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.[1] 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.

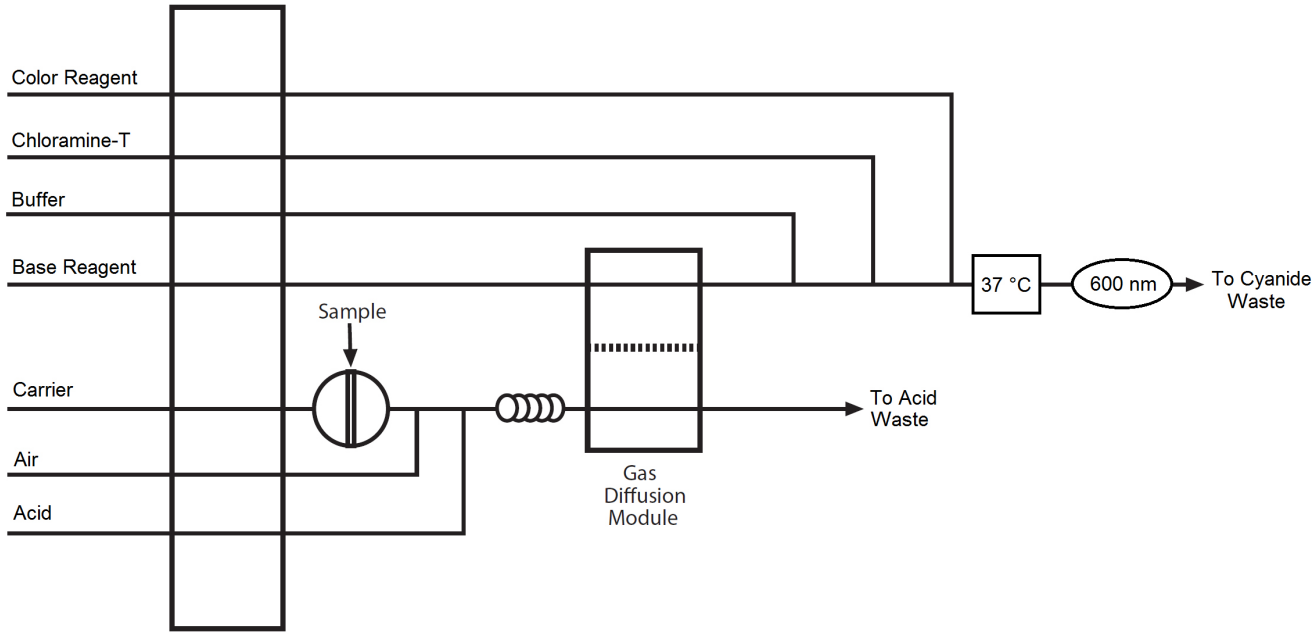


Figure 1. General Flow Diagram for Free Cyanide by ISO 14403

Reagents and Calibrants

Chemical Name	CAS #	Chemical Formula	Part Number	Used in Prep Guide
Barbituric acid	67-52-7	$C_4H_4N_2O_3$		A1, A2
Brij®-35	9002-92-0	$(C_2H_4O)_nC_{12}H_{26}O$	326126	A1, A2, A3
Citric acid	77-92-9	$C_6H_8O_7$		A3
Chloramine-T trihydrate	7080-50-4	$CH_3C_6H_4SO_2NNaCl \cdot 3H_2O$		A1, A2, A3
1,3-Dimethylbarbituric acid	769-42-6	$C_6H_8N_2O_3$		A3
Hydrochloric acid	7647-01-0	HCl		A1, A2, A3
Isonicotinic acid (Pyridine-4-carboxylic acid)	55-22-1	$C_6H_5NO_2$		A1, A3
Potassium cyanide	151-50-8	KCN		
Pyridine	110-86-1	C_5H_5N		A2
Sodium hydroxide	1310-73-2	NaOH		A1, A2, A3
Sodium phosphate, monobasic	7558-80-7	NaH_2PO_4		A1, A2
Succinic acid	110-15-6	$C_4H_6O_4$		A3
Tetraethylenepentamine	112-57-2	$C_8H_{23}N_5$		A1, A2, A3
Water, deionized		H_2O		A1, A2, A3
Additionally, the following chemicals may be needed for sample preservation or treatment.				
Acetic acid, glacial	64-19-7	$C_2H_4O_2$		
Acetone	67-64-1	C_3H_6O		
Ascorbic acid	50-81-7	$C_6H_8O_6$		
5-[4-Dimethylaminobenzylidene]rhodanine	536-17-4	$C_{12}H_{12}N_2OS_2$		
Ethylenediamine	107-15-3	$C_2H_8N_2$		
Silver nitrate	7761-88-8	$AgNO_3$		
Sodium acetate	127-09-3	$C_2H_3O_2Na$		
Sodium arsenite	7784-46-5	$NaAsO_2$		
Sulfamic acid	5329-14-6	H_3NSO_3		

Summary of ISO 14403

Method

1. Prior to analysis, samples are treated to remove potential interferences and 50 µL of tetraethylenepentamine solution is added for the liberation of cyanide from nickel complexes. Acid addition converts cyanide ions 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 one of the following:
   - isonicotinic acid (pyridine-4-carboxylic acid) and barbituric acid to form a red-colored complex. The absorbance is measured at 600 nm.
   - pyridine and barbituric acid to form a red-colored complex. The absorbance is measured at 570 nm.
   - isonicotinic acid (pyridine-4-carboxylic acid) and 1,3-dimethylbarbituric acid to form a red-colored complex. The absorbance is measured at 600 nm.

