Method Abstract

Scope

This method is used for determining phenolic materials in drinking water, surface water, and domestic and industrial wastes according to USEPA Method 420.4. Additionally, this method enables phenol index analysis according to ISO Method 14402.

Summary

Phenol reacts with 4-aminoantipyrine and alkaline ferricyanide to form a red complex that is measured at 505 nm.

Interferences

Interferences from sulfur compounds are eliminated by acidifying the sample to a pH of less than 4.0 with sulfuric acid, and aerating briefly by stirring and adding cupric sulfate.

Oxidizing agents such as chlorine, detected by the liberation of iodine upon acidification in the presence of potassium iodide, are removed immediately after sampling by the addition of an excess of ferrous ammonium sulfate. If chlorine is not removed, the phenolic compounds may be partially oxidized, and the results may be lower than expected.

Background contamination from reagent and sample containers can be eliminated by using glass containers. To further minimize contamination, shorten the pump tubing by cutting just before and after the Precision Pump; replace these segments of tubing with Teflon® or PET.

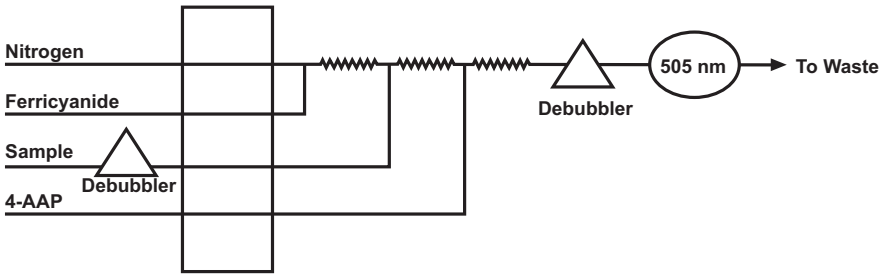
Performance Specifications

Range:	5.0–500 µg/L
Throughput:	30 samples/hour
Precision (at 5.0 µg/L):	~3% RSD
Precision (at 50 µg/L):	<1% RSD
Precision (at 500 µg/L):	<1% RSD
Method Detection Limit (MDL):	0.5 µg/L
ERA QC Sample Result:	106%

Chemicals

4-Aminoantipyrine, $C_{11}H_{13}N_3O$
Boric Acid, H_3BO_3
Deionized (DI) Water, ASTM Type I or II
DOWFAX® 2A1, part number A000080
Phenol, C_6H_5OH
Phosphoric Acid, concentrated, H_3PO_4
Potassium Chloride, KCl
Potassium Ferricyanide, $K_3Fe(CN)_6$
Sodium Hydroxide, NaOH

Basic Flow Diagram



Note

This method complies with USEPA Method 420.4.

Selected Reference

Phenolics, Total Recoverable (Colorimetric, Semi-Automated). *Methods for the Determination of Inorganic Substances in Environmental Samples*; EPA/600/R-93/100; U.S.

Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory: Cincinnati, OH, 1993; Method 420.4.

