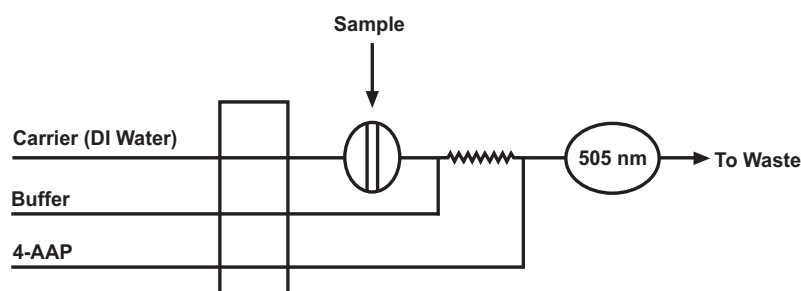


Method Abstract

Scope	This method is used for determining phenolic compounds in drinking water, surface water and and domestic and industrial wastes according to USEPA Method 420.4. Additionally, this method enables phenol index analysis according to ISO Method 14402.	
Summary	Prior to analysis, the phenol is distilled off-line from an acidic solution at 160 °C. Phenol reacts with 4-aminoantipyrine (4-AAP) and alkaline ferricyanide (FeCN) to form a red complex. The absorbance is measured at 505 nm.	
Interferences	Eliminate interferences from sulfur compounds by acidifying the sample to a pH of less than 4.0 with phosphoric acid, aerating briefly by stirring, and adding copper sulfate. Immediately remove oxidizing agents such as chlorine 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. Reduce background contamination from plastic tubing and sample containers by using glass sample tubes or acid-washed plastic cups for the samples and calibrants; run reagents through new pump tubes for at least an hour prior to running samples at low phenol concentrations.	
Performance Specifications	Range:	10.0–2,000 µg/L
	Throughput:	90 samples/hour
	Precision (at 100 µg/L):	<2% RSD
	Precision (at 500 µg/L):	<1% RSD
	Method Detection Limit (MDL):	2.0 µg/L
Chemicals	4-Aminoantipyrine, C ₁₁ H ₁₃ N ₃ O	Phosphoric Acid, concentrated, 85%, H ₃ PO ₄
	Boric Acid, H ₃ BO ₄	Potassium Chloride, KCl
	Deionized (DI) Water (ASTM Type I or II)	Potassium Ferricyanide, K ₃ Fe(CN) ₆
	Ferrous Ammonium Sulfate, (NH ₄) ₂ SO ₄ •FeSO ₄ •6H ₂ O	Sodium Hydroxide, NaOH
		Sulfuric Acid, concentrated, H ₂ SO ₄
	Phenol, C ₆ H ₅ OH	

Basic Flow Diagram



Selected Reference

Methods for Chemical Analysis of Water and Wastewater; EPA–600/4–79–020; U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory: Cincinnati, OH, 1984; Method 420.2.

Water Quality–Determination of Phenol Index by Flow Analysis (FIA and CFA), International Standard; ISO 14402:1999 (E); 1st ed.; Geneva, Switzerland, 1999.

