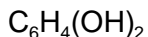


RESORCINOL

5701



MW: 110.1

CAS: 108-46-3

RTECS: VG9625000

METHOD: 5701, Issue 1

EVALUATION: PARTIAL

Issue 1: 15 January 1998

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		MEASUREMENT	
SAMPLER:	FILTER / SOLID SORBENT TUBE OVS tube: glass fiber filter, XAD-7	TECHNIQUE:	GAS CHROMATOGRAPHY, FID
FLOW RATE:	0.2 to 1 L/min	ANALYTE:	resorcinol
VOL-MIN:	5 L	DESORPTION:	2 mL methanol
-MAX:	160 L	INJECTION VOLUME:	1 µL
SHIPMENT:	routine; protect from exposure to light	TEMPERATURE-INJECTION:	250°C
SAMPLE STABILITY:	30 days at 5 °C (protect from light)	-DETECTOR:	300°C
BLANKS:	2 to 10 field blanks per set	-COLUMN:	80° -230°C (12°C/min)
ACCURACY		CARRIER GAS:	Helium, 2.4 mL/min
RANGE STUDIED:	not determined	COLUMN:	capillary, 30 m x 0.28-mm ID; 0.25-µm film 100% dimethyl polysiloxane Mtx-1™ or equivalent [1]
BIAS:	not determined	CALIBRATION:	solutions of resorcinol in methanol
OVERALL PRECISION (\hat{S}_{rr}):	not determined	RANGE:	12 to 5960 µg/sample [LOQ determined from XAD-7 solid sorbent DE recovery]
ACCURACY:	not determined	ESTIMATED LOD:	2 µg/sample
		PRECISION (\hat{S}_r):	0.032 [1]

APPLICABILITY: 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].

REAGENTS:

1. Resorcinol, reagent grade.*
2. Methanol, chromatographic grade.*
3. Helium, purified.
4. Hydrogen, prepurified.
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- μ L and other sizes as needed, readable to 0.1 μ L.
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: $\mu\text{g/mL} = \text{mg/m}^3$

EVALUATION OF METHOD:

This work extended the range of the OSHA stopgap method for resorcinol [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:

- [1] Pendergrass SM [1997]. Backup data report for Resorcinol Method Development. Cincinnati, OH: National Institute for Occupational Safety and Health, MRSB (NIOSH) (unpublished May).
 [2] Eide, ME [1994]. OSHA Stopgap Method for Resorcinol, Salt Lake City, UT: OSHA Technical Center, Organic Service Branch I (March, 1994).

METHOD WRITTEN BY:

Stephanie M. Pendergrass, DPSE/NIOSH

TABLE 1. RESORCINOL RECOVERY DATA

Spike Level (μg)	Glass Fiber Filter 13-mm			XAD-7 Sorbent			OVS-7 Tube		
	n	% Rec	S_r	n	% Rec	S_r	n	% Rec	S_r
40	6	48.4	0.035	6	100.4	0.041	6	82.9	0.023
80	--	--	--	--	--	--	6	83.9	0.030
120	6	72.9	0.067	6	92.2	0.027	6	93.4	0.028
200	6	85.6	0.029	6	96.3	0.036	6	96.3	0.042
\bar{S}_r			0.047			0.035			0.032