Method Abstract

Scope

This method is used for the determination of phenolic compounds in drinking water, surface water, and domestic and industrial wastes, according to U.S. EPA 420.4. Additionally, this method enables phenol index analysis following in-line distillation according to ISO Method 14402. The Method Detection Limit (MDL) is 0.05 mg/L–phenol. The applicable range of the method is 0.5–1,000 ppb phenol. The range may be extended to analyze higher concentrations by sample dilution.

Summary

Phenol is distilled in-line from an acidic solution at 185 °C. The phenol distillate reacts with 4-aminoantipyrine (4-AAP) and alkaline ferricyanide (FeCN) to form a red complex. The absorbance is measured at 505 nm. The quality of the analysis is assured through reproducible calibration and testing of the Segmented Flow Analysis (SFA) system.

Interferences

Sulfide interferes significantly at concentrations greater than 10 ppm. Eliminate these interferences by acidifying the sample to a pH less than 4 with sulfuric acid, aerating briefly by stirring, and adding copper sulfate. Allow the resulting precipitate to settle before decanting the supernatant.

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 tube to mitigate inherent contamination from PVC pump tubes.

Performance Specifications

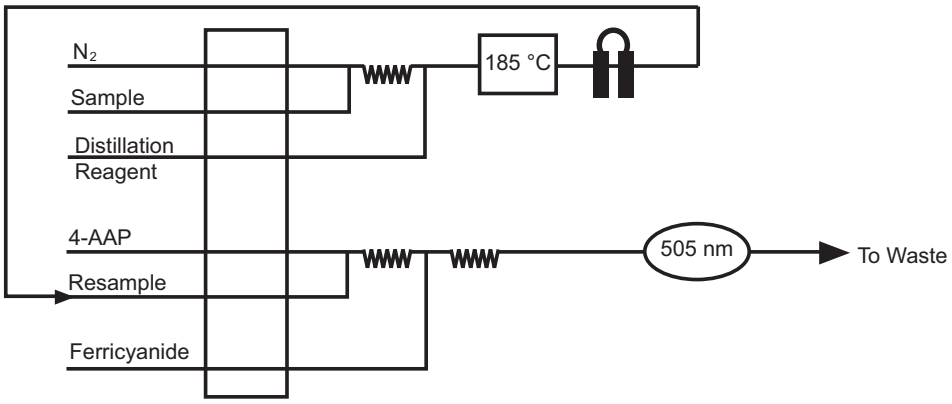
Range		0.10–4.0 mg/L
Throughput		22 samples/hr
Precision at	50 µg/L	~1% RSD
	500 µg/L	~1% RSD
Method Detection Limit (MDL)		0.05 mg/L
Accuracy*		96%

*ERA (Environmental Resource Associates) WasteWatR Cyanide and Phenol Quality Control Sample

Chemicals

4-Aminoantipyrine, $C_{11}H_{13}N_3O$ (FW 203.25)	Boric Acid, H_3BO_4 (FW 61.84)
Phenol, C_6H_5OH (FW 94.11)	Phosphoric Acid, concentrated, 85%, H_3PO_4 (FW 98.00)
Potassium Chloride, KCl (FW 74.55)	Potassium Ferricyanide, $K_3Fe(CN)_6$ (FW 329.25)
Sodium Hydroxide, NaOH (FW 40.00)	Sulfuric Acid, concentrated, H_2SO_4 (FW 98.08)

Basic Flow Diagram



Selected References

Phenolics, Total Recoverable (Colorimetric, Automated 4-AAP with Distillation). *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

