

TABLE 1 MW: TABLE 1 CAS: TABLE 1 RTECS: TABLE 1

METHOD: 1300, Issue 2

EVALUATION: FULL

Issue 1: 15 May 1989

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OSHA/NIOSH/ACGIH: Table 1

PROPERTIES: Table 1

COMPOUNDS: (SYNONYMS: Table 1)	acetone	2-hexanone
	cyclohexanone	methyl isobutyl ketone
	diisobutyl ketone	2-pentanone

SAMPLING		MEASUREMENT	
SAMPLER:	SOLID SORBENT TUBE (coconut shell charcoal, 100 mg/50 mg)	TECHNIQUE:	GAS CHROMATOGRAPHY, FID
FLOW RATE:	0.01 to 0.2 L/min	ANALYTE:	compounds above
VOL-MIN:	<u>acetone</u> 0.5 L <u>others</u> 1 L	DESORPTION:	1 mL CS ₂ ; stand 30 min
-MAX:	3 L 10 L	INJECTION VOLUME:	5 µL
SHIPMENT:	MIBK must be refrigerated [1]	TEMPERATURE-INJECTOR:	250 °C
SAMPLE STABILITY:	unknown	-DETECTOR:	300 °C
FIELD BLANKS:	2 to 10 field blanks per set	-COLUMN:	50 °C to 170 °C @ 10°/min
ACCURACY		CARRIER GAS:	N ₂ or He, 30 mL/min
RANGE STUDIED, BIAS, OVERALL PRECISION (\hat{S}_{IT}), and ACCURACY		COLUMN:	glass (3.5 m x 6-mm ID), packed with 10% SP2100 0.1% Carbowax 1500 on Chromosorb WHP
see EVALUATION OF METHOD		CALIBRATION:	standard solutions of analyte in CS ₂
		RANGE:	0.06 to 10 mg per sample [2]
		ESTIMATED LOD:	0.02 mg per sample
		PRECISION (\hat{S}_j):	see EVALUATION OF METHOD

APPLICABILITY: This is intended as a general method for the ketones listed above. If only certain compounds are of interest, the instrumental conditions can be changed to maximize instrument response for these compounds.

INTERFERENCES: None reported. Alternate columns, e.g., 10% SP-2100 or DB-1 fused silica capillary, can be used.

OTHER METHODS: This method combines and replaces Methods S1, S18, S19 and S20 [3], S178 and S358 [4], and 1300 (dated 2/15/84).

REAGENTS:

1. Carbon disulfide (CS₂) GC grade.*
2. Analytes, reagent grade.
3. Nitrogen, prepurified.
4. Hydrogen, dry.
5. Air, filtered, dry.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6-mm OD, 4-mm ID, flame-sealed ends with plastic caps, containing two sections of activated (600 °C) coconut shell charcoal (front = 100 mg; back = 50 mg) separated by a 2-mm urethane foam plug. A silylated glass wool plug precedes the front section and a 3-mm urethane foam plug follows the back section. Pressure drop across the tube at 1 L/min must be less than 3.4 kPa. Tubes are commercially available.
2. Personal sampling pump, 0.01 to 0.2 L/min, with flexible connecting tubing.
3. Gas chromatograph equipped with FID, integrator and column (page 1300-1).
4. Vials, 2-mL, glass, PTFE-lined crimp caps.
5. Syringe, 10-μL, readable to 0.1 μL.
6. Pipet, TD, 1-mL, with pipet bulb.
7. Volumetric flasks, 10-mL.

SPECIAL PRECAUTIONS: Carbon disulfide is toxic and an acute fire and explosion hazard (flash point = -30 °C); work with it only in a hood.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break the 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 0.5 to 3 L for acetone or 1 to 10 L for the other analytes.
4. Cap the samplers and pack securely for shipment.

SAMPLE PREPARATION:

5. Place the front and back sorbent sections of the sampler in separate vials. Discard the glass wool and foam plugs.
6. Add 1.0 mL CS₂ 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 0.02 to 10 mg analyte per sample.
 - a. Add known amounts of analyte to CS₂ 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 vs. mg analyte).
9. Determine desorption efficiency (DE) at least once for each lot of charcoal used for 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 analyte or of a standard solution of analyte in CS₂ 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. mg analyte 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 1300-1. 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 CS₂, reanalyze, and apply the appropriate dilution factor in calculations.
12. Measure peak area.