Figures

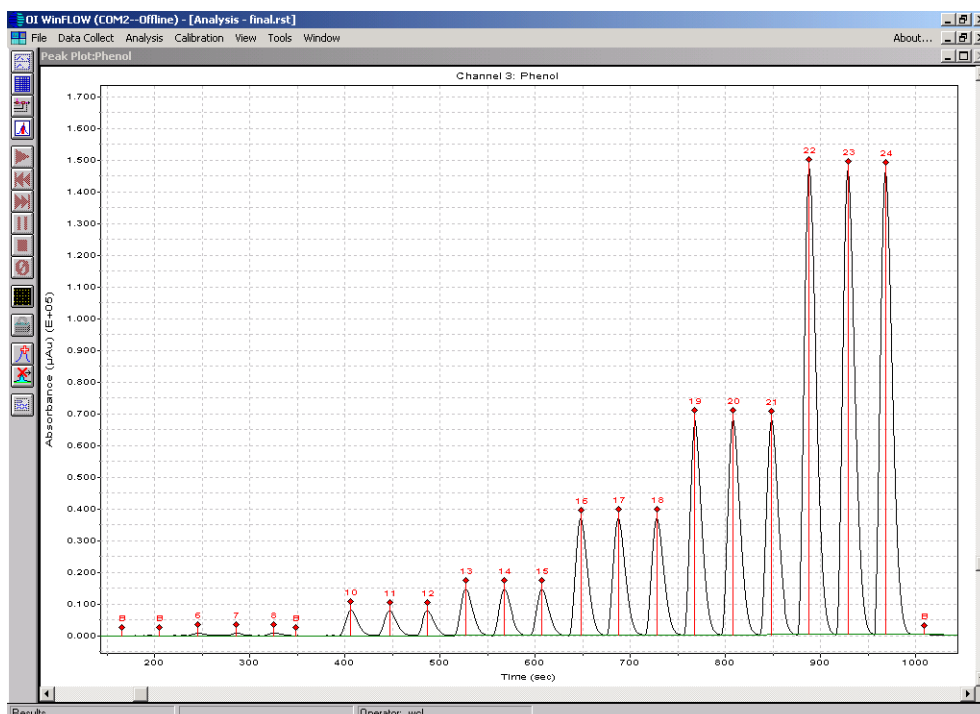


Figure 1. Post-Distillation Phenol Calibration (10–2,000 ppb)

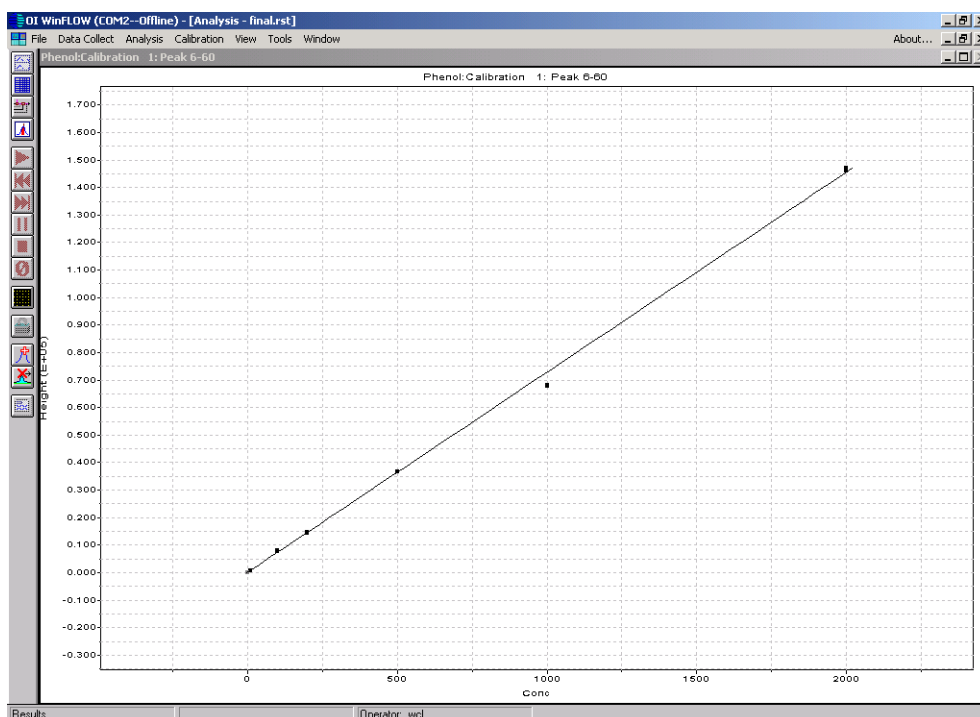


Figure 2. Post-Distillation Phenol Calibration Curve (10–2,000 ppb)

