

# Flow-based Analysis for Environmental Monitoring

## What is Flow-based analysis?

Flow-based analysis is the generic term which 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  $\mu$ l, 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 simple photometric and electrochemical detectors.
- Low instrumentation costs.
- Low scheduled maintenance requirement.
- Unattended automated operation over an extended period of time provides 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.



## Methodology Note : 95/2 Trace Nitrate in Water

### Global FIA, Inc.

PO Box 2297

Gig Harbor, WA, 98335

Support : (253)-265-8586

Fax : (253)-265-8587

# Methodology Note : 95/2

## Trace Nitrate in Water

### Summary

Nitrate in effluent samples is determined by injecting the sample into an imidazole buffer stream (pH 7.5). This stream carries the sample over a copperized cadmium reactor which reduces the nitrate to nitrite. This stream is merged with a coloring reagent comprising sulfanilamide and NED. A violet color, which is monitored photometrically at 555 nm, results.

### Application

This method is suitable for the determination of nitrate in effluent samples in the concentration range 0.05 to 1 mg/l. Higher levels can also be accommodated by including a dilution system into the manifold. The precision of the method, as determined by the relative standard deviation ( $s_r$ ), is 0.01 at the 0.5 mg/l level. A sampling frequency of 30 measurements per hour is obtained. No major interferences are anticipated.

### Equipment

Global FIA, Inc. has a full range of components necessary to configure an analyzer as depicted in the FIA manifold. The analyzer could either be a Bench top, Field Portable, or At-line analyzer and is controlled by the

FlowTEK™ device control and data acquisition software package.

### Reagents

1. Imidazole Buffer. 6.8 g +5 ml HCl dissolved in 1l dist. water **B**
2. 25 ml 1% Sulfanilamide, 25 ml 0.1% NED and 10 ml H<sub>3</sub>PO<sub>4</sub> dissolved in 100 ml dist water. **C**
3. Nitrate stock solution: 2000 mg/l. Working standards are prepared by suitable dilution of this stock solution. **S**

### Optimum conditions

Pump flow rate: 1.0 ml/ min.  
Reaction coil 1: 50 cm **RC**  
Injection loop: 100 µl **SL**

### Procedure

1. Assemble the manifold as set out in the diagram.
2. Prepare the reagents according to the procedure described above.
3. Program FlowTEK™ with the desired Procedures.
4. Ensure that the FlowTEK™ Method described has been defined.
5. Pump the reagents through the manifold for about 20 minutes in order to attain stable flow conditions and allow the detector to warm up.
6. Filter the sample and dilute it to within the analytical range.

7. Ensure that the selection valve is selecting the correct line. Either a standard for a calibration, or the sample.
8. Run the Main Procedure and allow FlowTEK™ to calculate the sample concentration.

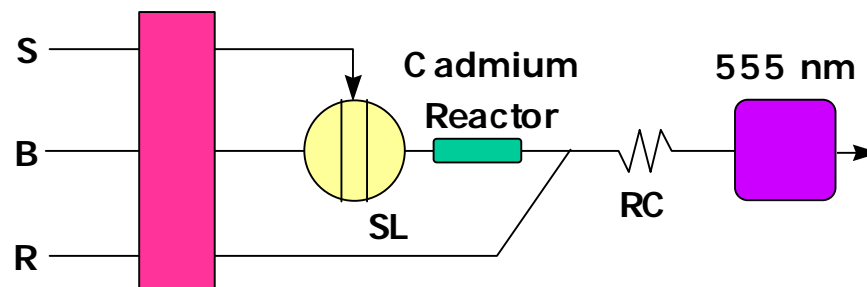
### FlowTEK™ method

Expt Time: 120 sec  
0 sec Inject, Pump forward  
5 sec AutoZero  
30 sec Load  
120 sec Pump Off

### Figures of merit

Working Range, mg/ l	0.05 - 1
Precision, $s_r$	0.01
Sampling rate, h <sup>-1</sup>	30

### Flow Injection manifold



### Notes

Care and re-coating the Cadmium reactor.

1. When not in use, the Cd reactor should be filled with the buffer solution.
2. To reactivate the reactor, inject a 5 mg/l nitrate solution into the analyzer.
3. To re-coat the Cd with copper, wash briefly with 0.1M HCl, rinse with water, then pump a 0.1M Copper solution through the reactor for 2 minutes.
4. Minimize the exposure of the cadmium to air to prolong the reactor life time.

Nov-95