones penetrate one another as they are carried to the detector by a suitable pump. Determination of the analyte of interest is analogous to FIA.

Wet chemical techniques form the basis of many analytical measurements. These proven chemistries, many approved by the EPA and other regulatory bodies, can be automated by using Flow-based techniques such as FIA and SIA.

Advantages of Flow-based analysis

- Applicable to a broad range of compounds by using a variety of chemistries and simple photometric and electrochemical detectors.
- Low cost and simple instrumentation.
- Low scheduled maintenance requirement.
- When an **on-line** analyzer is used**,** unattended automated operation over an extended period of time provides both real-time results and a detailed picture of analyte profiles. This detailed information is not available when grab samples, or even composites, are taken and analyzed at a later time and remote location. Unstable compounds can be measured *in-situ.*
- Accurate and precise sample manipulation.
- Quality control measures can be included to assure the integrity of the analytical results.
- A wide range of useful manifold components allow complex operations such as dilution, trace enrichment, gas permeation, dialysis, and several others to be incorporated into the analyzer manifold
- Constant flushing of the manifold lines minimizes the risk of fouling

How Global FIA, Inc. can help you

Global FIA, Inc. is a supplier of FIA / SIA components, supplies, and expertise and will gladly customize a Flow-based Analyzer to meet your specific requirements. Analyzers suitable for laboratory operation, continuous on-line process and field monitoring, and research and development are available.

Technology Note : 95/1
Partial Loop (Timed) Injection

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Summary

The dilution of samples in an FIA/SIA manifold has enjoyed much attention and is often cited as one of the most useful applications of FIA. This Note describes one of the most simple procedures for achieving dilutions of up to 1:100 with a precision at least as good as 1% RSD, and generally 2% RSD or better for higher dilutions. The procedure is based on the precise timed injection of < 10µl of the sample loop.

Application

This dilution procedure is applicable to all FIA manifolds and makes use of readily available FIA components. It is based on the well established fact that dispersion, and hence dilution, is strongly impacted by sample size.

Benefits

This approach offers several benefits:

- It allows the use of small sample volumes without the need to make use of tubing with an ID smaller than the rest of the manifold.
- It offers a simple means of precise dynamic sample dilution.
- Method development time can be reduced by allowing convenient and simple variation of sample volume.

Equipment

There are three critical components used to apply this technique. They are the valve, pump, and device control software. These components must meet certain criteria in order to ensure good reproducibility. The key component is a Valco Cheminert injection valve (the C22 and C22Z series) that is micro-processor controlled and has the unique capability of "timed injection" switching. This feature of the valve allows variable volume injection with a single sample loop, and also the reproducible injection of small volumes.

To use the timed injection mode, a variable in the actuator is loaded with the desired Delay Time, DT, via an RS-232 link. Actuation of the valve via an RS-232 command or contact closure causes the valve to switch from "Load" to "Inject", pause for a period equal to the pre-set DT, and then rapidly switch back to the "Load" position. DT can be set at any value between 0 and 10,000 msec.

The second important component is a highly reproducible and pulseless variable speed pump, such as the Cavro XP-3000 Syringe Pump.

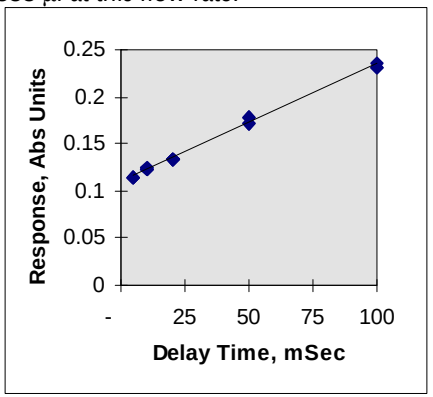
The third important component is a device control software package, such as FlowTEK™, distributed by Global FIA. This software has the capability of controlling the valve in the timed injection mode using both RS-232 and digital I/O.

Description

1. Assemble the desired manifold.
2. Set up the valve for mode 2 digital input (see Notes below).
3. Design the desired Method and Procedures in FlowTEK™
4. Select a pump speed and DT that will achieve the desired sample size or dilution. It may be necessary to carry out a few empirical experiments to determine the optimum settings.
5. Run the Main Procedure.

Results

By using different values for DT, it is possible to vary the injection volume. With flow rates commonly used in FIA, volumes as small as 1 µl can be injected using the timed injection mode. In the first test, the smallest volume that can be injected was determined by plotting the Response of triplicate readings against Delay Time. This graph and the pump flow rate were used to calculate the smallest possible injected volume. At a flow rate of 2.0 $mlmin^{-1}$, this was found to be less than 3 µl. The maximum delay time (10 sec) gives a sample volume of 333 µl at this flow rate.



The capability of this approach to perform precise dilutions was tested by the timed injections of a concentrated solution of Bromothymol Blue indicator from a 100µl injection loop into a borax buffer carrier stream. The concentration of the dye was such that a 1:100 dilution of the test solution gave an absorbance of about 0.300 AU. A pump flow-rate of 2.0 $mlmin^{-1}$ was found to give the best precision. Five injection times (10, 20, 50, 100, and 500 msec) were tested. The reaction coil used was 150 cm long. The results of these experiments are given in a tabular summary.

Notes

The new C22 and C22Z series of Valco Cheminert valves equipped with micro electronic actuators have two input modes. In this Note the conventional default mode used to load and inject samples will not be discussed. Although it is possible to use any serial communication package (9600,n,8,1) to utilize these two modes of operation, this Note will cover the use of FlowTEK™.

Inject Time msec	Pk Ht %RSD	Dilution
10	**1.13**	**1:228**
20	**0.93**	**1:208**
50	**0.75**	**1:163**
100	**0.62**	**1:120**
500	**0.52**	**1:57**

1. To set the valve to mode 2, use Directive 66 with the command string, **SM2**. The actuator is returned to the default digital control mode by issuing the command **SM1** in the same way.
2. In control mode 2, asserting pin 5 on the valve actuator causes the valve to toggle to the opposite position. Asserting pin 6 causes the valve to switch to the opposite position, wait a pre-set Delay Time, and then switch back. (Between each injection, it is necessary to set both pins low.) These settings can be programmed into a new device definition in FlowTEK™. See the Method chapter in the FlowTEK™ manual. (The digital output for timed injection is 2; to toggle to the opposite setting, 1; and to clear the valve, 0.)
3. Set the Delay Time by issuing the command **DTn** with n=1-10,000 msec. Use Directive 66 to set DT. The default is 100 msec.
4. At the start of each experiment you must use the Clear Valve setting to establish the Load position. Also, ensure that the valve is in the Load position at the start of the Run.
5. Once you have changed the parameters in the valve, these will remain in place at power down.

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