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

Figures

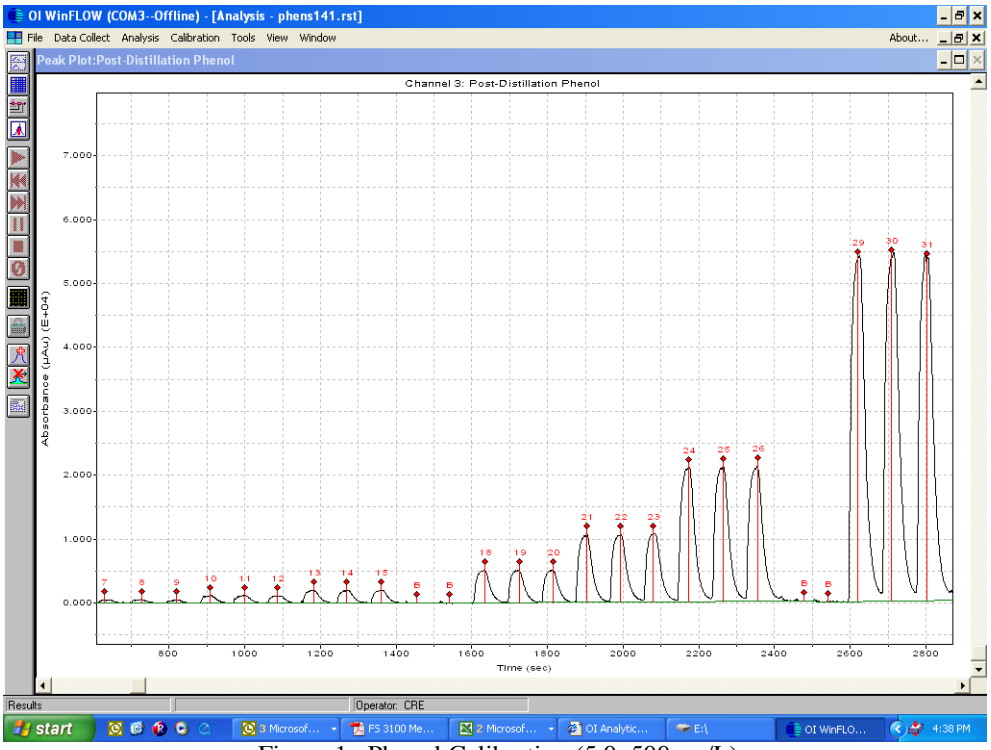


Figure 1. Phenol Calibration (5.0–500 µg/L)

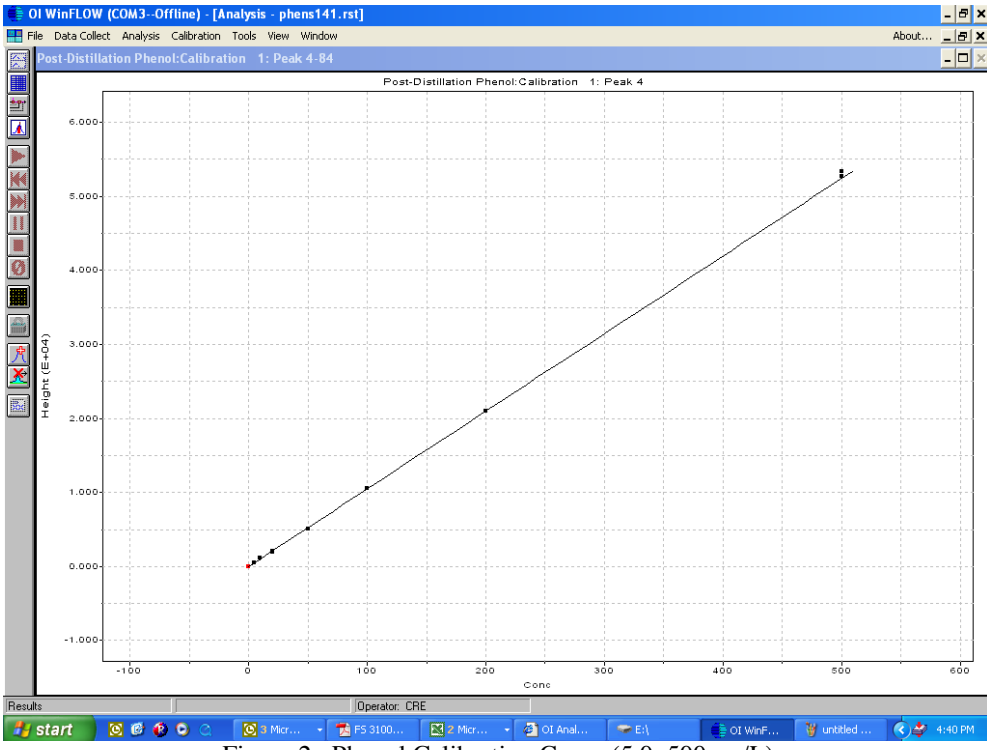


Figure 2. Phenol Calibration Curve (5.0–500 μg/L)

