

Flow Solution™ FS 3700 Automated Chemistry Analyzer

In-Line Distillation of Phenol, USEPA by Segmented Flow Analysis (SFA) USEPA 420.4

Cartridge Part Number 330364CT and Channel Part Number 330363

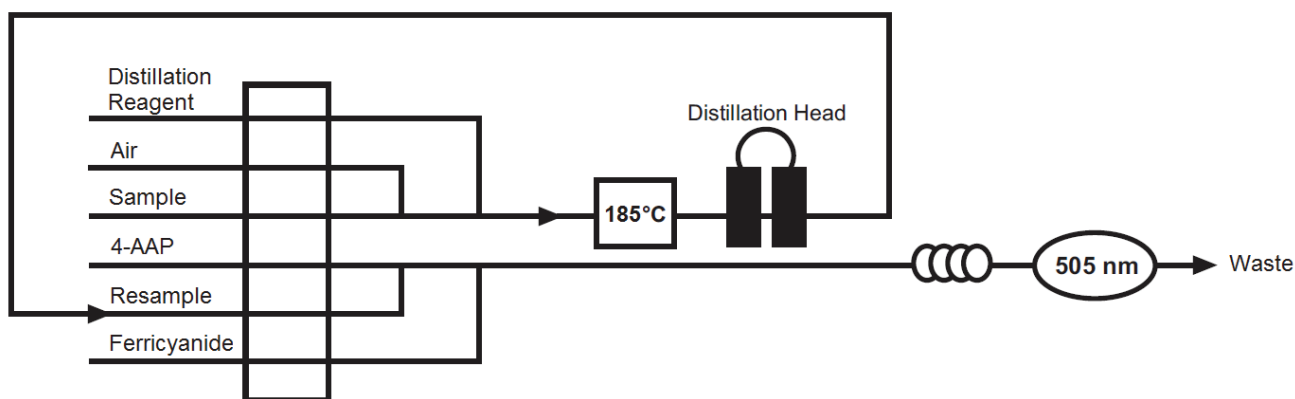
Scope and Application

This method is used for the determination of phenolic compounds in drinking water, surface water, and domestic and industrial wastes according to USEPA 420.4.¹ Additionally, this method enables phenol index analysis following in-line distillation according to ISO Method 14402.²

Method Performance

Range	1.0 µg/L – 500 µg/L
Rate	22 samples/hour
Precision	2% RSD at mid-point of range
Method Detection Limit (MDL)	0.5 µg/L

Figure 1. General flow diagram for In-Line Distillation of Phenol, USEPA/ISO



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_4	
DOWFAX 2A1	12626-49-2		328852
Kleenflow™ acidic			A001251
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 chemical(s) 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 USEPA Method 420.4

Method

1. 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.¹
2. 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

Interferences

1. Sulfide interferes 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.
2. 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.
3. 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.

4. 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.
5. Detergents in wastewater can affect negatively impact the distillation of phenol. For samples that contain detergents, the method should not be used to determine any concentration below 0.1 mg/L without verifying performance and/or adjusting cartridge plumbing as necessary.
6. Conditions or contaminants that suppress the boiling point of water (e.g. altitude >2000 meters) can negatively impact the distillation of phenol by co-distilling water. Adjust the distillation temperature and/or re-sample line + debubble pull as necessary to minimize the amount of water co-distilling with phenol.
7. Filter turbid samples prior to analysis.

