Flow Solution™ FS 3700 Automated Chemistry Analyzer

Post-distillation Phenol by Flow Injection Analysis and Photometric Detection USEPA 420.4
Cartridge Part Number 330083CT

Scope and Application

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.

Method Performance

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

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

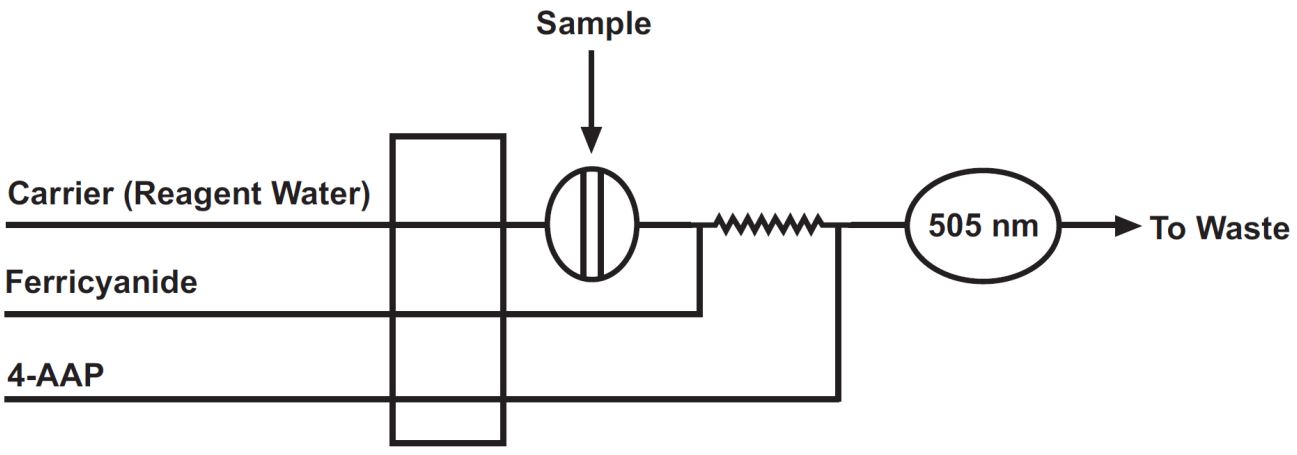

Figure 1. General flow diagram for Post-distillation Phenol by USEPA 420.4.

Reagents and Calibrants

Chemical Name	CAS #	Chemical Formula	Part Number
4-Aminoantipyrine	83-07-8	$C_{11}H_{13}N_3O$	
Boric acid	10043-35-3	H_3BO_3	
DOWFAX 2A1	12626-49-2		328852
Phenol	108-95-2	C_6H_5OH	
Phosphoric acid	7664-38-2	H_3PO_4	
Potassium chloride	7447-40-7	KCl	
Potassium ferricyanide	13746-66-2	$K_3Fe(CN)_6$	
Sodium hydroxide	1310-73-2	NaOH	
Sulfuric acid, concentrated	7664-93-9	H_2SO_4	
Water, deionized		H_2O	
Additionally, the following chemicals may be needed for sample preservation or treatment.			
Cupric sulfate pentahydrate	7758-99-8	$CuSO_4 \cdot 5H_2O$	
Ferrous ammonium sulfate	10045-89-3	$(NH_4)_2SO_4FeSO_4$	

Summary of US EPA Method 420.4

US EPA Method 420.4 summarizes the method and interferences as follows below.[1]

Method

- Prior to analysis, phenol is manually distilled from an acidic soluton. Phenol distillate reacts with 4-aminoantipyrine (4-AAP) and alkaline ferricyanide (FeCN) to form a red complex. The absorbance is measured at 505 nm.[1]

- Color response of phenolic materials with 4-aminoantipyrine is not the same for all compounds. Because phenolic type wastes usually contain a variety of phenols, it is not possible to duplicate a mixture of phenols to be used as a standard. For this reason, phenol has been selected as a standard and any color produced by the reaction of other phenolic compounds is reported as phenol. This value will represent the minimum concentration of phenolic compounds present in the sample.[1]

