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The Model FS3700 and Chemistries Part 2

APRIL 30TH, 2020

O·I·Analytical 
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FS3700 Training

Automated Chemistry Analyzers – FS3700

What we will cover:

- The FS3700
- FS3700 Chemistries
- Cyanides and Ammonia/TKN – Gas Diffusion

Presenter – Hank Hahn

I am currently the OI Analytical Senior Sales Specialist based in College Station, Texas. I have nearly 29 years of analytical instrumentation experience, including product lines for gas chromatography, automated flow chemistry and TOC analyzers. My sales colleagues refer to me as a valued resource, provide wealth of knowledge in not only my understanding the technical side of our instrumentation, but I also have an understanding how it best provides solutions for various applications in numerous industries.



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The FS3700

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Customers' Problem Statement

Wet chemical methods for ion analysis are inherently very sensitive and offer analysts the analytical capability that is demanded by regulatory methods. However, these methods are also inherently tedious, time-consuming, and generally require large volumes of chemical reagents and hazardous waste

Problem Examples for Ion Analysis

- Contract Service Laboratories
 - Wide range of sample matrices and required chemistries
- Municipal Drinking and Waste Water Laboratories
 - Ground water, surface water, and treated water with typical ion concentrations in the parts-per-billion range.
- Chemical or Petrochemical Laboratories
 - Treated process water contamination levels in the parts-per-million
 - Need for rapid Cyanide or other chemistries with on-site lab. Need numbers fast

Product Solution

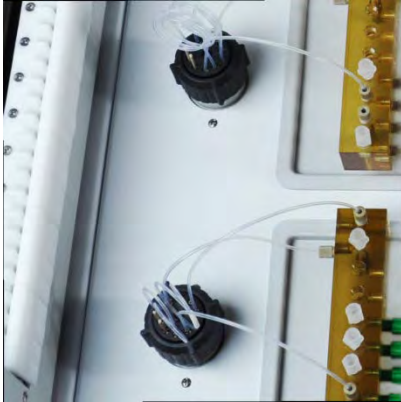
- The FS3700 platform offers the customer the flexibility, and analytical capability to meet their needs.
- Plug and Play
- The unique design features offer high sample throughput and low cost of ownership.
- Robust technologies to meet the demands of every type of sample matrix.
- Excellent precision and accuracy.
- Regulatory compliance to ASTM, ISO, SM, (US)EPA.

Automating Wet Chemistry for Laboratory Productivity

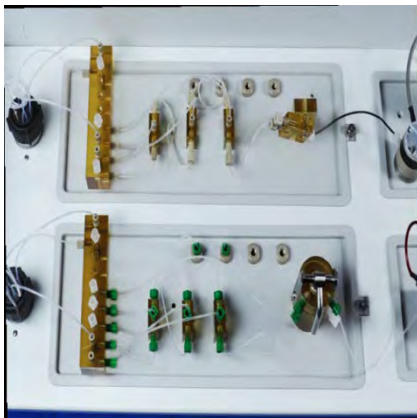


The FS 3700 Automated Chemistry Analyzer is an advanced continuous flow analyzer designed to improve laboratory productivity by automating wet chemistry test procedures.

Automating Wet Chemistry for Laboratory Productivity



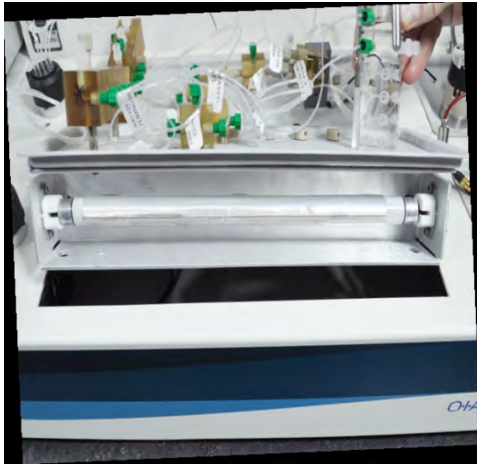
An automated injection valve is installed in the analysis module chassis when required to run a flow injection analysis (FIA) method.



Each chemistry cartridge is pre-assembled with all components needed to perform a validated analysis method – just attach the pump tubing and detector flow cell.

Ordering by ‘channel’ provides a convenient way to configure the 3700. Modular, flexible hardware provides a great platform for research, in-house or proprietary methods

Automating Wet Chemistry for Laboratory Productivity



In-line devices for reactions requiring heating or UV digestion are mounted on the underside of chemistry cartridges. FlowView software provides user programmable control of the UV lamp and cartridge heater set points in 1 °C increments.



Photometric and amperometric detector modules plug-in to the FS 3700 to support methods employing colorimetric chemistries or amperometric measurements. The Expanded Range™ photometric detector and auto-scaling software virtually eliminate off-scale samples. A single calibration curve can range from low ppb to high ppm concentrations.

Value Proposition

The Flow Solution 3700

The Flow Solution 3700 Automated Flow Chemistry Analyzer introduces state of the art technology with a flexible, innovative system that brings together advanced, icon-driven software and modular, plug-n-play components that allow you to easily customize your system for your chemistries and improve your laboratory workflow. Designed for ease of operation and low cost of ownership, this system provides high sample throughput, while handling complex matrices and a wide variety of chemistries.

Value Proposition

Flow Solution 3700 for Cyanide

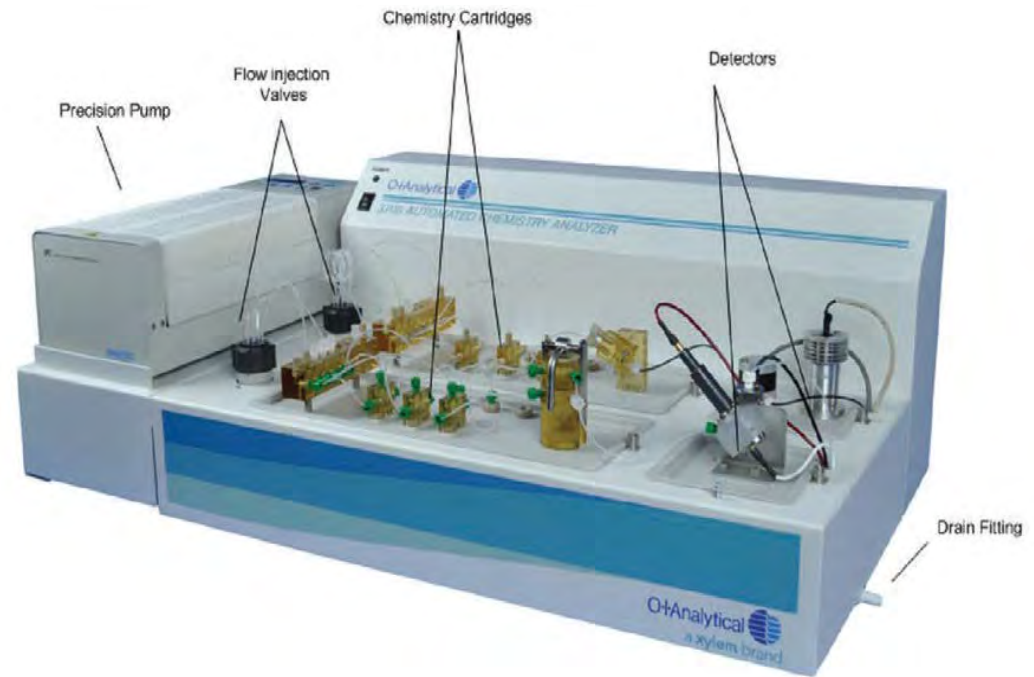
As the recognized leader in cyanide analysis, OI Analytical designs accurate, high-performance benchtop and online instrumentation for performing flow injection cyanide analysis on drinking water samples and wastewater samples from mining, metal plating, and other industrial operations. Designed for ease of operation and low cost of ownership, this system provides high sample throughput, while handling complex matrices for cyanide analysis.

FS3700 Layout



FS3700 Features

- For years, laboratories have turned to OI Analytical for accurate, reliable continuous flow analyzers, the Flow Solution™ 3700 is the next generation Automated Chemistry Analyzer in our family.
- Built incorporating the strengths of our previous platforms – ALPKEM, Perstorp, CNSolution, FS 3100 and Flow IV.
 - The ability to run SFA or FIA methods,
 - A new, user-friendly interface using the best of prior software (WinFLOW) and incorporated into now FlowView.
 - Compatibility with Windows 10
 - Connects via USB – can be run from any computer & many tablets.



Chemistries available

Available cyanide (EPA OIA-1677)

Available cyanide (ASTM D6888)

Total cyanide (ASTM D7511)

Ammonia/TKN by gas diffusion

Ammonia (Phenate/Nitrogen)

Nitrate/Nitrite (FIA /SFA)

Phosphate, all forms

Phosphate, all forms (low level)

Phenol, Post-distillation

Phenol, In-line distillation

Chloride

Sulfate

Cyanide – ISO Methods and Post

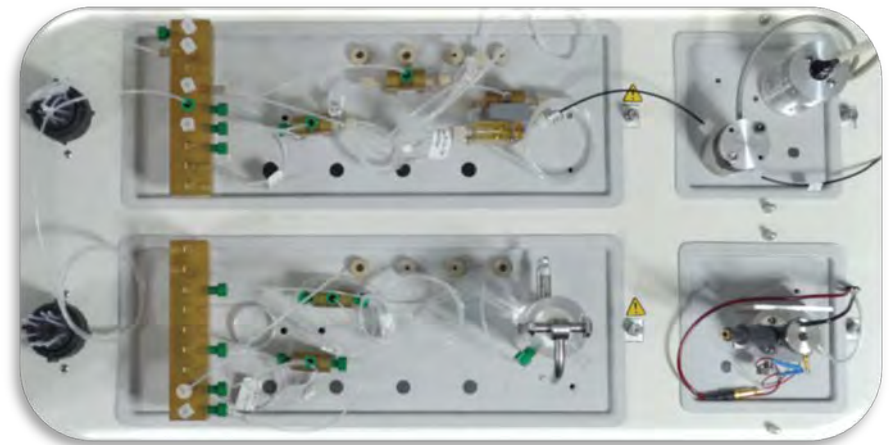
Distillation



Chemistries available

The chemistries released for the FS 3700 are the best method – selected from all configurations developed for previous generation analyzers and user-submitted methods.

- The 'best' method will be selected for use on the FS 3700 (regardless of if it is a FIA or SFA method).
- Chemistries may be customized as needed.
- Modular, flexible platform



Autosamplers



180 Position XYZ Autosampler
OI Part Number 330964



360+ Position XYZ Autosampler
OI Part Number 330965

3180 Autosampler



180 Position XYZ Autosampler

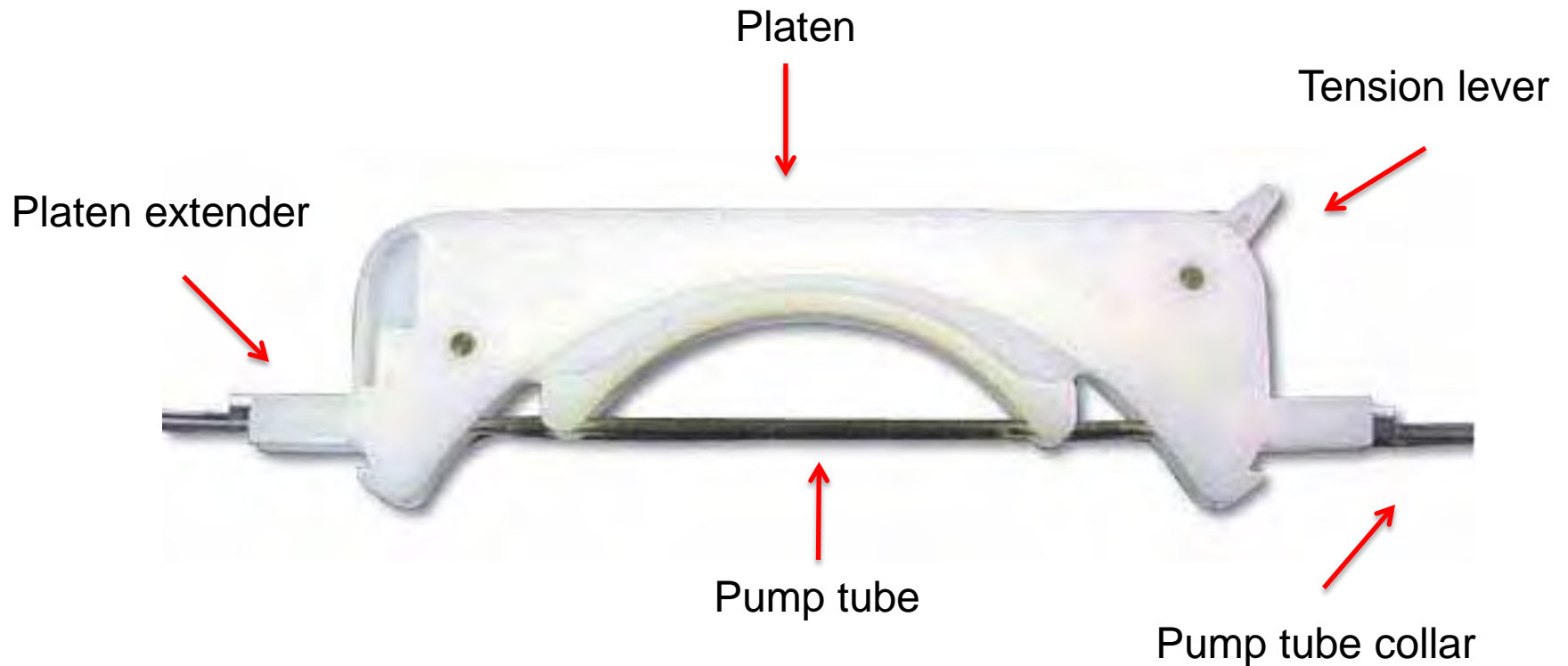
- 2 - 90 Position sample racks
- 10 Position standards rack(50-milliliter conical centrifuge vials with caps)
- Rinse Station, the flowing rinse station is located at the left end of the standards positions at the back of the sample base.
- On-board peristaltic pump. A two-channel peristaltic pump moves the rinse solution from the rinse source through the flowing rinse station.
- Sample Probe Kit. The kit includes the sample probe. The sample probe fits into the Z-drive.

Ismatec 24 Channel Pump



For Autoanalyzers, these Ismatec pumps use multi colored collared pump tubes that vary the amount of volume that gets produced. To allow for a continual flow of solutions, the pump tube roller heads gently rotate around the tubing material. The compression of the rollers/cams on the peristaltic tubing acts like a check valve in the pump keeping liquid flowing smoothly in one direction and offering a repeatable and consistent flow with each revolution. The pump tubes are held in place with an easy to remove platen that allows for micro control of each pump tube. There are controls on the pump but it is also mainly controlled by the FlowView software.

Ismatec 24 Channel Pump Platen



Platen, platen extender and tension lever
Tube material is Phthalate free PVC or tygon

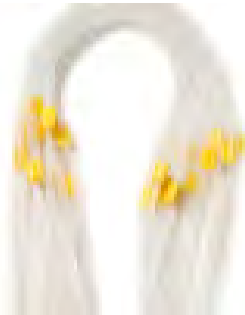
Pump Tubes

Pump Tube Type	Internal Diameter (inches)	Flow at 40% (ml/min)
<u>Orange/Blue</u>	0.01	0.03
<u>Orange/Green</u>	0.015	0.1
<u>Orange/Yellow</u>	0.02	0.18
<u>Orange/White</u>	0.025	0.25
<u>Black/Black</u>	0.03	0.32
<u>Orange/Orange</u>	0.035	0.41
<u>White/White</u>	0.04	0.56
<u>Red/Red</u>	0.045	0.71
<u>Gray/Gray</u>	0.051	0.84
<u>Yellow/Yellow</u>	0.056	1.01
<u>Yellow/Blue</u>	0.06	1.12
<u>Blue/Blue</u>	0.065	1.35
<u>Green/Green</u>	0.073	1.57

Pump Tubes – Example of Ammonia/TKN



Orange/Green Air Injection



Yellow/Yellow Sample Line Pump



Gray/Gray



Black/Black Debubbler Pull



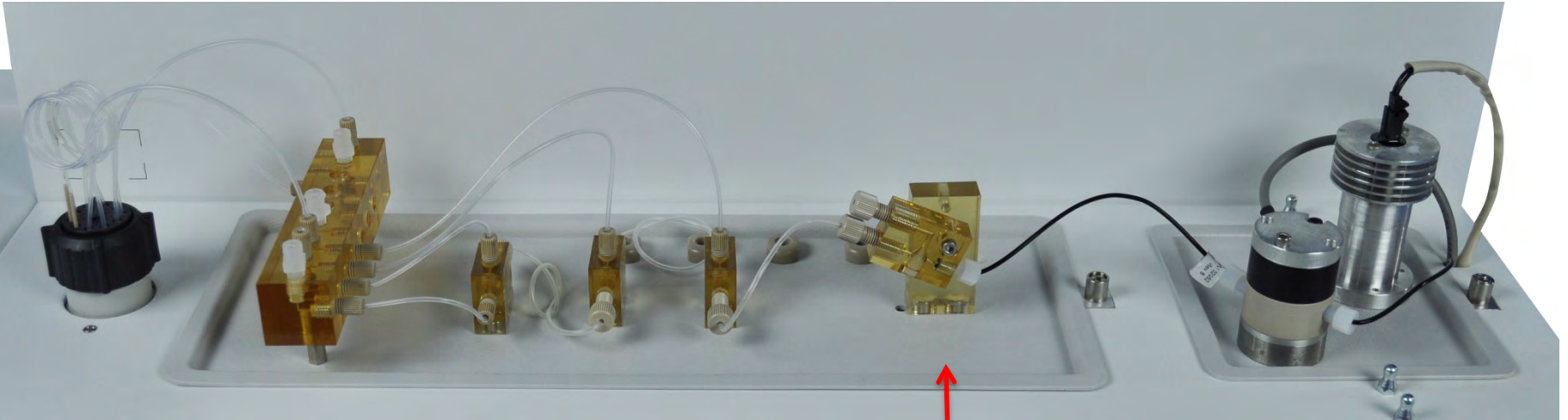
Orange/Yellow

What's in a Channel



- Everything needed to perform analysis is included in a channel.
- FIA Valve, Chemistry Plate and Detector (Amperometric or Photometric)
- Cartridges include gas diffusion manifolds, heaters or UV lamps (as needed) all tubing, pump tubing and the chemistry kit.

What's in a Channel (Colorimetric Chemistry)

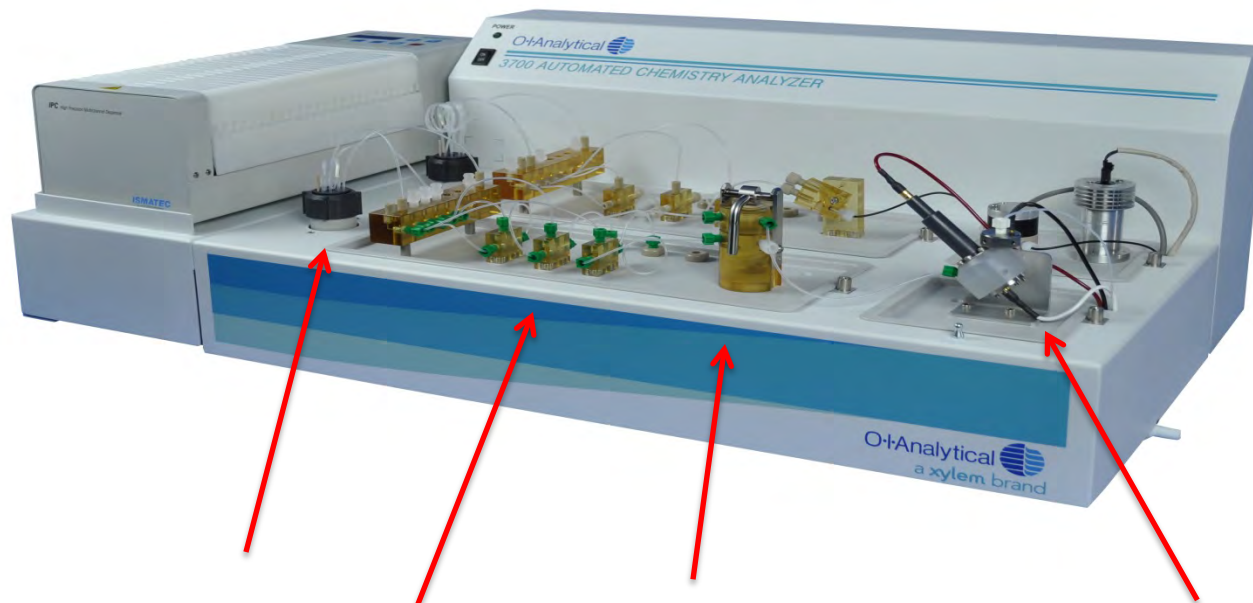


FIA Valve
(Chemistry
specific)

Chemistry Plate with
Mixing coils

Photometric Detector
Includes Filter specific
to chemistry

What's in a Channel (Amperometric Cyanide Chemistry)



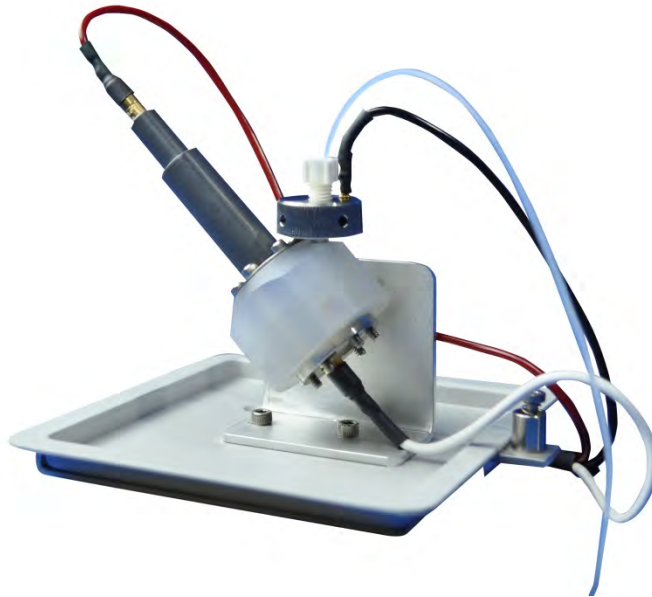
FIA Valve

Gas Diffusion Module

Amperometric Flow Cell with Reference Electrode

Chemistry Plate with mixing tee's and pump tubes

Amperometric Detector (Cyanide Chemistries Only)



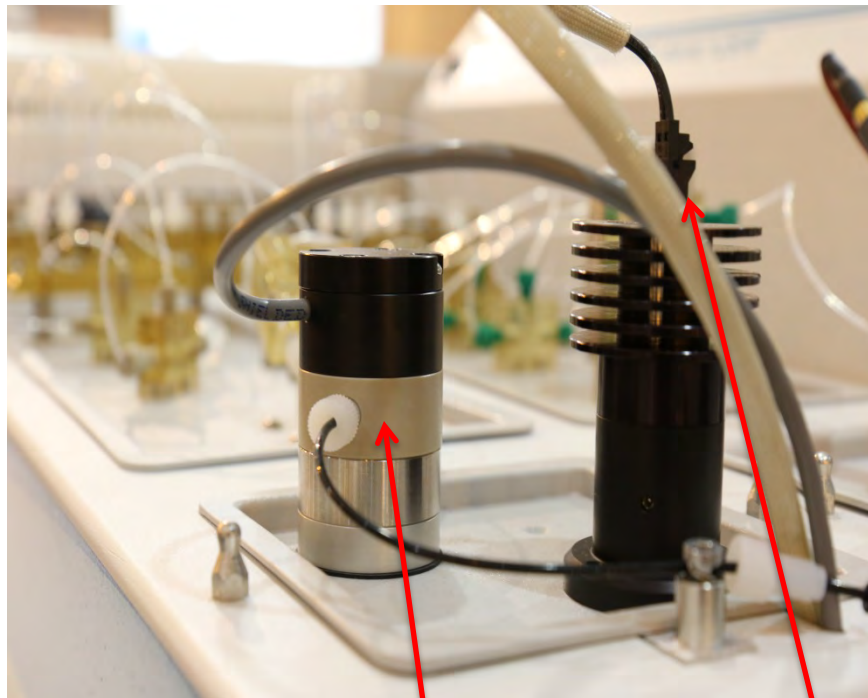
- Best amperometric detector on the market further refined to reduce noise.
- Not a 3rd Party Detector
- Improved cell design & upgraded electronics.
- Made up of: Reference Electrode (Red), Working Electrode (White) and
- Counter Electrode (Black) closes the loop

Photometric Detector – Expanded Range Detection



- 10x increase in signal-to-noise for some photometric methods.
- Improved (or equivalent) detection limits of the FS 3100.
- MDLs down to low ppb, limitation is now flow noise.
- Eliminates need for Auto-dilution
- Wide Range capability - The benefit of using a wide-range detector lies in its inherently high sensitivity. Because of its high sensitivity, it can detect very small changes in the signal without increasing the gain. This allows detection of very low concentrations. Because the gain is not increased, it can also detect high-level peaks extremely accurately in the same run without topping out.

Photometric Detector



Flow cell –
5mm standard

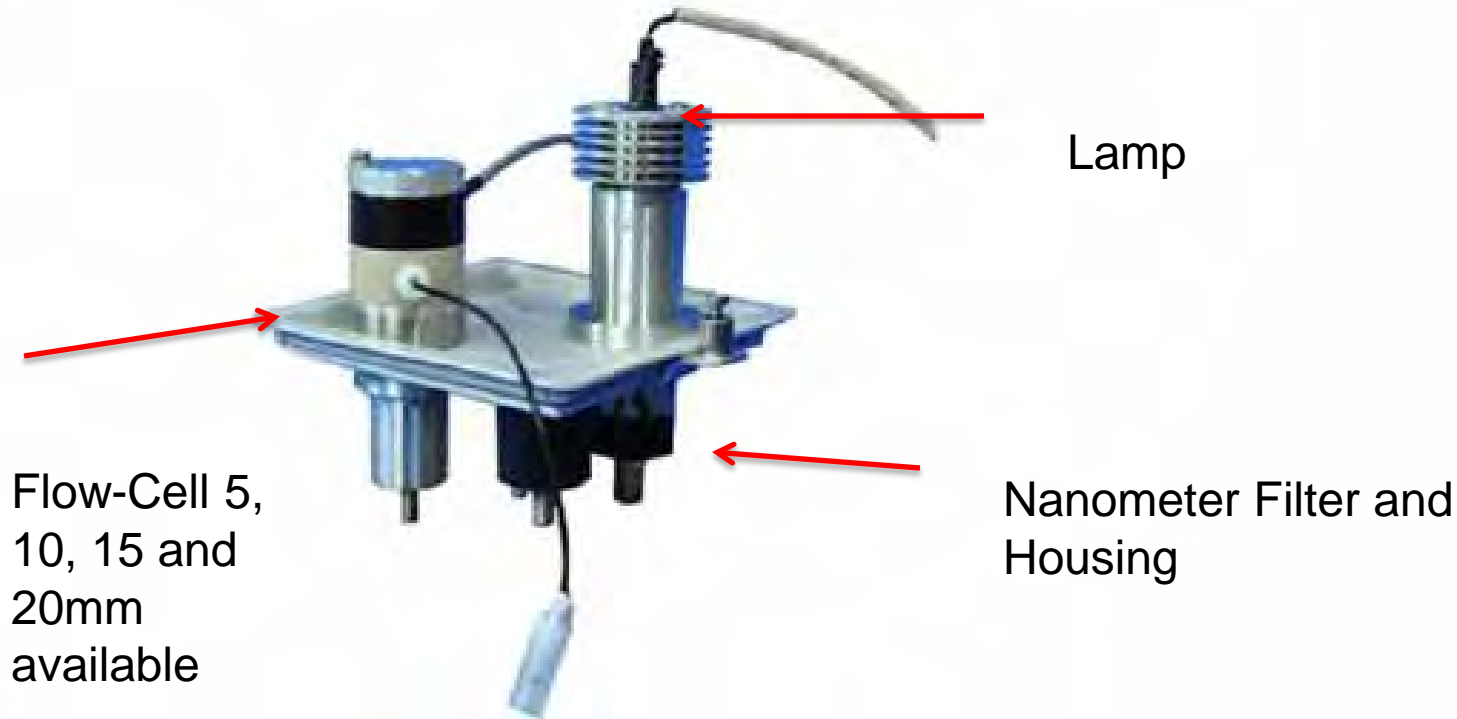
Lamp

Sample stream flows through the detector required for the chemistry. As the sample flows through you will see a peak rise on the graph (FlowView software).

After the samples passes through the peak will settle back to baseline until the next sample begins to flow through the flow cell.

The peak is compared to the calibration curve and a result is calculated against the curve.