Interferences

1. The practices in the most current ASTM D7365 “Standard Practices for Sampling, Preservation and Mitigating Interferences in Water Samples for Analysis of Cyanide” should be referred to and for best practices for sampling, preservation and interference mitigation.

2. Some of the known interferences to this method include aldehydes, nitrite, oxidizing agents, sulfide, sulfite, surfactants, and thiocyanate. Multiple interferences may require the analysis of a series of laboratory fortified sample matrices (LFM) to verify the suitability of the chosen treatment. See below for details on some of these interferences and refer to **Sample Handling and Preservation** for treatments for mitigating various interferences.

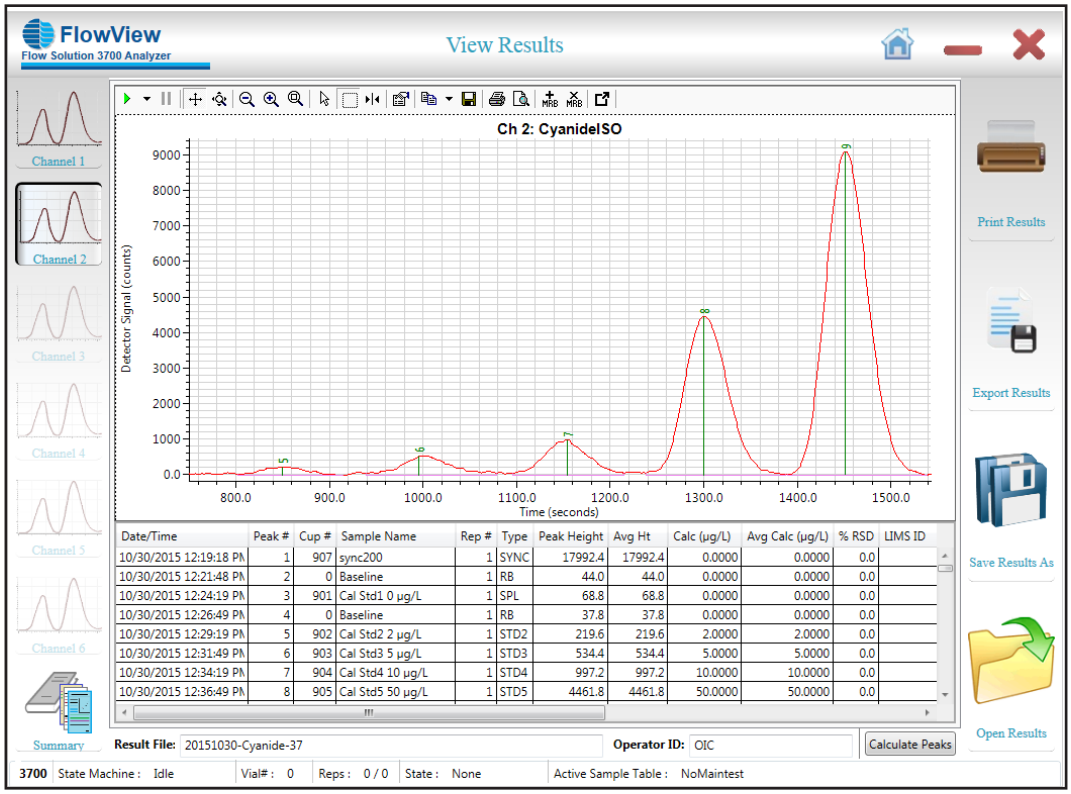
3. Samples that have been disinfected by UV irradiation are likely to contain aldehydes. Sample treatment is described in **Sample Handling and Preservation** for samples containing water soluble aldehydes, such as formaldehyde or acetaldehyde.

4. Nitrite interferes with this method above concentrations of 5 ppm. Sample treatment is described in **Sample Handling and Preservation**.

5. Oxidizing agents such as chlorine decompose most cyanides. Remove oxidizing agents that decompose cyanides. Sample treatment is described in **Sample Handling and Preservation**.

6. Other compatible procedures for removing or suppressing interferences may be used, provided they do not adversely affect overall method performance.

7. Method interferences can be caused by contaminants in the reagents, reagent water, and glassware, which may bias the results. Take care to keep all such items free of contaminants.