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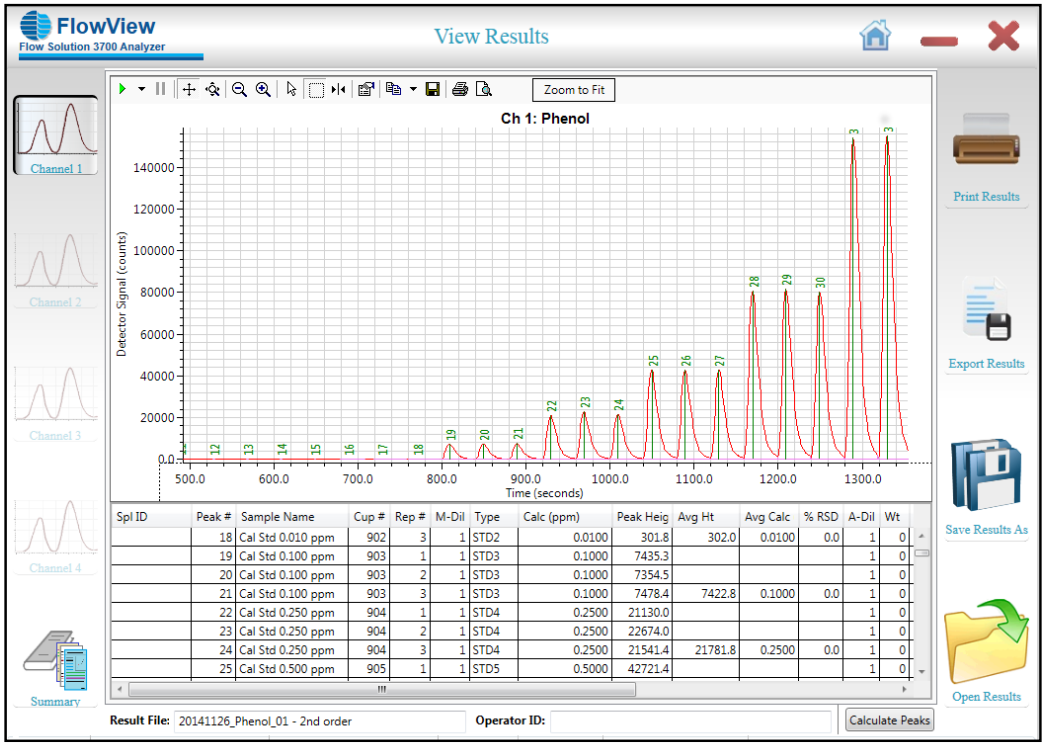
Interferences

- Sulfide interferences significantly at concentrations greater than 10 ppm. Eliminate these interferences by acidifying the sample to a pH < 2 with sulfuric acid, aerating briefly and stirring. Copper sulfate may also be used. Allow the resulting precipitate to settle before decanting the supernatant. This method is able to tolerate 200 ppb sulfide.

- Remove oxidizing agents such as chlorine immediately after sampling by adding an excess of ferrous ammonium sulfate. Oxidizing agents can be detected by the liberation of iodine upon acidification in the presence of potassium iodide. If chlorine is not removed, the phenolic compounds may be partially oxidized, and the results may be low.