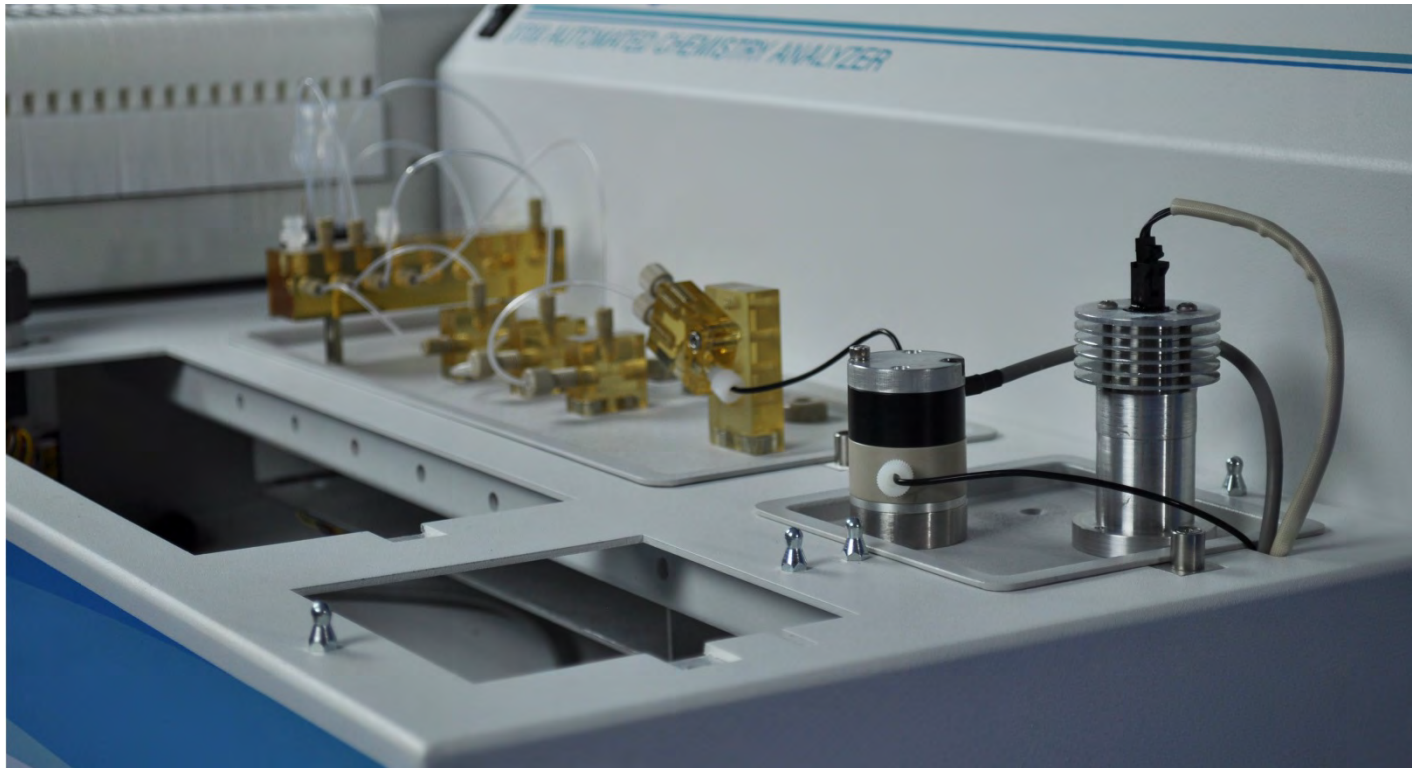
Photometric Detector – Flow Cell




This is where everything coalesces and the results of the sample collection, reagent preparation, and instrument operation come together to produce the final analytical conclusions. The FS3700 detector design allows for the flow cell windows to be removed, cleaned and/or replaced if required without a costly replacement of the entire flow cell.

Photometric Detector – Expanded Range Detection


Refinements in detector design improved signal to noise ratio and increase sensitivity. This led to minimizing noise and found with 8 port valve plumbed to utilize a bypass loop equal to size to the injection loop while in load position alleviated pressure differential and reduced detector noise related to the valve actuation.




FlowView Software

 **O.I. Analytical**
Flow Solution 3700 Analyzer


Home — ✕




Configure Analyzer




Build Method




Build Sample Table



Run Samples



View Results



Perform Maintenance

Beta3	State Machine : Idle	Signal Poller : Idle	Vial# : 0	Reps : 0 / 0	State : None	Active Sample Table : NO2-Brant
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FlowView Software

Intuitive interface

32 and 64 bit, Windows Pro 7, 8 10 Pro

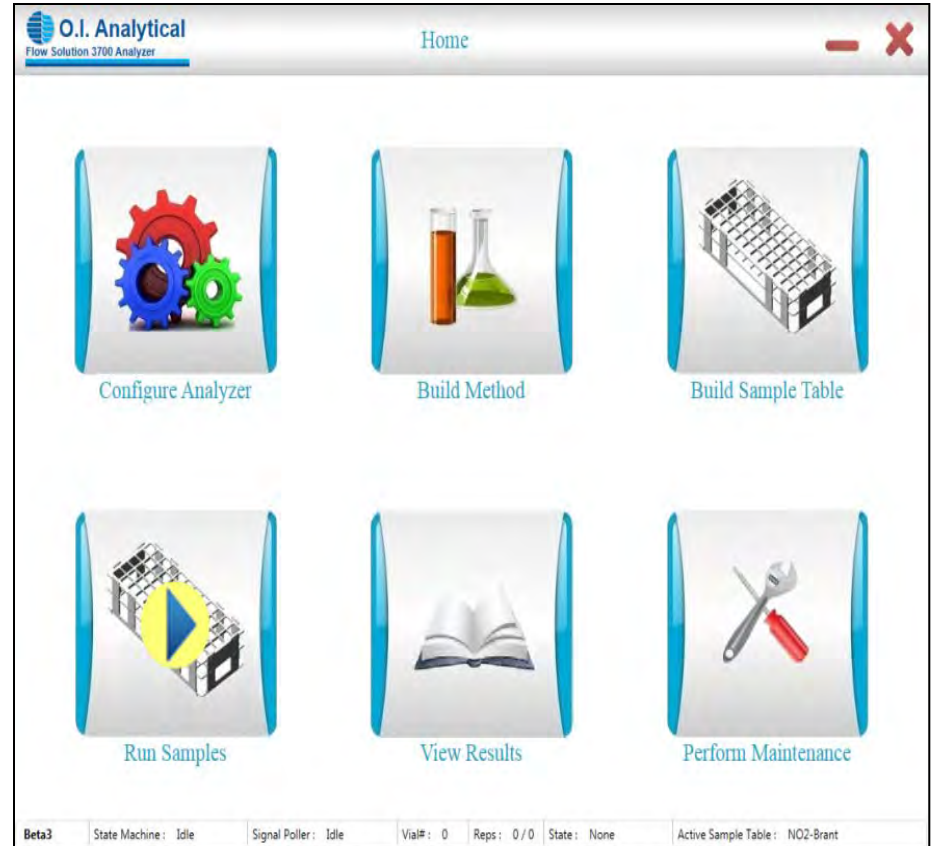
Performs dynamic auto-scaling keeping analyte peaks on scale over a large dynamic range (3–4 orders of magnitude)

Allows viewing and editing results in real-time

Easy LIMS import/export configuration

Re-prioritize or edit the sample table on the fly during analysis

Software control of cartridge heaters in 1 °C increments



FlowView Software – Configure Analyzer

Click a highlighted component on the chassis diagram to configure and/or review analyzer settings:

- **Pump** Run speed and timing of peristaltic pump for run and post-run
- **Valves** Enable/disable for each channel and set timing for FIA load/inject
- **Channel** Enable/disable channels, cycle duration and channel name
- **UV** Enable/disable UV digestion/amps, if equipped
- **Heater** Enable/disable each, and define heater setpoints, if equipped
- **Detectors** Configure mode and settings for photometric, amperometric
- **Sampler** Set-up for either 180 Position or 360 Position

O.I. Analytical
Flow Solution 3700 Analyzer

Configure Analyzer

Configure System - Detectors

Primary Chassis

Sampler

Photometric CH 1

Photometric CH 2

Enable Detector 1

Mode: Photometric

Sample Gain: Photometric

Polarity: Amperometric

ISE

A/D

Cell Potential while in Standby mode (volts)

Enable Detector 2

Mode: Photometric

Sample Gain: 8

Reference Gain: 8

Polarity: Positive

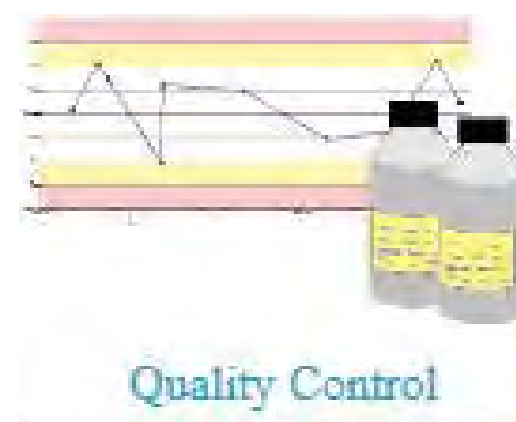
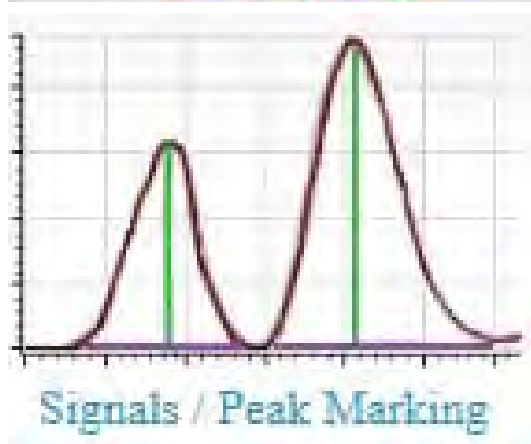
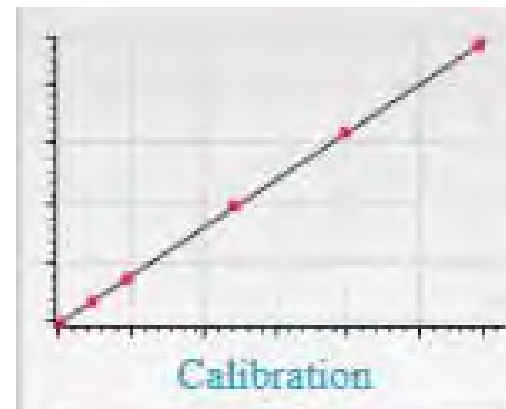
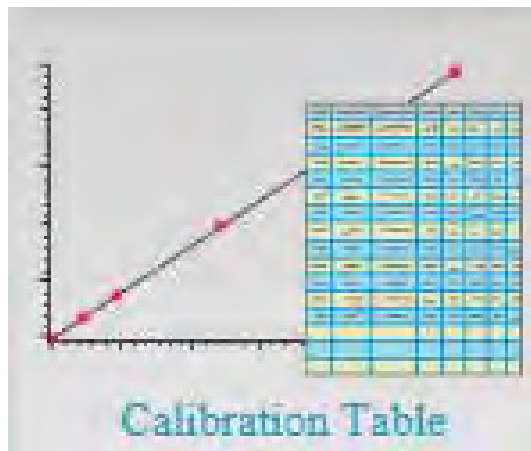
Cell Potential while in Standby mode (volts)

Save

FS3700 State Machine: Idle Signal Poller: Idle Vial#: 0 Reps: 0/0 State: None Active Sample Table: colortest

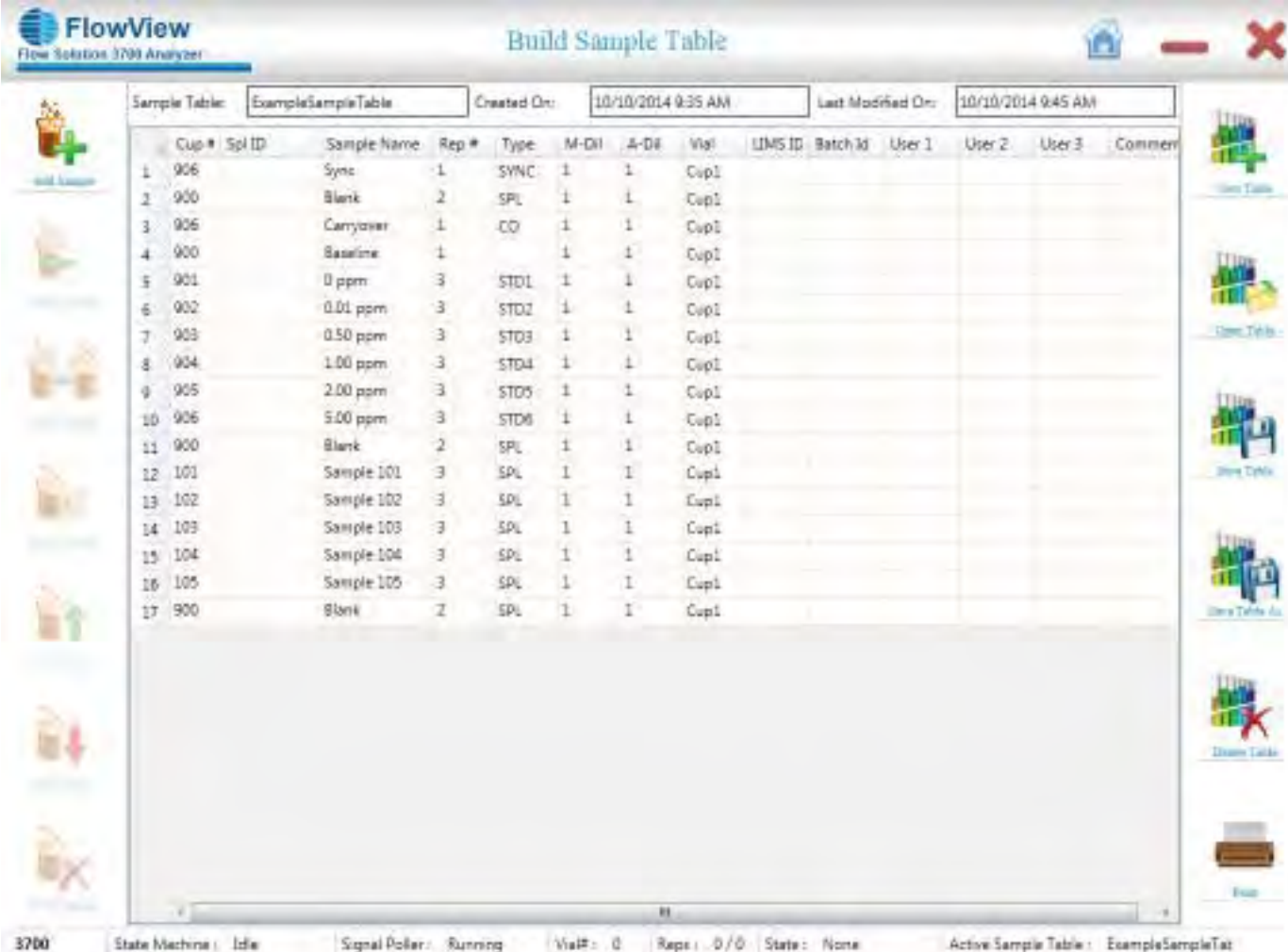
FlowView – Build Method

This screen is divided into four sections that allow the user to define the method and calibration parameters assigned for each channel..



FlowView – Build Sample Table

This screen allows the user to define a sequence of samples to be analyzed



FlowView
Flow Solution 3700 Analyzer

Build Sample Table

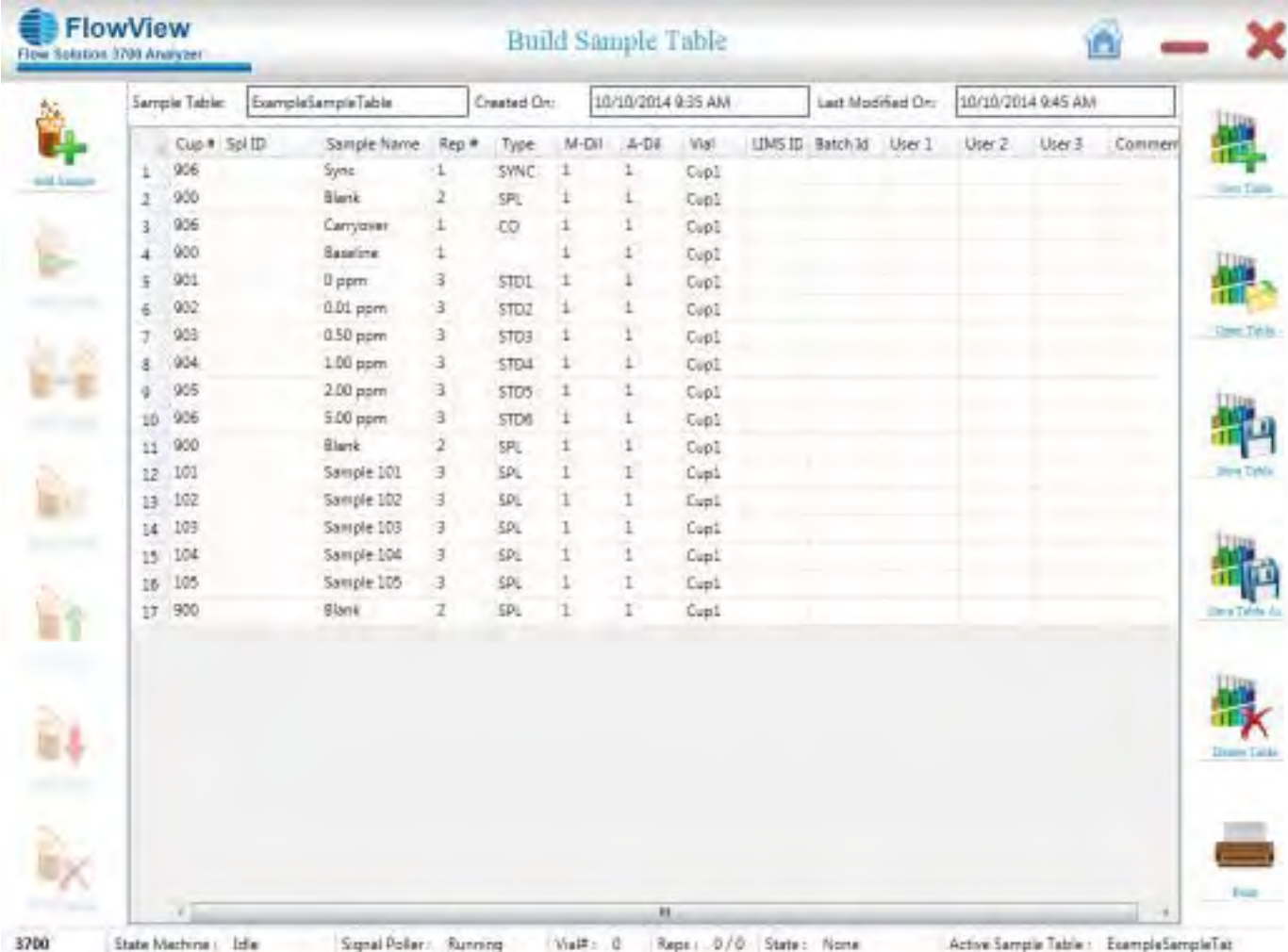
Sample Table: ExampleSampleTable Created On: 10/10/2014 9:35 AM Last Modified On: 10/10/2014 9:45 AM

Cup #	Spl ID	Sample Name	Rep #	Type	M-Dil	A-Dil	Vial	LIMS ID	Batch Id	User 1	User 2	User 3	Comment
1	906	Sync	1	SYNC	1	1	Cup1						
2	900	Blank	2	SPL	1	1	Cup1						
3	906	Carryover	1	CO	1	1	Cup1						
4	900	Baseline	1		1	1	Cup1						
5	901	0 ppm	3	STD1	1	1	Cup1						
6	902	0.01 ppm	3	STD2	1	1	Cup1						
7	903	0.50 ppm	3	STD3	1	1	Cup1						
8	904	1.00 ppm	3	STD4	1	1	Cup1						
9	905	2.00 ppm	3	STD5	1	1	Cup1						
10	906	5.00 ppm	3	STD6	1	1	Cup1						
11	900	Blank	2	SPL	1	1	Cup1						
12	101	Sample 101	3	SPL	1	1	Cup1						
13	102	Sample 102	3	SPL	1	1	Cup1						
14	103	Sample 103	3	SPL	1	1	Cup1						
15	104	Sample 104	3	SPL	1	1	Cup1						
16	105	Sample 105	3	SPL	1	1	Cup1						
17	900	Blank	2	SPL	1	1	Cup1						

3700 State Machine: Idle Signal Poller: Running Vial#: 0 Repe: 0/0 State: None Active Sample Table: ExampleSampleTab

FlowView – Build Sample Table

This screen allows the user to define a sequence of samples to be analyzed



FlowView
Flow Solution 3700 Analyzer

Build Sample Table

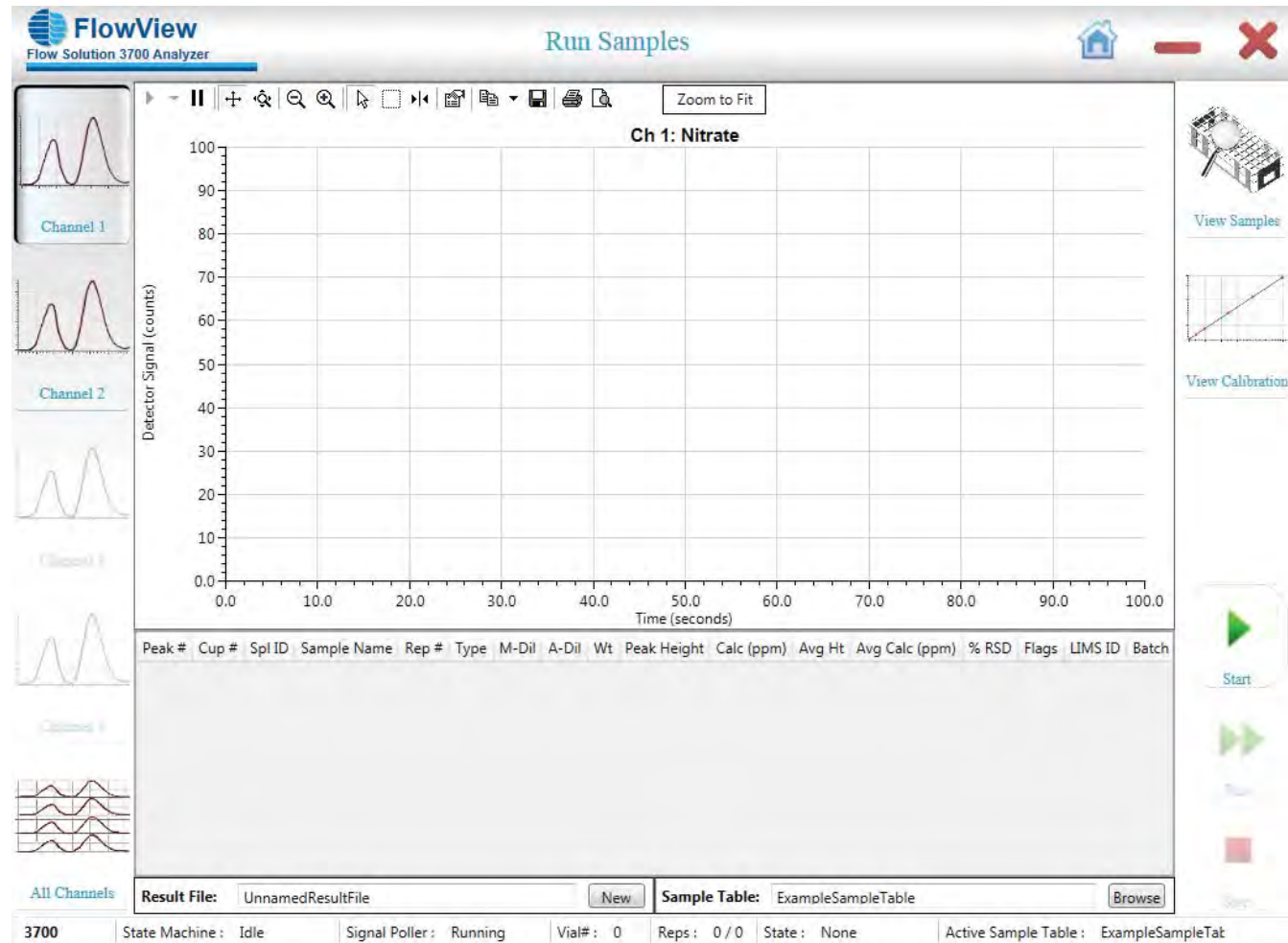
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2	900	Blank	2	SPL	1	1	Cup1						
3	906	Carryover	1	CO	1	1	Cup1						
4	900	Baseline	1		1	1	Cup1						
5	901	0 ppm	3	STD1	1	1	Cup1						
6	902	0.01 ppm	3	STD2	1	1	Cup1						
7	903	0.50 ppm	3	STD3	1	1	Cup1						
8	904	1.00 ppm	3	STD4	1	1	Cup1						
9	905	2.00 ppm	3	STD5	1	1	Cup1						
10	906	5.00 ppm	3	STD6	1	1	Cup1						
11	900	Blank	2	SPL	1	1	Cup1						
12	101	Sample 101	3	SPL	1	1	Cup1						
13	102	Sample 102	3	SPL	1	1	Cup1						
14	103	Sample 103	3	SPL	1	1	Cup1						
15	104	Sample 104	3	SPL	1	1	Cup1						
16	105	Sample 105	3	SPL	1	1	Cup1						
17	900	Blank	2	SPL	1	1	Cup1						

3700 State Machine: Idle Signal Poller: Running Vial#: 0 Repe: 0/0 State: None Active Sample Table: ExampleSampleTab

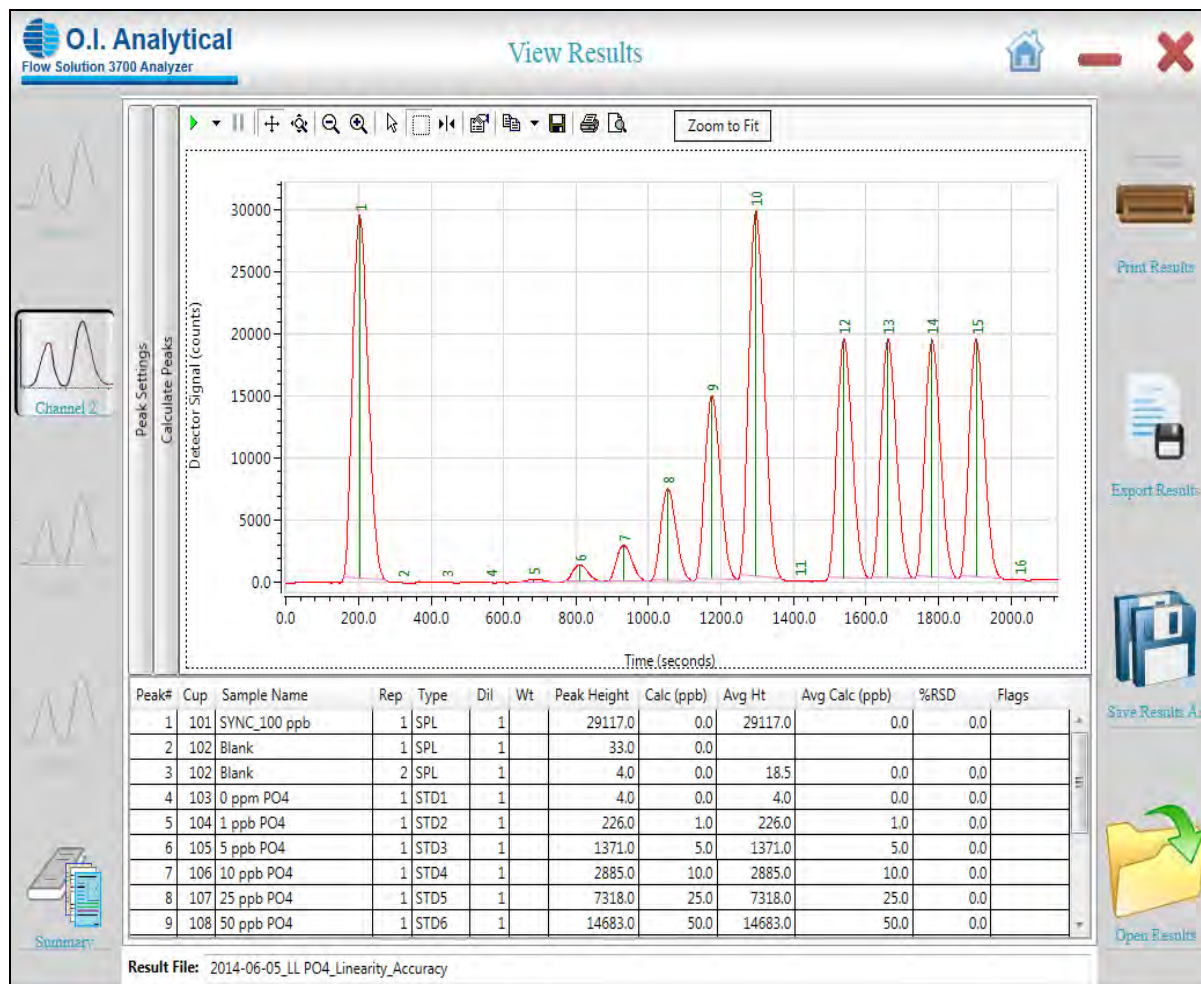
FlowView – Run Samples

Open the **Run Samples** screen to control the sample sequence and monitor data as it is collected




FlowView – View Results

Open the **View Results** screen to review results data. Results may be reviewed from active or completed sequence





FlowView – View Results



FlowView
Flow Solution 3700 Analyzer

View Results

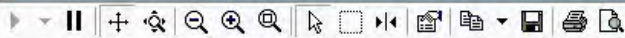



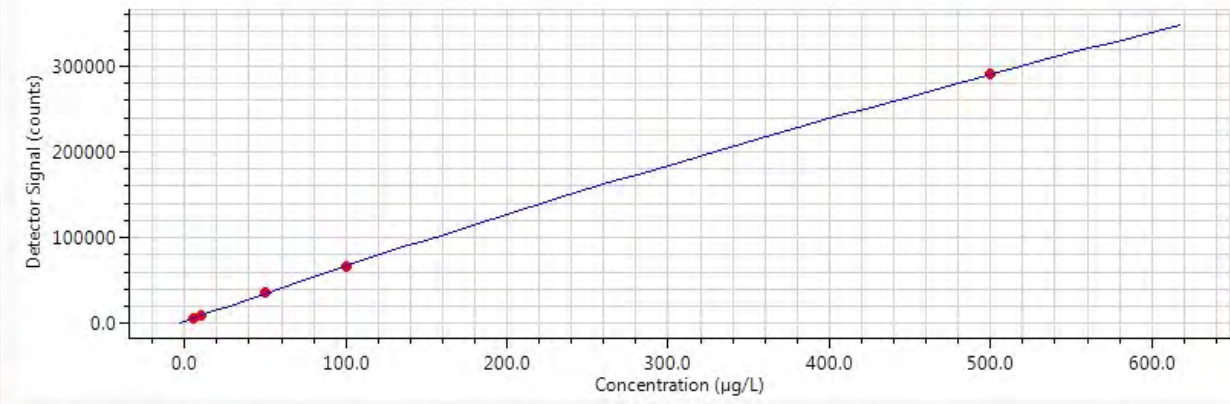
Configuration
Methods
Calibration Results
Sample Table

Channel1
Channel2
Channel3
Channel4
Channel5
Channel6

Curve Fit:	R ² : 0.99999652	RF: n/a	Offset: n/a	HLCAVG: 0.00000000
2nd Order	a: 8.387033E-010	b: 1.490329E-003	c: -3.391394E+000	LHCAVG: 0.00000000

Name	Type	Std. Conc (µg/L)	Avg Height	Calc. Avg Conc (µg/L)	%RSD	Flags
Cal Std3 5 ppb	STD3	5.0000	5960.5	5.5214	0.0	
Cal Std4 10 ppb	STD4	10.0000	8558.0	9.4243	0.0	
Cal Std5 50 ppb	STD5	50.0000	35164.8	50.0529	0.0	
Cal Std6 100 ppb	STD6	100.0000	66860.5	100.0020	0.0	
Cal Std8 500 ppb	STD8	500.0000	290334.0	499.9994	0.0	

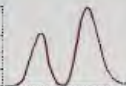











Result File: 20151203-TotalCN-02


Operator ID: OIC

Calculate Peaks


 Channel 1
 Channel 2
 Channel 3
 Channel 4
 Channel 5
 Channel 6
 Summary




Print Results



Export Results



Save Results As



Open Results

FlowView – Maintenance

This screen allows the user to view information and test the operation of all components attached to the FS 3700 from a single screen it may be accessed at any time and provides real-time information on raw detector outputs, FIA valve operation, UV lamps, heaters, and other devices.

