
Phenols Determination in Drinking Water and Industrial Wastes

Reference: APAT CNR IRSA n° 5070A1, EPA N° 9065 and ISO 6439:1990

Tested with **VELP Scientifica UDK 149 Distillation Unit** (code F30200140)



Introduction

Phenolic chemicals are of growing concern as water pollutants with their very low taste and odor thresholds, high persistence and toxicity. The compounds exist both in raw water and in treated water.

Although phenols can be quickly dispersed in the air, they may persist in water for a week or more.

Furthermore, phenols that remain in soil may be degraded by bacteria or other microorganisms.

In particular, chlorophenols are dangerous to human health even when they are present in water at low concentrations.

The determination of phenols is one of the routine analysis in order to assess the quality in drinking and ground water and in domestic and industrial wastes, even at low concentration (0.005-0.100 mg /l phenols).

The method described allows the phenols determination by steam distillation, followed by a colorimetric reaction with 4-aminoantipyrine.

Equipment

- Photometer for use at 460 nm
- 50 mm glass cuvette
- pH meter
- 500 ml volumetric flasks
- 500 or 1000 ml separatory funnels
- Filter papers

Chemicals

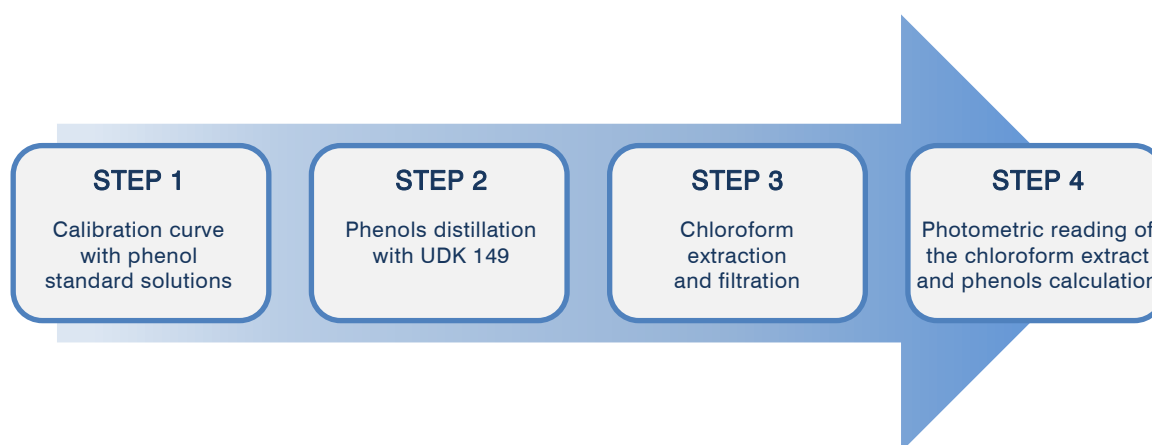
- Distilled water Type II, previously boiled and cooled
- Aminoantipyrine solution: dissolve 2 g of 4-aminoantipyrine in 100 ml of distilled water
- Potassium ferricyanide solution: dissolve 8 g of $K_3Fe(CN)_6$ in 100 ml of distilled water
- Ammonium chloride solution 2%: dissolve 20 g of NH_4Cl in 1000 ml of distilled water
- Ammonia 25 %
- Concentrated phenol solution (1000 mg/l): dissolve 250 mg of phenol in 250 ml of freshly boiled and cooled distilled water
- Diluted phenol solution (10 mg/l): dilute 2.5 ml of the concentrated phenol solution to 250 ml
- Chloroform, analytical grade

Sample

Phenols standard solution, 0.005 mg/l concentrated.

Procedure

The phenols quantification requires 4 steps:



STEP 1 - Calibration Curve

Using the diluted phenol solution (10 mg/l), create the calibration curve in the range 0.005-0.100 mg/l. 500 ml of different concentrations of standard solutions may be processed as in Step 3 and 4.

STEP 2 - Phenols Distillation

Condition the UDK 149 unit by performing a Wash down. Set a new method with the following parameters:

- H₂O (dilution water): none
- NaOH (32 %): none
- H₃BO₃: none
- Steam power: 50 %
- Distillation time: 8 min

Position the test tube containing 10 ml of sample into the unit UDK 149 and start the distillation. About 200 ml of distillate is collected into a 500 ml volumetric flask.

Blank: 10 ml of distilled water Type II.

Once accepted the blank average, the samples are distilled in series.

STEP 3 - Chloroform Extraction

Dilute the distillation solution to 500 ml using water type II and transfer the solution into a 1000 ml beaker.

Add 25 ml of ammonium chloride solution 2 % by using a pipette and adjust the pH to 10.0 ± 0.2 with some drops of ammonia 25 %. Transfer the solution into a 500 or 1000 ml separatory funnel and add 3.0 ml of Aminoantipyrine solution and 3.0 ml of potassium ferricyanide solution. Shake the separatory funnel and after 5 minutes add 25 ml of chloroform with a pipette. Shake vigorously for about 2 minutes and let the organic phase settle.

Filter chloroform extract through a filter paper and collect the filtrate in a 100 ml flask.

STEP 4 - Photometric Reading and Calculation

Transfer the filtrate solution in a 50 mm glass cuvette and read the extinction values at 460 nm, against distilled water.

The phenols concentration has been calculated by using the calibration curve equation:

$$y = 19.676 x + 0.22$$

Results

Phenols Standard Solution	y	x
	Extinction value (EXT)	Phenol concentration (mg/l)
Blank 1	0.278	
Blank 2	0.277	
Std 0.005 mg/L	0.312	0.005
Std 0.005 mg/L	0.316	0.005
Std 0.005 mg/L	0.310	0.005
Std 0.005 mg/L	0.310	0.005
Std 0.005 mg/L	0.317	0.005
Average ± SD		0.005 ± 0.000

In case of ground water analysis, drinking, surface and saline water and domestic and industrial wastes, interferences from sulfur compounds are eliminated by acidifying the sample to a pH < 4 with sulphuric or phosphoric acid.

Conclusion

The obtained results are reliable and in accordance with the expected values.

This makes the UDK 149 suitable for phenol quantification and is able to provide precise and reliable results.

The same procedure could be performed with VELP distillation units UDK 129, 139 and 159 after the disconnection of the titration vessel.