Date/Time	Peak #	Cup #	Sample Name	Rep #	Type	Peak Height	Avg Ht	Calc (µg/L)	Avg Calc (µg/L)	% RSD	LIMS ID
10/30/2015 12:19:18 PM	1	907	sync200	1	SYNC	17992.4	17992.4	0.0000	0.0000	0.0	
10/30/2015 12:21:48 PM	2	0	Baseline	1	RB	44.0	44.0	0.0000	0.0000	0.0	
10/30/2015 12:24:19 PM	3	901	Cal Std1 0 µg/L	1	SPL	68.8	68.8	0.0000	0.0000	0.0	
10/30/2015 12:26:49 PM	4	0	Baseline	1	RB	37.8	37.8	0.0000	0.0000	0.0	
10/30/2015 12:29:19 PM	5	902	Cal Std2 2 µg/L	1	STD2	219.6	219.6	2.0000	2.0000	0.0	
10/30/2015 12:31:49 PM	6	903	Cal Std3 5 µg/L	1	STD3	534.4	534.4	5.0000	5.0000	0.0	
10/30/2015 12:34:19 PM	7	904	Cal Std4 10 µg/L	1	STD4	997.2	997.2	10.0000	10.0000	0.0	
10/30/2015 12:36:49 PM	8	905	Cal Std5 50 µg/L	1	STD5	4461.8	4461.8	50.0000	50.0000	0.0	

Figure 2. Free Cyanide Calibration Series

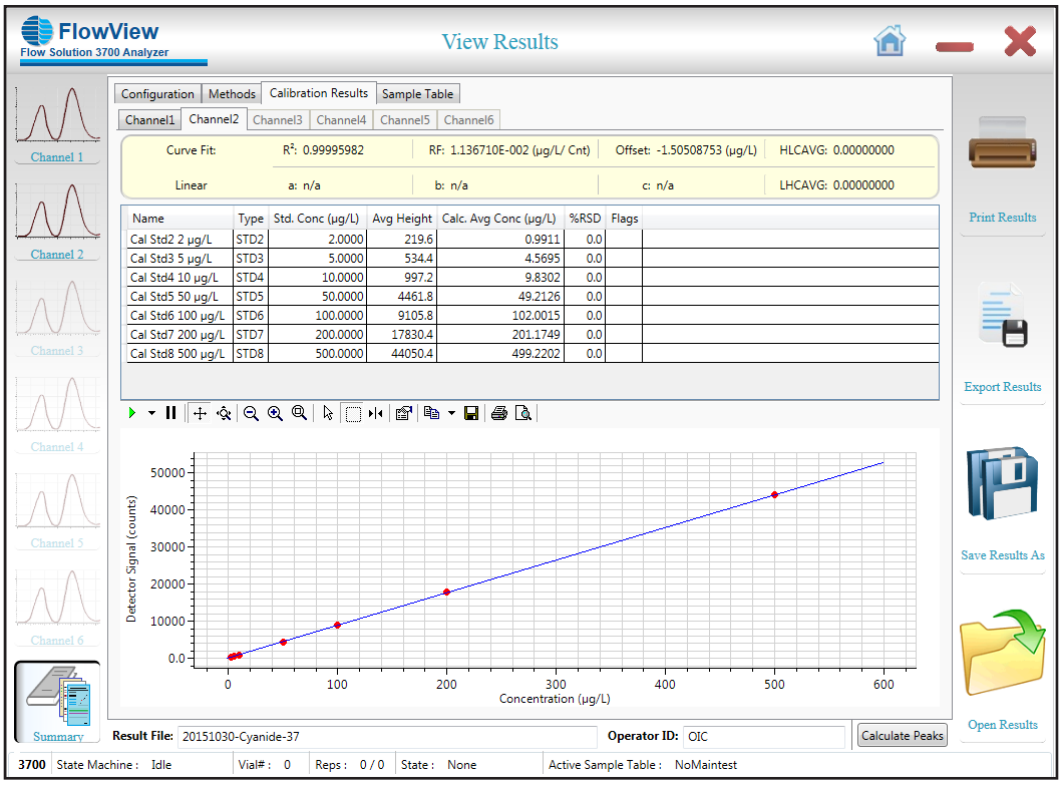


Figure 3. Calibration Curve and Statistics

Table 2. Consumables, spare parts and accessories for Free Cyanide, ISO 14403

Consumable	Part Number
Pump tubes kit - Free Cyanide, ISO 14403	330372TK
Gas Diffusion Membrane – Cyanide (5 pk)	A001520
400 µL Injection/Bypass Loop	319334
PEEK Autosampler Probe for RA/3090/3360 Sampler	325331
Brij®-35	326126

Optional Accessories	Part Number
Amperometric Detector	330077
OIA-1677/ASTM D7511 Control Standard	328942

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.

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