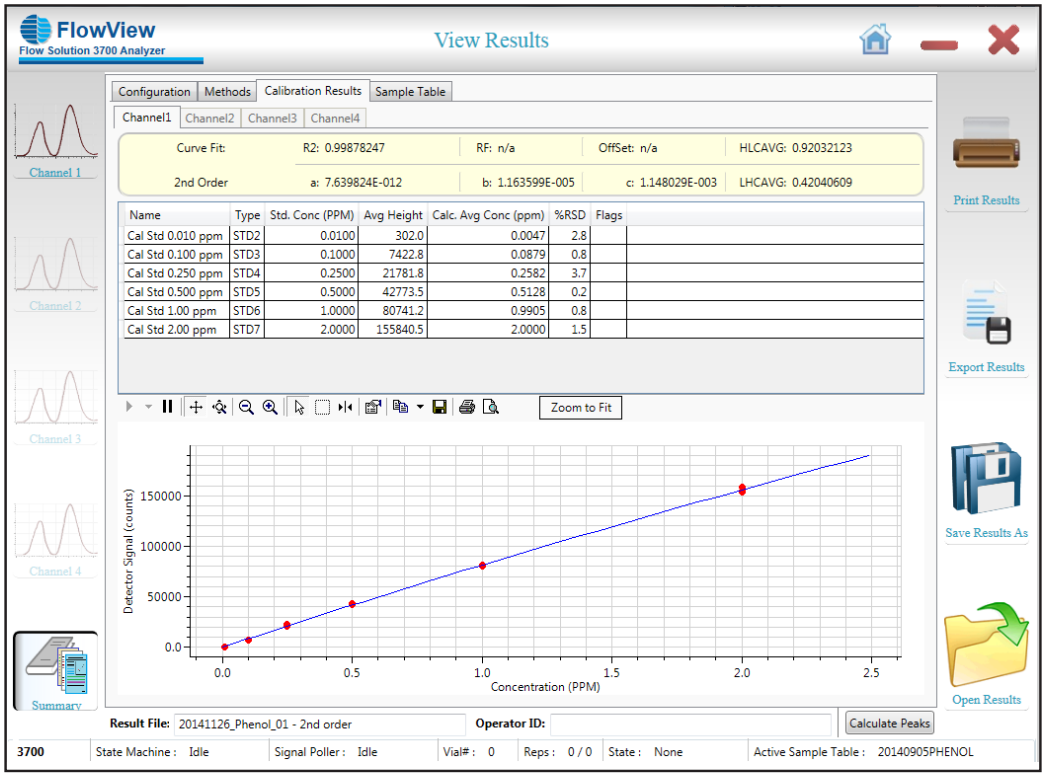
- Use glass tubes or acid-washed plastic cups for the samples and calibrants to eliminate background contamination from plastic tubes and sample containers. Use sample line pump tubes to mitigate inherent contamination from PVC pump tubes.

- 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.



Spl ID	Peak #	Sample Name	Cup #	Rep #	M-Dil	Type	Calc (ppm)	Peak Heig	Avg Ht	Avg Calc	% RSD	A-Dil	Wt
	18	Cal Std 0.010 ppm	902	3	1	STD2	0.0100	301.8	302.0	0.0100	0.0	1	0
	19	Cal Std 0.100 ppm	903	1	1	STD3	0.1000	7435.3				1	0
	20	Cal Std 0.100 ppm	903	2	1	STD3	0.1000	7354.5				1	0
	21	Cal Std 0.100 ppm	903	3	1	STD3	0.1000	7478.4	7422.8	0.1000	0.0	1	0
	22	Cal Std 0.250 ppm	904	1	1	STD4	0.2500	21130.0				1	0
	23	Cal Std 0.250 ppm	904	2	1	STD4	0.2500	22674.0				1	0
	24	Cal Std 0.250 ppm	904	3	1	STD4	0.2500	21541.4	21781.8	0.2500	0.0	1	0
	25	Cal Std 0.500 ppm	905	1	1	STD5	0.5000	42721.4				1	0

Figure 2. Post-distillation Phenol Calibration Series



Name	Type	Std. Conc (PPM)	Avg Height	Calc. Avg Conc (ppm)	%RSD	Flags
Cal Std 0.010 ppm	STD2	0.0100	302.0	0.0047	2.8	
Cal Std 0.100 ppm	STD3	0.1000	7422.8	0.0879	0.8	
Cal Std 0.250 ppm	STD4	0.2500	21781.8	0.2582	3.7	
Cal Std 0.500 ppm	STD5	0.5000	42773.5	0.5128	0.2	
Cal Std 1.00 ppm	STD6	1.0000	80741.2	0.9905	0.8	
Cal Std 2.00 ppm	STD7	2.0000	155840.5	2.0000	1.5	

Figure 3. Calibration curve and statistics

Table 1. Consumables, spare parts and accessories for Post-distillation Phenol

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

Pump tubes should be replaced monthly, or as-needed to maintain system performance. Maximum life expectancy for pump tubes is approximately 800 hours. Refer to the operating notes section for details regarding the use of surfactant and Kleenflow™ for maintaining system performance.

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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