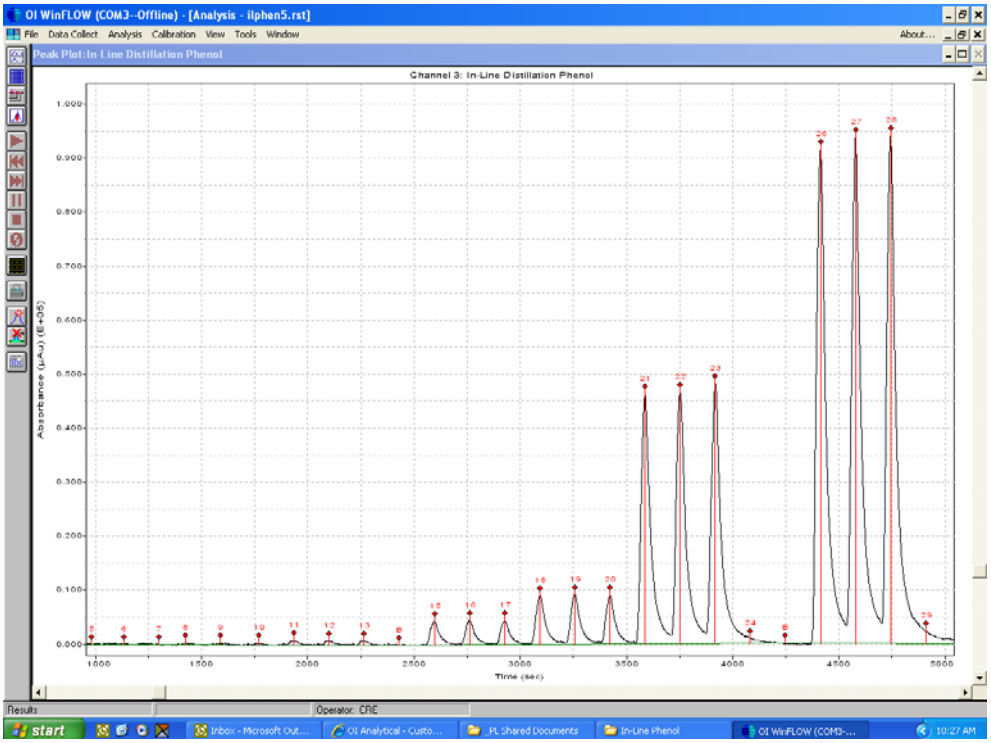


Figure 1. In-line Distillation Phenol Calibration (0.5–1,000 ppb)

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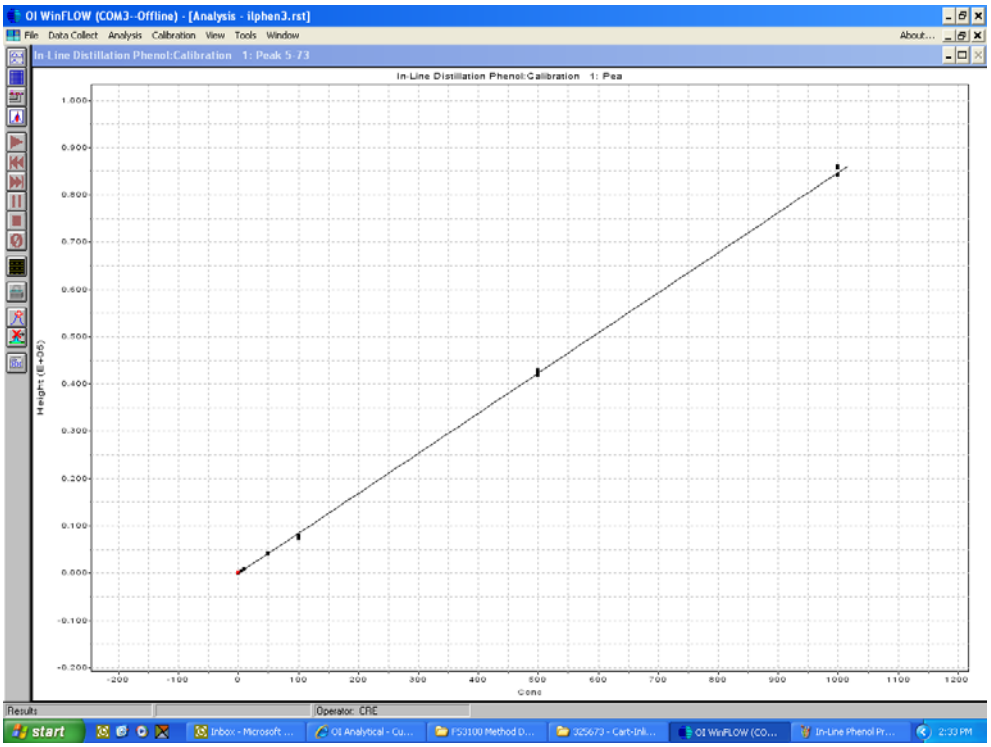

Figure 2. In-line Distillation Phenol Calibration Curve (0.5–1,000 ppb)