FlowView
Flow Solution 3700 Analyzer

Perform Maintenance

System Information

	F/W Rev	PCA Rev
Main PIC (Primary) :	----	----
Main PIC (aux) :	----	----
Comms PIC (primary) :	----	----
Comms PIC (aux) :	----	----
Pump (Primary) :		n/a
Pump (Aux) :		n/a
Auto Sampler :		n/a
SW Rev/Date :	V1.0.2 Nov 20 2014	

Ready Error Busy Reset

FIA Valves

Inject Load

Channel#1 Channel#2

Command

Command: Send

Response:

Heated Zones

Enable Zone	Error	Actual	Desired
<input type="checkbox"/> Channel1		<input type="text"/>	<input type="text"/>
<input type="checkbox"/> Channel2		<input type="text"/>	<input type="text"/>
<input type="checkbox"/> PCA Ambient		<input type="text"/>	Set

Detectors

	F/W Rev	PCA Rev	Status
Detector1 :			0
Detector2 :			0
Detector3 :	Disabled	Disabled	-
Detector4 :	Disabled	Disabled	-

Raw Detector Outputs

	Sample (@gain)	Ref (@gain)
Ch1:		
Ch2:		

UV Lamp

UV Lamp

Diagnostics

Enable Debug Log

3700_Debug.txt

Relay Outputs **Relay Inputs**

Relay #1 Input #1

Relay #2

Auto-Sampler

Go To HOME Pump ON Needle UP

Go To WASH Pump OFF Needle DOWN

Depth:

Move to Vial Vial:

Manual Pump Control

Pump ON Pump OFF

Speed Go

Auto-Sampler: Tray Offset Correction

	60 Pos		90 Pos	
	X-Offset	Y-Offset	X-Offset	Y-Offset
Tray1:	0	0	0	0
Tray2:	1095	0	1075	0
Tray3:	2190	0	2170	0
Tray4:	3285	0	3270	0

Save Offsets

3700 State Machine: Ready Signal Poller: Running Vial#: 0 Reps: 0/0 State: None Active Sample Table: ExampleSampleTab

FlowView – Additional Features

Sample Table Import From CSV file

A CSV file containing sample ID's, cup numbers, sample types, and other information may be imported into the Build Sample Table screen. The user-provided CSV file must conform to a format compatible with the FlowView Build Sample Table editor. Customizable import format via Import screen (Similar to MS excel text file import).

Full Screen GUI

Ability to Pause and Resume the run between samples, emergency samples capability.

Supports Multi Languages

xylem

2

FS3700
Chemistries

O·Analytical 
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FS3700 Chemistry Analyzer Methods



FS 3700 Automated Chemistry Analyzer Methods

Analyte	Technique	Method	Operating Range	MDL ¹	Throughput	Channel ² Part #	Cartridge Part #
Ammonia	SFA, Gas Diffusion	USEPA 350.1	0.01-20.0 ppm 10.0-20,000 ppb	0.001 ppm 1.0 ppb	40 samples per hour	330109	330094
Ammonia, Nitrogen (Phenate)	FIA	USEPA 350.1	0.01-20 ppm	0.002 ppm	51 samples per hour	330353	330354
Chloride	SFA	Standard Methods 4500-Cl-E	1.0-200 ppm	0.12 ppm	60 samples per hour	330360	330361
Cyanide Available (1677)	FIA	OIA-1677-09	0.002-5.00 ppm 2.0-5,000 ppb	0.0005 ppm 0.5 ppb	30 samples per hour	330107	330092
Cyanide Available (D6888) (Sulfide abatement)	FIA	ASTM D6888-09	0.005-0.5 ppm 5.0-500 ppb	0.002 ppm 2.0 ppb	30 samples per hour	330106	330091
Cyanide Free (D7237)	FIA	ASTM D7237-10	2.0-500 ppb	0.5 ppb	30 samples per hour	330355	330356
Cyanide Free	Photometric Detection	ISO 14403	2.0-500 ppb	0.4 ppb	30 samples per hour	330371	330372
Cyanide Post-Distillation	FIA, Photometric Detection	USEPA 335.4	5.0-500 ppb	0.5 ppb	30 samples per hour	330351	330352
Cyanide Total	SFA, UV Digestion	ASTM D7511-09	0.003-0.5 ppm 3.0-500 ppb	0.0001 ppm 1.0 ppb	30 samples per hour	330076	330090
Cyanide Total	Photometric Detection	ISO 14403	2.0-500 ppb	0.4 ppb	30 samples per hour	330366	330367
Hexavalent Chromium	FIA	USEPA 600/4-79-020	0.01-10 mg/L	0.0011 mg/L	48 samples per hour	331543	331544

FS3700 Chemistry Analyzer Methods

Analyte	Technique	Method	Operating Range	MDL ¹	Throughput	Channel ² Part #	Cartridge Part #
MBAS	Continuous Flow	ISO 16265	0.025-2.0 mg/L as LAS	0.008 mg/L as LAS	24 samples per hour	330357	330358
Nitrate/Nitrite	FIA	USEPA 353.2	0.01-10.0 ppm 10.0-10,000 ppb	0.001 ppm 1.0 ppb	60 samples per hour	330108	330093
	SFA		0.005-10.0 ppm	0.001 ppm	40 samples per hour	331377	331376
Nitrate/Nitrite in Milk	FIA w/ In-line Dialysis	ISO 14673-3	Nitrate 0.5 mg/L - 5.0 mg/L Nitrite 0.025 µg/L - 0.400 µg/L	Nitrate 0.016 mg/L Nitrite 0.0016 mg/L	30 samples per hour	331534	331535
Phenol In-line distillation	SFA	USEPA 420.2	1.0-500 ppb	0.5 ppb	22 samples per hour	330363	330364
Phenol Post-Distillation	FIA	USEPA 420.4	0.01-2.00 ppm 10.0-2,000 ppb	0.002 ppm 2.0 ppb	90 samples per hour	330110	330083
Phosphorus All Forms	FIA	USEPA 365.1	0.01-5.0 ppm 10.0-5,000 ppb	0.001 ppm 1.0 ppb	60 samples per hour	330111	330096
Phosphorus All Forms - Low Level	FIA	USEPA 365.1	0.001-0.1 ppm 1.0-100 ppb	0.0003 ppm 0.3 ppb	45 samples per hour	330112	330095
Sulfate	FIA Photometric	USEPA 375.2	1.0 mg/L - 25 mg/L	0.1 mg/L	40 samples per hour	331385	331386
TKN Total Kjeldahl Nitrogen	SFA, Gas Diffusion	USEPA 351.2	0.01-20.0 ppm 10.0-20,000 ppb	0.001 ppm 1.0 ppb	40 samples per hour	330109	330094

¹ Method Detection Limit (MDL) determined in accordance with 40 CFR Part 136 Appendix B

² Channels include the cartridge, detector, and valve (if required).



151 Cimarron Road
PO Box 9010
College Station, Texas
77842-9010

(979) 690-1711
(800) 653-1711 USA/Canada
FAX (979) 690-0480

www.oico.com
E-mail: oi-info@xyleminc.com

Publication 40750517

Publication 40750517

Ammonia Nitrogen (Phenate) – US EPA 350.1

Channel P/N: 330353

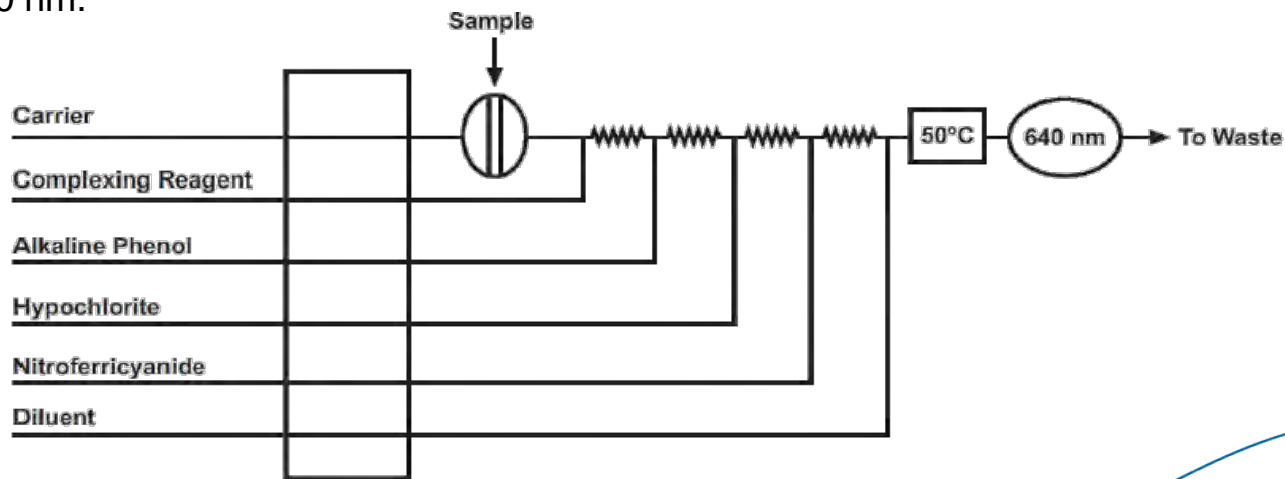
Cartridge P/N: 330354

This method is used for the determination of ammonia in drinking water, surface water, and domestic and industrial wastes according to **USEPA Method 350.1** and **Standard Methods 4500-NH₃H**. This method can also be used for the determination of ammonia nitrogen in potassium chloride (KCl) extracts of soils and plants.

Prior to analysis, the ammonia is buffered at a pH of 9.5 and distilled into a solution of boric acid. Ammonia reacts with alkaline phenol and hypochlorite to form indophenol blue in an amount proportional to the ammonia concentration. The blue color is intensified with sodium nitroferricyanide, and the absorbance is measured at 640 nm.

Method Performance

Range	0.01-20 mg/L ammonia as nitrogen
Rate	51 samples/hour
Precision	1% RSD at mid-point of range
Method Detection Limit (MDL)	0.002 mg/L



Ammonia Nitrogen (Phenate) – US EPA 350.1

Channel P/N: 330353

Cartridge P/N: 330354

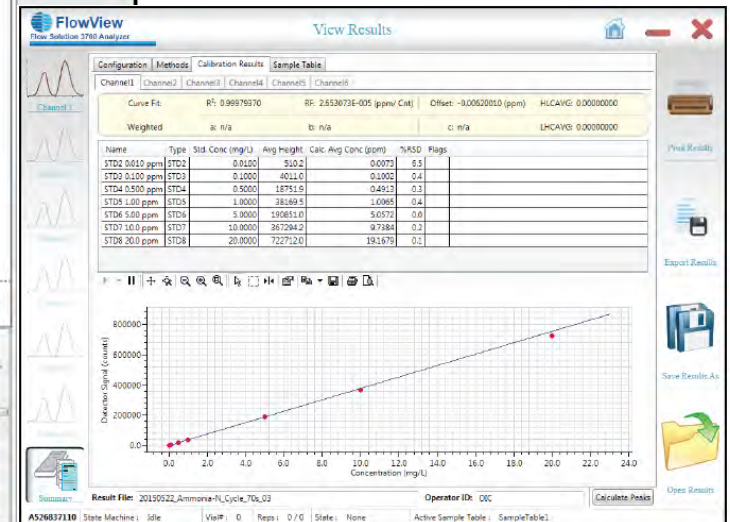
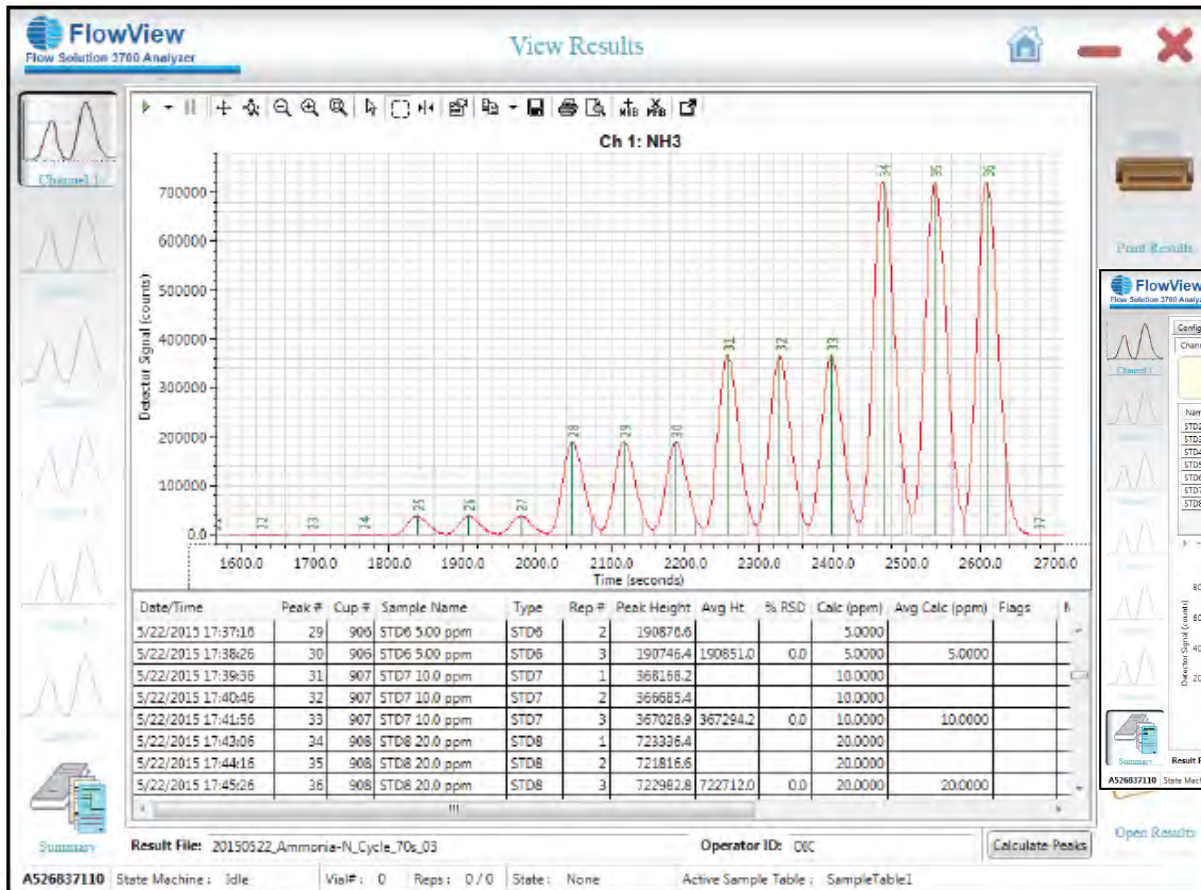
Results

	0.01 ppb	0.5 ppb	1.00 ppb
Replicate 1	0.0095	0.5062	1.0269
Replicate 2	0.0098	0.5059	1.0253
Replicate 3	0.0094	0.5073	1.0240
Replicate 4	0.0088	0.5076	1.0273
Replicate 5	0.0095	0.5061	1.0231
Replicate 6	0.0090	0.5065	1.0206
Replicate 7	0.0107	0.5049	1.0219
Replicate 8	0.0101	0.5061	1.0223
Replicate 9	—	0.5059	1.0292
Replicate 10	—	0.5066	1.0263
Mean	0.0100	0.5063	1.0247
Standard Deviation	0.000605	0.000759	0.002753
%RSD	6.30%	0.15%	0.27%
%Accuracy	96.0%	101.3%	102.5%
MDL	0.0018 ppm	—	—

Ammonia Nitrogen (Phenate) – US EPA 350.1

Channel P/N: 330353
 Cartridge P/N: 330354

Graph of Results and Calibration Curve



Ammonia/TKN by Gas Diffusion

Channel P/N: 330108
Cartridge P/N: 330094

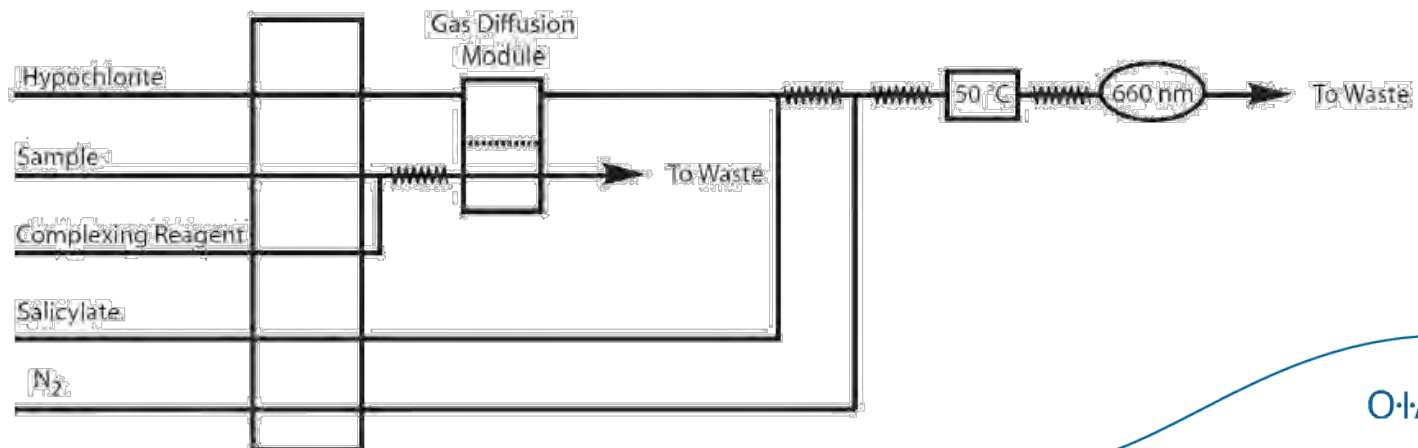
This method is used for determining Total Kjeldahl Nitrogen (TKN) in drinking water, surface water, municipal and industrial wastewater, according to **EPA 351.2**. A gas diffusion step is used to separate TKN from particulates, ions, and copper-containing digest matrices.

Method Performance

Range	0.01 – 20 mg/L N
Rate	40 samples/hour
Precision	2% RSD at mid-point of range
Method Detection Limit (MDL)	0.001 mg/L N as ammonia

This method can also be used to determine ammonia nitrogen according to **USEPA method 350.1**.

The sample pH is raised to a pH of >11, and the ammonia molecules generated pass through a gas diffusion membrane and are absorbed into an alkaline hypochlorite solution to form chloramine. The chloramine reacts with salicylate to form indophenol blue in an amount that is proportional to the ammonia concentration. Sodium nitroferricyanide intensifies the blue color. Measure the absorbance at 660 nm.



Ammonia/TKN by Gas Diffusion

Channel P/N: 330108

Cartridge P/N: 330094

Results

	Calibrant 0.01 mg/L	Calibrant 0.1 mg/L	Calibrant 1.0 mg/L	Calibrant 10.0 mg/L
Replicate 1	0.0164	0.0987	1.000	10.8048
Replicate 2	0.0154	0.0977	1.014	10.6499
Replicate 3	0.0161	0.0999	1.011	10.8464
Replicate 4	0.0165	0.1015	1.025	10.6782
Replicate 5	0.0161	0.0981	1.003	10.6677
Replicate 6	0.0162	0.0977	1.007	10.6621
Replicate 7	0.0161	0.1012	0.983	10.5252
Replicate 8	0.0156	0.0971	0.985	10.4613
Replicate 9	0.0160	0.0984	0.993	10.5411
Replicate 10	—	0.0999	1.001	10.6293
Mean	0.0160	0.0990	1.002	10.6466
Standard Deviation	0.000350	0.001526	0.012962	0.118858
%RSD	2.18%	1.54%	1.29%	1.12%
%Accuracy	—	—	—	—
MDL	0.0010	—	—	—

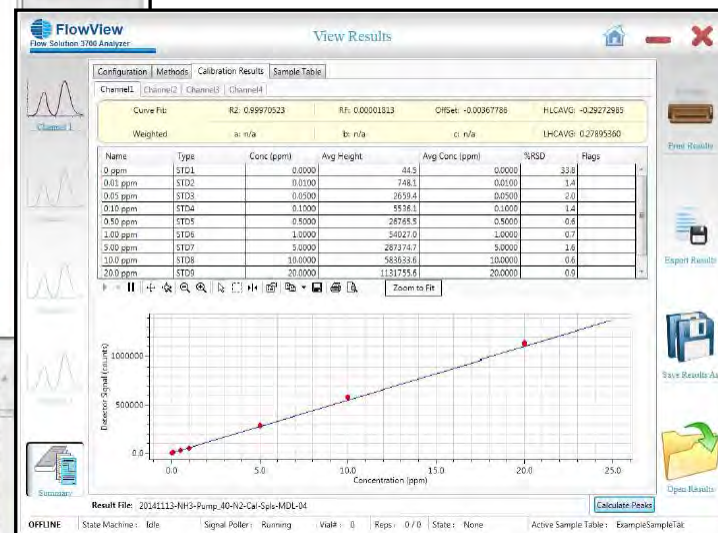
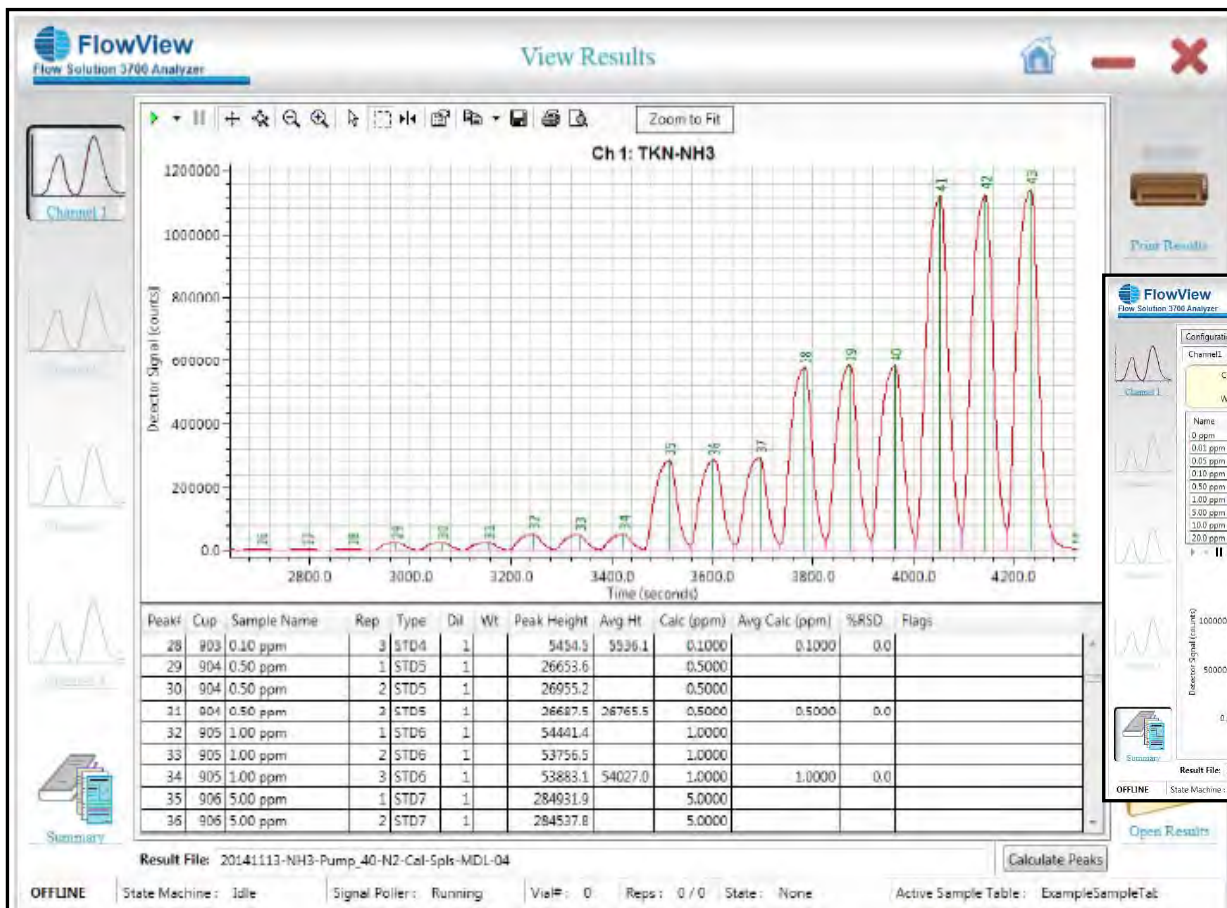
Digest TKN samples prior to analysis in the presence of sulfuric acid, potassium sulfate, and a copper catalyst at a final temperature of 380 °C. Free ammonia and organic nitrogen compounds convert to ammonium sulfate under these conditions.

A digestion step is not carried out when analyzing ammonia singly by **USEPA 350.1**.

