

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.

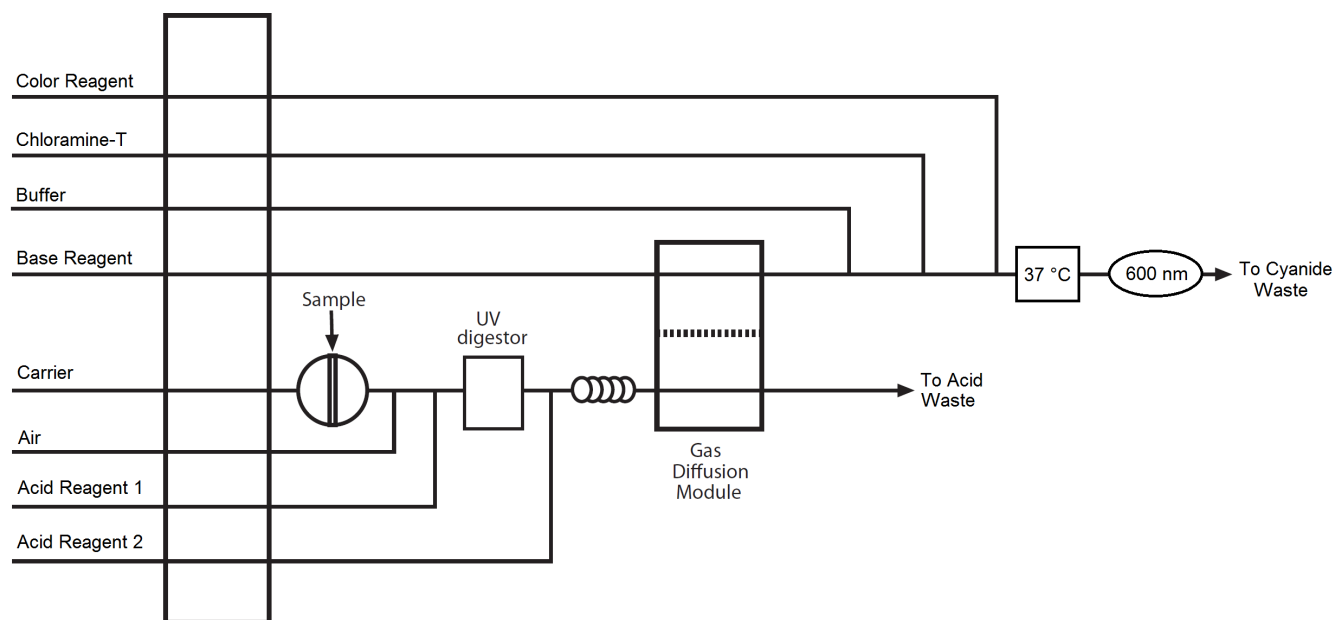


Figure 1. General Flow Diagram for Total Cyanide by ISO 14403.

Reagents and Calibrants

Chemical Name	CAS #	Chemical Formula	Part Number	Used in Prep Guide
Ascorbic acid	50-81-7	$C_6H_8O_6$		A1, A2
Barbituric acid	67-52-7	$C_4H_4N_2O_3$		A1, A2
Brij®-35	9002-92-0	$(C_2H_4O)_n C_{12}H_{26}O$	326126	A1, A2, A3
Bismuth nitrate pentahydrate	10035-06-0	$Bi(NO_3)_3 \cdot 5H_2O$		A1, A2
Challenge Matrix, ASTM D7365			327788	
Citric acid	77-92-9	$C_6H_8O_7$		A1, A2, 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
OIA-1677/ASTM D7511 Control Standard			328942	
Flow Solution – Total Acid 1 (TA1)			A001505	A1, A2
Flow Solution – Total Acid 2 (TA2)			A001872	A1, A2
Flow Solution – Total Carrier			A001668	A1, A2
Flow Solution – Base Reagent			A001103	A1, A2
Hydrochloric acid	7647-01-0	HCl		A3
Hypophosphorous acid	6303-21-5	H_3PO_2		A1, A2
Isonicotinic acid (Pyridine-4-carboxylic acid)	55-22-1	$C_6H_5NO_2$		A1, A3
Potassium cyanide	151-50-8	KCN		
Potassium ferricyanide	13746-66-2	$K_3Fe(CN)_6$		
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
Sulfuric Acid	7664-93-9	H_2SO_4		A1, A2
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, 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.