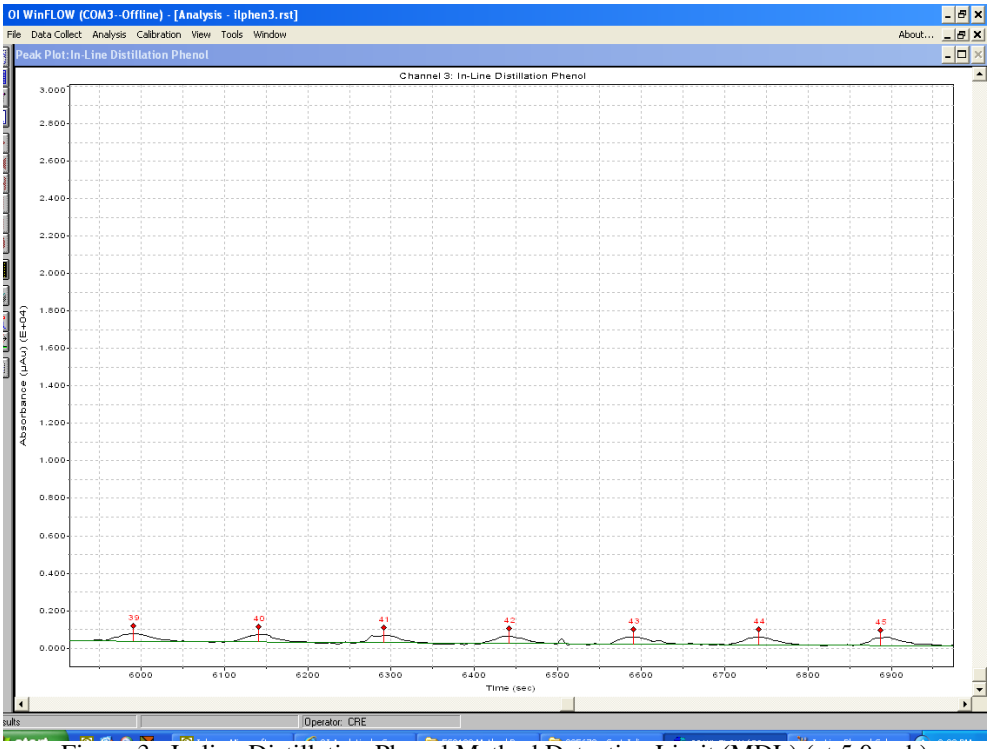

Figure 3. In-line Distillation Phenol Method Detection Limit (MDL) (at 5.0 ppb)

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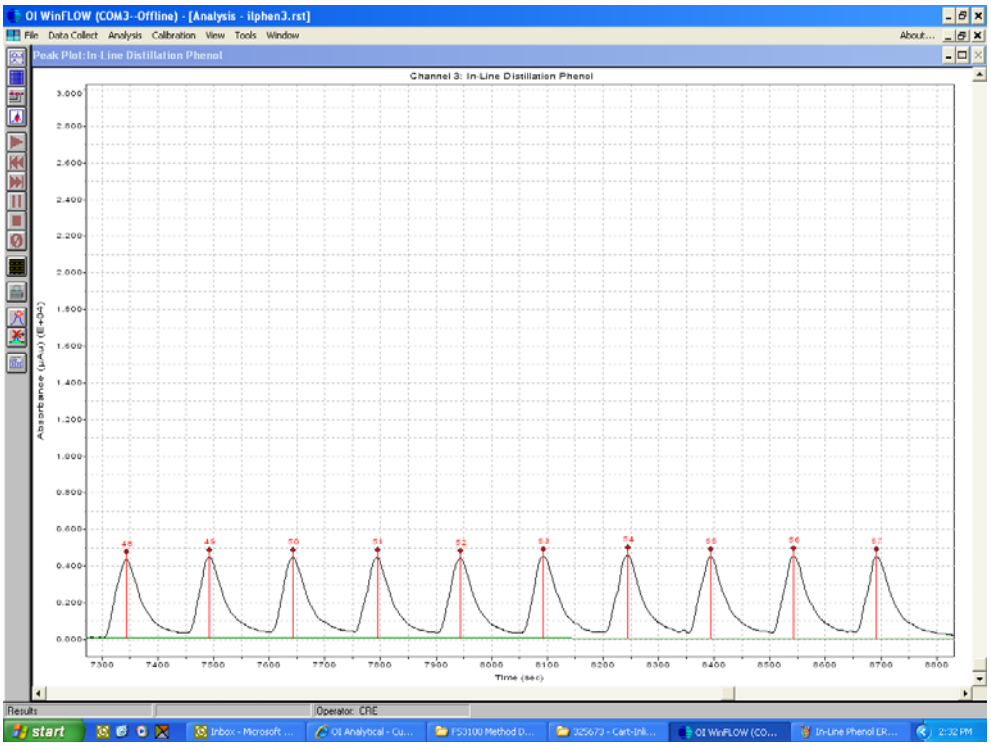

Figure 4. In-line Distillation Phenol Precision (at 50 ppb)