Ammonia/TKN by Gas Diffusion

Channel P/N: 330108
Cartridge P/N: 330094

Graph of Results and Calibration Curve



Chloride – Standard Methods

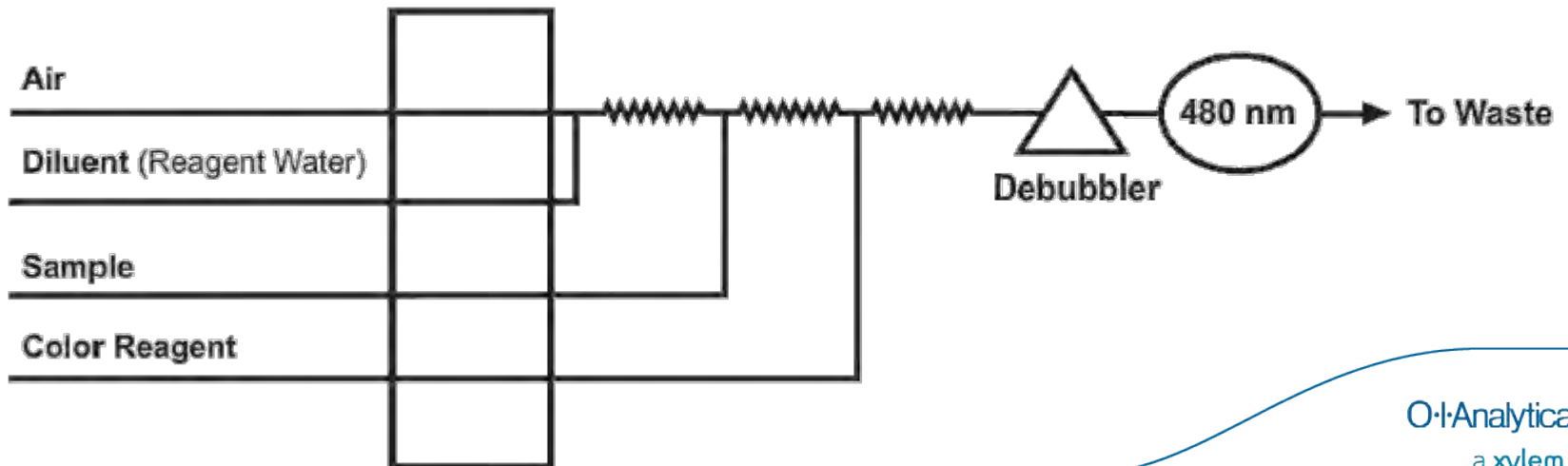
Channel P/N: 330360
Cartridge P/N: 330361

This method is used for the determination of chloride in drinking water, surface water, and domestic and industrial waste according to **Standard Methods 4500–Cl-E**. Additionally, this method enables chloride analysis according to **ISO Method 15682**.

Method Performance

Range	1.0–200 mg/L
Rate	60 samples/hour
Precision	1% RSD at mid-point of range
Method Detection Limit (MDL)	0.12 mg/L

Chloride reacts with mercuric thiocyanate, liberating thiocyanate ion by the formation of soluble mercuric chloride. In the presence of ferric ion, free thiocyanate ion forms a highly colored ferric thiocyanate complex. The colored complex is measured at 480 nm.



Chloride – Standard Methods

Channel P/N: 330360
Cartridge P/N: 330361

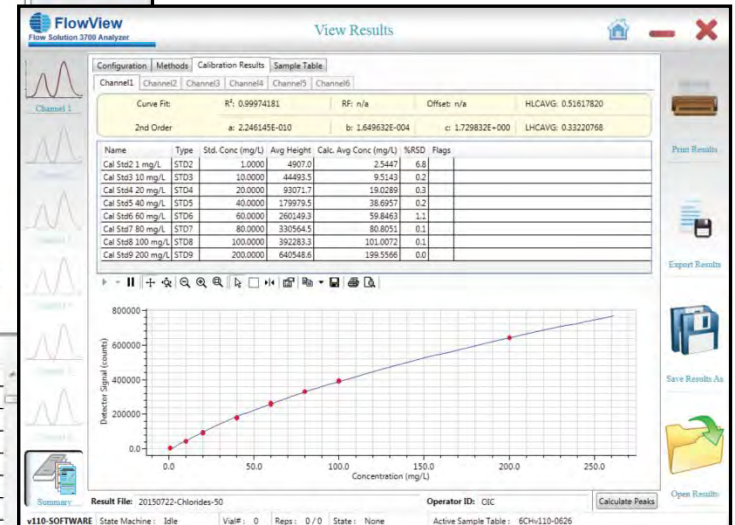
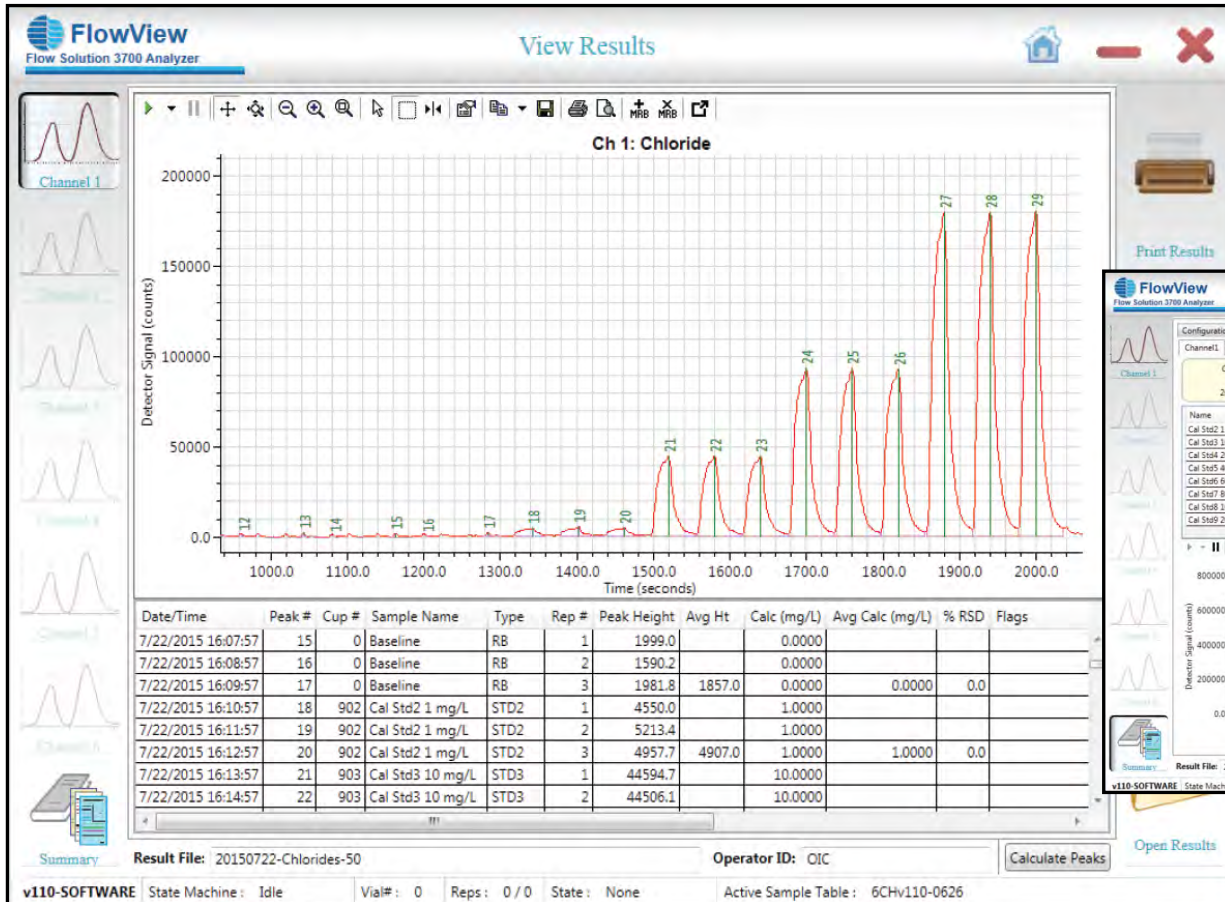
Results

	0.5 mg/L	1 mg/L	10 mg/L	100 mg/L
Replicate 1	0.6752	1.0329	9.7626	99.7625
Replicate 2	0.6822	1.0672	9.7847	100.2298
Replicate 3	0.6496	1.0017	9.8320	100.0321
Replicate 4	0.7375	1.0777	9.8945	100.1124
Replicate 5	0.6740	1.0932	9.9176	100.0661
Replicate 6	0.6798	1.0550	9.9516	100.2666
Replicate 7	0.6975	1.1361	9.9969	99.9733
Replicate 8	—	1.0705	10.1044	100.7019
Replicate 9	—	1.1044	10.0625	100.2342
Replicate 10	—	—	10.0483	100.4492
Mean	0.685 mg/L	1.071 mg/L	9.94 mg/L	100.2 mg/L
Standard Deviation	0.027134	0.039396	0.118677	0.261223
%RSD	3.96%	3.68%	1.19%	0.26%
%Accuracy	—	107.1%	99.4%	100.2%
MDL	0.0852 mg/L	—	—	—

Chloride – Standard Methods

Channel P/N: 330360
Cartridge P/N: 330361

Graph of Results and Calibration Curve



Cyanide, Available – ASTM D6888

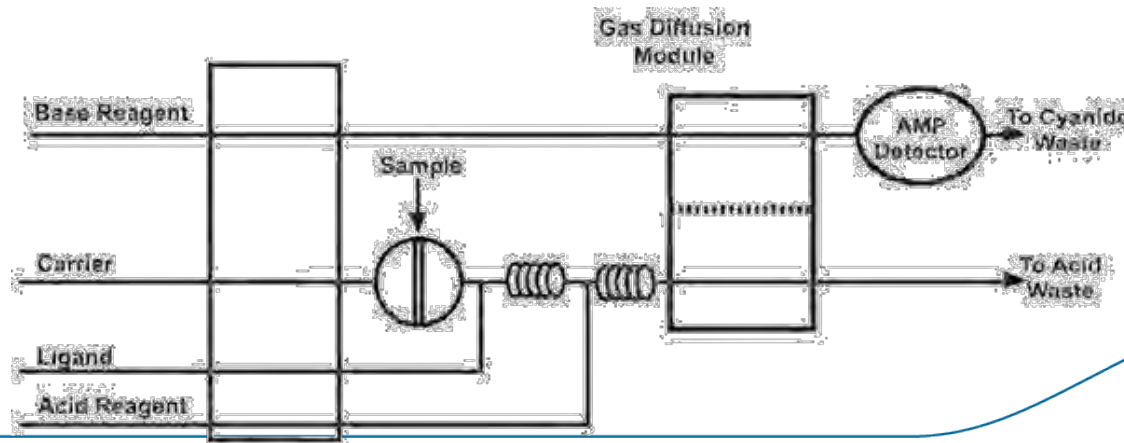
Channel P/N: 330106
 Cartridge P/N: 330091

This method is used for determining available cyanide in water and wastewater by automated ligand exchange, flow injection analysis, and amperometric detection according to **ASTM Method D6888-09**. This method is approved for use in the USEPA's data gathering and monitoring programs associated with the Clean Water Act.

Method Performance

Range	5.0 µg/L–0.5 mg/L
Rate	30 samples/hour
Precision at 50 µg/L	<2% RSD
Method Detection Limit (MDL)	1.0 µg/L

Ligand exchange reagents form thermodynamically stable complexes with transition metal ions, releasing the cyanide ion from the cyano-complexes. Addition of acid converts the cyanide ion to hydrogen cyanide gas (HCN), which passes under a gas diffusion membrane. The hydrogen cyanide gas diffuses through the membrane into an alkaline receiving solution where it converts back to cyanide ion. The cyanide ion is monitored amperometrically with a silver working electrode, silver/silver chloride reference electrode, and platinum/stainless steel counter electrode at an applied potential of zero volt. The current generated is proportional to the cyanide concentration present in the original sample.



Cyanide, Available – ASTM D6888

Channel P/N: 330106
Cartridge P/N: 330091

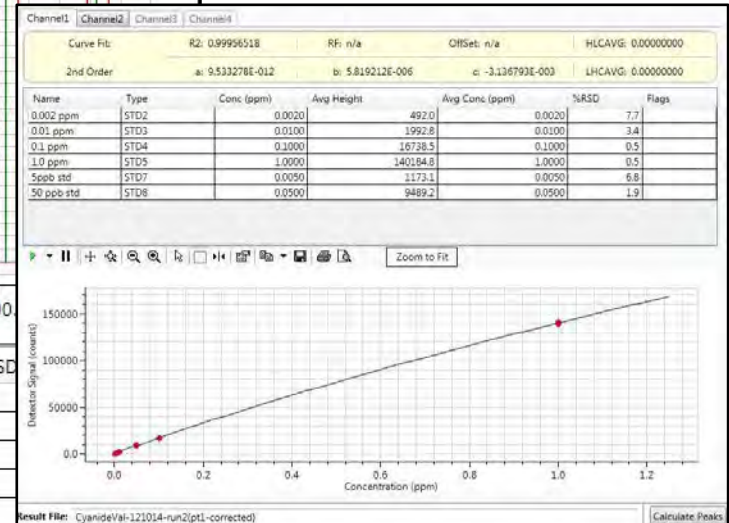
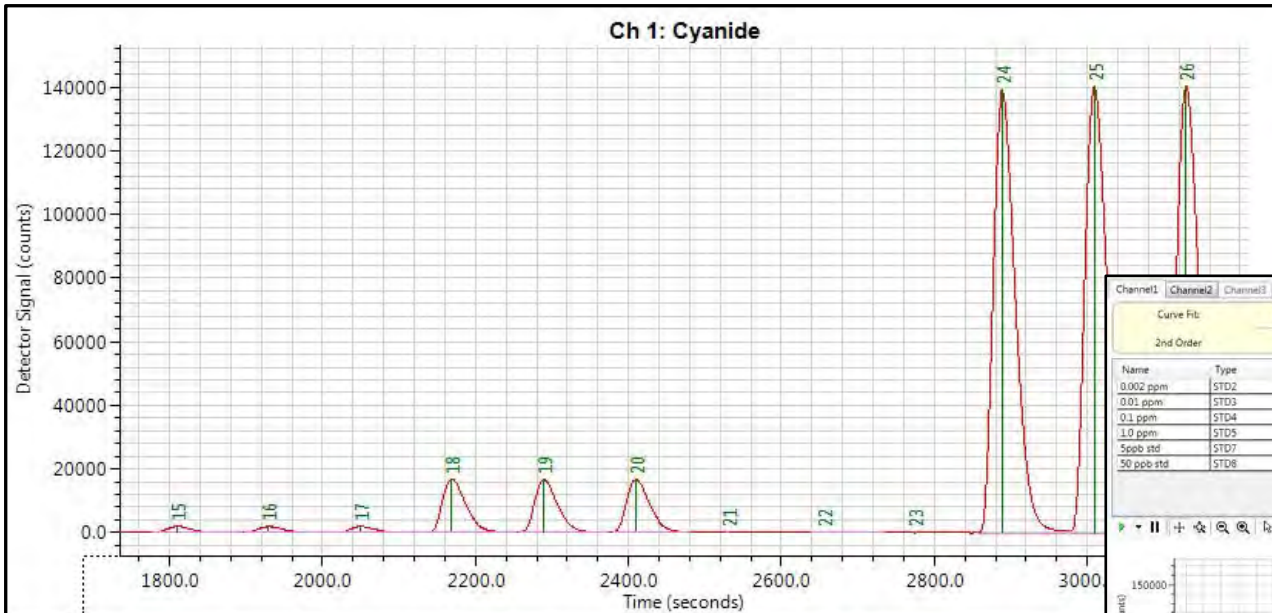
Results

	Calibrant 0.002 mg/L	Calibrant 0.010 mg/L	Calibrant 0.050 mg/L	Calibrant 0.100 mg/L
Replicate 1	0.0014	0.0108	0.0532	0.1044
Replicate 2	0.0016	0.0108	0.0520	0.1044
Replicate 3	0.0016	0.0107	0.0517	0.1036
Replicate 4	0.0013	0.0106	0.0515	0.1037
Replicate 5	0.0010	0.0105	0.0514	0.1032
Replicate 6	0.0013	0.0104	0.0514	0.1033
Replicate 7	0.0015	0.0102	0.0510	0.1027
Replicate 8	0.0014	0.0108	0.0513	0.1029
Replicate 9	—	0.0104	0.0514	0.1035
Replicate 10	—	0.0102	0.0508	0.1039
Mean	0.0014	0.0105	0.0516	0.1036
Standard Deviation	0.000196	0.000237	0.000669	0.000570
%RSD	14.12%	2.25%	1.30%	0.55%
%Accuracy	—	—	—	—
MDL	0.000587	—	—	—

Cyanide, Available – ASTM D6888

Channel P/N: 330106
Cartridge P/N: 330091

Graph of Results and Calibration Curve



Peak#	Cup	Sample Name	Rep	Type	Dil	Wt	Peak Height	Calc (ppm)	Avg Ht	Avg Calc (ppm)	%RSD
17	904	0.01 ppm	3	STD3	1		2000.0	0.0100	1992.8	0.0100	
18	905	0.1 ppm	1	STD4	1		16828.0	0.1000			
19	905	0.1 ppm	2	STD4	1		16734.9	0.1000			
20	905	0.1 ppm	3	STD4	1		16652.6	0.1000	16738.5	0.1000	
21	901	blank	1	SPL	1		316.1	-0.0064			
22	901	blank	2	SPL	1		370.2	-0.0058	343.1	-0.0061	-6.3
23	901	baseline	1	RB	1		301.9	-0.0065	301.9	-0.0065	0.0
24	907	1.0 ppm	1	STD5	1		139406.7	1.0000			
25	907	1.0 ppm	2	STD5	1		140382.2	1.0000			

Cyanide, Available – OIA-1677

Channel P/N: 330107
Cartridge P/N: 330092

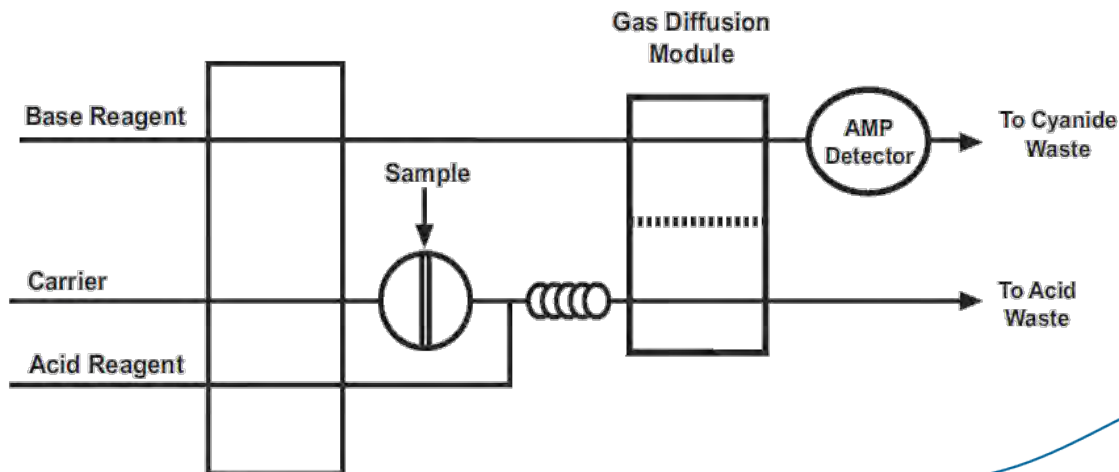
This method is used for determining available cyanide in water and wastewater by ligand exchange, flow injection analysis, and amperometric detection according to **USEPA OIA-1677-09, USEPA OIA-1677-DW and ASTM Method D6888-09.**

This method is used in the USEPA's data gathering and monitoring programs associated with the Clean Water Act, Resource Conservation and Recovery Act, Comprehensive Environmental Response, Compensation and Liability Act, and Safe Drinking Water Act.

Ligand exchange reagents are added to samples prior to analysis to release the cyanide ion from the cyano-complexes. An aliquot of the treated sample is injected into the FIA system. Addition of acid converts the cyanide ion to hydrogen cyanide gas (HCN), which passes under a gas diffusion membrane. The hydrogen cyanide gas diffuses through the membrane into an alkaline receiving solution where it converts back to cyanide ion. The cyanide ion is monitored amperometrically with a silver working electrode, silver/silver chloride reference electrode, and platinum/stainless steel counter electrode at an applied potential of zero volt. The current generated is proportional to the cyanide concentration present in the original sample.

Method Performance

Range	2.0 µg/L–5.0 mg/L
Rate	30 samples/hour
Precision	3% RSD
Method Detection Limit (MDL)	0.5 µg/L



Cyanide, Available – OIA-1677

Channel P/N: 330107
Cartridge P/N: 330092

Results

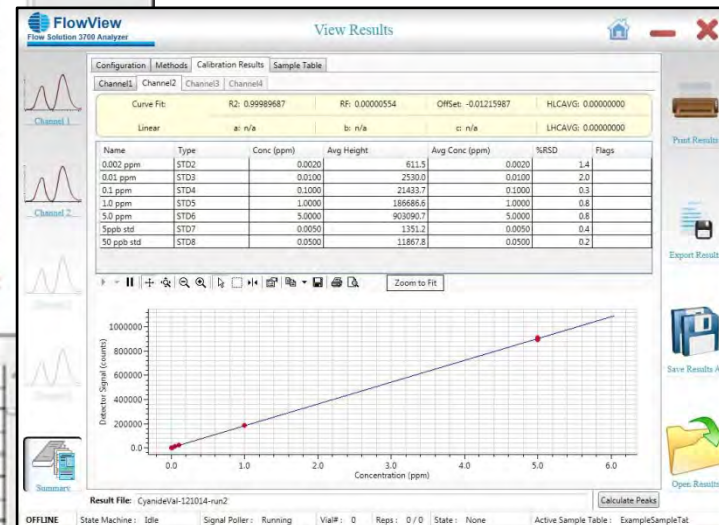
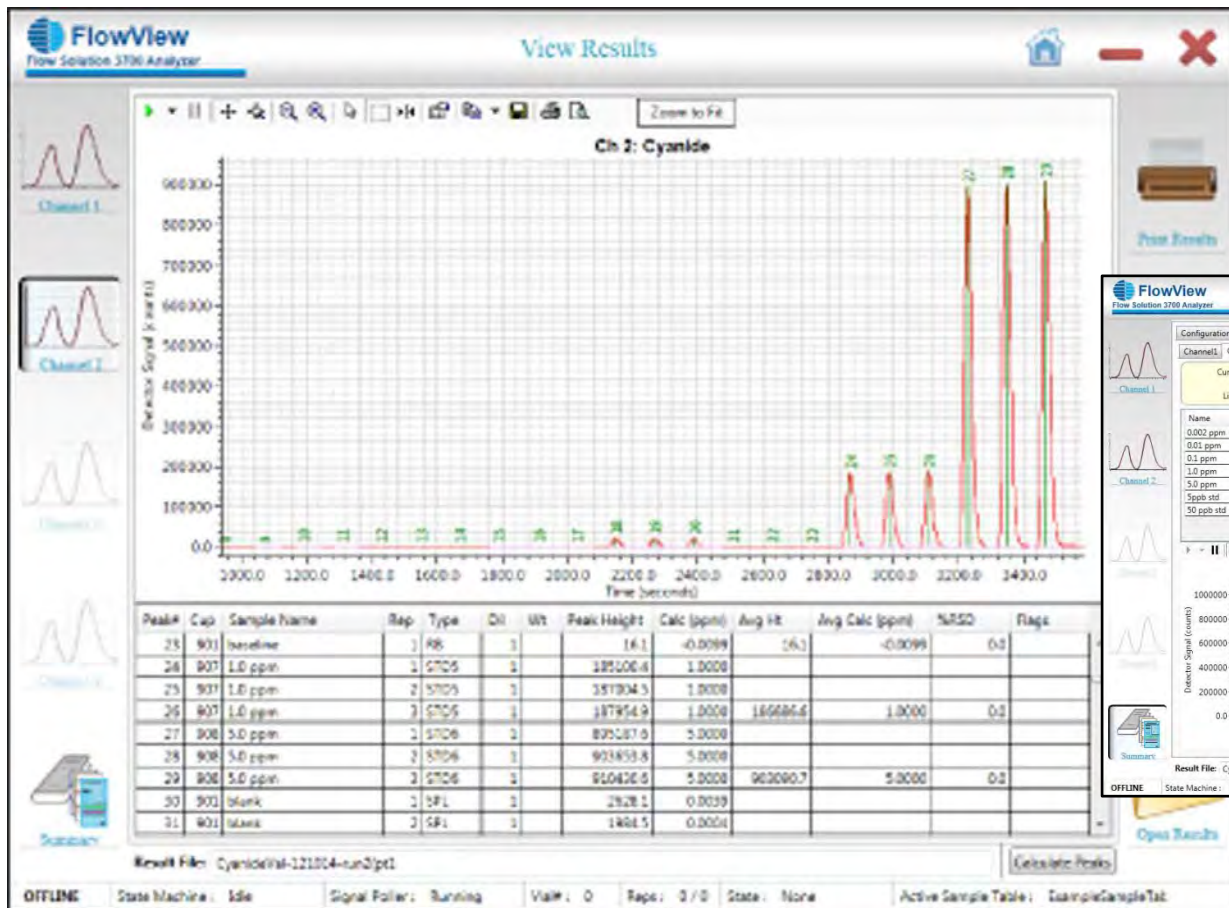
	Calibrant 0.002 mg/L	Calibrant 0.010 mg/L	Calibrant 0.050 mg/L	Calibrant 0.100 mg/L
Replicate 1	0.0011	0.0107	0.0527	0.1021
Replicate 2	0.0011	0.0106	0.0520	0.1012
Replicate 3	0.0011	0.0106	0.0522	0.1020
Replicate 4	0.0011	0.0106	0.0522	0.1015
Replicate 5	0.0008	0.0105	0.0520	0.1013
Replicate 6	0.0009	0.0105	0.0521	0.109
Replicate 7	0.0009	0.0104	0.0520	0.1018
Replicate 8	0.0009	0.0104	0.0518	0.1006
Replicate 9	0.0009	0.0104	0.0519	0.1007
Replicate 10	0.0008	0.0102	0.0519	0.1007
Mean	0.0010	0.0105	0.0520	0.1013
Standard Deviation	0.000114	0.000127	0.000139	0.000536
%RSD	11.71%	1.22%	0.27%	0.53%
%Accuracy	—	—	—	—
MDL	0.000363	—	—	—

The “OIA” in OIA-1677 stands for “OI Analytical”

Cyanide, Available – OIA-1677

Channel P/N: 330107
 Cartridge P/N: 330092

Graph of Results and Calibration Curve



Total Cyanide by ASTM D7511-12

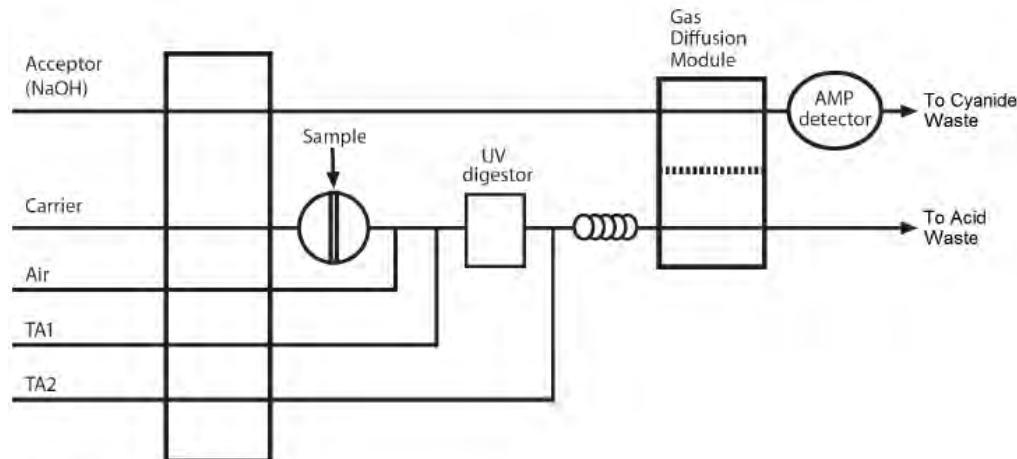
Channel P/N: 330076
Cartridge P/N: 330090

This method is used for determining total cyanide in drinking and surface waters, as well as domestic and industrial wastewaters by **ASTM D7511-12**. Cyanide ion (CN^-), hydrogen cyanide in water ($\text{HCN}(\text{aq})$), and the cyano-complexes of zinc, copper, cadmium, mercury, nickel, silver, and iron may be determined by this method.

Prior to analysis, treat the sample to remove potential interferences. Ultraviolet (UV) digestion releases cyanide from cyanide complexes. Acid addition converts cyanide ion to hydrogen cyanide gas (HCN), which passes under a gas diffusion membrane. The hydrogen cyanide gas diffuses through the membrane into an alkaline receiving solution, where it converts back to cyanide ion. A silver working electrode, silver/silver chloride reference electrode, and platinum/stainless steel counter electrode at an applied potential of zero volt amperometrically monitor the cyanide ion. The current generated is proportional to the cyanide concentration present in the original sample.

