Flow Solution™ FS 3700 Automated Chemistry Analyzer

Post-distillation Cyanide by Flow Injection Analysis and Photometric Detection USEPA 335.4

Cartridge Part Number 330352CT

Scope and Application

This method is used for the determination of cyanide in distilled samples, including water, wastewater, soil, and sludge, in accordance with **USEPA method 335.4**. This method also applies to the determination of total cyanide in samples distilled by other methods, such as **Standard Methods 4500-CN⁻** and **ASTM D2036**, with the assumption that 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 at mid-point of range
Method Detection Limit (MDL)	0.5 µg/L

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

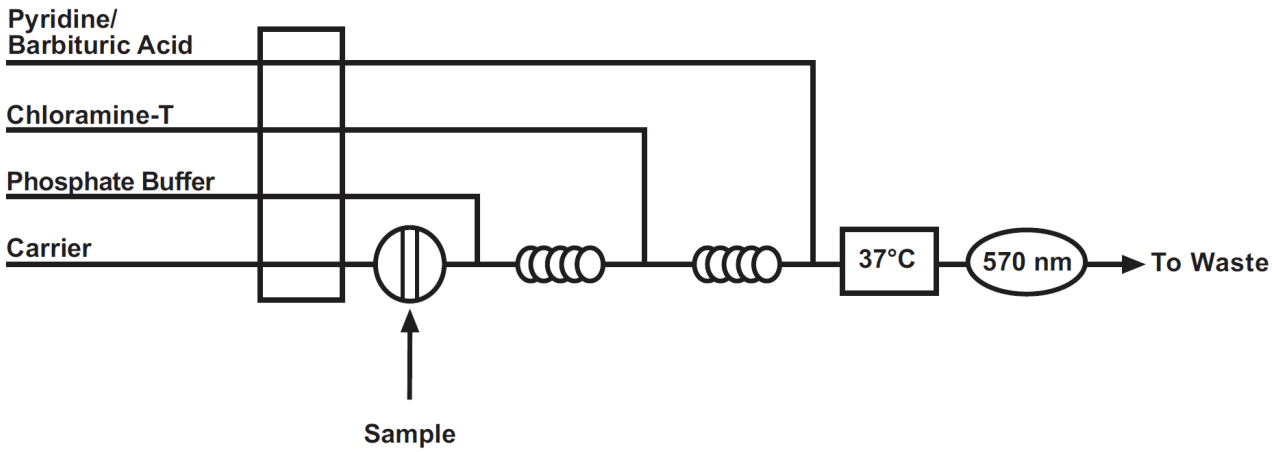


Figure 1. General flow diagram for Post-distillation Cyanide by USEPA 335.4

Reagents and Calibrants

Chemical Name	CAS #	Chemical Formula	Part Number
Barbituric acid	67-52-7	$C_4H_4N_2O_3$	
Brij®-35	9002-92-0	$(C_2H_4O)_nC_{12}H_{26}O$	326126
Challenge Matrix, ASTM D7365			327788
Chloramine-T trihydrate	7080-50-4	$CH_3C_6H_4SO_2NNaCl \cdot 3H_2O$	
Hydrochloric acid	7647-01-0	HCl	
Potassium cyanide	151-50-8	KCN	
Potassium hydroxide	1310-58-3	KOH	
Pyridine	110-86-1	C_5H_5N	
Sodium hydroxide	1310-73-2	NaOH	
Sodium phosphate, monobasic	7558-80-7	NaH_2PO_4	
Water, deionized		H_2O	
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 arsenite	7784-46-5	$NaAsO_2$	

Summary of USEPA Method 335.4

Method

1. 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.

Interferences

1. Several interferences are encountered with this method. Some of the known interferences to this method include aldehydes; nitrate/nitrite; and oxidizing agents, such as chlorine, thiocyanate, thiosulfate and sulfide. Multiple interferences may require the analysis of a series of laboratory-fortified sample matrices (LFM) to verify the suitability of the chosen treatment. Some interferences are eliminated or reduced by the distillation.

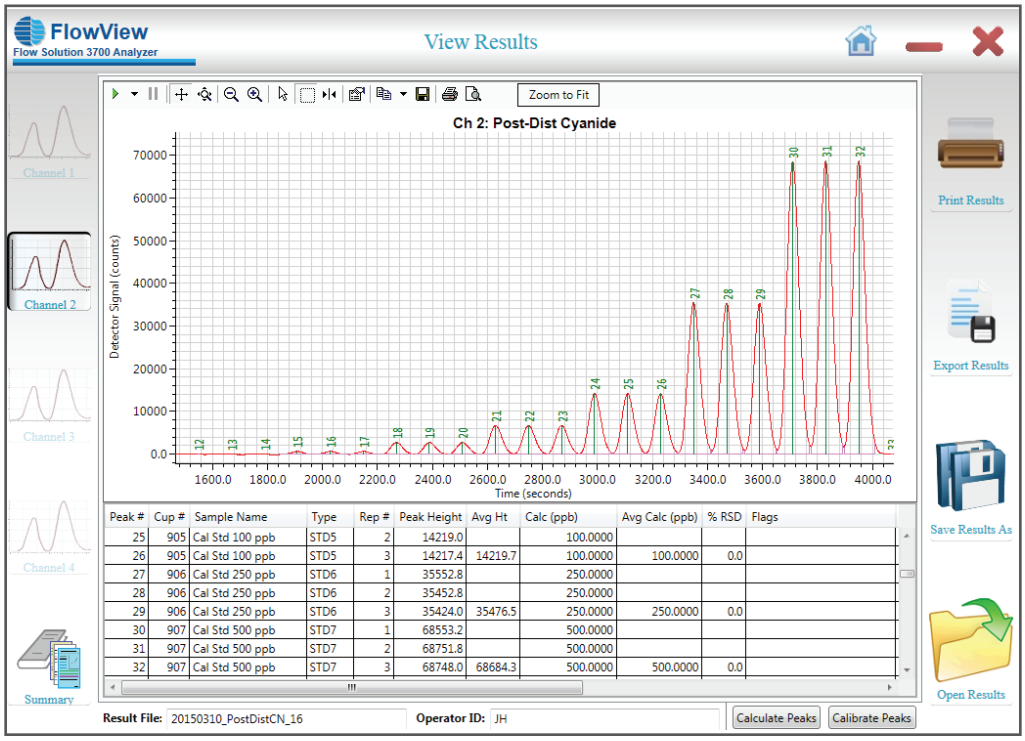
2. Sulfides adversely affect the procedure by producing hydrogen sulfide during distillation. Sample treatment is described in **Sample Handling and Preservation**.