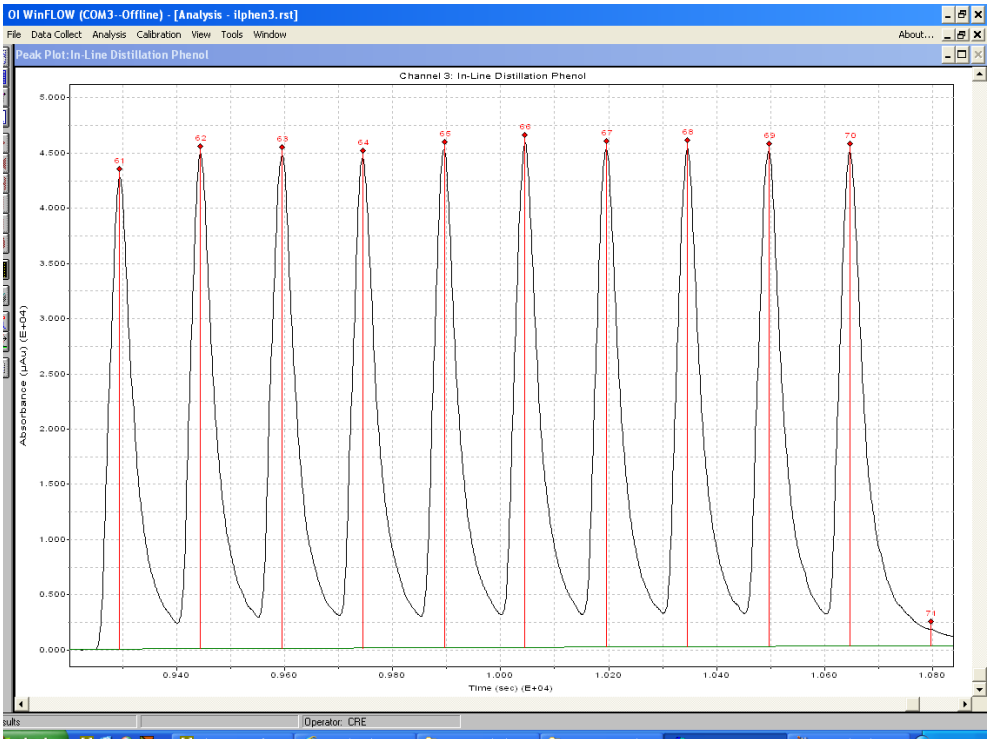

Figure 5. In-line Distillation Phenol Precision (at 500 ppb)

Table 1: In-line Distillation Phenol Validation Results

Parameter	Calibrant 5.0 µg/L	Calibrant 50 µg/L	Calibrant 500 µg/L	ERA QC Standard 717 µg/L
Rep 1	5.4014	51.4071	505.3069	692.1567
Rep 2	4.9648	51.3472	515.1608	698.6903
Rep 3	4.9733	51.3769	512.8530	697.4190
Rep 4	4.8754	51.2417	508.9299	702.0557
Rep 5	4.7845	50.6147	518.0547	—
Rep 6	5.3818	51.9511	524.3877	—
Rep 7	5.1404	52.7628	517.2325	—
Rep 8	—	51.9297	517.2325	—
Rep 9	—	52.2523	513.8435	—
Rep 10	—	52.1105	513.4267	—
Average	5.0702213	51.699387	514.71146	697.58043
Standard Deviation	0.2444358	0.6165759	5.2639552	4.1110927
%RSD	4.8210087	1.1926173	1.0227002	0.589336
MDL	0.7675284	—	—	—
%Accuracy	—	—	—	96.42

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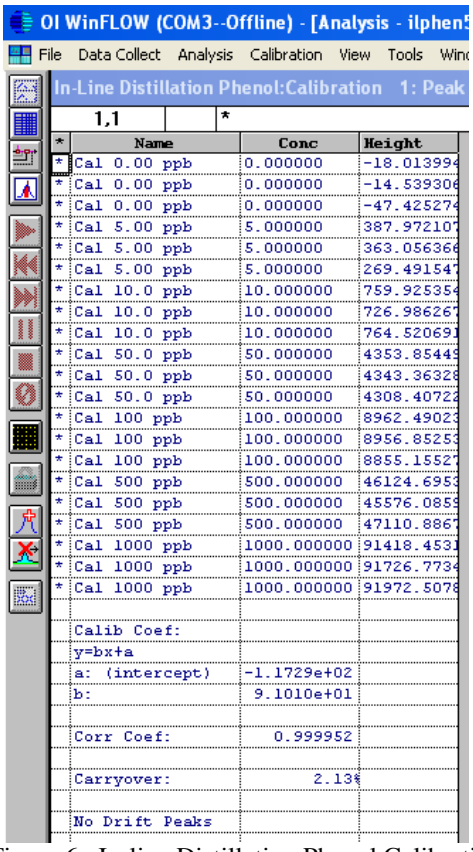

Figure 6. In-line Distillation Phenol Calibration Results (0.5–1,000 ppb)