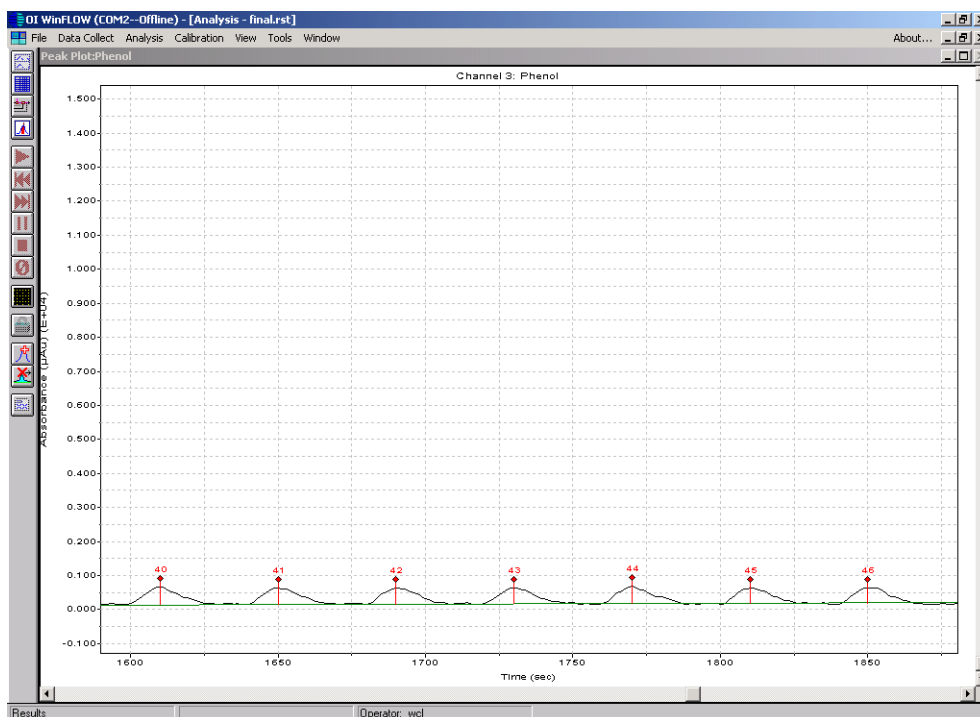


Figure 3. Post-Distillation Phenol Method Detection Limit (at 5 ppb)

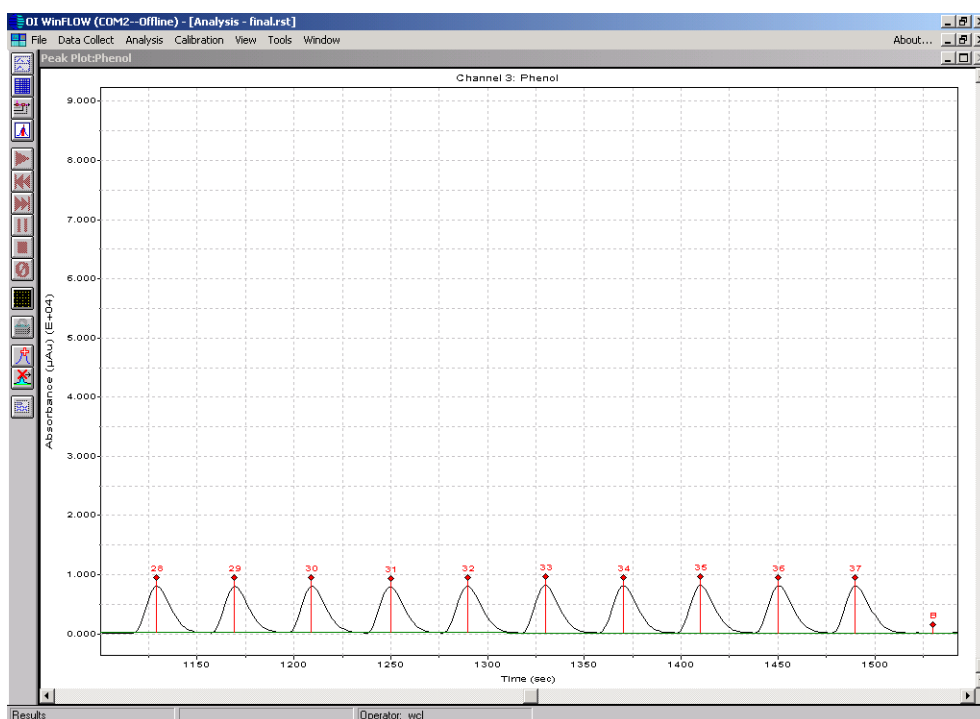


Figure 4. Post-Distillation Phenol Precision (at 100 ppb)

