

# METHYL ETHYL KETONE

2500



MW: 72.11

CAS: 78-93-3

RTECS: EL6475000

METHOD: 2500, Issue 2

EVALUATION: FULL

Issue 1: 15 February 1984

Issue 2: Rev., 15 May 1996

OSHA : 200 ppm  
 NIOSH: 200 ppm: STEL 300 ppm  
 ACGIH: 200 ppm: STEL 300 ppm  
 (1 ppm = mg/m<sup>3</sup>)

PROPERTIES: liquid; d 0.805 g/mL @ 20 °C; BP 79.6 °C;  
 MP -86 °C; VP 13 kPa (100 mm Hg; 13%  
 v/v) @ 25 °C; explosive range 2 to 10%  
 v/v in air

SYNONYMS: 2-butanone; MEK

SAMPLING		MEASUREMENT	
<b>SAMPLER:</b>	SOLID SORBENT TUBE (beaded carbon, ~150 mg/ ~75 mg)	<b>TECHNIQUE:</b>	GAS CHROMATOGRAPHY, FID
<b>FLOW RATE:</b>	0.01 to 0.2 L/min	<b>ANALYTE:</b>	methyl ethyl ketone (MEK)
<b>VOL-MIN:</b>	0.25 L @ 200 ppm	<b>DESORPTION:</b>	1 mL CS <sub>2</sub> ; stand 30 min
<b>-MAX:</b>	12 L	<b>INJECTION VOLUME:</b>	5 µL
<b>SHIPMENT:</b>	routine	<b>TEMPERATURE-INJECTION:</b>	250 °C
<b>SAMPLE STABILITY:</b>	at least 90 days @ -5 °C [1]	<b>-DETECTOR:</b>	300 °C
<b>BLANKS:</b>	2 to 10 field blanks per set	<b>-COLUMN:</b>	55 to 75 °C
<b>ACCURACY</b>		<b>CARRIER GAS:</b>	N <sub>2</sub> or He, 25mL/min
<b>RANGE STUDIED:</b>	295 to 1180 mg/m <sup>3</sup>	<b>COLUMN:</b>	glass or stainless steel, 4m x 2-mm ID; 20% SP-2100/0.1% Carowax 1500 on Supelcoport 100/120
<b>BIAS:</b>	6.21%	<b>CALIBRATION:</b>	MEK solutions in CS <sub>2</sub>
<b>OVERALL PRECISION (Ŝ<sub>r</sub>):</b>	0.069 [2]	<b>RANGE:</b>	0.15 TO 5 mg per sample
<b>ACCURACY:</b>	± 17.83%	<b>ESTIMATED LOD:</b>	4 µg per sample [2,3]
		<b>PRECISION (Ŝ<sub>s</sub>):</b>	0.04 [1]

**APPLICABILITY:** The working range is 17 to 560 ppm (50 to 1650 mg/m<sup>3</sup>) for a 3-L air sample. The method is applicable to 15-min sample. This method was developed to give improved sample stability compared to conventional charcoal tubes [3,4]. Side-by-side comparisons of this method and Method S3 were made in sporting goods manufacturing plant in which MIBK, THF, AND toluene were also present. This method has also been used successfully for methyl isobutyl ketone [1] and acetone [5].

**INTERFERENCES:** Under the given conditions, acetone and isopropanol have retention times similar to MEK. Mass spectrometry and other GC column, e.g., SP-1000, or 30 mx 0.32-mm WCOT capillary coated with 1 µm DB-1, are aids to resolving interferences.

**OTHER METHODS:** This method is similar, except for the sampler, to Methods P&CAM 127 [6] and S3 [7,8], which it replaces.

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### REAGENTS:

1. Eluent: Carbon disulfide\*, chromatographic quality, containing 0.1% (v/v) benzene\* or other suitable internal standard.
2. Methyl ethyl ketone
3. Nitrogen or helium, purified.
4. Hydrogen, prepurified.
5. Air, filtered

\* See SPECIAL PRECAUTIONS

### EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6-mm OD, 4-mm ID. Two sections of 20/40 mesh beaded carbon separated by 2-mm foam plug (front = ~ 150 mg, back = ~75 mg). Tubes are commercially available: Anasorb 747 (SKC 226-81), Anasorb CMS (SKC 226-121), Carboxen-564 (Supelco ORBO-90), or equivalent.
2. Personal sampling pump, 0.01 to 0.2 L/min, with flexible connecting tubing.
3. Gas chromatograph, FID, integrator and column (page 2500-1).
4. Vials, glass, 2-mL, PTFE-lined crimp caps.
5. Syringe, 10- $\mu$ L, readable to 0.1  $\mu$ L.