Method Performance

Range	2.0–500 $\mu\text{g/L}$
Rate	30 samples/hour
Precision	<2% RSD at mid-point of range
Method Detection Limit (MDL)	1.0 $\mu\text{g/L}$



Total Cyanide by ASTM D7511

Channel P/N: 330076
Cartridge P/N: 330090

Results

	2.00 µg/L	100.0 µg/L
Replicate 1	2.6434	114.8519
Replicate 2	2.7488	112.6534
Replicate 3	2.7458	114.7577
Replicate 4	2.5968	114.9816
Replicate 5	2.4553	116.7331
Replicate 6	2.5314	115.4497
Replicate 7	2.7471	114.6902
Replicate 8	2.4054	114.1623
Replicate 9	2.3064	118.9074
Replicate 10	2.4239	116.1910
Mean	2.5604 µg/L	115.3378 µg/L
Standard Deviation	0.1606	1.6712
%RSD	6.27%	1.45%
%Recovery	128.0%	115.3%
MDL	0.4529 µg/L	—

Part Numbers

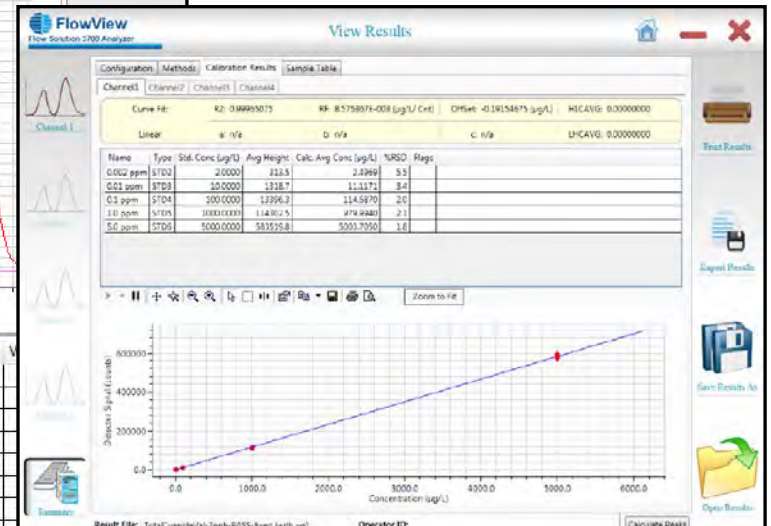
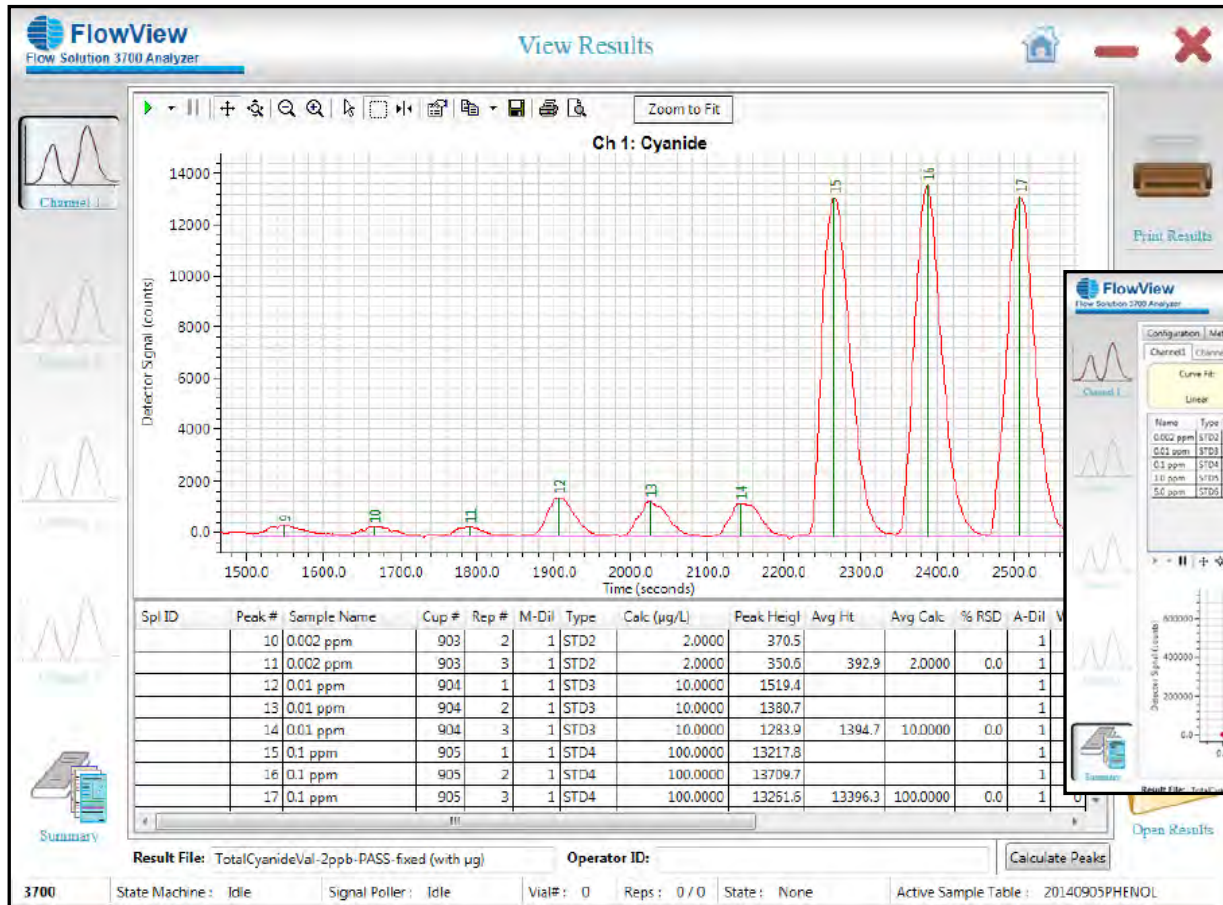
Consumable	Part Number
Pump tubes kit - Total Cyanide, ASTM D7511	330090TK
Gas Diffusion Membrane – Cyanide (5 pk)	A001520
Flow Solution - Base Reagent	A001103
Flow Solution - Total Acid 1 (TA1)	A001505
Flow Solution - Total Acid 2 (TA2)	A001872
Flow Solution - Total Carrier	A001668
200 µL Injection/Bypass Loop	285684
Amperometric Cell, tested	330001
Amperometric Detector – Reference Electrode	329513
Teflon UV Digestion Coil	311084
UV Lamp, 312 nm	A001682
PEEK Autosampler Probe for RA/3090/3360 Sampler	325331

Optional Accessories	Part Number
Challenge Matrix, ASTM D7365	327788
Teflon Heater Coil Assembly	329486
SFA customization kit – Total Cyanide D7511	330375

Total Cyanide by ASTM D7511

Channel P/N: 330076
Cartridge P/N: 330090

Graph of Results and Calibration Curve



Cyanide, Post-distillation – EPA 335.4

Channel P/N: 330110

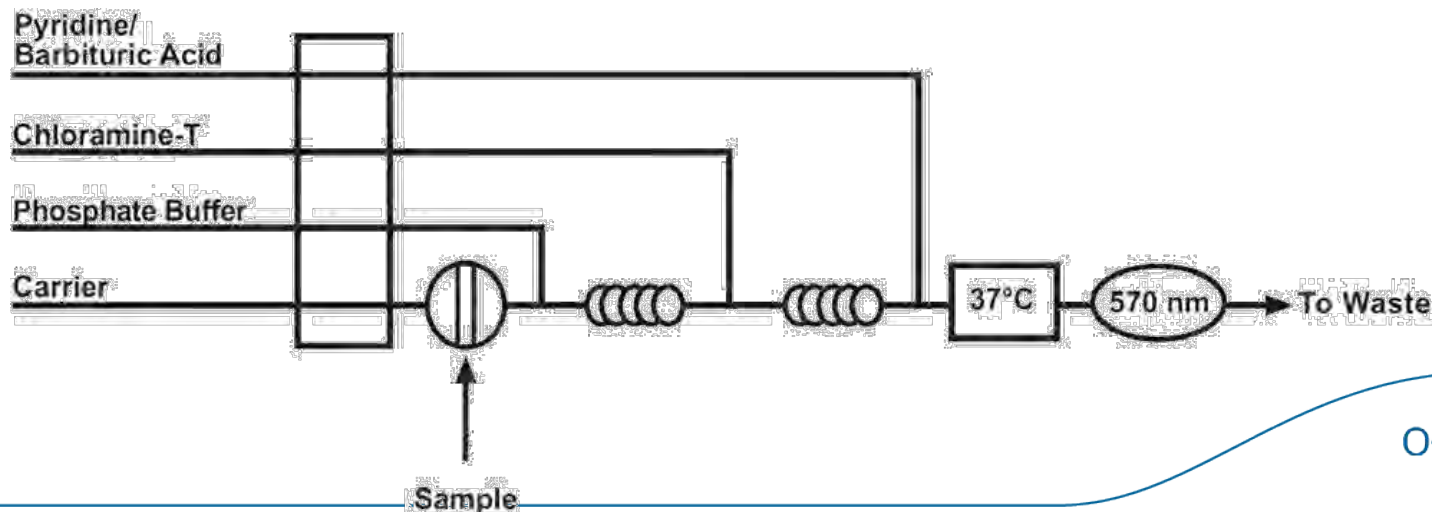
Cartridge P/N: 330352

This method is used for the determination of cyanide in distilled samples that includes water, wastewater, soil, and sludge, according to **USEPA method 335.4**. This method also applies to determining total cyanide in samples distilled by other methods, such as **Standard Methods 4500-CN** and **ASTM D2036**, as long as calibration standards are prepared with the same sodium hydroxide concentration used for samples.

Method Performance

Range	5.0–500 µg/L
Rate	30 samples/hour
Precision	1% RSD
Method Detection Limit (MDL)	0.5 µg/L

Prior to analysis, off-line manual distillation releases cyanide from cyanide complexes. Sodium cyanide is converted to cyanogen chloride by reaction with chloramine-T at a pH less than 8. The cyanogen chloride then reacts with pyridine-barbituric acid to form a red-colored complex. The absorbance is measured at 570 nm.



Cyanide, Post-distillation – EPA 335.4

Channel P/N: 330110

Cartridge P/N: 330352

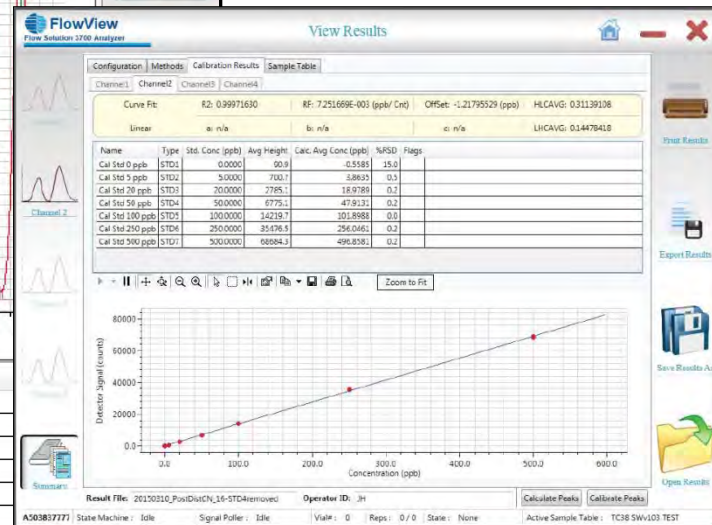
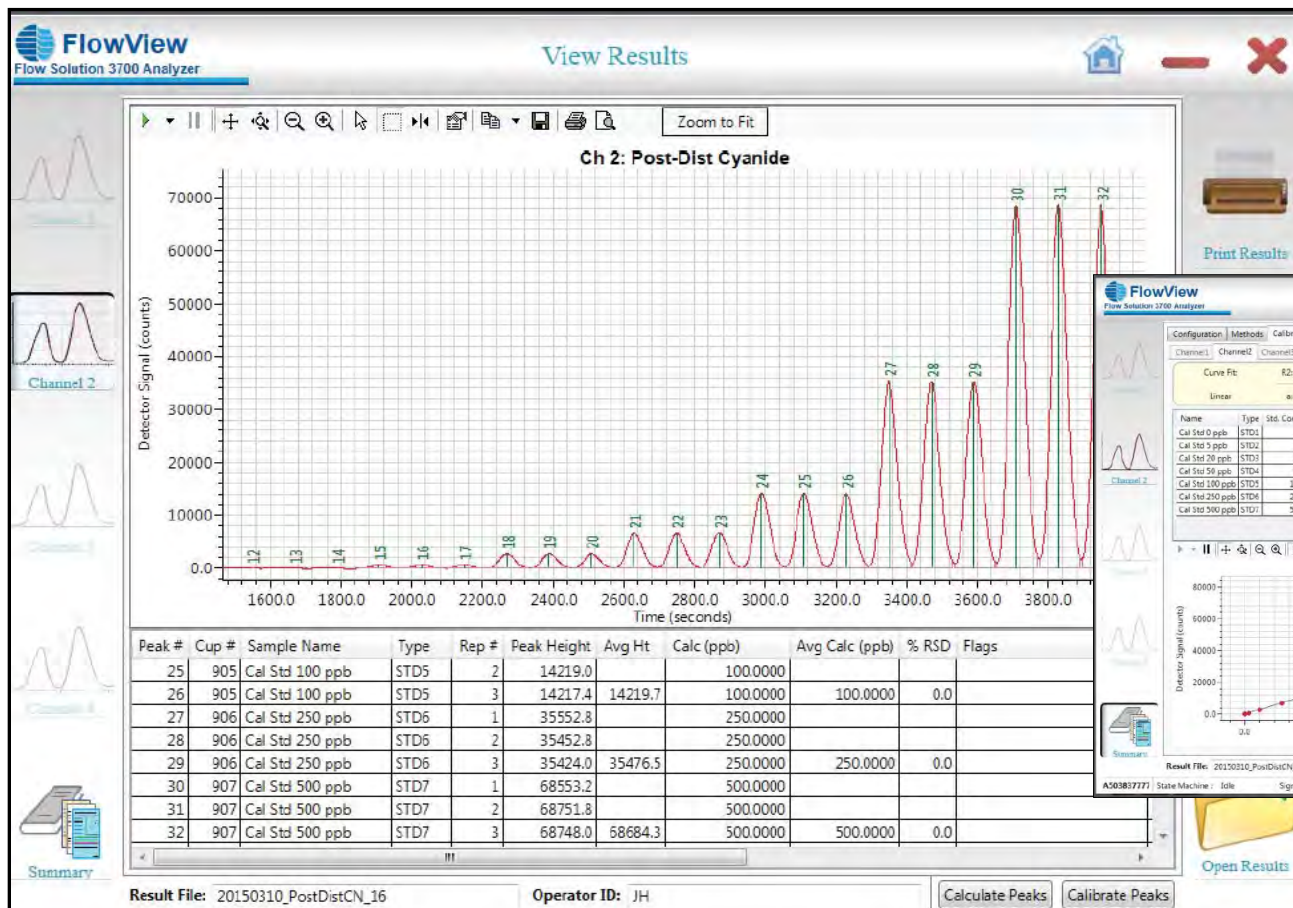
Results

	2 ppb	5 ppb	500 ppb
Replicate 1	1.4623	4.6182	487.7388
Replicate 2	1.4391	4.5515	486.2203
Replicate 3	1.426	4.5384	485.4604
Replicate 4	1.4652	4.5123	483.3835
Replicate 5	1.5957	4.5065	483.1268
Replicate 6	1.5522	4.5761	485.5778
Replicate 7	1.3955	4.6124	484.4727
Replicate 8	1.4855	4.5051	481.2515
Replicate 9	—	4.5718	483.0252
Replicate 10	—	—	483.7127
Mean	1.478	4.5547	484.3970
Standard Deviation	0.066384	0.043257	1.882898
%RSD	4.49%	0.95%	0.39%
%Accuracy	73.9%	91.1%	96.9%
MDL	0.199 ppb	—	—

Cyanide, Post-distillation – EPA 335.4

Channel P/N: 330110
Cartridge P/N: 330352

Graph of Results and Calibration Curve



Nitrate/Nitrite – EPA 353.2 FIA and SFA



Method
Nitrate/Nitrite by USEPA 353.2
Document #43770916

Flow Solution™ FS3700 Automated Chemistry Analyzer

Nitrate plus Nitrite Nitrogen or Nitrite Nitrogen by Segmented Flow Analysis (SFA) or Flow Injection Analysis (FIA), USEPA 353.2

Cartridge Part Numbers 331376CT (SFA) and 330093CT (FIA)

Scope and Application

This method is used for the determination of nitrate (NO_3^-) plus nitrite (NO_2^-) or nitrite singly in drinking water, groundwater, surface water, and domestic and industrial wastes according to **USEPA Method 353.2**, **Standard Methods 4500- NO_3I** and **4500- NO_3F** and **ISO Method 13395**.^{1,2,3} This method includes information for both FIA and SFA.

Nitrate/Nitrite – EPA 353.2 FIA and SFA

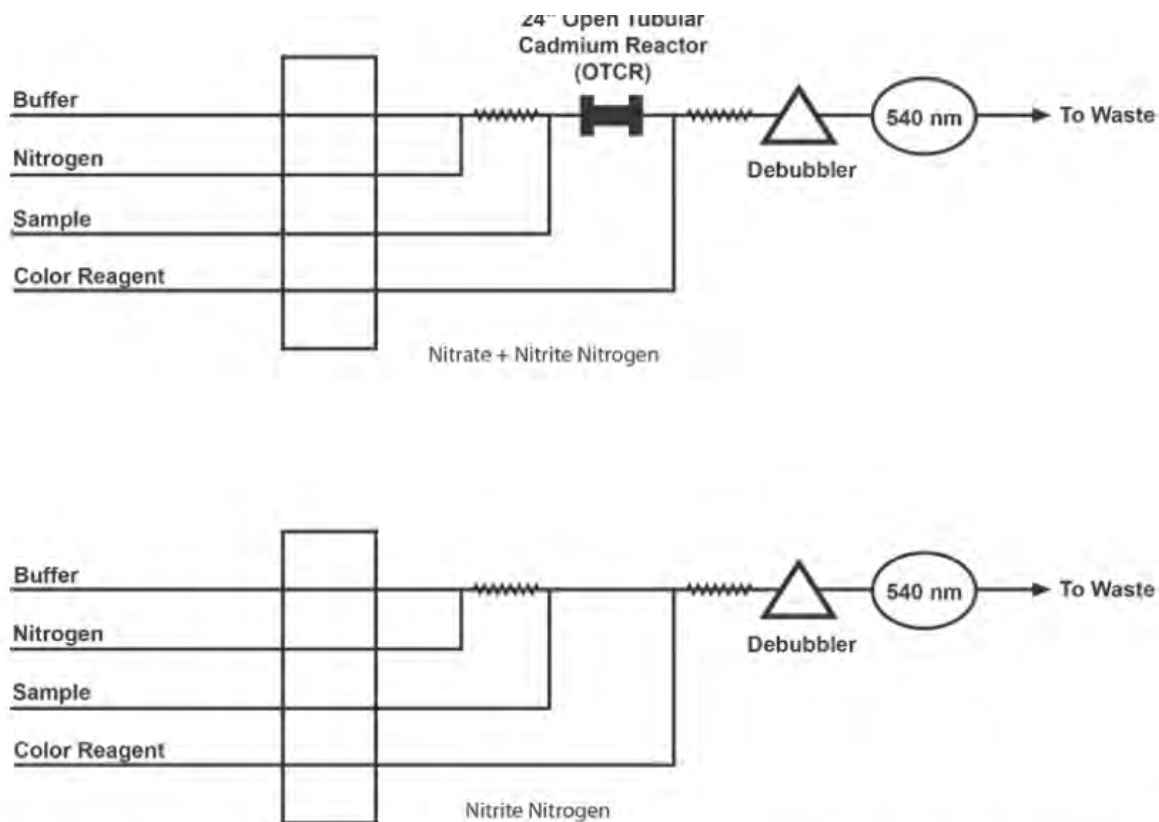


Figure 1. General SFA Flow Diagram for Nitrate plus Nitrite Nitrogen and Nitrite Nitrogen by USEPA 353.2

Cartridge Part Numbers 331376CT (SFA) & 330093CT (FIA)

Nitrate/Nitrite – EPA 353.2

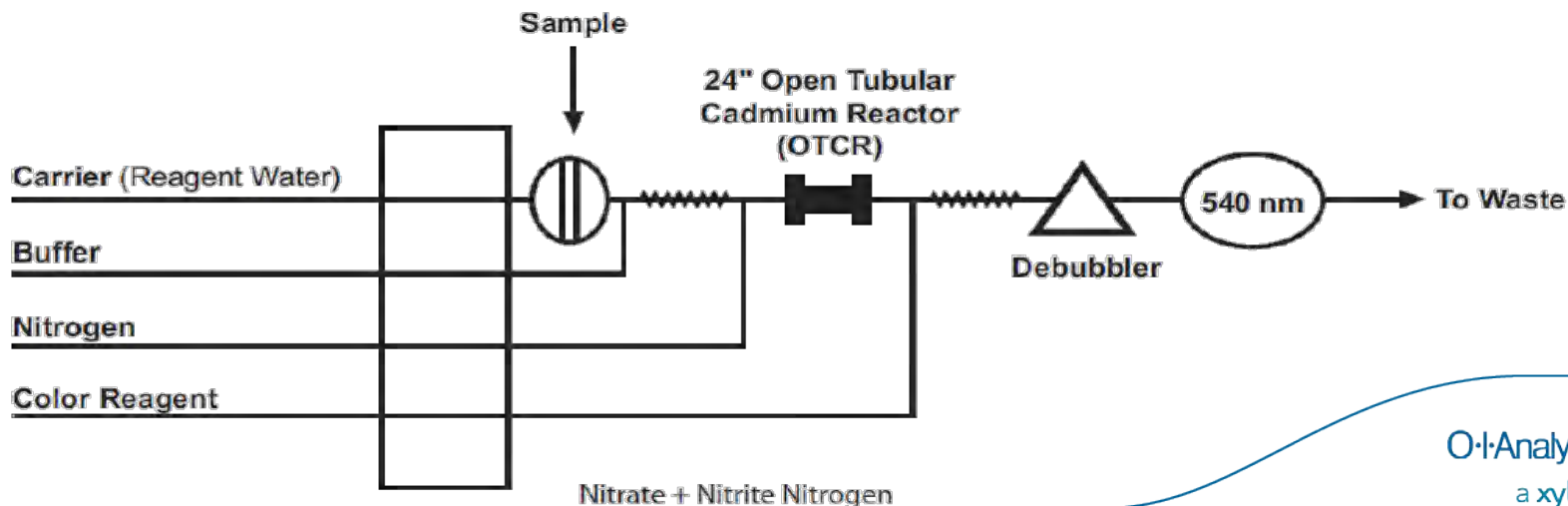
Channel P/N: 330109
Cartridge P/N: 330093

This method is used for the determination of nitrate (NO_3^-) plus nitrite (NO_2^-) or nitrite singly in drinking water, groundwater, surface water, and domestic and industrial wastes according to **US EPA Method 353.2**, **Standard Methods 4500- NO_3^- -1**, and **ISO Method 13395**.

Method Performance

Range	0.005 – 10 mg/L
Rate	60 samples/hour
Precision	1% RSD at mid-point of range
Method Detection Limit (MDL)	0.001 mg/L

A filtered sample is passed through a column to quantitatively reduce nitrate to nitrite using cadmium metal. Nydahl provides a good discussion of nitrate reduction by cadmium metal. The nitrite (that was originally present plus reduced nitrate) is determined by diazotizing with sulfanilamide and subsequently coupling with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye that is measured colorimetrically at 540 nm.



Nitrate/Nitrite – EPA 353.2

Channel P/N: 330109
Cartridge P/N: 330093

Results

	NO ₃ 0.050 mg N/L	NO ₃ 0.100 mg N/L	NO ₃ 5 mg N/L	NO ₃ 10 mg N/L	NO ₂ 5 mg N/L	NO ₂ 10 mg N/L
Replicate 1	0.0054	0.1021	4.8783	9.5327	4.9975	9.7191
Replicate 2	0.0054	0.1017	4.8717	9.5269	5.0029	9.7073
Replicate 3	0.0053	0.1011	4.8796	9.5144	5.0000	9.7011
Replicate 4	0.0054	0.1006	4.8728	9.4945	4.9786	9.6795
Replicate 5	0.0050	0.0997	4.8593	9.4838	4.9680	9.6468
Replicate 6	0.0049	0.0993	4.8208	9.4725	4.9074	9.6382
Replicate 7	0.0049	0.0983	4.8504	9.4361	4.9044	9.6305
Replicate 8	—	—	—	—	4.9030	—
Replicate 9	—	—	—	—	—	—
Replicate 10	—	—	—	—	—	—
Mean (mg N/L)	0.00519	0.1004	4.86184	9.49441	4.9577	9.6746
Standard Deviation	0.000241	0.001367	0.020918	0.033946	0.045233	0.036098
%RSD	4.65%	1.36%	0.43%	0.36%	0.91%	0.37%
%Recovery	103.7%	100.4%	97.2%	94.9%	99.2%	96.7%
MDL	0.00076 mg N/L	—	—	—	—	—

Part Numbers

Consumable	Part Number
Pump tubes kit - Nitrate/Nitrite by USEPA 353.2	330093TK
Brij [®] -35	326126
24" OTCR (Cadmium coil assembly) – with nut and ferrule	A000897
Nitrogen Pillow Assembly	A000811
100 µL Injection/Bypass Loop	285676
PEEK Autosampler Probe for RA/3090/3360 Sampler	325331

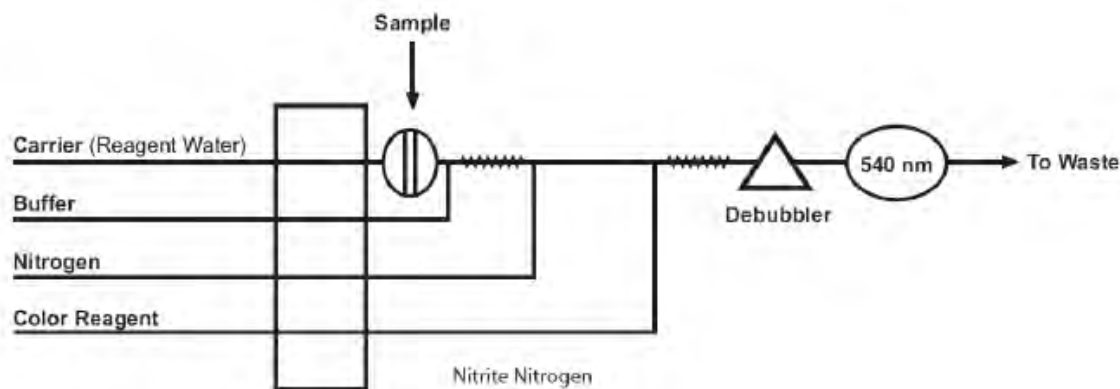
Nitrate/Nitrite – EPA 353.2

Channel P/N: 330109

Cartridge P/N: 330093

Results

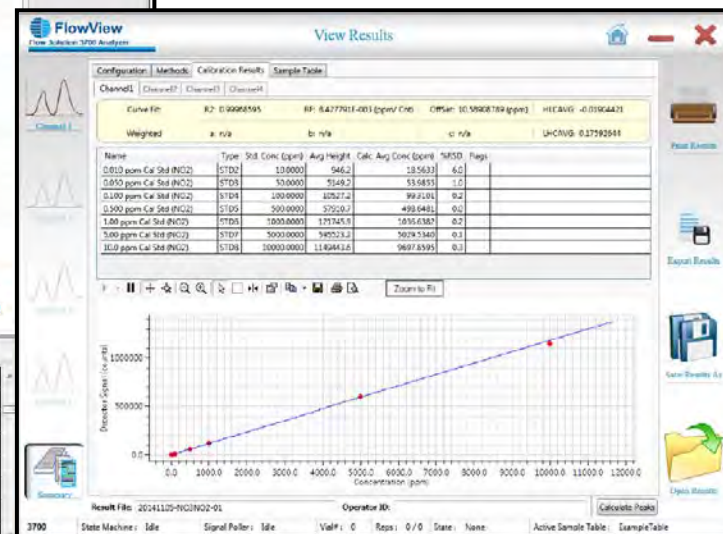
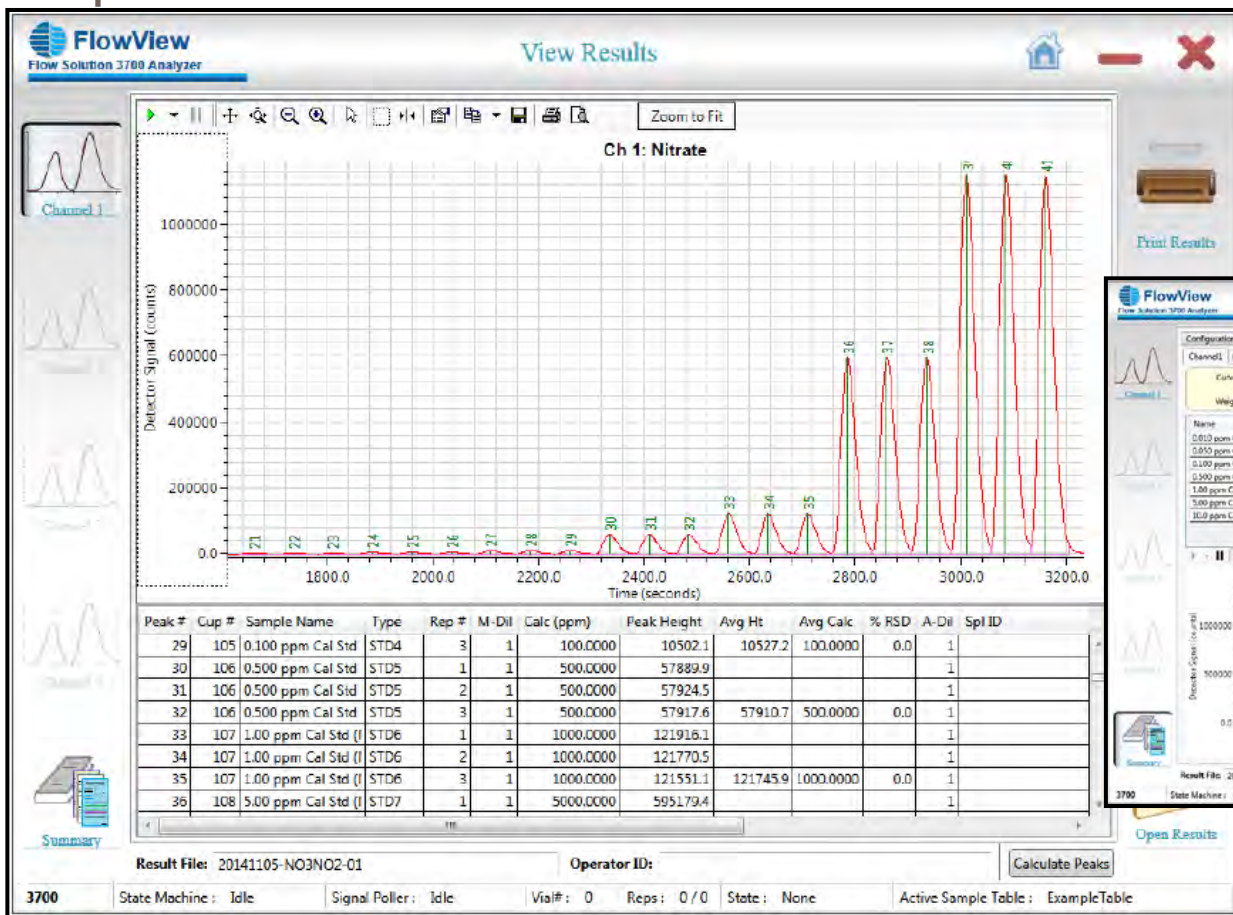
	NO ₂ 0.010 mg N/L	NO ₂ 0.100 mg N/L	NO ₂ 5 mg N/L	NO ₂ 10 mg N/L
Replicate 1	0.0102	0.1058	4.9975	9.7191
Replicate 2	0.0101	0.1055	5.0029	9.7073
Replicate 3	0.0102	0.1048	5.0000	9.7011
Replicate 4	0.0098	0.1046	4.9786	9.6795
Replicate 5	0.0099	0.1039	4.9680	9.6468
Replicate 6	0.0101	0.1035	4.9074	9.6382
Replicate 7	0.0099	0.1021	4.9044	9.6305
Replicate 8	0.0098	0.1014	4.9030	—
Replicate 9	0.0097	—	—	—
Replicate 10	0.0099	—	—	—
Mean (mg N/L)	0.00996	0.1040	4.9577	9.6746
Standard Deviation	0.000178	0.001563	0.045233	0.036098
%RSD	1.78%	1.50%	0.91%	0.37%
%Recovery	99.6%	104.0%	99.2%	96.7%
MDL	0.00050 mg N/L	—	—	—