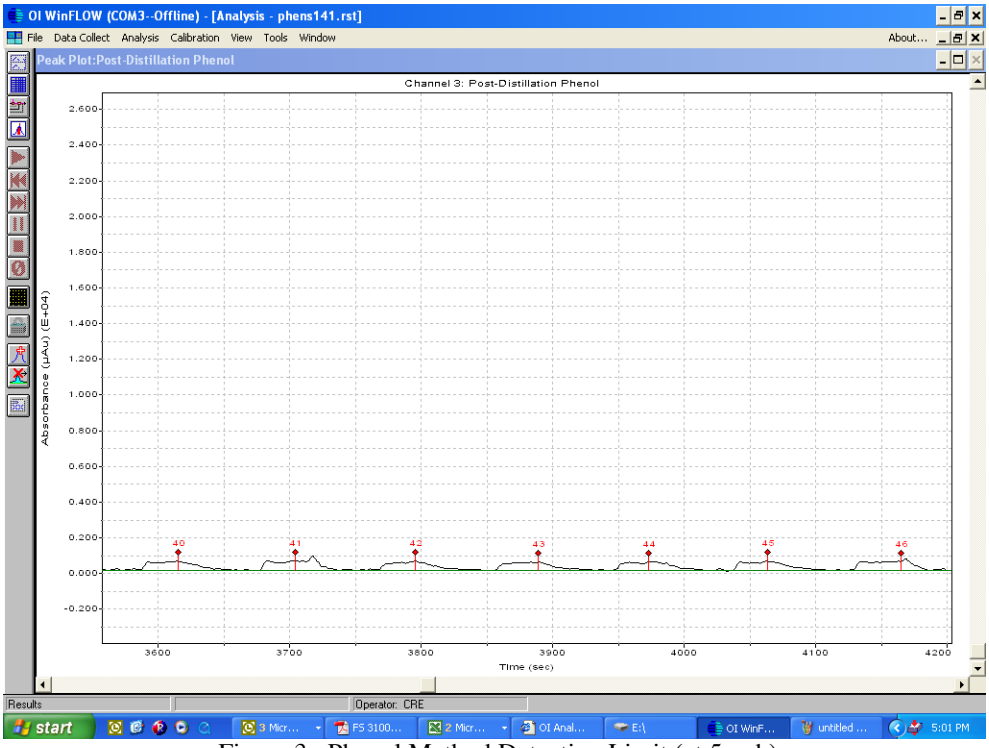


Figure 3. Phenol Method Detection Limit (at 5 ppb)

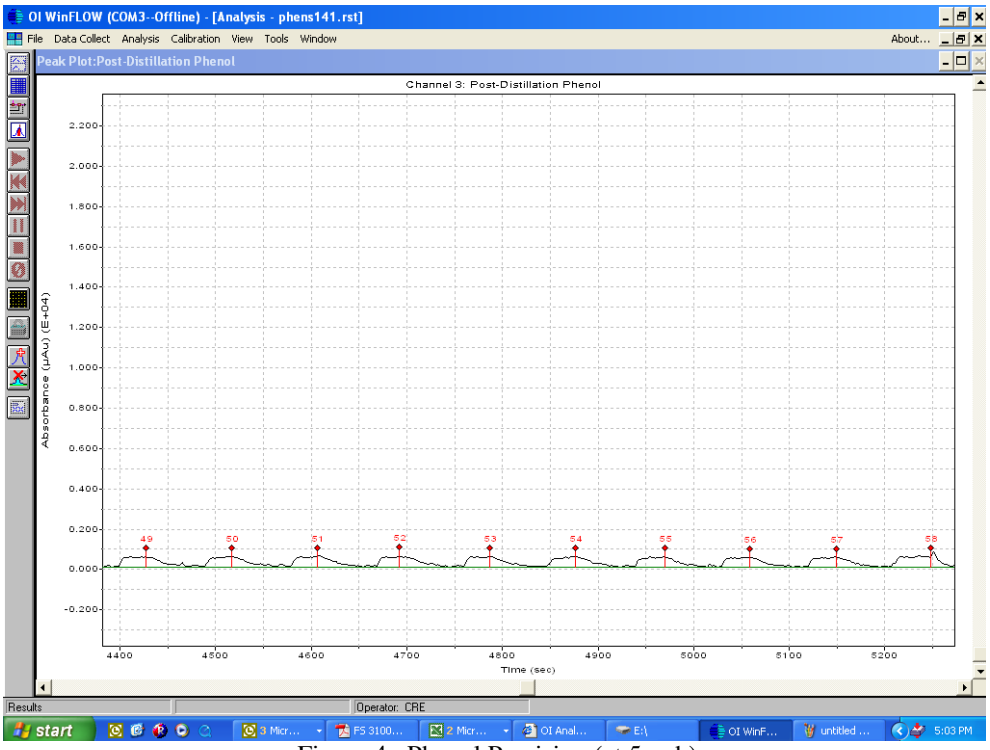


Figure 4. Phenol Precision (at 5 ppb)