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**SPECIAL PRECAUTIONS:** Carbon disulfide is toxic and an acute fire and explosion hazard (flash point = -30 °C. Work with it only in a hood. Benzene is a human carcinogen.

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### SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break with ends of the sampler immediately before sampling. Attach sampler to personal sampling pump with flexible tubing.
3. Sample at an accurately known flow rate between 0.01 and 0.2 L/min for a total sample size of 0.25 to 12 L.
4. Cap the samplers with plastic (not rubber) caps and pack securely for shipment.

### SAMPLE PREPARATION:

NOTE: Store samples in a freezer.

5. Place the front and back sorbent sections of the sampler tube in separate vials. Discard the glass wool and foam plugs.
6. Add 1.0 mL eluent to each vial. Attach crimp cap to each vial.
7. Allow to stand 30 min with occasional agitation.

### CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards over the range 4 to 500  $\mu$ g MEK per sample.
  - a. Add known amounts of MEK to eluent 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 (ratio or peak area of analyte to peak area of internal standard vs.  $\mu$ g MEK).

9. Determine desorption efficiency (DE) at least once for each batch of sorbent used to sampling in the calibration range (step 8). Prepare three tubes at each of five levels plus three media blanks.
  - a. Remove and discard back sorbent section of a media blank sampler.
  - b. Inject a known amount of MEK directly onto front sorbent section with a microliter syringe.
  - c. Cap the tube. Allow to stand overnight.
  - d. Desorb (steps 5 through 7) and analyze together with working standards (steps 11 and 12).
  - e. Prepare a graph of DE vs. µg MEK recovered.
10. Analyze three quality control blind spikes and three analyst spikes to insure that the calibration graph and DE graph are in control.

**MEASUREMENT:**

11. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 2500-1. Inject sample aliquot manually using solvent flush technique or with autosampler.  
NOTE: If peak area is above the linear range of the working standards, dilute with eluent, reanalyze and apply the appropriate dilution factor in calculation.
12. Measure peak area. Divide the peak area of analyte by the peak area of internal standard on the same chromatogram.

**CALCULATIONS:**

13. Determine the mass, µg (corrected for DE) of MEK 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 MEK in the air volume sampled,  $V$  (L):

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

**EVALUATION OF METHOD:**

The method was evaluated with spiked samplers and with atmospheres generated by syringe pump/air dilution, verified by infrared absorption. For Amborsorb XE-347 samplers, breakthrough (80% RH, 200 ppm, 0.3 L/min) = 16.4 L; DE (4 to 18 mg per sample) = 1.03; storage stability (0.7 to 4 mg per sample) = 90% after six weeks at 25 °C; precision and accuracy as given on page 2500-1 (15 samples) [2]. A user check gave an estimated LOD of 4 µg MEK per sample [3].

Storage stability of MEK and methyl isobutyl ketone (MIBK) on Anasorb CMS samplers was determined for the Proficiency Analytical Testing (PAT) program [1]. Stability of MEK (0.125, 0.622, and 2.282 mg per sample) was 94 to 99% after 90 days at -5 °C. Only the highest level (2.282 mg) was stable (96% recovery) at ambient temperature for 30 days, and was stable for 180 days (98% recovery) at freezer temperatures (-5 °C). Results were similar for MIBK, although all concentration levels (0.103, 0.516, and 2.064 mg per sample) were stable for 30 days at ambient temperatures.

**REFERENCES:**

- [1] NIOSH [1996]. Six-month storage stability data for methyl ethyl ketone and methyl isobutyl ketone. Proficiency Analytical Testing program, Jensen Groff, unpublished data.
- [2] Slick, E.J. [1983]. NIOSH, unpublished data.
- [3] User check, UBTL, NIOSH Seq. # 3990-J(unpublished, August 29, 1983
- [4] User check, DataChem Labs., NIOSH Seq. # 7053-L (unpublished, August 9, 1990).
- [5] DataChem Labs, NIOSH Seq. # 7373-L (unpublished, Nov.27, 1991).
- [6] NIOSH [1977]. Organic solvents in air: Method P&CAM 127. In: Eller PM, ed. NIOSH Manual of Analytical Methods, 2<sup>nd</sup> ed., V. 1, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-A.
- [7] Ibid., V. 2, 2-Butanone: Method S3. U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B.
- [8] NIOSH [1977]. Documentation of the NIOSH Validation Tests, S3, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185.

**METHOD REVISED BY:**

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