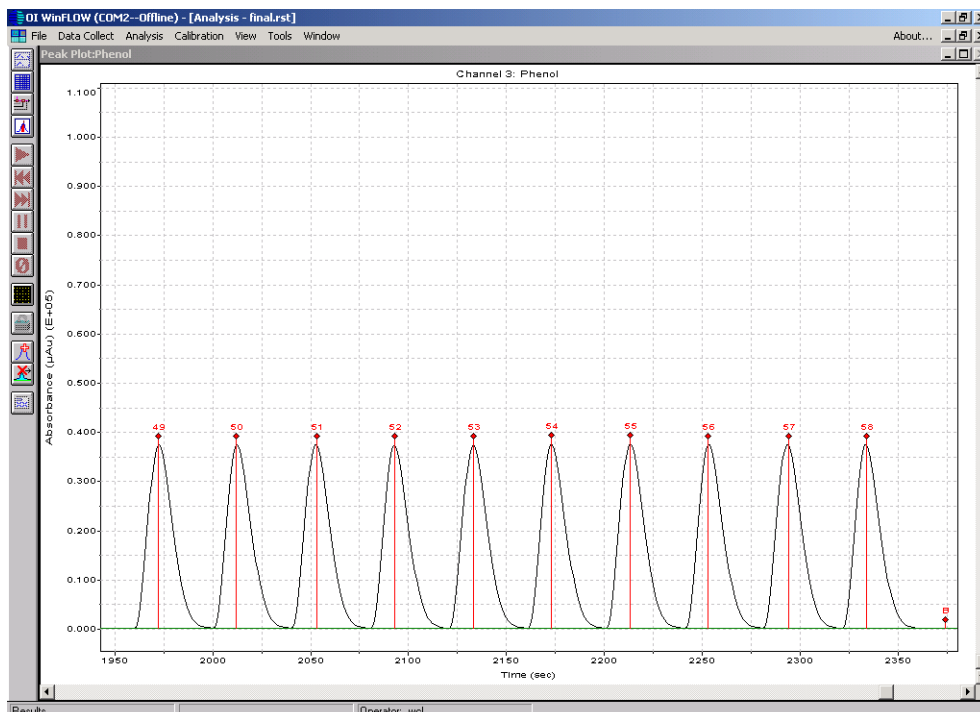


Figure 5. Post-Distillation Phenol Precision (at 500 ppb)

Phenol:Calibration 1: Peak 6-60

Name	Conc	Height
* cal 10.00 ppb	10.000000	802.800171
* cal 10.00 ppb	10.000000	792.794800
* cal 10.00 ppb	10.000000	847.402344
* cal 100.0 ppb	100.000000	8060.19873
* cal 100.0 ppb	100.000000	7784.73533
* cal 100.0 ppb	100.000000	7789.34570
* cal 200.0 ppb	200.000000	14577.7949
* cal 200.0 ppb	200.000000	14527.9560
* cal 200.0 ppb	200.000000	14459.8037
* cal 500.0 ppb	500.000000	36781.3164
* cal 500.0 ppb	500.000000	36837.1289
* cal 500.0 ppb	500.000000	36834.0312
* cal 1000 ppb	1000.000000	68100.8828
* cal 1000 ppb	1000.000000	67911.5546
* cal 1000 ppb	1000.000000	67714.9296
* cal 2000 ppb	2000.000000	147005.656
* cal 2000 ppb	2000.000000	146332.203
* cal 2000 ppb	2000.000000	146068.812

Calib Coef:	
y=bx+a	
a: (intercept)	1.7593e+02
b:	7.2739e+01
Corr Coef:	
0.999220	
Carryover:	
0.134%	
No Drift Peaks	

Figure 6. Post-Distillation Phenol Calibration Results (10–2,000 ppb)

Table 1. Total Phenolics Precision Calculations

	500 µg/L	100 µg/L	5.00 µg/L
Rep 1	510	104	4.82
Rep 2	508	104	4.18
Rep 3	510	104	3.96
Rep 4	509	104	4.06
Rep 5	508	104	4.47
Rep 6	512	107	3.66
Rep 7	512	106	3.56
Rep 8	509	107	—
Rep 9	508	106	—
Rep 10	509	106	—
Mean	510	105	4.10
Standard Deviation	1.6633	1.4662	0.439982
% RSD	0.33	1.39	10.7
% Recovery	102	105	82
MDL	—	—	1.4