N-METHYL-2-PYRROLIDINONE 1302

C_5H_9NO MW: 99.13 CAS: 872-50-4 RTECS: UY5790000

METHOD: 1302, Issue 1 **EVALUATION: PARTIAL** **Issue 1: 15 January 1998**

OSHA: None
NIOSH: None
ACGIH: None
(1 ppm = 4.05 mg/m^3 @ NTP)

PROPERTIES: Clear liquid, amine-like odor; d = 1.027 g/mL@ 25 °C; BP = 202 °C @ 760 mm Hg; FP = -24.4 °C; VP = 0.29 mm Hg (0.039 kPa) @ 20 °C; explosive limits = 1.3% to 9.5% (v/v).

SYNONYMS: 1-methyl-2-pyrrolidone, *N*-methyl-γ-pyrrolidone, *N*-methyl-γ-butyrolactone, NMP, 1-methylazacyclopentan-2-one, MP, M-Pyrol

SAMPLING

SAMPLER: SOLID SORBENT TUBE (Coconut shell charcoal, 100/50 mg)

FLOW RATE: 0.05 to 0.2 L/min

VOL-MIN: 0.5 L
-MAX: 125 L

SHIPMENT: Keep cold. Protect from prolonged exposure to light.

SAMPLE STABILITY: 14 days at 5 °C [1]

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: not determined

BIAS: not determined

OVERALL PRECISION ($\hat{S}_{rT}$): not determined

ACCURACY: not determined

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, NPD or FID

ANALYTE: *N*-methyl-2-pyrrolidinone (NMP)

DESORPTION: 1 mL methylene chloride/methanol (95:5)

INJECTION VOLUME: 1 µL

TEMPERATURE-INJECTION: 250 °C
-DETECTOR: 300 °C
-COLUMN: 60 to 200 °C (10 °C/min)

CARRIER GAS: Helium, 2.4 mL/min

COLUMN: amine capillary, 30 m, 0.32-mm ID, 1-µm film, crossbonded® 5% diphenyl-95% dimethyl polysiloxane

CALIBRATION: solutions of NMP in solvent

RANGE: 0.063 to 25.8 µg/sample (NPD) [1]
0.662 to 2066 µg/sample (FID) [1]

ESTIMATED LOD: 0.02 µg/sample (NPD) [1]
0.3 µg/sample (FID) [1]

PRECISION ($\bar{S}_r$): 0.05 (NPD) [1]
0.01 (FID) [1]

APPLICABILITY: Under the GC parameters given in the method *N*-methyl-2-pyrrolidinone 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 represents an improvement over an OSHA In-House method for *N*-methyl-2-pyrrolidone [2]. Method 1302 employs a shorter capillary column, improved sensitivity and recovery at lower sample levels, and a choice of two detection systems.

REAGENTS:

1. *N*-methyl-2-pyrrolidinone, reagent grade.*
2. Methanol, chromatographic grade. *
3. Methylene chloride, chromatographic grade*
4. Helium, purified.
5. Hydrogen, prepurified.
6. Air, filtered.
7. Desorption Solvent. 5% methanol in 95% methylene chloride.
8. Calibration Stock Solution. Add *N*-methyl-2-pyrrolidinone to desorption solvent in a 10-mL volumetric flask. Protect from light.

* See SPECIAL PRECAUTIONS

EQUIPMENT:

1. Sampler: Glass tube, 70 mm, 6-mm OD, containing two sections of coconut shell charcoal (100 mg front, 50 mg back section) separated by polyurethane foam plug. A glass wool plug precedes the front section and a polyurethane foam plug follows the back section. Tubes are commercially available (SKC 226-01, Supelco ORBO-32).
2. Personal sampling pump, 0.05 to 2 mL/min, with flexible connecting tubing.
3. Gas chromatograph, nitrogen phosphorous detector and/or flame ionization detector, integrator, and amine capillary column, Restek Rtx-5 or equivalent (page 1302 -1).
4. Vials, autosampler, with PTFE-lined caps.
5. Microliter syringes, 10-µL and other sizes as needed, readable to 0.1 µL.
6. Flasks, volumetric, various sizes.
7. Pipets, various sizes.

SPECIAL PRECAUTIONS: *N*-methyl-2-pyrrolidinone is an irritant with possible teratogenic properties. Methanol is flammable and a dangerous fire risk. Methylene chloride is a potential occupational carcinogen. 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. Break ends of tubes immediately before sampling. Attach sampler to personal sampling pump with flexible tubing.
3. Sample at an accurately known flow rate of 0.05 to 0.2 L/min for a total sample size of 0.5 to 125 L.
4. Cap the samplers and pack securely for shipment. Protect samplers from prolonged exposure to light.

SAMPLE PREPARATION:

5. Place front sorbent section including glass wool plug and back sorbent section in separate autosampler vials. Discard foam plugs.
6. Add 1 mL desorption solvent to each vial and cap.
7. Let each vial stand with occasional agitation for 30 min to aid desorption.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards over the range from below the LOD to 10 times the LOQ. The calibration graph may be extended if sample concentrations dictate.
   a. Add known amounts of calibration stock solution to solvent in 10-mL volumetric flasks and dilute to the mark. Prepare additional standards by serial dilution. Prepare fresh daily.
   b. Analyze together with samples and blanks (steps 11 and 12).
   c. Prepare calibration graph (peak area or height vs. µg *N*-methyl-2-pyrrolidinone).
9. Determine desorption efficiency (DE) at least once for each lot of charcoal tubes used for sampling in the calibration range (step 8).
   a. Prepare three samplers at each of six levels plus three media blanks.
   b. Remove the back section of the charcoal tubes. Inject a known amount of calibration stock

solution directly onto the front sorbent bed of each charcoal 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 (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 1302-1. 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 desorption solvent, reanalyze, and apply the appropriate dilution factor in the calculations.
12. Measure peak areas.

CALCULATIONS:

13. Determine the mass, µg (corrected for DE), for *N*-methyl-2-pyrrolidinone 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 *N*-methyl-2-pyrrolidinone 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/m^3

EVALUATION OF METHOD:

The method was evaluated for *N*-methyl-2-pyrrolidinone using GC-NPD and GC-FID over a range of 0.662 to 2066 µg/sample. Precision was determined to be 0.05 using GC-NPD and 0.01 using GC-FID. For GC-NPD, a DE graph was developed over a range of 0.310 µg to 10.8 µg. GC-FID was used to verify the results. The mean recovery was determined to be 98.8% with a pooled relative standard deviation ($\bar{S}_r$) of 0.051. An extended DE graph of the upper level of the method (0.103 to 2.07 mg) was previously determined by OSHA [2]. When spiked onto charcoal tubes at the 10.8 µg level and stored at ambient temperature, N-methyl-2- pyrrolidinone was stable on the charcoal tube for 7 days. OSHA reported that *N*-methyl-2-pyrrolidinone, when spiked at the 2070 µg level, was stable for 15 days at ambient temperature [2]. Refrigerated storage is recommended.

REFERENCES:

[1] Pendergrass, SM [1997]. Backup data report for N-methyl-2-pyrrolidinone (unpublished). Cincinnati, OH: National Institute for Occupational Safety and Health.
[2] Eide M [1991]. OSHA Stopgap Method for N-methyl-2-pyrrolidinone. Salt Lake City, UT: OSHA Salt Lake City Technical Center.

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