Nitrate/Nitrite – EPA 353.2

Channel P/N: 330109
Cartridge P/N: 330093

Graph of Results and Calibration Curve



Phenol, In-line distillation – EPA 420.4

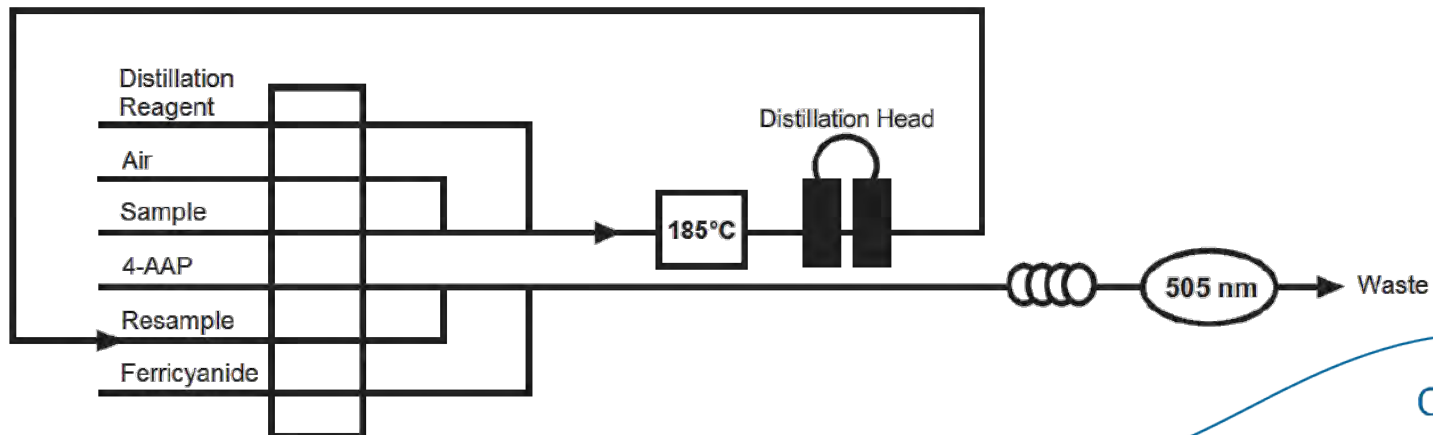
Channel P/N: 330363
Cartridge P/N: 330364

This method is used for the determination of phenolic compounds in drinking water, surface water, and domestic and industrial wastes according to **USEPA 420.4**. Additionally, this method enables phenol index analysis following in-line distillation according to **ISO Method 14402**.

Method Performance

Range	1.0 – 500 µg/L
Rate	22 samples/hour
Precision	2% RSD at mid-point of range
Method Detection Limit (MDL)	0.5 µg/L

Phenol is distilled in-line from an acidic solution at 185 °C. The phenol distillate reacts with 4-aminoantipyrine (4-AAP) and alkaline ferricyanide (FeCN) to form a red complex. The absorbance is measured at 505 nm.



Phenol, In-line distillation – EPA 420.4

Channel P/N: 330363

Cartridge P/N: 330364

Results

	1 µg/L	5 µg/L	50 µg/L
Replicate 1	1.1941	4.5274	49.4971
Replicate 2	1.259	4.4309	49.7406
Replicate 3	1.2604	4.7959	50.0485
Replicate 4	1.3147	4.5577	49.7794
Replicate 5	1.3493	4.4695	49.1596
Replicate 6	1.2715	4.7114	49.4735
Replicate 7	1.3361	4.2635	49.6329
Replicate 8	—	4.3804	49.5348
Replicate 9	—	4.5998	49.7715
Replicate 10	—	4.3411	48.4288
Mean	1.284 µg/L	4.508 µg/L	49.51 µg/L
Standard Deviation	0.053759	0.165622	0.446343
%RSD	4.19%	3.67%	0.90%
%Accuracy	128.4%	90.2%	99.0%
MDL	0.169 µg/L	—	—

Part Numbers

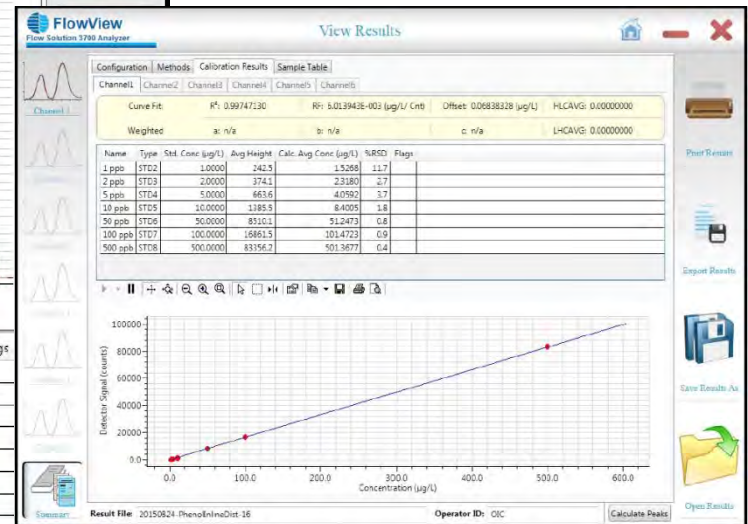
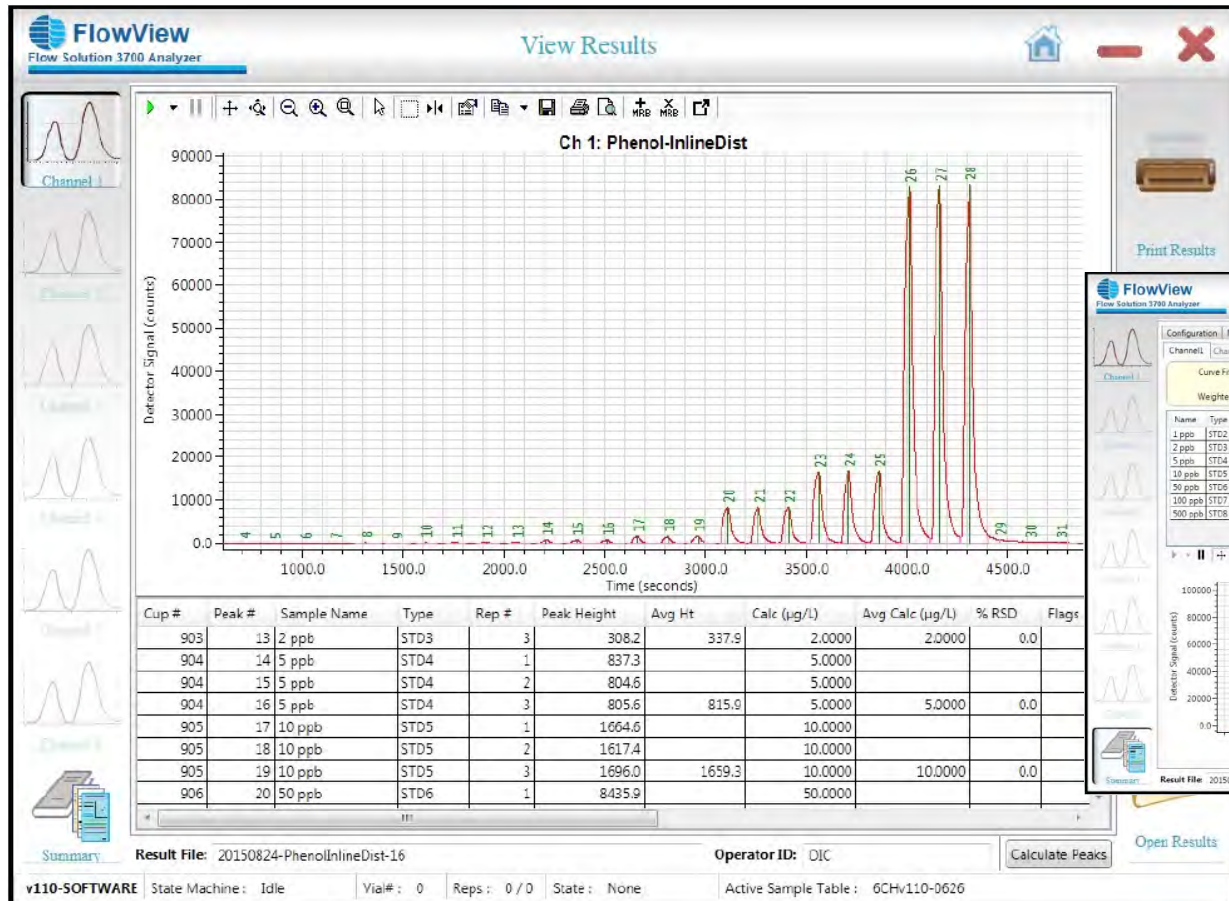
Consumable	Part Number
Pump tubes kit - Phenol, in-line distillation	330364TK
Purple/White pump tubes (12 pack) - for cooling kit	A000360
KleenFlow Acidic	A002295
DOWFAX 2A1	328852
Sample Vials, Glass 8-mL (13 x 100 mm) (pack of 1000)	A000514
Tubing ASSY-Distillation head to cartridge	330795
Distillation coil assembly	A515017
Distillation head assembly	A000677*

Optional Accessories	Part Number
Refrigerated recirculating chiller (115V)	261909
Chiller tubing kit	302810

Phenol, In-line distillation – EPA 420.4

Channel P/N: 330363
Cartridge P/N: 330364

Graph of Results and Calibration Curve



Phenol, Post-distillation – EPA 420.4

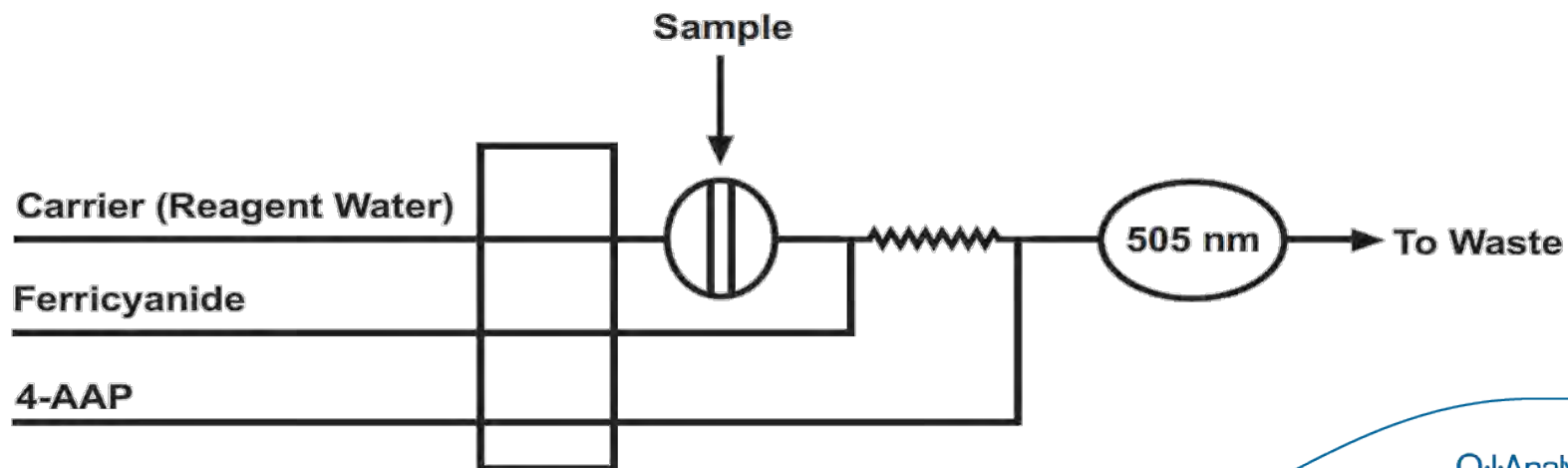
Channel P/N: 330110
Cartridge P/N: 330083

This method is used for the determination of phenolic compounds in drinking water, surface water, and domestic and industrial wastes according to **USEPA Method 420.4**. Additionally, this method enables phenol index analysis following distillation according to **ISO Method 14402**.

Prior to analysis, phenol is manually distilled from an acidic solution. Phenol distillate reacts with 4-aminoantipyrine (4-AAP) and alkaline ferricyanide (FeCN) to form a red complex. The absorbance is measured at 505 nm.

Method Performance

Range	10.0 – 2000 µg/L
Rate	90 samples/hour
Precision	2% RSD at mid-point of range
Method Detection Limit (MDL)	2.0 µg/L



Phenol, Post-distillation – EPA 420.4

Channel P/N: 330110

Cartridge P/N: 330083

Results

	2 µg/L	10 µg/L	500 µg/L	1000 µg/L	2000 µg/L
Replicate 1	2.6	11.8	509.2	1070.4	1977.7
Replicate 2	2.8	13.5	498.8	1084.5	1984.7
Replicate 3	2.5	11.8	505.5	1101.0	1975.7
Replicate 4	2.8	12.5	517.2	1075.4	1973.0
Replicate 5	2.4	12.7	514.6	1073.5	1956.9
Replicate 6	2.7	11.9	508.7	1069.2	1956.6
Replicate 7	2.4	13.1	519.5	1051.1	1949.2
Replicate 8	2.3	12.2	499.8	1046.9	1915.0
Replicate 9	2.3	12.2	502.2	1039.2	1922.1
Replicate 10	2.7	–	501.1	1067.2	1950.6
Mean (µg/L)	2.55	12.41	507.66	1067.84	1956.15
Standard Deviation	0.194789	0.596750	7.458061	18.300285	23.281430
%RSD	7.68%	4.81%	1.47%	1.71%	1.19%
%Accuracy	127.5%	124.1%	101.5%	106.8%	97.8%
MDL	0.5523 µg/L	–	–	–	–

Part Numbers

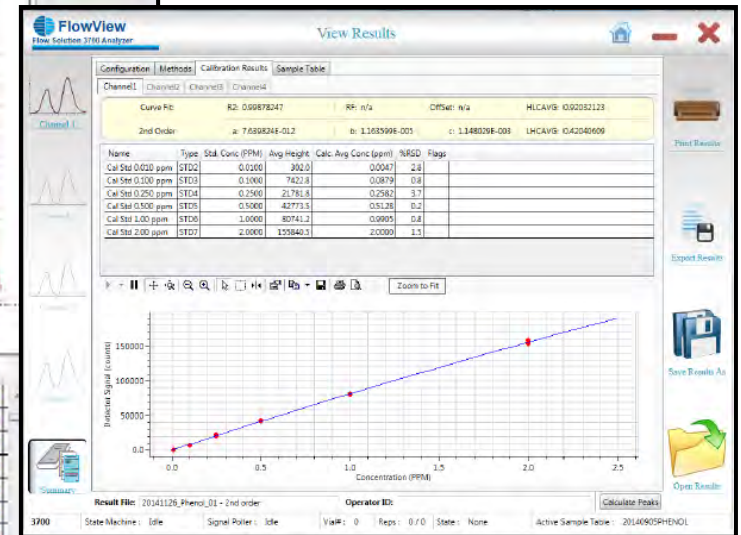
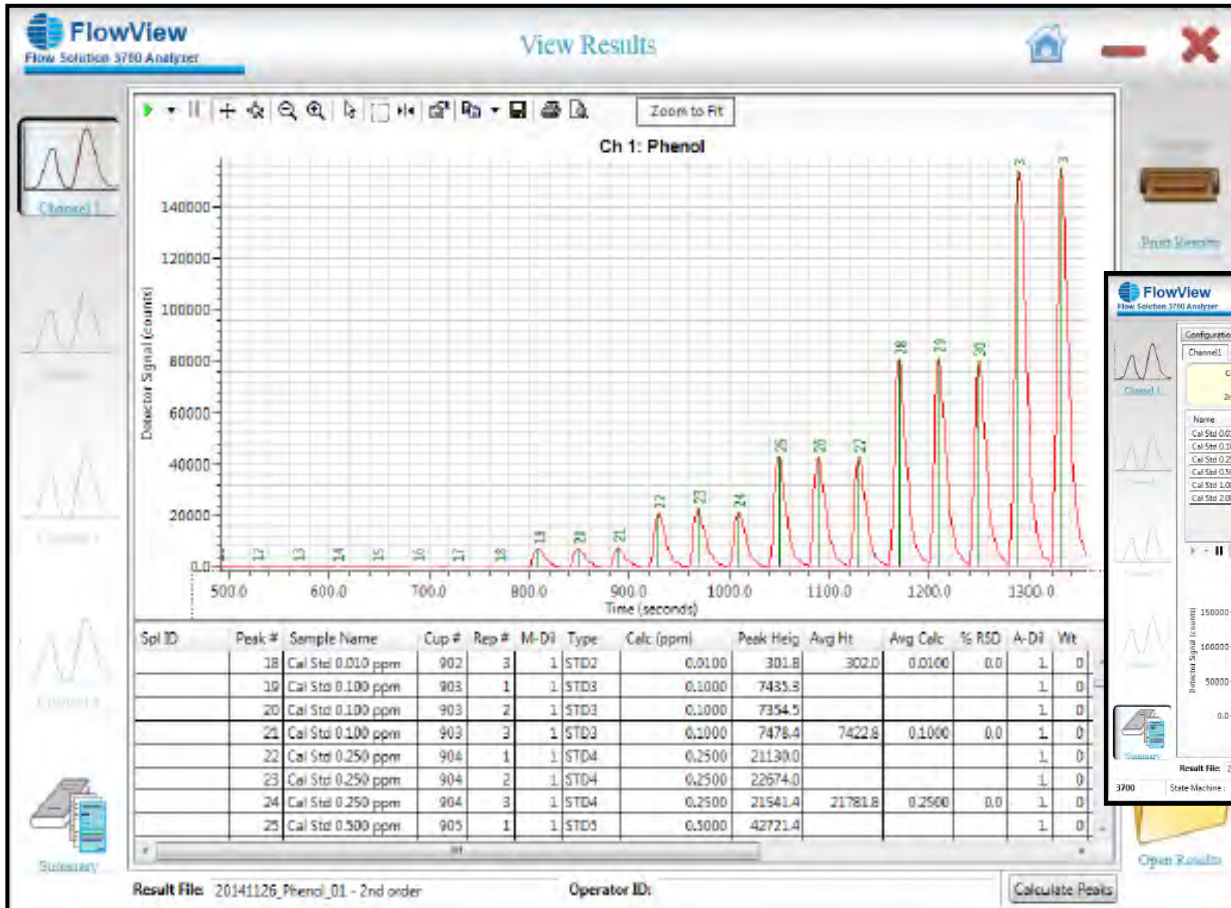
Consumable	Part Number
Pump tubes kit - Phenol, post-distillation	330083TK
KleenFlow™ Acidic	A002295
DOWFAX 2A1	328852
Sample Vials, Glass 8 mL (13 X 100 mm) (pack of 1000)	A000514
Sample/Bypass Loop – 200 µL	285684
PEEK Autosampler Probe for RA/3090/3360 Sampler	325331

Optional Accessories	Part Number
Glass Rinse Station – with tubing kit	330789
Nitrogen Gas Pillow Assembly	A000811
Phenol, in-line distillation Upgrade Kit	330374

Phenol, Post-distillation – EPA 420.4

Channel P/N: 330110
Cartridge P/N: 330083

Graph of Results and Calibration Curve



Phosphate, All Forms – EPA 365.1

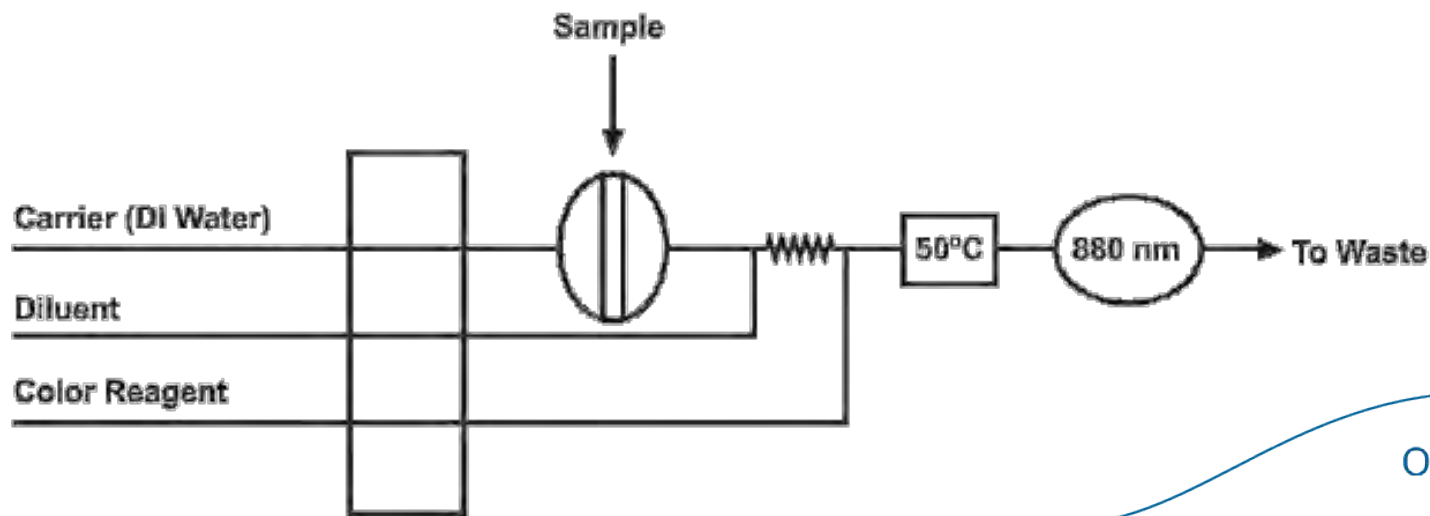
Channel P/N: 330112
Cartridge P/N: 330096

This method is used for the determination of orthophosphate in drinking, ground, and surface waters, and domestic and industrial wastes according to **USEPA Method 365.1**, **Standard Method 4500-P G**, and **ISO 15681-1**.

Method Performance

Range	0.01–5.0 mg/L P
Rate	60 samples/hour
Precision	1% RSD
Method Detection Limit (MDL)	0.001 mg/L

Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form an antimony-phospho-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentration.



Phosphate, All Forms – EPA 365.1

Channel P/N: 330112

Cartridge P/N: 330096

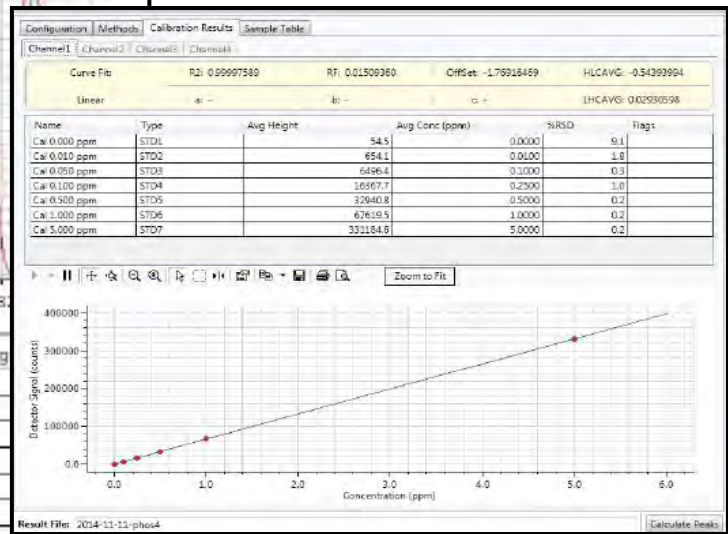
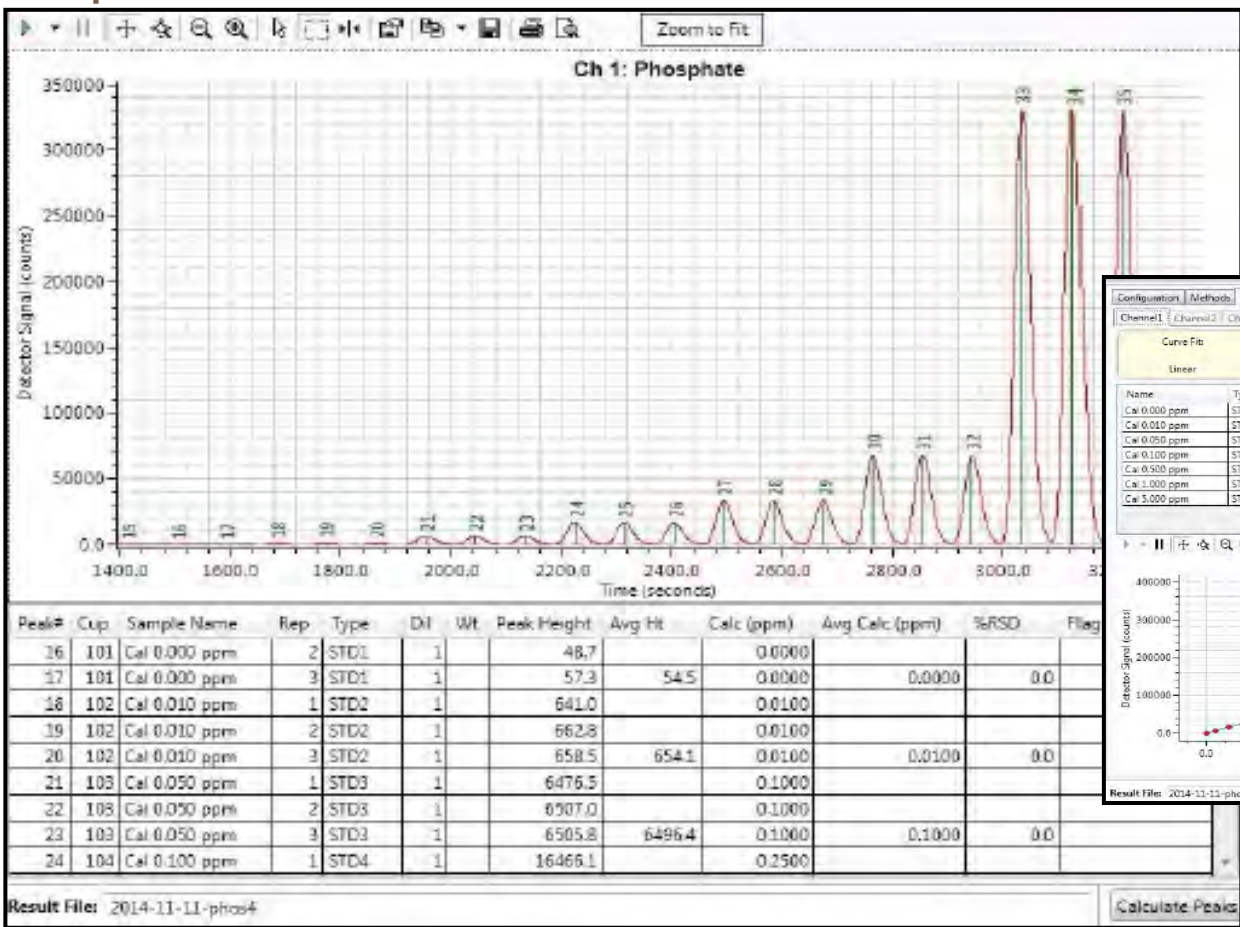
Results

	0.500 mg P/L	0.100 mg P/L	0.010 mg P/L
Replicate 1	0.489	0.0963	0.0080
Replicate 2	0.490	0.0965	0.0080
Replicate 3	0.490	0.0965	0.0081
Replicate 4	0.482	0.0964	0.0080
Replicate 5	0.488	0.0961	0.0078
Replicate 6	0.487	0.0959	0.0080
Replicate 7	0.486	0.0958	0.0079
Replicate 8	0.484	0.0953	—
Replicate 9	0.481	0.0949	—
Replicate 10	0.480	0.0948	—
Mean	0.486	0.0959	0.0080
Standard Deviation	0.003715	0.000643	0.000095
%RSD	0.76%	0.67%	1.19%
%Recovery	97.1%	95.9%	79.7%
MDL	—	—	0.00028

Phosphate, All Forms – EPA 365.1

Channel P/N: 330112
Cartridge P/N: 330096

Graph of Results and Calibration Curve



Phosphate, All Forms (low level) – EPA 365.1

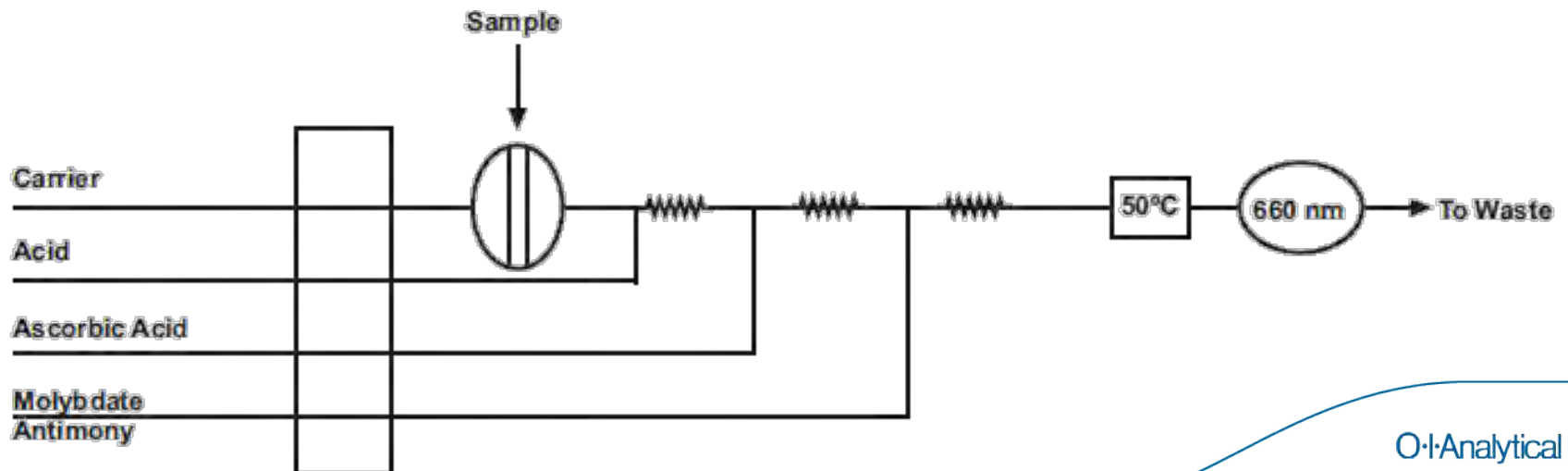
Channel P/N: 330111
Cartridge P/N: 330095

This method is used for the determination of orthophosphate in surface, and domestic and industrial wastes according to **USEPA Method 365.1**

Method Performance

Range	1.0 – 100 µg/L
Rate	45 samples/hour
Precision	1% RSD at mid-point of range
Method Detection Limit (MDL)	0.3 µg/L

Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form an antimony-phospho-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentration.



