PHENOL

C_6H_5OH MW: 94.11 CAS: 108-95-2 RTECS: SJ3325000

METHOD: 3502, Issue 2 **EVALUATION: FULL** **Issue 1: 15 February 1984**
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SYNONYMS: carbolic acid.

SAMPLING

SAMPLER: BUBBLER
(0.1 N sodium hydroxide)

FLOW RATE: 0.2 to 1 L/min

VOL-MIN: 26 L
-MAX: 240 L

SHIPMENT: hand delivery or special bubbler shipping cases

SAMPLE STABILITY: at least 5 days @ 25 °C [1]

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 9.5 to 38 mg/m^3 [1]
(100-L samples)

BIAS: -5.87% [1]

OVERALL PRECISION ($\hat{S}_{rT}$): 0.068 [1]

ACCURACY: ± 16.5%

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

ANALYTE: phenol

pH ADJUSTMENT: 0.1 mL conc. sulfuric acid; pH < 4

INJECTION VOLUME: 5 µL

TEMPERATURE-INJECTION: 215 °C
-DETECTOR: 225 °C
-COLUMN: 200 °C

CARRIER GAS: N_2 or He, 50 mL/min

COLUMN: stainless steel, 1.2 m x 6-mm OD, 35/60 mesh Tenax or equivalent

CALIBRATION: phenol in 0.1 N sodium hydroxide

RANGE: 0.5 to 6 mg [2]

ESTIMATED LOD: 10 µg per sample [3]

PRECISION ($\hat{S}_r$): 0.044 [1]

APPLICABILITY: The working range is 5 to 60 mg/m^3 for a 100-L air sample [4]. This method may be used for STEL measurements (15-L samples).

INTERFERENCES: None identified. Ethanol does not interfere.

OTHER METHODS: This is Method S330 in a revised format [2]; the method also appears in the phenol criteria document [4].

REAGENTS:

1. Phenol.*
2. Water, distilled.
3. Sulfuric acid, conc.
4. Calibration stock solution, 0.5 mg/mL. Dissolve 50 mg phenol (accurately weighed) in 0.1 N sodium hydroxide in a 100-mL volumetric flask. Dilute to 100 mL.
5. Collection medium, 0.1 N sodium hydroxide. Dissolve 4.0 g sodium hydroxide in distilled water. Dilute to 1 L.
6. Nitroge or Helium, purified.
7. Hydrogen, purified.
8. Air, filtered, compressed.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass midget bubbler.*
2. Personal sampling pump, 0.2 to 1 L/min, with splashover protection and flexible connecting tubing.
3. Gas chromatograph with glass-lined injection port, FID, integrator and column (page 3502-1).
4. Syringe, 10-µL, readable to 0.1 µL.
5. Volumetric flasks, 25- to 100-mL.*
6. Pipets, 0.1-, 0.5-, 1-, 2-, 4-, 8-, and 15-mL, graduated in 0.1-mL increments, with pipet bulb.*
7. pH paper.
8. Glass wool.

*Wash with detergent; rinse thoroughly with distilled water.

SPECIAL PRECAUTIONS: Phenol is a severe poison and is corrosive [3]. Use protective equipment when handling. All work should be performed in a hood.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Pipette 15 mL 0.1 N sodium hydroxide into each midget bubbler.
3. Connect a splashover tube between the midget bubbler and the personal sampling pump using short pieces of flexible tubing.
4. Sample at 0.2 to 1 L/min for a total sample size of 26 to 240 L.
5. After sampling, remove and tap the bubbler stem gently against the inside wall of the bubbler bottle. Rinse the stem with 1 mL distilled water, adding the wash to the bubbler. Seal the bubbler with a hard, non-reactive stopper (PTFE or glass, not rubber) to prevent leakage during shipping.

SAMPLE PREPARATION:

6. Transfer the solution from the bubbler to a 25-mL volumetric flask.
7. Rinse the bubbler twice with 1 mL distilled water and add the rinses to the flask.
8. Add 0.1 mL conc. H_2SO_4 to the flask and mix. Use pH paper to make sure that the pH is less than 4.
9. Dilute to the mark with distilled water and mix.

CALIBRATION AND QUALITY CONTROL:

10. Calibrate daily with at least five working standards covering the range of the samples:
    a. Pipet, e.g., 0, 0.5, 1.0, 2.0, 4.0, 8.0 and 15 mL calibration stock solution into 25-mL volumetric flasks. Add the appropriate amount of 0.1 N sodium hydroxide with a graduated pipette to reach a 15-mL volume for each of the working standards.
    b. Add 0.1 mL conc. H_2SO_4 to each standard. Make up to 25 mL with distilled water.
    c. Check the solution with pH paper to make sure that its pH is less than 4.

d. Analyze the working standards and blanks under the same GC conditions and during same time period as samples.
e. Prepare a calibration graph by plotting peak area vs. µg phenol in the 25 mL volume.

MEASUREMENT:

11. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 3502-1.
12. Inject a sample aliquot using solvent flush technique. Make duplicate injections of samples and standards. Measure peak area.
13. Clean the glass inlet on the GC at the end of each day with water and acetone rinses. Reinsert the glass inlet into the injection port and let it bake out overnight.

CALCULATIONS:

14. Read the mass (µg) of phenol found in the sample bubbler (W) and average blank bubblers (B) corresponding to each peak area from the calibration graph.
15. Calculate concentration, C (mg/m ^{3}), of phenol in the air volume sampled, V (L):

$$C = \frac{(W - B)}{V}, \ mg/m^3.$$

EVALUATION OF METHOD:

Method S357 [2] was issued on August 1, 1975, and validated over the range 9.46 to 37.8 mg/m ^{3} at 22 °C and 760 mm Hg, using a 100-L sample [1]. Overall precision, $\hat{S}_{rT}$, was 0.068, determined by sampling and analyzing generated atmospheres containing 9.46, 18.9 and 37.8 mg/m ^{3} phenol in air. The concentrations were determined from the delivery rate of the syringe drive pump and the flow rates of the dilution air (20.1 L/min). The collection efficiency of the bubbler was determined at 37.8 mg/m ^{3} and found to be 1.00 $\pm$ 0.01. No concentration changes occurred after five days storage of six samples of 15 mL 0.1 N sodium hydroxide in 25-mL volumetric flasks spiked with 1.9 mg phenol. Recovery at the OSHA standard level was 97.4%; this was not considered to be a significant bias.

REFERENCES:

[1] Documentation of the NIOSH Validation Tests, S330, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977).
[2] NIOSH Manual of Analytical Methods, 2nd. ed., V. 3, S330, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).
[3] UBTL Memorandum, Analytical Laboratory Report for Phenol, Sequence #2660-N and 2340-J (December 8, 1980 and June 25, 1980).
[4] Criteria for a Recommended Standard...Occupational Exposure to Phenol, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 76-196 (1976).

METHOD REVISED BY: Julie R. Okenfuss, NIOSH/DPSE; S357 originally validated under NIOSH