

# MONOMETHYLANILINE

3511



MW: 107.16

CAS: 100-61-8

RTECS: BY4550000

METHOD: 3511, Issue 1

EVALUATION: PARTIAL

Issue 1: 15 August 1994

**OSHA :** 2 ppm (skin)  
**NIOSH:** 0.5 ppm (skin)  
**ACGIH:** 0.5 ppm (skin)  
 (1 ppm = 4.38 mg/m<sup>3</sup> @ NTP)

**PROPERTIES:** liquid, discolors on standing; MP - 57 °C;  
 BP 194 - 196 °C; d = 0.989 g/mL @ 20 °C;  
 VP 40 Pa (0.3 mm Hg) @ 20 °C

**SYNONYMS:** N-methylaniline; N-methylbenzenamine

SAMPLING		MEASUREMENT	
<b>SAMPLER:</b>	MIDGET BUBBLER (0.05 M sulfuric acid, 10 mL)	<b>TECHNIQUE:</b>	GAS CHROMATOGRAPHY, FID
<b>FLOW RATE:</b>	0.2 to 1.0 L/min	<b>ANALYTE:</b>	monomethylaniline
<b>VOL-MIN:</b>	11 L @ 2 ppm	<b>EXTRACTION:</b>	neutralize with 1 mL 4M NaOH
<b>-MAX:</b>	100 L	<b>INJECTION VOLUME:</b>	5 µL
<b>SHIPMENT:</b>	take care to prevent spillage	<b>CARRIER GAS:</b>	N <sub>2</sub> , 50 mL/min
<b>SAMPLE STABILITY:</b>	at least 7 days @ 25 °C	<b>TEMPERATURE-INJECTOR:</b>	245 °C
<b>BLANKS:</b>	2 to 10 field blanks per set	<b>-DETECTOR:</b>	275 °C
<b>BULK SAMPLE:</b>	required	<b>-COLUMN:</b>	170 °C
<b>ACCURACY</b>		<b>COLUMN:</b>	60/80 mesh Chromosorb 103, 2ft x 1/8"-O.D., with an Ascarite precolumn
<b>RANGE STUDIED:</b>	4.38 to 17.52 mg/m <sup>3</sup> [1] (100-L samples)	<b>CALIBRATION:</b>	standard solutions of monomethylaniline in sulfuric acid
<b>BIAS:</b>	none [1]	<b>RANGE:</b>	0.1 to 3 mg per sample [1]
<b>OVERALL PRECISION (<math>\hat{S}_{rT}</math>):</b>	0.089 [1]	<b>ESTIMATED LOD:</b>	8 µg per sample
<b>ACCURACY:</b>	± 26.38%	<b>PRECISION (<math>\hat{S}_p</math>):</b>	0.024 [1]

**APPLICABILITY:** The working range is 0.2 to 7 ppm (1 to 30 mg/m<sup>3</sup>) for a 100-L air sample. Using a nitrogen-specific detector instead of a FID will greatly increase sensitivity. This alternate detector has been used for amines with a 30-m x 0.25- mm x 0.25-µm film DB-5 (5% methyl, phenyl-50% dimethyl-polysiloxane) fused-silica capillary column with a liner packed with KOH-coated glass wool.

**INTERFERENCES:** None known.

**OTHER METHODS:** This revises Method S153 [2].

**REAGENTS:**

1. Monomethylaniline <sup>\*</sup>, ACS reagent grade.
2. Sulfuric acid, 0.05 M, ACS reagent grade.
3. Sodium hydroxide,<sup>\*</sup> 4 M.
4. Calibration stock solution.

\* See Special Precautions

**EQUIPMENT:**

1. Sampler: Midget bubbler containing 10 mL 0.05 M sulfuric acid.
2. Personal sampling pump, 0.2 to 1 L/min, with flexible polyethylene or PTFE tubing.
3. pH paper or meter
4. Vials, glass, 20-mL with PTFE-lined caps.
5. Gas chromatograph with a flame ionization detector, recorder, integrator and column (page 3511-1).
6. Graduated cylinders, 10-mL
7. Syringes, 10- and 500- $\mu$ L.
8. Volumetric flasks, 10-mL.
9. Pipets, 1- and 10-mL glass, delivery, with pipet bulb.