Phosphate, All Forms (low level) – EPA 365.1

Channel P/N: 330111

Cartridge P/N: 330095

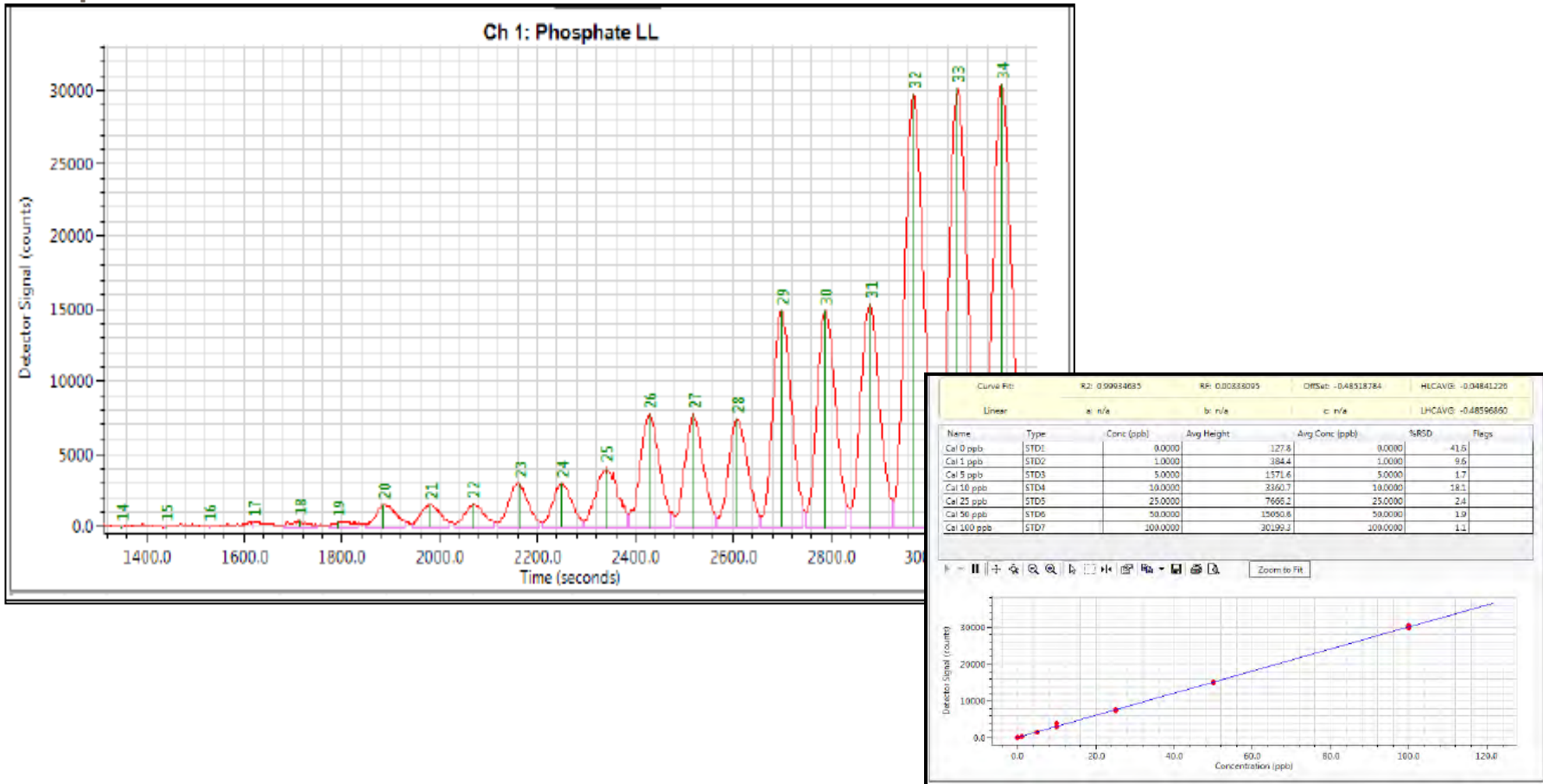
Results

	1.0 ppb P/L	10 ppb P/L	100 ppb P/L
Replicate 1	0.784	9.4232	96.8956
Replicate 2	0.791	9.4385	97.0946
Replicate 3	0.823	9.4336	97.0692
Replicate 4	0.873	9.3336	97.1350
Replicate 5	0.930	9.3917	97.2281
Replicate 6	0.835	9.2723	97.0092
Replicate 7	0.758	9.3166	96.7844
Replicate 8	0.793	9.4579	96.9658
Replicate 9	—	9.2479	96.9955
Replicate 10	—	9.3381	96.6994
Mean	0.823	9.3653	97.0309
Standard Deviation	0.55817	0.073933	0.160379
%RSD	6.78%	0.79%	0.17%
Recovery	—	—	—
MDL	0.167	—	—

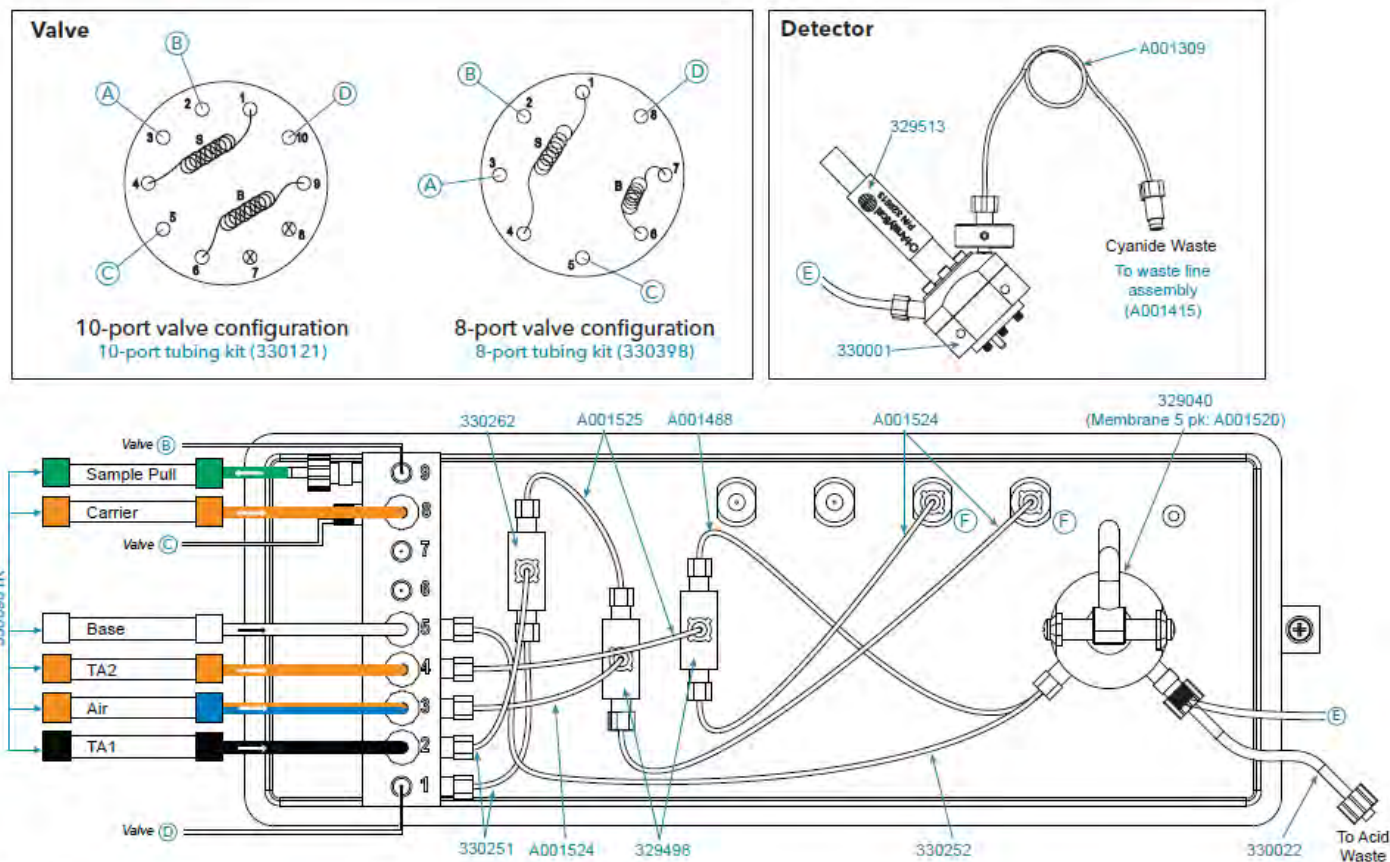
Phosphate, All Forms (low level) – EPA 365.1

Channel P/N: 330111
 Cartridge P/N: 330095

Graph of Results and Calibration Curve



Example of Valve, Detector and Pump Tube and Pump Rates with a method



Notes

Pump Speed: 40%

Sample Loop: 200 μ L

Bypass Loop: 200 μ L

(A) Autosampler transfer line

(F) Connection to/from UV digester assembly (311084)
312 nm lamp (A001682)

Figure 2. Diagram of 330090CT (Total Cyanide Cartridge) and 330076 (Total Cyanide Channel)

Consumables per Chemistry

Table 2. Consumables, spare parts and accessories for Total Cyanide

Consumable	Part Number
Pump tubes kit - Total Cyanide, ASTM D7511	330090TK
Gas Diffusion Membrane – Cyanide (5 pk)	A001520
Flow Solution - Base Reagent	A001103
Flow Solution - Total Acid 1 (TA1)	A001505
Flow Solution - Total Acid 2 (TA2)	A001872
Flow Solution - Total Carrier	A001668
200 µL Injection/Bypass Loop	285684
Amperometric Cell, tested	330001
Amperometric Detector – Reference Electrode	329513
Teflon UV Digestion Coil	311084
UV Lamp, 312 nm	A001682
PEEK Autosampler Probe for RA/3090/3360 Sampler	325331

Optional Accessories	Part Number
Challenge Matrix, ASTM D7365	327788
Teflon Heater Coil Assembly	329486
SFA customization kit – Total Cyanide D7511	330375

Pump tubes should be replaced monthly, or on an as-needed to maintain system performance. The resample line may need to be replaced weekly. Maximum life expectancy for pump tubes is approximately 800 hours.

ISO Methods Cyanide

These methods are used for the determination of cyanide in ground water, drinking water, surface water, leachate and waste water.

- Free Cyanide by Gas Diffusion and Photometric Detection, ISO 14403

Flow Solution™ FS 3700 Automated Chemistry Analyzer

Free Cyanide by Gas Diffusion and Photometric Detection, ISO 14403
Cartridge Part Number 330372CT

Scope and Application

This method is used for the determination of cyanide in ground water, drinking water, surface water, leachate and waste water, according to ISO method 14403. Seawater can be analyzed with possible changes in sensitivity and adaptation of the reagent and calibration solutions to the salinity of the samples.

Method Performance

Range	2.0–500 ppb
Rate	30 samples/hour
Precision	1% RSD at mid-point of range
Method Detection Limit (MDL)	0.4 ppb

The range may be extended to analyze other concentrations by changing the size of the sample loop.

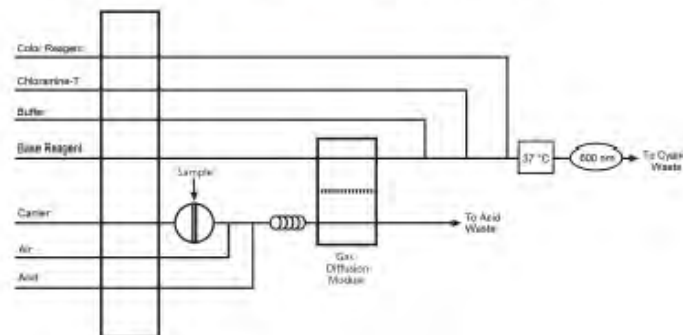


Figure 1. General Flow Diagram for Free Cyanide by ISO 14403

ISO Methods - Cyanide

- Total Cyanide by In-Line Ultraviolet Digestion, Gas Diffusion, and Photometric Detection, ISO 14403

Flow Solution™ FS 3700 Automated Chemistry Analyzer

Total Cyanide by In-Line Ultraviolet Digestion, Gas Diffusion, and Photometric Detection, ISO 14403
Cartridge Part Number 330367CT

Scope and Application

This method is used for the determination of cyanide in ground water, drinking water, surface water, leachate and waste water, according to ISO method 14403. Seawater can be analyzed with possible changes in sensitivity and adaptation of the reagent and calibration solutions to the salinity of the samples.

Method Performance

Range	2.0–500 ppb
Rate	30 samples/hour
Precision	1% RSD at mid-point of range
Method Detection Limit (MDL)	0.4 ppb

The range may be extended to analyze other concentrations by changing the size of the sample loop.

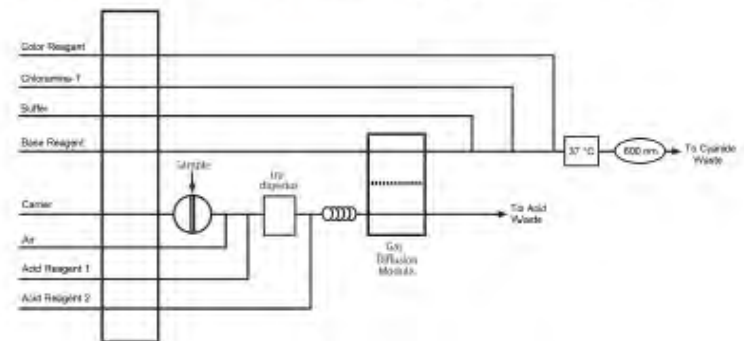


Figure 1. General Flow Diagram for Total Cyanide by ISO 14403.

ISO Methods - Cyanide

Total Cyanide by In-Line

Prior to analysis, treat the sample to remove potential interferences. Ultraviolet (UV) digestion releases cyanide from cyanide complexes. Acid addition converts cyanide ion to hydrogen cyanide gas (HCN), which passes under a gas diffusion membrane. The hydrogen cyanide gas diffuses through the membrane and is absorbed in a sodium hydroxide solution. Sodium cyanide is converted to cyanogen chloride by reaction with chloramine-T at a pH less than 8. The cyanogen chloride then reacts with either:

- isonicotinic acid (pyridine-4-carboxylic acid) and barbituric acid to form a red-colored complex. The absorbance is measured at 600 nm. See Prep Guide A1.
- pyridine-barbituric acid to form a red-colored complex. The absorbance is measured at 570 nm. See Prep Guide A2.
- isonicotinic acid (pyridine-4-carboxylic acid) and 1,3-dimethylbarbituric acid to form a red-colored complex. The absorbance is measured at 600 nm. See Prep Guide A3.



Method Abstract
Total Cyanide by ISO 14403
Document #42800118

Flow Solution™ FS 3700 Automated Chemistry Analyzer

Total Cyanide by In-Line Ultraviolet Digestion, Gas Diffusion, and Photometric Detection, ISO 14403
Cartridge Part Number 330367CT

Scope and Application

This method is used for the determination of cyanide in ground water, drinking water, surface water, leachate and waste water, according to ISO method 14403. Seawater can be analyzed with possible changes in sensitivity and adaptation of the reagent and calibration solutions to the salinity of the samples.

Method Performance

Range	2.0–500 ppb
Rate	30 samples/hour
Precision	1% RSD at mid-point of range
Method Detection Limit (MDL)	0.4 ppb

The range may be extended to analyze other concentrations by changing the size of the sample loop.

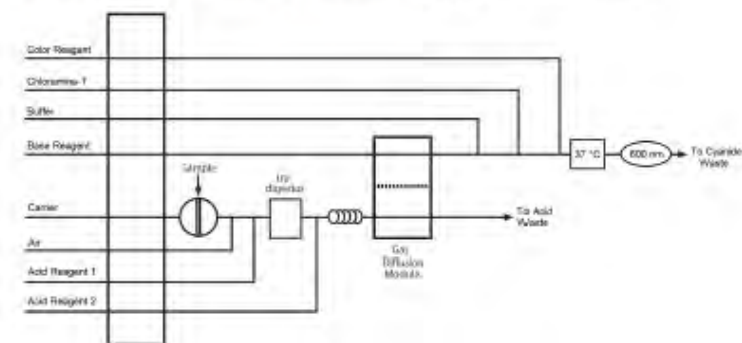


Figure 1. General Flow Diagram for Total Cyanide by ISO 14403.

ISO Methods - MBAS

Methylene Blue Active Substances (MBAS) Index using Continuous Flow Analysis ISO 16265

This method is used for the determination of the methylene blue active substances (MBAS) index in drinking water, ground water, surface water, domestic and industrial. Anionic surfactants are the most important substances showing methylene blue activity. This method is, therefore, useful for estimating the anionic surfactant content (e.g. soaps) of water.

Flow Solution™ FS 3700 Automated Chemistry Analyzer

Methylene Blue Active Substances (MBAS) Index using Continuous Flow Analysis ISO 16265
Cartridge Part Number 330358CT

Scope and Application

This method is used for the determination of the methylene blue active substances (MBAS) index in drinking water, ground water, surface water, domestic and industrial wastes according to ISO Method 16265. Anionic surfactants are the most important substances showing methylene blue activity. This method is, therefore, useful for estimating the anionic surfactant content (e.g. soaps) of water.

Method Performance

Range	0.025 - 2.0 mg/L as LAS
Rate	24 samples/hour
Precision	<5% RSD at mid-point of range
Method Detection Limit (MDL)	0.008 mg/L as LAS

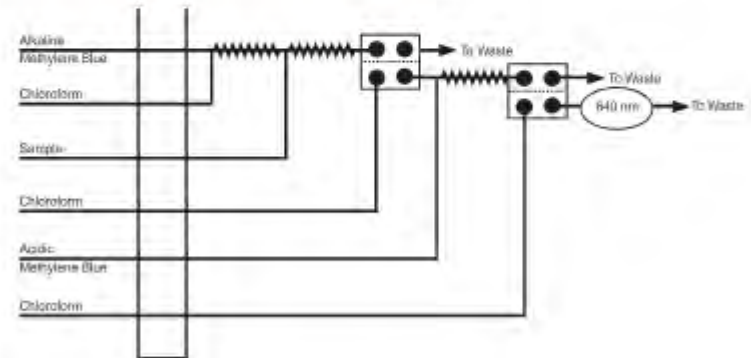


Figure 1. General Flow Diagram for Methylene Blue Active Substances by ISO 16265

ISO Methods - Milk

Though nitrate and nitrite occur naturally in plants and soil and are essential for good health, too much can cause blue baby syndrome and has been linked to birth defects. It is possible for nitrate and nitrite to be introduced to milk and milk products (whey, powdered milk, infant formula, etc.) in the manufacturing process. Thus, dairy products should always be tested for these contaminants for QA/QC and for reporting values for exported products.

Automated Analysis of Nitrates/Nitrites in Milk and Milk Products

Though nitrate and nitrite occur naturally in plants and soil and are essential for good health, too much can cause methemoglobinemia (blue baby syndrome) and has been linked to birth defects. In the human body, nitrates found in vegetables and other foods are converted to nitrites and then into nitric oxide, which helps regulate blood pressure and supports the immune and nervous systems. However, nitrate is also found in fertilizer, which through run-off can contaminate drinking water supplies.

It is also possible for nitrate and nitrite to be introduced to milk and milk products (whey, powdered milk, infant formula, etc.) in the manufacturing process. Thus, dairy products should always be tested for these contaminants for QA/QC and for reporting values for exported products.

The **Flow Solution™ 3700 Automated Chemistry Analyzer** offers a safe, cost-effective alternative to labor-intensive traditional testing. The FS 3700 automates ISO Method 14673-3, offering accurate results while significantly increasing sample throughput.

Standard Features

- Accurate results
- Cost-effective
- High sample throughput
- Automated - set it up and go!
- Easy-to-use, icon-driven software



ISO Methods - Milk

This method is used for the determination of nitrate and nitrite in milk and milk products using cadmium reduction and FIA with in-line dialysis, according to **ISO 14673-3**. This method is also applicable to cheeses (hard, semi-hard, soft, and processed), milk powder, whey powder, liquid milk, and milk-based infant food.

Document #43890417



Method Abstract
Nitrate/Nitrite in Milk by ISO 14673-3
Document #43890417

Flow Solution™ FS 3700 Automated Chemistry Analyzer

Determination of Nitrate and Nitrite in Milk and Milk Products Using Cadmium Reduction and FIA with In-line Dialysis per ISO 14673-3

Cartridge Part Number 331535CT

Scope and Application

This method is used for the determination of nitrate and nitrite in milk and milk products using cadmium reduction and FIA with in-line dialysis, according to ISO 14673-3. This method is also applicable to cheeses (hard, semi-hard, soft, and processed), milk powder, whey powder, liquid milk, and milk-based infant food.¹

Method Performance

Nitrate

Range	0.5 mg/L – 5.0 mg/L
Rate	30 samples/hour
Precision	≤ 2% RSD at mid-range
Method Detection Limit (MDL)	0.016 mg/L

Nitrite

Range	0.025 µg/L – 0.400 µg/L
Rate	30 samples/hour
Precision	≤ 2% RSD at mid-range
Method Detection Limit (MDL)	0.0016 mg/L

Tobacco Methods

Potassium by Flame Emission Spectrometry and Continuous Flow Analysis (CFA)

- This method describes the configuration, calibration, and operation of the Flow Solution® 3700 system equipped with a flame photometer, which is used for the analysis of potassium in drinking water, surface water, saline water, and domestic and industrial wastes

Volatile Base in Tobacco by Online Distillation and Segmented Flow Analysis (SFA)

- This method is used for the determination of volatile base in tobacco leaf samples.

Total Sugar in Tobacco by Segmented Flow Analysis (SFA)

- This method is used for the determination of total sugar in tobacco extracts.

Reducing Sugars in Tobacco by Segmented Flow Analysis (SFA)

- This method is used for the determination of reducing sugars in tobacco extracts.

Total Alkaloids (As Nicotine) in Tobacco by Segmented Flow Analysis

- This method is used for the determination of total alkaloids (as nicotine) in tobacco extracts.

Chloride in Tobacco Extracts by Segmented Flow Analysis

- This method is used for the determination of chloride in tobacco extracts.

xylem

3

Cyanide, Ammonia/TKN by Gas Diffusion

O·I·Analytical 
a xylem brand

Cyanide

Cyanide compounds are used in a wide ranged of industrial applications.

- Hydrogen cyanide is used in the production of nylon 6/6 and methyl methacrylate plastics.
- Cyanide salts are used in metal plating baths for electroplating of brass, bronze, cadmium, copper, gold, silver and zinc.
- Other industrial processes employing cyanide include; petroleum refining, steel production, microelectronics manufacturing, specialty chemical and pharmaceutical production.
- Potassium or sodium cyanide are used in mining operations to leach gold, and other metals, from ore.

Cyanide

Cyanide Analysis... NO Distillation.

ALPKEM
A Division of O-I Analytical

DRAFT Method 1677
Available Cyanide by Flow Injection / Liquid Exchange

Scope and Application

1. The method is for the determination of free and weakly dissociable cyanide in water and wastewater by flow injection and liquid exchange... (text continues)

2. Summary of Method

2.1 The method involves... (text continues)

Measure CATC (Cyanide Amenable to Chlorination) and WAD (Weak and Dissociable) Cyanide in 90 Seconds WITHOUT INTERFERENCES and NO DISTILLATION!

Uses Less Sample
Uses Less Reagents
Uses Significantly Less Dangerous Reagents (no pyridine or barbituric acid)
Has a Low MDL (1.3 µg/L)
Has a wide dynamic range (2 - 5000 µg/L) and is adjustable upward.
NO INTERFERENCES
NO DISTILLATION
Total Cyanide Results in 7 Minutes!

CYANIDE (CATC or WAD) RESULTS IN 90 SECONDS! NO DISTILLATION!



DRAFT METHOD 1677 as presented at EPA's 20th Annual Conference on ANALYSIS OF POLLUTANTS IN THE ENVIRONMENT - May 1997.

ALPKEM cyanide analysis...beyond the limits of distillation.

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*Designed specifically for the needs of the Precious Metal Mining Industry...
CNSolution Model 3202*

CYANIDE (CATC or WAD) RESULTS IN 90 SECONDS! NO DISTILLATION!

Validate your CYANIDE DESTRUCTION PROCESS and Make INTELLIGENT ENVIRONMENTAL DECISIONS with:

- FAST, ACCURATE & INTERFERENCE-FREE RESULTS!

In use AROUND-THE-WORLD. Rapidly becoming the INDUSTRY STANDARD for CYANIDE monitoring...the CNSolution.

Call 1-800-653-1711 for more information.

* DRAFT METHOD 1677 as presented at EPA's 20th Annual Conference on ANALYSIS OF POLLUTANTS IN THE ENVIRONMENT - May 1997.

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From Models 3202, CN3000, CN3100 to FS3700, Alpkem / OI Analytical the innovators in Cyanide Analysis.

