RESORCINOL 5701

C₆H₄(OH)₂  MW: 110.1  CAS: 108-46-3  RTECS: VG9625000


OSHA: no PEL  NIOSH: 10 ppm  ACGIH: 10 ppm
(1 ppm = 4.49 mg/m³ @ NTP)

PROPERTIES: white crystals, hygroscopic; d 1.272 g/mL @ 20 °C; BP 280 °C; MP 111 °C; VP 2.66 x 10⁻⁵ kPa (2 x 10⁻⁴ mm Hg) @ 25 °C, vapor density 3.79 (air=1)

SYNONYMS: 1,3-benzenediol, m-benzenediol, 1,3-dihydroxybenzene, m-dihydroxybenzene, 3-hydroxyphenol, m-hydroxyphenol

SAMPLING

SAMPLER: FILTER / SOLID SORBENT TUBE
OVS tube: glass fiber filter, XAD-7
FLOW RATE: 0.2 to 1 L/min
VOL-MIN: 5 L
VOL-MAX: 160 L
SHIPEMENT: routine; protect from exposure to light
SAMPLE STABILITY: 30 days at 5 °C (protect from light)
BLANKS: 2 to 10 field blanks per set

TECHNIQUE: GAS CHROMATOGRAPHY, FID
ANALYTE: resorcinol
DESORPTION: 2 mL methanol
INJECTION VOLUME: 1 µL
TEMPERATURE-INJECTION: 250 °C
-DETECTOR: 300 °C
-COLUMN: 80 °C - 230 °C (12 °C/min)
CARRIER GAS: Helium, 2.4 mL/min
COLUMN: capillary, 30 m x 0.28-mm ID; 0.25-µm film
100% dimethyl polysiloxane Mtx-1™ or equivalent [1]
CALIBRATION: solutions of resorcinol in methanol
RANGE: 12 to 5960 µg/sample [LOQ determined from XAD-7 solid sorbent DE recovery]
ESTIMATED LOD: 2 µg/sample
PRECISION (σr): 0.032 [1]

RANGE STUDIED: not determined
BIAS: not determined
OVERALL PRECISION (σr): not determined
ACCURACY: not determined

APPLICATION: The working range is 0.04 to 22.12 ppm (0.2 mg/m³ to 99.3 mg/m³) for a 60-L air sample. Under the GC parameters given in the method, resorcinol can be identified based upon retention time and quantified.

INTERFERENCES: No specific interferences were identified. However, any compound with a similar retention time may interfere.

OTHER METHODS: This method is based on the OSHA stopgap method for resorcinol [2].

NIOSH Manual of Analytical Methods (NMAM), Fourth Edition
REAGENTS:

1. Resorcinol, reagent grade.*
2. Methanol, chromatographic grade. *
3. Helium, purified.
5. Air, filtered.
6. Calibration stock: Dissolve mg amount of resorcinol in 10 mL of solvent. Prepare additional standards by serial dilution.

* See SPECIAL PRECAUTIONS

EQUIPMENT:

1. Sampler: XAD-7 OVS tube, 13-mm OD, containing two sections of XAD-7 (200 mg front section/100 mg back section) separated by polyurethane foam plug. A glass fiber filter precedes the front section and is held in place with a Teflon® ring. A polyurethane foam plug follows the back section. Tubes are commercially available (SKC # 226-57).
2. Personal sampling pump, 0.2 to 1.0 mL/min, with flexible connecting tubing.
3. Gas chromatograph, flame ionization detection, integrator, and Mtx-1 capillary column (page 5701-1).
4. Ultrasonic bath.
5. Vials, autosampler, with PTFE-lined caps.
6. Vials, 4-mL, with screw caps.
7. Microliter syringes, 10-µL and other sizes as needed, readable to 0.1 µL.
8. Flasks, volumetric, various sizes.
9. Pipets, various sizes.

SPECIAL PRECAUTIONS: Resorcinol is a toxic irritant and is also air and light sensitive. Methanol is flammable and a dangerous fire risk. Wear appropriate protective clothing and work with these compounds in a well ventilated hood.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Remove front and rear caps from each tube immediately before sampling. Attach sampler to personal sampling pump with flexible tubing.
3. Sample at an accurately known flow rate between 0.2 and 1.0 L/min for a total sample size of 5 to 160 L.
4. Cap the samplers and pack securely for shipment. Protect samplers from exposure to light.