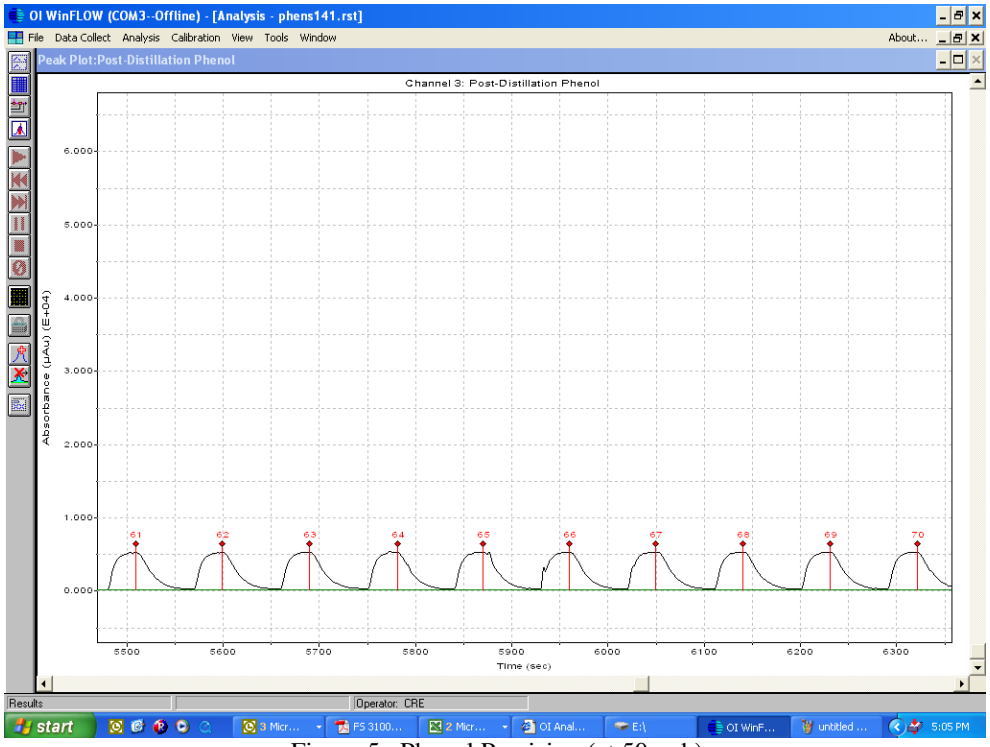


Figure 5. Phenol Precision (at 50 ppb)

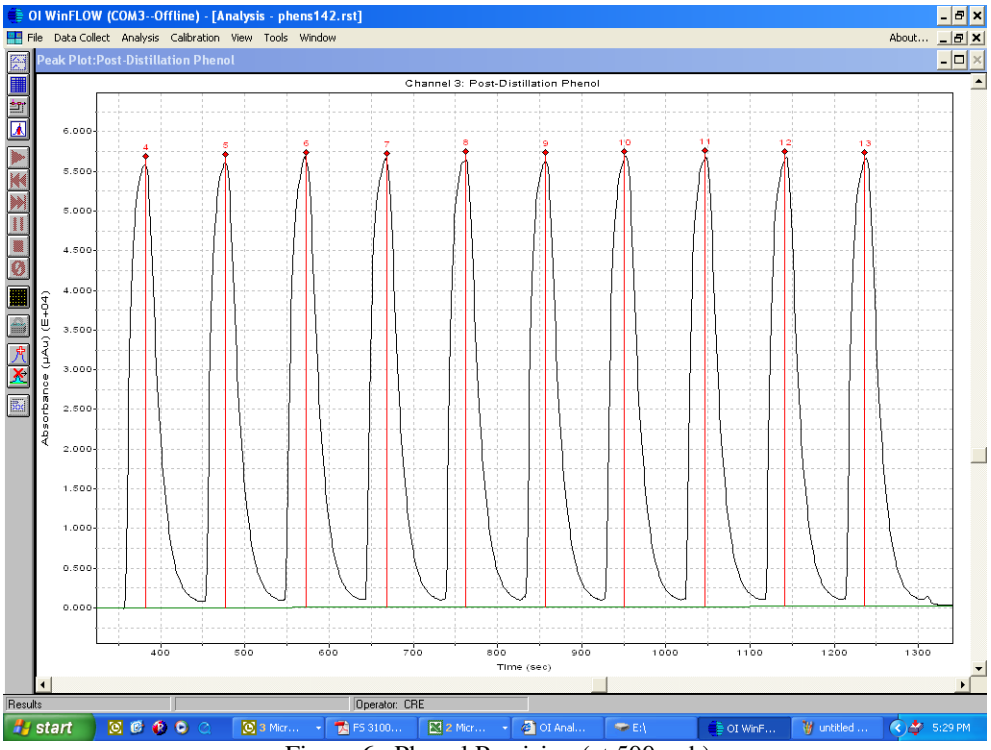


Figure 6. Phenol Precision (at 500 ppb)