Cyanide

ASTM D2036 - 09(2015) Standard Test Methods for Cyanides in Water



Designation: D2036 - 09

Standard Test Methods for Cyanides in Water¹

This standard is issued under the fixed designation D2036; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last approval. A superscripted epsilon (ϵ) indicates an editorial change since the last revision or approval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These test methods cover the determination of cyanides in water. The following test methods are included:

Test Method	Sections
Test Method A—Total Cyanides after Distillation	12 to 18
Test Method B—Cyanides Amenable to Chlorination ² by Difference	19 to 20
Test Method C—Weak Acid Dissociable Cyanides	26 to 32
Test Method D—Cyanides Amenable to Chlorination without Distillation (Shen-Qu Method)	33 to 35

1.2 Cyanogen halides may be determined separately.

1.3 Nitro 1—Cyanogen chloride is the most common of the cyanogen halide complexes as it is a reaction product and is usually present when chlorinating cyanide-containing industrial waste water. For the presence or absence of CNCl, the spot test method given in *Annex A1* can be used.

1.3 These test methods do not distinguish between cyanide ions and metalocyanide compounds and complexes. Furthermore, they do not detect the cyanates. Cyanates can be determined using ion chromatography without digestion.

Nitro 2—The cyanate complexes are decomposed when the sample is acidified in the distillation procedure.

1.4 The cyanide in cyanocomplexes of gold, platinum, cobalt and some other transition metals is not completely recovered by these test methods. Refer to Test Method D6994 for the determination of cyanometal complexes.

1.5 Cyanide from only a few organic cyanides are recovered, and those only to a minor extent.

1.6 Part or all of these test methods have been used successfully with reagent water and various waste waters. It is the user's responsibility to assure the validity of the test method for the water matrix being tested.

¹ These test methods are under the jurisdiction of ASTM Committee D19 on Water and are the direct responsibility of Subcommittee D19.06 on Methods for Analysis for Organic Substances in Water. Current edition approved Oct. 1, 2009. Published October 2009. Originally approved in 1964. Last previous edition approved in 2006 as D2036 - 06. DOI: 10.1520/D2036-09.

² For an explanation of the term cyanides amenable to alkaline chlorination, see Lacey, L. E. and Zablan, W., "Analytical Methods and Instrumentation for Determining Cyanogen Compounds," *Papers on Industrial Water and Industrial Waste Water*, ASTM STP 337, 1962, pp. 32-45.

1.7 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific hazard statements are given in sections 5.1, 8.8, 8.18, 9, 11.3, and 16.1.9.

2. Referenced Documents

2.1 *ASTM Standards*³

D1329 Terminology Relating to Water

D1193 Specification for Reagent Water

D2777 Practice for Determination of Precision and Bias of

Applicable Test Methods of Committee D19 on Water

D3370 Practices for Sampling Water from Closed Conduits

D5788 Guide for Spiking Organics into Aqueous Samples

D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis

D6696 Guide for Understanding Cyanide Species

D6888 Test Method for Available Cyanide with Ligand

Displacement and Flow Injection Analysis (FIA) Utilizing

Gas Diffusion Separation and Amperometric Detection

D6994 Test Method for Determination of Metal Cyanide

Complexes in Wastewater, Surface Water, Groundwater

and Drinking Water Using Anion Exchange Chromatog-

raphy with UV Detection

D7284 Test Method for Total Cyanide in Water by Micro

Distillation followed by Flow Injection Analysis with Gas

Diffusion Separation and Amperometric Detection

D7365 Practice for Sampling, Preservation and Mitigating

Interferences in Water Samples for Analysis of Cyanide

D7511 Test Method for Total Cyanide by Segmented Flow

Injection Analysis, In-Line Ultraviolet Digestion and Am-

perometric Detection

E60 Practice for Analysis of Metals, Ores, and Related

Materials by Spectrophotometry

E275 Practice for Describing and Measuring Performance of

Ultraviolet and Visible Spectrophotometers

³ For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

Cyanide

ASTM D7365 - Standard Practice for Sampling, Preservation and Mitigating Interferences in Water Samples for Analysis of Cyanide



Designation: D7365 – 09a

Standard Practice for Sampling, Preservation and Mitigating Interferences in Water Samples for Analysis of Cyanide¹

This standard is issued under the fixed designation D7365; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last approval. A superscript number (n) indicates an editorial change since the last revision or approval.

1. Scope

1.1 This practice is applicable for the collection and preservation of water samples for the analysis of cyanide. This practice addresses the mitigation of known interferences prior to the analysis of cyanide. Responsibilities of field sampling personnel and the laboratory are indicated.

1.2 The sampling, preservation and mitigation of interference procedures described in this practice are recommended for the analysis of total cyanide, available cyanide, weak acid dissociable cyanide, and free cyanide by Test Methods D2036, D4282, D4374, D6888, D6994, D7237, D7284, and D7511. The information supplied in this practice can also be applied to other analytical methods for cyanide, for example, EPA Method 335.4.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

- D1129 Terminology Relating to Water
- D1193 Specifications for Reagent Water
- D2036 Test Methods for Cyanides in Water
- D3370 Practices for Sampling Water from Closed Conduits
- D3694 Practices for Preparation of Sample Containers and for Preservation of Organic Constituents

¹ This practice is under the jurisdiction of ASTM Committee D19 on Water and in the direct responsibility of Subcommittee D19.06 on Methods for Analysis for Organic Substances in Water.

Current edition approved Oct. 1, 2009. Published October 2009. Originally approved in 2007. Last previous edition approved in 2009 as D7365 – 09. DOI: 10.1520/D7365-09a.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

D3856 Guide for Management Systems in Laboratories Engaged in Analysis of Water

D4282 Test Method for Determination of Free Cyanide in Water and Wastewater by Microdiffusion

D4374 Test Methods for Cyanides in Water—Automated Methods for Total Cyanide, Weak Acid Dissociable Cyanide, and Thiocyanate

D4411 Guide for Sampling Fluvial Sediment in Motion

D4840 Guide for Sample Chain-of-Custody Procedures

D4841 Practice for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents

D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis

D6888 Test Method for Available Cyanide with Ligand Displacement and Flow Injection Analysis (FLA) Utilizing Gas Diffusion Separation and Amperometric Detection

D6994 Test Method for Determination of Metal Cyanide Complexes in Wastewater, Surface Water, Groundwater and Drinking Water Using Anion Exchange Chromatography with UV Detection

D6696 Guide for Understanding Cyanide Species

D7237 Test Method for Free Cyanide with Flow Injection Analysis (FLA) Utilizing Gas Diffusion Separation and Amperometric Detection

D7284 Test Method for Total Cyanide in Water by Micro Distillation followed by Flow Injection Analysis with Gas Diffusion Separation and Amperometric Detection

D7511 Test Method for Total Cyanide by Segmented Flow Injection Analysis, In-Line Ultraviolet Digestion and Amperometric Detection

2.2 U.S. EPA Methods:³

- EPA OIA-1677
- EPA Method 335.2
- EPA Method 335.4

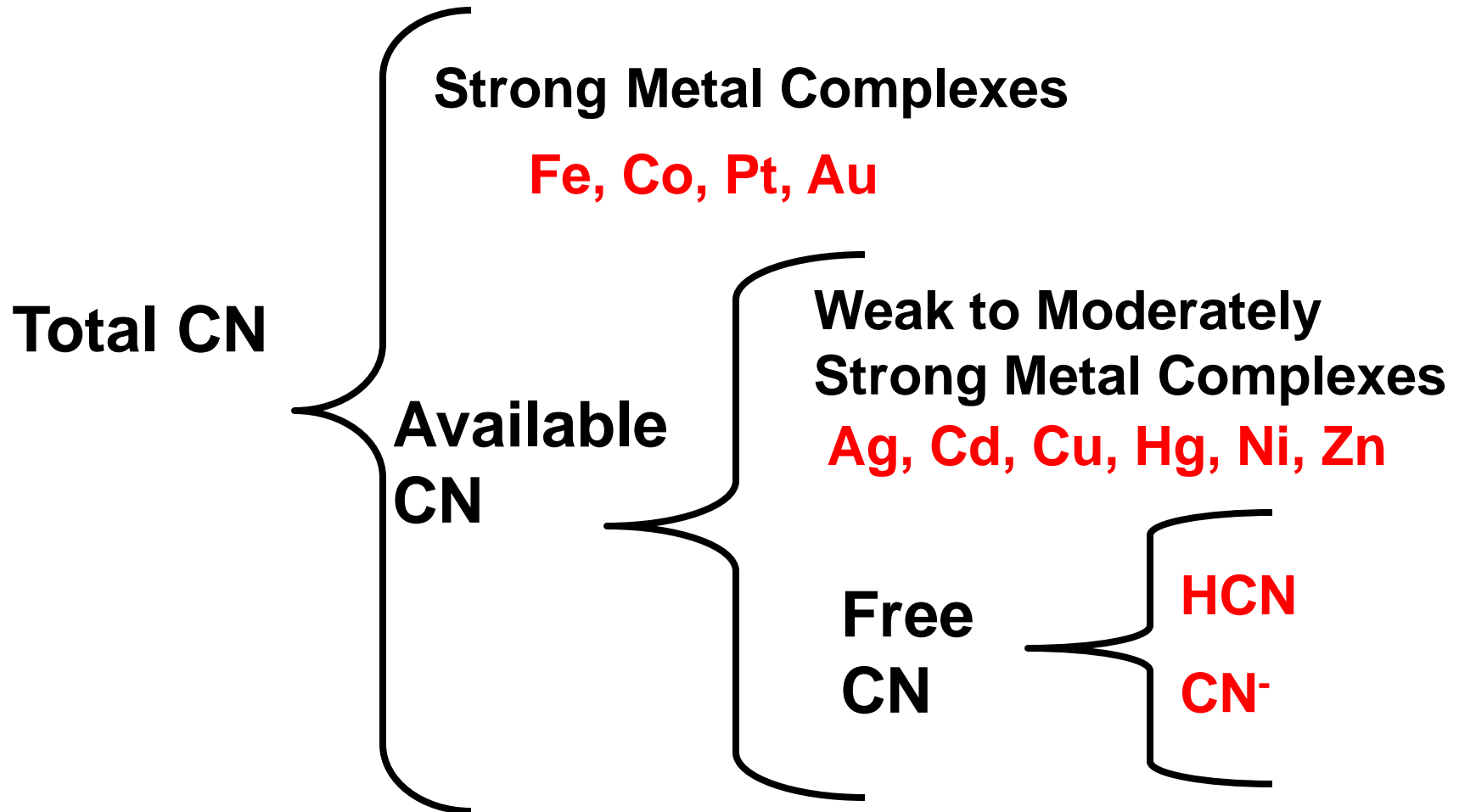
2.3 USGS Methods:⁴

- USGS 1-3300-85
- USGS 1-4302-85

³ Available from United States Environmental Protection Agency (EPA), Acid Rain Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, <http://www.epa.gov>.

⁴ Available from United States Geological Survey, 12201 Sunrise Valley Drive, Reston, VA, 20192, www.usgs.gov.

Cyanide methods measure the various cyanide “species”



Cyanide

Free cyanide refers to the sum of HCN and CN ion in a sample. Free cyanide is bioavailable and about 1000 times more toxic to aquatic organisms than it is to humans. Weak to moderately strong metal-cyanide complexes are compounds that dissociate and release HCN under mildly acidic conditions. The WAD, CATC, and OIA1677 (D6888) methods are developed to quantify available cyanide. These methods measure the weak and moderately strong metal cyanide complexes plus free cyanide.

Strong metal cyanide complexes require strongly acidic conditions to dissociate and release hydrogen cyanide gas. Strong metal cyanide species include complexes of iron, cobalt, and platinum group metals.

The EPA method defined “Total cyanide D7511” includes the strong metal-cyanides, weak to moderately strong metal-cyanides, and free cyanide. The EPA defined “total cyanide” does not include thiocyanate or organic cyanides.

Cyanide

Available Cyanide

Method OIA-1677: Available Cyanide by Ligand Exchange and Flow Injection Analysis (FIA)

This method is for determination of available cyanide in water and wastewater by flow injection, ligand exchange, and amperometric detection. The method is for use in EPA's data gathering and monitoring programs associated with the Clean Water Act, Resource Conservation and Recovery Act, Comprehensive Environmental Response, Compensation and Liability Act, and Safe Drinking Water Act.

Cyanide detection is accomplished using a flow-injection analysis (FIA) system. A 200- μ L aliquot of the pre-treated sample is injected into the flow injection manifold of the system. The addition of hydrochloric acid converts cyanide ion to hydrogen cyanide (HCN) that passes under a gas diffusion membrane. The HCN diffuses through the membrane into an alkaline receiving solution where it is converted back to cyanide ion. The cyanide ion is monitored amperometrically with a silver working electrode, silver/silver chloride reference electrode, and platinum/stainless steel counter.

Cyanide – D6888



Designation: D 6888 – 09

Standard Test Method for Available Cyanide with Ligand Displacement and Flow Injection Analysis (FIA) Utilizing Gas Diffusion Separation and Amperometric Detection¹

This standard is issued under the fixed designation D 6888; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last approval. A superscript symbol (¹) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This method is used to determine the concentration of available inorganic cyanide in an aqueous wastewater or effluent. The method detects the cyanides that are free (HCN and CN⁻) and metal-cyanide complexes that are easily dissociated into free cyanide ions. The method does not detect the less toxic strong metal-cyanide complexes, cyanides that are not “amenable to chlorination.”

1.2 Total cyanide can be determined for samples that have been distilled as described in Test Methods D 2036, Test Method A, Total Cyanides after Distillation. The cyanide complexes are dissociated and absorbed into the sodium hydroxide capture solution, which can be analyzed with this test method; therefore, ligand exchange reagents from Sections 8.12 and 8.13 would not be required when determining total cyanide after distillation.

1.3 This procedure is applicable over a range of approximately 2 to 400 µg/L (parts per billion) available cyanide. Higher concentrations can be analyzed by dilution or lower injection volume.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific hazard statements are given in Note 2 and Section 9.*

2. Referenced Documents

2.1 ASTM Standards:²

D 1129 Terminology Relating to Water

¹ This test method is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.06 on Methods for Analysis for Organic Substances in Water.

Current edition approved Oct. 1, 2009. Published October 2009. Originally approved in 2003. Last previous edition approved in 2004 as D 6888 - 04.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

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D 1193 Specification for Reagent Water
D 2036 Test Methods for Cyanides in Water
D 2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water
D 3556 Guide for Good Laboratory Practices in Laboratories Engaged in Sampling and Analysis of Water
D 4375 Practice for Basic Statistics in Committee D19 on Water
D 5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis
D 6696 Guide for Understanding Cyanide Species
D 7365 Practice for Sampling, Preservation and Mitigating Interferences in Water Samples for Analysis of Cyanide
E 60 Practice for Analysis of Metals, Ores, and Related Materials by Molecular Absorption Spectrometry
E 275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D 1129 and Guide D 6696.

3.2 *available cyanide, n*—Inorganic cyanides that are free (HCN and CN⁻) and metal-cyanide complexes that are easily dissociated into free cyanide ions. Available cyanide does not include the less toxic strong metal-cyanide complexes, cyanides that are not “amenable to chlorination.”

4. Summary of Test Method

4.1 Complex cyanides bound with nickel or mercury are released by ligand displacement by the addition of a ligand displacement agent prior to analysis.

4.2 Other weak and dissociable cyanide species do not require ligand displacement.

4.3 The treated sample is introduced into a flow injection analysis (FIA) system where it is acidified to form hydrogen cyanide (HCN). The hydrogen cyanide gas diffuses through a hydrophobic gas diffusion membrane, from the acidic donor stream into an alkaline acceptor stream.

4.4 The captured cyanide is sent in an amperometric flow-cell detector with a silver-working electrode. In the presence of cyanide, silver in the working electrode is oxidized at the

Cyanide – Sour Water Application

Cyanide Generation and Corrosion

Cracking organic nitrogen compounds in petroleum feedstocks liberates hydrogen cyanide (HCN), ammonia, and other nitrogen compounds.

The formation and downstream effects of cyanide are a major concern in fluid catalytic cracking and hydrocracking operations.

Publication 37410114



Presented at the 2014 Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, Chicago, Illinois, March 2 - March 6, 2014

Introduction

Fluid catalytic cracking (FCC) is a major unit operation in refineries around the world. FCC is used to convert low-value, high molecular weight feedstocks such as shale oil, tar sands oil, and coker gas oils into lighter, high-value products by "cracking" C-C bonds. These feedstocks may contain high levels of organic nitrogen compounds such as indole, carbazole, pyridine, and quinoline (Figure 1), which form ammonia and cyanide in the reactor of FCC units. The nitrogen content of crude petroleum is generally in the range of 0.1 - 0.9%, however, some crude may contain up to 2% nitrogen. The more asphaltic the oil the higher the nitrogen content.⁽¹⁾

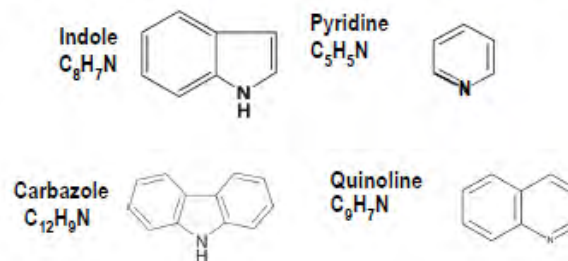


Figure 1. Common Organic Nitrogen Compounds in Petroleum

Cyanide

Total Cyanide:

ASTM D 7511-09e2 uses narrow-band, low-watt UV irradiation to decompose cyanide complexes in samples at ambient temperature in a continuously flowing acidic stream. Reducing and complexing reagents, combined with ambient temperature UV digestion minimizes the formation of matrix interferences. Elimination of the sample distillation step enables measurement of cyanide at lower concentrations with improved precision.

Higher sample throughput:

- Lower labor costs
- Lower cost per analysis for reagents and consumables
- Eliminates analyst exposure to hazardous reagents (boiling, concentrated sulfuric acid and pyridine)

Whitepaper No. 3687 - Total Cyanide Analysis of NPDES Wastewater Samples by ASTM D 7511-09e2

Cyanide – Soil and Sediment Samples

Insoluble cyanide complexes, such as Prussian Blue, are bound to particulate matter or soil particles and are not quantitatively recovered by distillation procedures. A sodium hydroxide extraction followed by total cyanide analysis using ASTM D 7511-12 recovers cyanides.

Publication 39440513



Extraction and Analysis of
Cyanide In Soil and
Sediment Samples

Introduction

The toxicity and mobility of cyanide in soil is governed by its chemical form. Simple cyanide, or the cyanide ion (CN^-), can be weakly adsorbed onto soil particles at $\text{pH} > 9.2$. Weak metal-cyanide complexes ($[\text{M}(\text{CN})_2]^{2-}$) and strong metal – cyanide complexes ($[\text{M}(\text{CN})_4]^{3-}$ or $[\text{M}(\text{CN})_6]^{4-}$) have an affinity for metal oxides and organic matter that decreases with increasing pH, however, other salts in solution tend to inhibit adsorption¹. Simple cyanide, weak metal-cyanide complexes, and strong-metal cyanide complexes are readily soluble in water. Metal – Metal cyanide complexes, such as Prussian Blue ($\text{Fe}_4[\text{Fe}(\text{CN})_6]_3$), are insoluble in water and are the most common forms of cyanide found in sediment and soil². Metal-metal cyanide complexes are insoluble in acid solution, and solubility increases with pH.

A common practice for the extraction of cyanide in soil and sediment is acid distillation³. This approach is valid for all cyanide forms except the metal-metal cyanide complexes that are most likely to be present. The quality control practice of spiking a soil sample with simple cyanides, or strong metal-cyanide complexes to validate acid distillation is misleading because these cyanide complexes are readily soluble. Acid distillation of cyanide in soil results in low and irreproducible recoveries for the metal-metal cyanide complexes most likely to be present. Acid distillation of cyanide in soil is ineffective and does not accurately measure “total” cyanide nor does it estimate cyanide toxicity.

Regulatory Status of RCRA SW-846 Cyanide Analysis Methods

The U.S. EPA has issued a comprehensive set of cyanide analysis methods based on gas-diffusion amperometry for Safe Water Drinking Act and Clean Water Act compliance testing and reporting.^{4,5,6,7,8,9} These employ ligand

Cyanide – Simultaneous Analysis of Available and Total Cyanide

USEPA methods OIA-1677(1) and ASTM D 7511-12(2) have the advantage of determining available cyanide and total cyanide respectively without a preliminary distillation step. These methods are usually run separately due to the manual ligand addition step required in OIA-1677 to release cyanide from certain metal-cyanide complexes. In this study, Method OIA-1677 was modified to automatically inject a diluted ligand exchange reagent into the available cyanide method. Automatic ligand injection enables total and available cyanide to be determined simultaneously from the same aliquot and using the same reagents.

Application Note 37930312



Application Note 37930312

Keywords

ASTM D 7511-12
Available Cyanide
Gas-Diffusion Amperometry
Total Cyanide
USEPA OIA-1677

Simultaneous Analysis of Available and Total Cyanide by Gas Diffusion Amperometry Methods USEPA OIA-1677 and ASTM D 7511-12

Introduction

USEPA methods OIA-1677⁽¹⁾ and ASTM D 7511-12⁽²⁾ have the advantage of determining available cyanide and total cyanide respectively without a preliminary distillation step. These methods are usually run separately due to the manual ligand addition step required in OIA-1677 to release cyanide from certain metal-cyanide complexes. In this study, Method OIA-1677 was modified to automatically inject a diluted ligand exchange reagent into the available cyanide method. Automatic ligand injection enables total and available cyanide to be determined simultaneously from the same aliquot and using the same reagents.

Experimental

A new cartridge based on the acidification reagents described in ASTM D 6888-09⁽³⁾ was designed to automatically add a diluted ligand exchange reagent solution to each sample injection. The ligand exchange reagent is added in such small quantity that, even though flowing continuously, the approach saves money over manual addition.

The total cyanide cartridge for ASTM D 7511 was modified to use a single FEP Teflon UV-digestion coil. This requires a lower flow rate for the TA1 acidification reagent. The TA1 reagent recipe was also modified from the original formulation to achieve higher recoveries for total cyanide, and to decrease interferences from thiocyanate plus nitrate.

These modifications (allowed by 40 CFR Part 136.6 Method Flexibility), enable the analyst to share carrier, acidification, and acceptor reagents decreasing the overall complexity of the analysis. Sample solutions are merely poured into autosampler vials, and injected without pretreatment. A tee splits the sample in half with one half directed to the total cyanide cartridge, and the other half directed to the available cyanide cartridge. A schematic diagram of a dual channel system configured for simultaneous

Cyanide – Model 9310 On-Line CN Analyzer

The Model 9310 analyzer can be used as a benchtop analyzer for grab samples or deployed for on-line measurements and process control.

Measures available cyanide in precious metal leaching solutions by U.S. EPA Method OIA-1677 and ASTM D 6888-09.

Measurement Ranges

- 0.2 to 50 ppm CN
- 2.0 to 500 ppm CN
- 20 to 2,000 ppm CN



Cyanide – Model 9310 On-Line CN Analyzer

The CNSolution 9310 supports the measurement and control of cyanide in multiple cyanidation unit operations

1. Cyanide Addition
2. Leaching
3. Cyanide Recycle
4. Detoxification
5. Effluent Discharge/Tailings



Gas Diffusion - Ammonia

The Method Update Rule passed in January 2012 modifies 40 CFR Part 136 and allows diffusion in place of distillation for the analysis of ammonia.

We utilize the continuous flow automated diffusion that passes ammonia through a hydrophobic membrane into an absorber solution that is automatically color developed and measured by the continuous flow analyzer method. Gas diffusion eliminates the need for, distillation. This allows facilities with NPDES permits to use gas diffusion to test wastewater samples for TKN/Ammonia and Clean Water Act compliance reporting.

- Higher sample throughput
- Lower labor costs
- Lower cost per analysis for reagents and consumables
- Eliminates analyst exposure to hazardous reagents (boiling concentrated base reagents (TKN) or boiling Borate buffer solutions (ammonia))
- Ability to analyze both ammonia and TKN using a single chemistry cartridge with the same reagents.*

* *OI recommends using separate calibrations that match the sample matrix.*

Whitepaper No. 3904 - Analysis of TKN and Ammonia in NPDES
Wastewater Samples by In-line Gas Diffusion

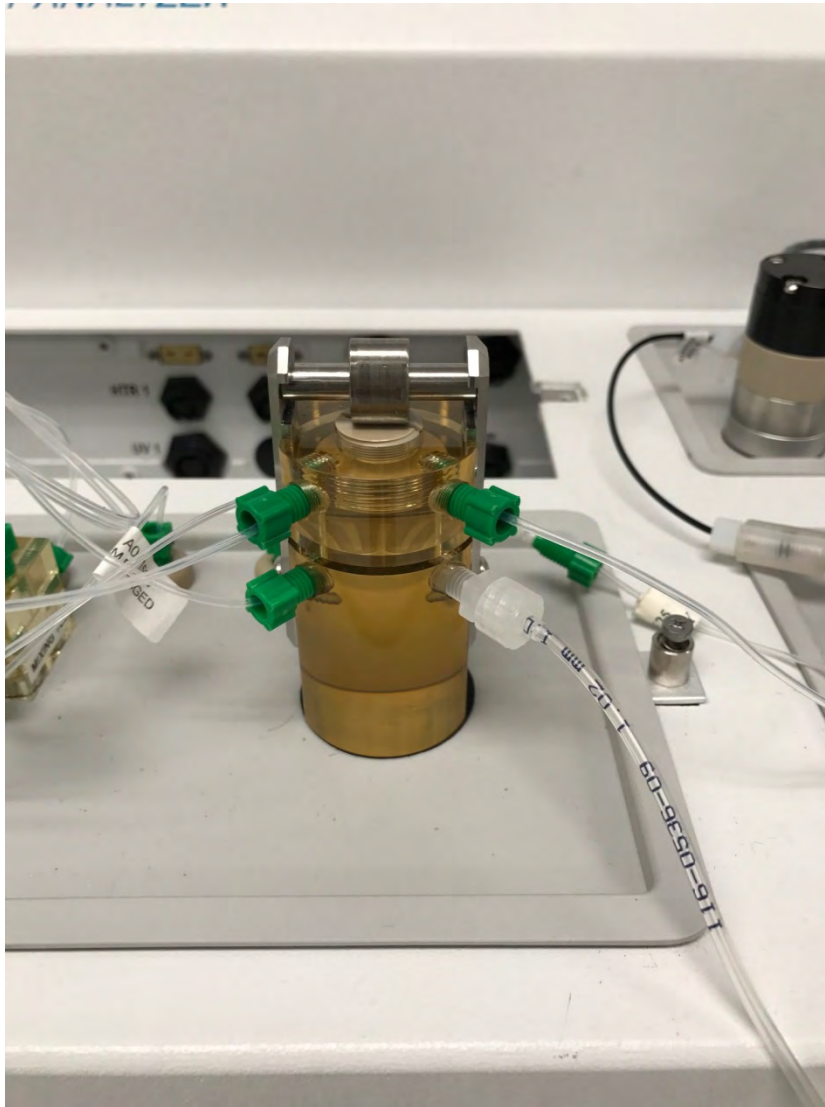
Gas Diffusion Module - Simplicity



OI Analytical Gas Diffusion Module Ammonia/TKN

The purpose of the gas diffusion module is to function as an in-line cleanup eliminating particulates, potential ionic interferences and chemical complexes. The target analyte permeates through the membrane into a separate flow path going on to further analysis while all the other components of the stream go off to waste. Without separation these interferences can cause problems with both amperometric and photometric flow cells by disrupting the electronic potential of the target molecule, causing precipitation in the colorimetric reagents, or simply creating problems with large particulates.

Gas Diffusion Module - Simplicity

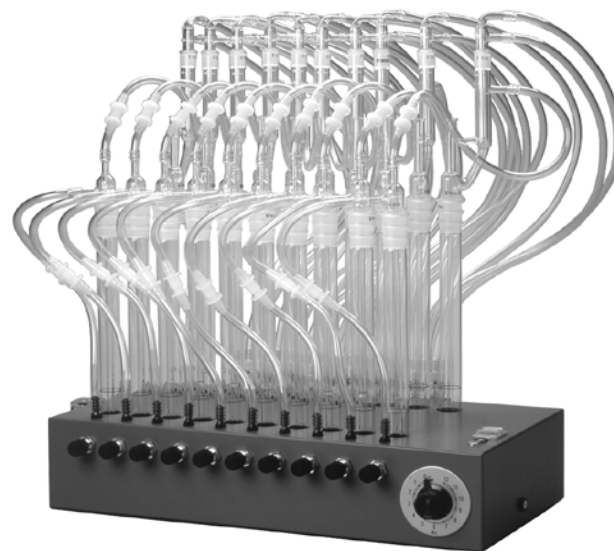


OI Analytical Gas Diffusion Module
Cyanide's (OIA-1677, D6888, and
D7511-12).

With Gas Diffusion farewell to Distillations



Macro Distillation



**MIDI
Distillations**

FS3700 Automated Chemistry Analyzer

Staff based in College Station, TX

Hank Hahn

Senior Sales Specialist

Xylem Lab Solutions, North America

M:979-204-4002

Hank.Hank@xylem.com

Tim Smith

ACA Product Specialist/Technical Support

Xylem Lab Solutions, North America

O: 979-690-5504

Tim.Smith@xylem.com

Mark Terrell

Product Manager, OI Analytical

Xylem Lab Solutions, North America

O: (979) 690-5580

Mark.Terrell@xylem.com

Thank you for joining us today!

For more information, please contact

Hank.Hahn@Xyleminc.com

info.apac@xyleminc.com



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