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. Thiocyanates can produce positive interference when they decompose to cyanide by UV radiation.⁴ This method uses a narrowband 312 nm irradiation wavelength, which keeps thiocyanate interference minimal. A modified TA1 reagent lowers thiocyanate interference.
7. Other compatible procedures for removing or suppressing interferences may be used, provided they do not adversely affect overall method performance.
8. Method interferences can be caused by contaminants in the reagents, reagent water, and glassware, which may

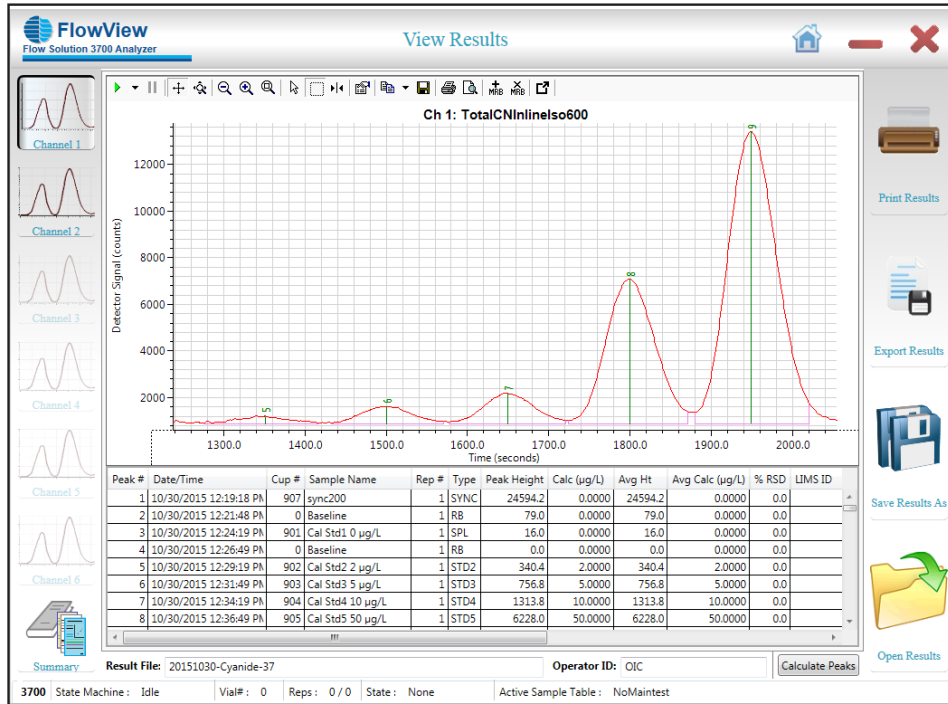


Figure 2. Total Cyanide Calibration Series

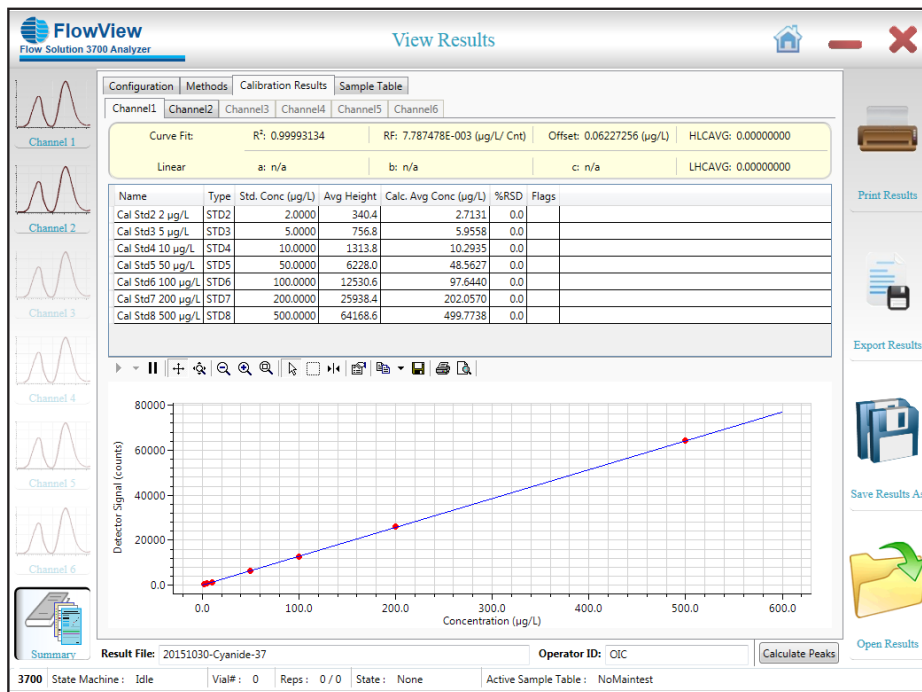


Figure 3. Calibration Curve and Statistics

Table 2. Consumables, Spare Parts and Accessories for Total Cyanide, ISO 14403

Consumable	Part Number
Pump tubes kit - Total Cyanide, ISO 14403	330367TK
Gas Diffusion Membrane – Cyanide (5 pk)	A001520
400 µL Injection/Bypass Loop	319334
UV Digestion Coil, Teflon	311084
UV Lamp, 312 nm	A001682
PEEK Autosampler Probe for RA/3090/3360 Sampler	325331
Brij®-35	326126
Flow Solution - Base Reagent	A001103
Flow Solution - Total Acid 1 (TA1)	A001505
Flow Solution - Total Acid 2 (TA2)	A001872
Flow Solution - Total Carrier	A001668

Optional Accessories	Part Number
Amperometric Detector	330077
OIA-1677/ASTM D7511 Control Standard	328942
Challenge Matrix, ASTM D7365	327788

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



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

(979) 690-1711
(800) 653-1711 USA/Canada
(979) 690-0440 Fax

www.oico.com
E-mail: OI-Mail@Xyleminc.com