

## SIDE BY SIDE COMPARISON TABLE FOR HACH METHOD NUMBER 10266 COLORIMETRIC DETERMINATION OF PHENOL

TOPIC	EPA METHOD NUMBER 420.1 (1978) Direct Photometric Method 50 - 1000 µg phenol/L	HACH METHOD NUMBER 10266 (1 January 2015) 5 - 150 mg phenol/L
SCOPE AND APPLICATION	<ol style="list-style-type: none"> <li>1. The method is applicable to the analysis of drinking, surface and saline waters, domestic and industrial wastes.</li> <li>2. The method is capable of measuring phenolic materials at the 5 µg/L level when the colored end product is extracted and concentrated in a solvent phase using phenol as a standard.</li> <li>3. The method is capable of measuring phenolic materials that contain more than 50 µg/L in the aqueous phase (without solvent extraction) using phenol as a standard.</li> <li>4. It is not possible to use this method to differentiate between different kinds of phenols.</li> </ol>	<ol style="list-style-type: none"> <li>1. For wastewater, seawater, drinking water, surface water and process water. For the exhaust air (after absorption) and exhaust air condensates that form during the manufacture and processing of benzene, petroleum products, glass and mineral fibres, hardboard, coke, oil shale, hazardous waste, town gas, coal and brown coal products, tar, asphalt and bitumen.</li> <li>2. The method is capable of measuring phenolic materials from 5 to 150 mg phenol/L in the aqueous phase using phenol as a standard. The method detection limit is 0.124 mg phenol/L for the LR and 0.569 mg phenol/L.</li> <li>3. It is not possible to use this method to differentiate between different kinds of phenols.</li> </ol>
SUMMARY OF METHOD	Phenolic materials react with 4-aminoantipyrine in the presence of potassium ferricyanide at a pH of 10 to form a stable reddish-brown colored antipyrine dye. The amount of color produced is a function of the concentration of phenolic material which is measured at 460 or 510 nm.	When an oxidizing agent is in the sample, ortho- and meta-substituted phenols form colored complexes with 4-aminoantipyrine (AAP). The measurement wavelength is 510 nm.

<p><b>COMMENTS</b></p>	<p>1. For most samples a preliminary distillation is required to remove interfering materials.</p> <p>2. Color response of phenolic materials with 4-amino antipyrine 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.</p>	<p>Same</p>
<p><b>SAFETY</b></p>	<p>1. The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials.</p> <p>2. Each laboratory is responsible for maintaining a current awareness file of the Occupational Health and Safety Act (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data sheets (MSDS) should be made available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advisable.</p> <p>3. The following chemicals have the potential to be highly toxic or hazardous, for detailed explanation consult the MSDS.</p>	<p>Same</p>

<p><b>INTERFERENCES</b></p>	<ol style="list-style-type: none"> <li>1. Interferences from sulfur compounds are eliminated by acidifying the sample to a pH of less than 4 with H<sub>3</sub>PO<sub>4</sub> and aerating briefly by stirring and adding CuSO<sub>4</sub>.</li> <li>2. 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 low.</li> </ol>	<ol style="list-style-type: none"> <li>1. Larger quantities of cobalt, iron(III), chromium(III) and sulfide interfere (high-bias results).</li> <li>2. Higher volume percentages of water-soluble organic solvents interfere (high-bias results or low-bias results with different phenols types).</li> <li>3. High concentrations of strong oxidizing and reducing agents in the sample interfere with the reaction process. Remove strong oxidizing and reducing agents from the sample before analysis.</li> <li>3. Other substances that combine with 4-aminoantipyrine (e.g., naphthols and aromatic amines) are also analyzed, which results in a higher phenol concentration.</li> <li>4. Verify the measurement results with sample dilutions or standard additions.</li> </ol>
<p><b>APPARATUS</b></p>	<ol style="list-style-type: none"> <li>1. Distillation apparatus, all glass consisting of a 1 liter pyrex distilling apparatus with Graham condenser.</li> <li>2. pH meter.</li> <li>3. Spectrophotometer, for use at 460 or 510 nm.</li> <li>4. Funnels.</li> <li>5. Filter paper.</li> <li>6. Membrane filters.</li> <li>7. Separatory funnels, 500 or 1000 mL.</li> <li>8. Nessler tubes, short or long form.</li> </ol>	<ol style="list-style-type: none"> <li>1. Micro Dist distillation block or DRB200 reactor block with Micro Dist distillation tubes.</li> <li>2. Phenol TNTplus reagent set TNT 868.</li> <li>3. Pipets with tips.</li> <li>4. Spectrophotometer suitable for measurements at 510 nm with a 13-mm round vial sample cell.</li> </ol>
<p><b>REAGENTS AND STANDARD</b></p>	<p>Potassium ferricyanide  Phenol  Phosphoric acid  Sulfuric acid  Copper sulfate  Ferrous ammonium sulfate  Chloroform  Ammonium chloride  Ammonium hydroxide  4-aminoantipyrine</p>	<p>Phenol  4-aminoantipyrine  Potassium sodium tartrate  Ammonium chloride  Ammonium hydroxide  Sodium persulfate  EDTA  Sulfuric acid</p>