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**SPECIAL PRECAUTIONS:** Monomethylaniline is toxic by inhalation, ingestion, and skin absorption [3,4]. Sodium hydroxide is corrosive to tissue. Wear gloves and work in a hood.

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**SAMPLING:**

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Attach outlet of impinger to the sampling pump. Place an empty bubbler or impinger in line behind the sampler to catch any splashover during sampling.
3. Sample 11 to 100 L of air at an accurately known flow rate in the range 0.2 to 1.0 L/min.
4. The temperature and pressure of the atmosphere being sampled should be recorded.
5. Transfer samples to 20 mL vial for shipping. Place in a suitable shipping container to prevent damage or spilling during transit.
6. Collect a bulk sample (ca. 1 g) in a glass vial and ship it separately.

**SAMPLE PREPARATION:**

7. Bring the volume of each sample to 10 mL with 0.05 M sulfuric acid.
8. Add 1 mL 4 M sodium hydroxide and mix thoroughly. The pH of the resulting solution should be greater than 10.
9. Analyze sample immediately to avoid loss of amine as the free base.

**CALIBRATION AND QUALITY CONTROL:**

10. Prepare at least six working standards (0 to 300  $\mu$ g/ mL of monomethylaniline).
  - a. Add appropriate aliquots of calibration stock solution to 0.05 M sulfuric acid. Follow neutralization procedure as in step 8.
  - b. Analyze working standards together with samples and blanks (steps 13 through 15).
  - c. Prepare a calibration graph of area vs. amount of monomethylaniline,  $\mu$ g per mL.
11. Determine recovery for each batch of sulfuric acid made up in the concentration range of interest. Prepare four 10 mL portions of sulfuric acid at each of five monomethylaniline levels plus three media blanks.
  - a. Put a 10 mL portion of sulfuric acid in a 20-mL vial.
  - b. Spike aliquot of calibration solution into 10 mL of 0.05 M sulfuric acid with a microliter syringe.
  - c. Cap and let stand overnight.

- d. Neutralize following step 8 and analyze along with working standards and blanks (steps 13 through 15).
- e. Prepare graph of recovery vs.  $\mu\text{g}$  monomethylaniline.
12. Analyze three quality control spikes and three analyst spikes to ensure that calibration graph and recovery graph are in control.

**MEASUREMENT:**

13. Set gas chromatograph according to manufacturer's recommendations and to conditions given page 3511-1.
14. Inject 5- $\mu\text{L}$  sample aliquot.  
NOTE: If peak area is above the linear range of the working standards, dilute, reanalyze, and apply appropriate dilution factor in calculations.
15. Measure peak area.

**CALCULATIONS:**

16. Determine mass,  $\mu\text{g}$  (corrected for recovery), of monomethylaniline (W) found in the sample and the average media blank.
17. Calculate concentration of monomethylaniline in the air volume sampled V (L):

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

**EVALUATION OF METHOD:**

This method was evaluated over the range 4.38 to 17.52  $\text{mg/m}^3$  using 100-L samples [1,2]. Overall sampling and measurement precision,  $\hat{S}_{rT}$ , was 0.089, with essentially 100% recovery, representing a non-significant bias. Sample stability during storage was evaluated at 791  $\mu\text{g}$  n-methylaniline per sample. Samples showed 98.3% recovery after seven days of storage.

**REFERENCES:**

- [1] Backup Data Report for n-methylaniline, Documentation of NIOSH Validation Tests. U.S. Department of Health, Education, and Welfare, (NIOSH) Publication 77-185 (April, 1977).
- [2] NIOSH Manual of Analytical Methods, 2nd. ed., V. 3, S153, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-175 (1978).
- [3] Hawley's Condensed Chemical Dictionary, 11th ed. Sax, N.I. and Lewis, R.J., Eds. Van Nostrand Reinhold Co., New York, NY (1987).
- [4] NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards, US DHHS (NIOSH) Publ. 81-123 (1981).

**METHOD REVISED BY:**

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