3. High results may be obtained for samples that contain nitrate or nitrite. During distillation, nitrate and nitrite form nitrous acid that reacts with some organic compounds to form oximes. These oximes decompose under test conditions to generate HCN. Pretreatment with sulfamic acid eliminates nitrate and nitrite interferences. Sample treatment is described in **Sample Handling and Preservation**.

4. Oxidizing agents such as chlorine decompose most cyanides. Remove oxidizing agents that decompose cyanides by adding ascorbic acid if analysis is to be perfomed within 24 hours; otherwise, use sodium arsenite. Sample treatment is described in **Sample Handling and Preservation**.

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

6. 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.



Peak #	Cup #	Sample Name	Type	Rep #	Peak Height	Avg Ht	Calc (ppb)	Avg Calc (ppb)	% RSD	Flags
25	905	Cal Std 100 ppb	STD5	2	14219.0		100.0000			
26	905	Cal Std 100 ppb	STD5	3	14217.4	14219.7	100.0000	100.0000	0.0	
27	906	Cal Std 250 ppb	STD6	1	35552.8		250.0000			
28	906	Cal Std 250 ppb	STD6	2	35452.8		250.0000			
29	906	Cal Std 250 ppb	STD6	3	35424.0	35476.5	250.0000	250.0000	0.0	
30	907	Cal Std 500 ppb	STD7	1	68553.2		500.0000			
31	907	Cal Std 500 ppb	STD7	2	68751.8		500.0000			
32	907	Cal Std 500 ppb	STD7	3	68748.0	68684.3	500.0000	500.0000	0.0	

Figure 2. Post-distillation Cyanide Calibration Series

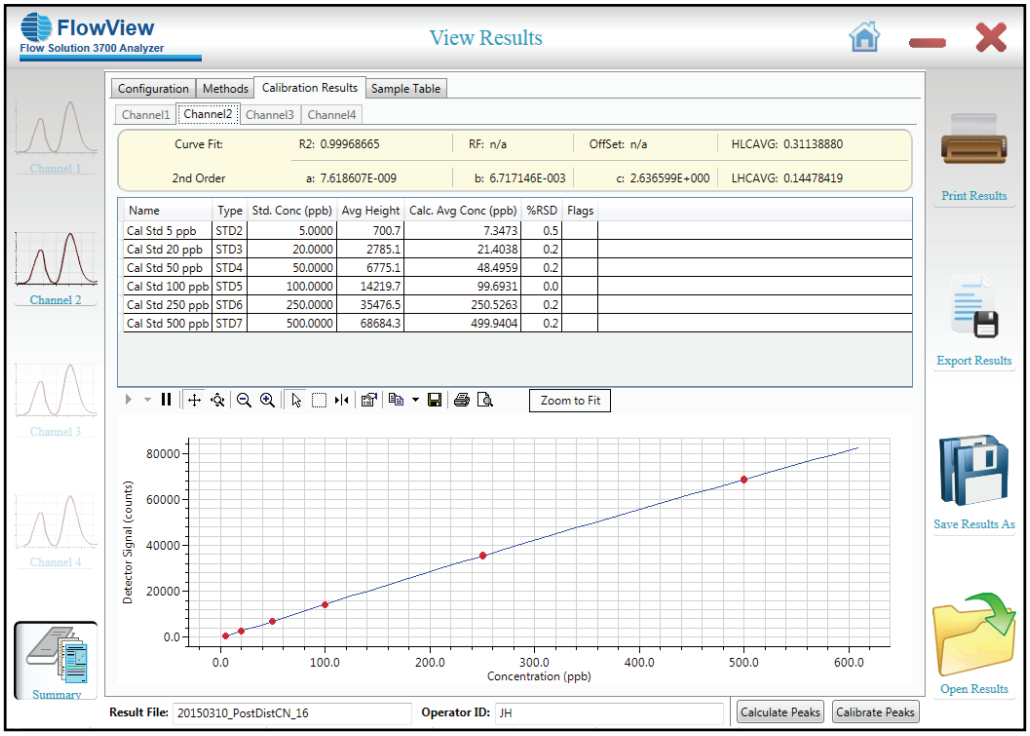


Figure 3. Calibration curve and statistics

O·I·Analytical
a xylem brand
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

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