Figure 2. In-Line Distillation of Phenol Calibration Series

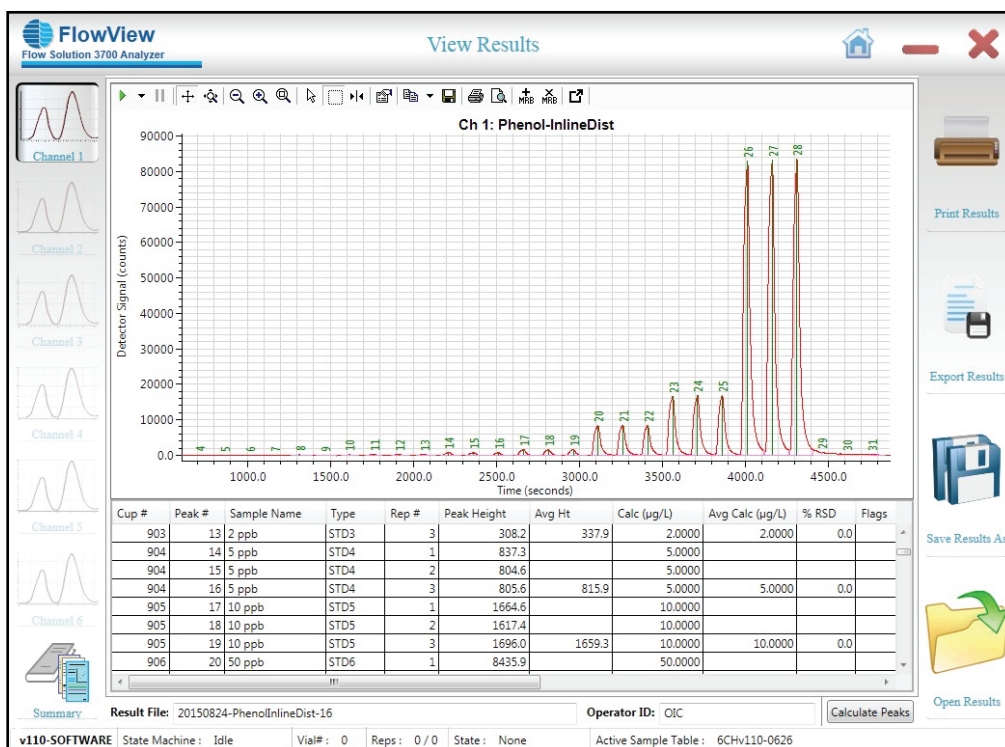
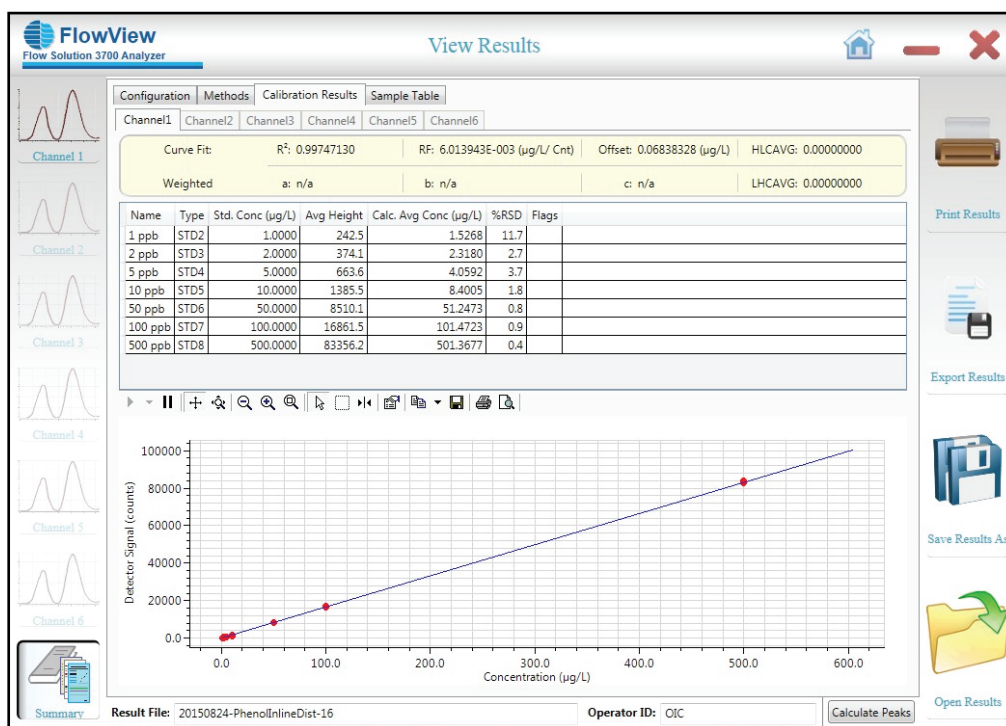


Figure 3. Calibration Curve and Statistics**Figure 4.** Consumables and Spare Parts for the In-Line Distillation of Phenol

Consumable	Part Number
Pump tubes kit - Phenol, in-line distillation	330364TK
Purple/White pump tubes (12 pack) - for cooling kit	A000360
KleenFlow Acidic	A002295
DOWFAX 2A1	328852
Sample Vials, Glass 8-mL (13 x 100 mm) (pack of 1000)	A000514
Tubing ASSY-Distillation head to cartridge	330795
Distillation head assembly	A000677*

Pump tubes should be replaced monthly, or on an as-needed basis to maintain system performance. The tubing connecting the distillation head to cartridge may need to be replaced weekly. Maximum life expectancy for pump tubes is approximately 800 hours. Refer to operating notes section for details regarding the use of surfactant and KleenFlow for maintaining system performance.

**The distillation head is not a consumable and does not need to be replaced regularly; however, it is recommended that a spare be kept on site.*



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