<p><b>SAMPLE COLLECTION PRESERVATION</b></p>	<p>Biological degradation is inhibited by the addition of 1 g/L of copper sulfate to the sample and acidification to a pH of less than 4 with phosphoric acid. The sample should be kept at 4°C and analyzed within 24 hours after collection.</p>	<ol style="list-style-type: none"> <li>1. Analyze the samples as soon as possible for best results.</li> <li>2. Collect samples in clean glass bottles.</li> <li>3. Rinse the sample bottle several times with the sample to be collected.</li> <li>4. Collect a sufficient quantity of sample to get a representative sample, for replicate analysis (if necessary) and for minimum waste disposal.</li> <li>5. At the time of collection, adjust the sample pH to 2 or less with sulfuric acid.</li> <li>6. If prompt analysis is not possible, keep the sample at or below 6 °C (43 °F) for a maximum of 28 days.</li> </ol>
<p><b>CALIBRATION AND STANDARDIZATION</b></p>	<p>2 - 500 µg phenol/L Before distillation standards should be adjusted to a pH of 4 with H<sub>2</sub>SO<sub>4</sub>.</p>	<p>5 - 40 mg phenol/L for the low range and 20 - 150 mg phenol/L for the high range. Before distillation standards should be adjusted to a pH of 4 with H<sub>2</sub>SO<sub>4</sub>.</p>
<p><b>PROCEDURE</b></p>	<p>Distillation procedure:</p> <ol style="list-style-type: none"> <li>1. Measure 500 mL sample into a beaker. Lower the pH to approximately 4 with 1 + 9 H<sub>3</sub>PO<sub>4</sub>, add 5 mL CuSO<sub>4</sub> solution and transfer to the distillation apparatus. Omit adding H<sub>2</sub>PO<sub>4</sub> and CuSO<sub>4</sub> if sample was preserved.</li> <li>2. Distill 450 mL of sample, stop the distillation, and when boiling ceases, add 50 mL of warm reagent water to the flask and resume distillation until 500 mL have been collected.</li> <li>3. If the distillate is turbid, filter through a prewashed membrane filter.</li> </ol>	<p>Distillation procedure:</p> <p>Distill all samples and standards with a Micro Dist distillation block. Refer to the Micro Dist documentation for the distillation procedure. Always distill the standards with the samples. Adjust the sample and the standard to approximately a pH of 4 with 1 M NaOH or 10% H<sub>2</sub>SO<sub>4</sub> before distillation.</p>

<p><b>DATA ANALYSIS AND CALCULATIONS</b></p>	<p>1. Prepare a standard curve by plotting the absorbance value of standards versus the corresponding phenol concentrations. 2. Obtain concentration value of sample directly from standard curve.</p>	<p>Same</p>
<p><b>Precision and Accuracy</b></p>	<p>1. Using the extraction procedure for concentration of color, six laboratories analyzed samples at concentrations of 9.6, 48.3, and 93.5 µg/L. Standard deviations were ±0.99, ±3.1 and ±4.2 µg/L, respectively. 2. Using the direct photometric procedure, six laboratories analyzed samples at concentrations of 4.7, 48.2 and 97.0 mg/L. Standard deviations were ±0.18, ±0.48 and ±1.58 mg/L, respectively.</p>	<p><u>Precision, Single Lab:</u> LR: x = 20.08 mg phenol/L SD = 0.13 mg phenol/L RSD = 0.63% HR: x = 98.55 mg phenol/L SD = 0.35 mg phenol/L RSD = 0.36%</p> <p><u>Method Detection Limit:</u> LR: 0.124 mg phenol/L SD = 0.04 mg phenol/L x = 5.07 mg phenol/L HR: 0.57 mg phenol/L SD = 0.18 mg phenol/L x = 19.3 mg phenol/L</p> <p><u>Spike Recoveries:</u> Industrial wastewater recovery spike duplicate 101.5% and 100.5% Domestic wastewater recovery spike duplicate 100.8% and 99.8% Produced wastewater recovery spike duplicate 100% and 101%</p>

**REFERENCES**

1. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p574-581, Method 510 through 510C, (1975).
2. Annual Book of ASTM Standards, Part 31, "Water", Standard D 1783-70, p553 (1976).
3. US EPA Methods for the Determination of Inorganic Substances in Environmental Samples, method **420.1** Revised August 1978.

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