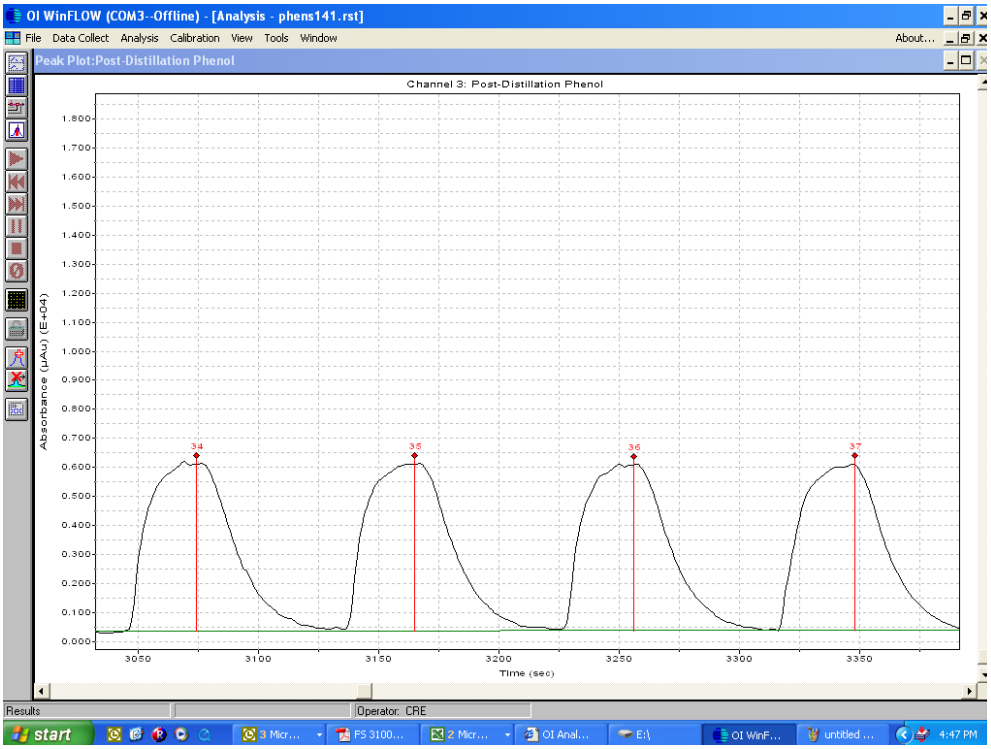


Figure 7. Phenol ERA QC Sample Precision (ERA 51.2 ppb)

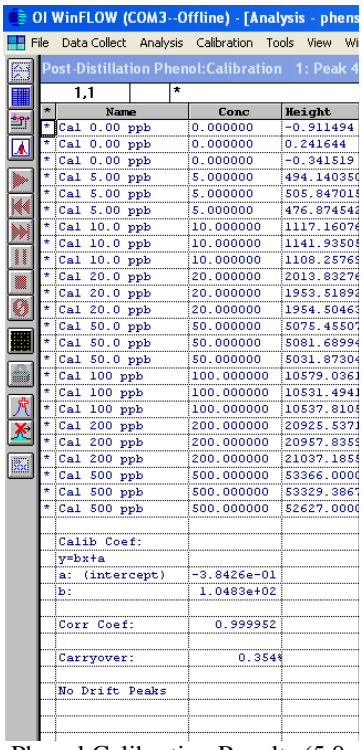


Figure 8. Phenol Calibration Results (5.0–500 µg/L)

Table 1: Phenol Method Data

Parameter	Calibrant (5.0 µg/L)	Calibrant (5.0 µg/L)	Calibrant (50 µg/L)	Calibrant (500 µg/L)	ERA QC Standard (51.2 µg/L)
Rep 1	5.25	4.81	48.88	533.25	54.78
Rep 2	5.34	4.89	48.71	533.36	54.57
Rep 3	5.46	5.18	49.25	535.21	54.19
Rep 4	4.53	5.23	49.25	533.94	54.42
Rep 5	4.92	5.06	49.35	535.57	—
Rep 6	5.02	5.17	49.34	533.86	—
Rep 7	4.68	5.05	49.53	535.53	—
Rep 8	—	4.77	49.58	535.83	—
Rep 9	—	4.85	49.55	534.52	—
Rep 10	—	5.13	49.53	534.08	—
Average	5.028	5.055	49.188	534.515	54.489
Standard Deviation	0.3441	0.1574	0.2866	0.2930	0.2453
% RSD	6.84	3.11	0.58	0.055	0.45
MDL	1.08	—	—	—	—
% Accuracy	—	—	—	—	106.42%

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