SAMPLE PREPARATION:

5. Place front sorbent section and glass fiber filter in a 4-mL screw cap vial. Place backup sorbent section in a separate vial. Discard foam plugs.
6. Add 2 mL of methanol to each vial and cap.
7. Place vials in an ultrasonic bath for 30 min to aid desorption.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards over the range of interest. Three standards (in duplicate) should cover the range from LOD to LOQ.
   a. Add known amounts of calibration stock solution to methanol in 10-mL volumetric flasks and dilute
to the mark.
b. Analyze together with samples and blanks (steps 11 and 12).
c. Prepare calibration graph (peak area or height vs. µg resorcinol).

9. Determine desorption efficiency (DE) at least once for each lot of OVS tubes used for sampling in the calibration range (step 8).
   a. Prepare three samplers at each of six levels plus three media blanks.
   b. Raise the Teflon holding ring to prevent wicking, and inject a known amount of calibration stock solution directly onto the front sorbent bed of each OVS tube.
   c. Allow the tubes to air equilibrate for several minutes, then cap the ends of the tubes and allow to stand overnight.
   d. Desorb the samples (steps 5 through 7) and analyze together with standards and blanks (steps 11 and 12).
   e. Prepare a graph of DE vs. µg analyte recovered.

10. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration and DE graphs are in control.

MEASUREMENT:

11. Set gas chromatograph according to manufacturer’s recommendations and to conditions given on page 5701-1. Inject a 1-µL sample aliquot manually using solvent flush technique or with an autosampler.  
    NOTE: If peak area is above the linear range of the working standards, dilute with methanol, reanalyze and apply the appropriate dilution factor in the calculations.

12. Measure peak areas.

CALCULATIONS:

13. Determine the mass, µg (corrected for DE), for resorcinol found in the sample front (W_f) and back (W_b) sorbent sections, and in the average media blank front (B_f) and back (B_b) sorbent sections.
    NOTE: If W_b > W_f/10, report breakthrough and possible sample loss.

14. Calculate concentration, C, of resorcinol in the air volume sampled, V (L):

\[
C = \frac{W_f + W_b - B_f - B_b}{V}, \text{ mg/m}^3
\]

    NOTE: µg/mL = mg/m³

EVALUATION OF METHOD:

This work extended the range of the OSHA stopgap method for recorcinol [2] to lower concentration levels.  The method was evaluated for resorcinol over the range of 0.040 to 5.96 mg per sample.  Table 1 shows the desorption efficiencies for each of the sampler components, glass fiber filter (alone) and XAD-7 sorbent, as well as the complete OVS tube.  The 40 µg, 80 µg, 120 µg, and 200 µg levels were evaluated by NIOSH [1].  Higher concentration levels (298 to 5860 µg) were evaluated by OSHA [2].  The OSHA study tested retention efficiency by spiking filters with 5.96 mg resorcinol and pulling humidified air through the tubes.  The mean recovery from these samples was 99.7%, with no resorcinol found on the back up section.  When stored at ambient temperature, resorcinol samples spiked at 120 µg were stable on the XAD-7 sorbent for 28 days,
having a mean recovery of 97.5%. However, refrigerated storage is recommended.

REFERENCES:


METHOD WRITTEN BY:

Stephanie M. Pendergrass, DPSE/NIOSH

TABLE 1. RECORCINOL RECOVERY DATA

<table>
<thead>
<tr>
<th>Spike Level (µg)</th>
<th>Glass Fiber Filter 13-mm</th>
<th>XAD-7 Sorbent</th>
<th>OVS-7 Tube</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>n</td>
<td>% Rec</td>
<td>S_r</td>
</tr>
<tr>
<td>40</td>
<td>6</td>
<td>48.4</td>
<td>0.035</td>
</tr>
<tr>
<td>80</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>120</td>
<td>6</td>
<td>72.9</td>
<td>0.067</td>
</tr>
<tr>
<td>200</td>
<td>6</td>
<td>85.6</td>
<td>0.029</td>
</tr>
<tr>
<td>S_r</td>
<td></td>
<td>0.047</td>
<td></td>
</tr>
</tbody>
</table>