CALCULATIONS:

13. Determine the mass, mg (corrected for DE) of analyte 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 analyte in the air volume sampled, V (L):

$$C = \frac{(W_f + W_b - B_f - B_b) \cdot 10^3}{V}, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

The methods on which this method was based were validated under NIOSH Contract CDC-99-74-45 [5]. Desorption efficiency was checked by spiking known amounts of the compounds (either neat or in solutions with CS₂) on coconut shell charcoal. Samples were generated for acetone, cyclohexanone, 2-pentanone, and methyl isobutyl ketone by heating a quantity of the liquid to just below its boiling point in a 3-necked, 500-mL round bottom flask. The compound was carried through a fixed-temperature condenser to the concentrations. Samples were generated for diisobutyl ketone and 2-hexanone using a syringe pump which delivered the compounds to a heated glass-lined inlet which was swept with nitrogen, carrying the vapor to the mixing chamber. Low-level desorption efficiency checks showed the DEs to be 70% and 76% for 0.024 and 0.096 mg/sample respectively for cyclohexanone and 87% for 0.052 mg/sample of MIBK [2]. Results of the evaluation experiments are given below.

Compound	Method [2-5]	Range, mg/m ³ Overall Precision (\bar{S}_r)	Breakthrough ¹ (L)	Measurement Range, mg/sample (\bar{S}_r)	DE ²
Acetone	S1, 127	1200 to 4500 (0.082)	4.3	2.4 to 14.2 (0.024)	0.86
Cyclohexanone	S19	98 to 392 (0.062)	65	3.8 to 18.0 (0.025)	0.82
Diisobutyl ketone	S358	145 to 582 (0.070)	44	1.8 to 7.0 (0.032)	0.97
2-Hexanone	S178	188 to 790 (0.053)	>45	1.5 to 8.1 (0.018)	0.81
MIBK	S18	208 to 836 (0.064)	17	2.1 to 8.3 (0.008)	0.91
2-Pentanone	S20	395 to 1570 (0.063)	19	3.5 to 14.0 (0.011)	0.90

¹ 5% breakthrough, 0.2 L/min at high end of concentration range in dry air.

² Averaged over mass range shown.

REFERENCES:

- [1] UBTL Report, Sequence #5528-J (NIOSH, unpublished, Oct. 2, 1986).
- [2] Williams, Karen, Desorption Efficiency Determination for Cyclohexanone and Methyl Isobutyl Ketone, NIOSH/MRSB, Unpublished (1986).
- [3] NIOSH Manual of Analytical Methods, 2nd. ed., V. 2, S1, S18, S19 and S20, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).
- [4] Ibid, V. 3, S178 and S358, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).
- [5] Documentation of the NIOSH Validation Tests, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977), available as Stock No. PB 274-248 from NTIS, Springfield, VA 22161.
- [6] NIOSH Manual of Analytical Methods, 2nd. ed., V. 1, P&CAM 127, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-A (1977).

METHOD REVISED BY:

Ardith Grote, NIOSH/DPSE; methods originally validated under NIOSH Contract CDC-99-74-45.

Table 1. General Information

Compound (Synonyms)	Formula (M.W.)	Properties*	Exposure Limits, ppm		mg/m ³ / ppm @ NTP
			OSHA NIOSH ACGIH		
Acetone 2-Propanone; (CAS #67-64-1) RTECS AL3150000	CH ₃ COCH ₃ (58.08)	liquid; BP 56 °C; d 0.791 g/mL; VP 35.5 kPa (266 mm Hg)	750; STEL 1000 250: Group III Pesticide 750; STEL 1000		2.37
Cyclohexanone Cyclohexyl ketone (CAS #108-94-1) RTECS GW1050000	C ₆ H ₁₀ O (98.15)	liquid; BP 155 °C; d 0.947 g/mL; VP 0.3 kPa (2 mm Hg)	25 (skin) 25 (skin); Group III Pesticide 25 (skin)		4.01
Diisobutyl ketone (2,6-dimethyl- 4-heptanone; CAS #108-83-8) RTECS MJ5775000	((CH ₃) ₂ CHCH ₂) ₂ CO (142.24)	liquid; BP 169 °C; d 0.847 g/mL; VP 0.23 kPa (1.7 mm Hg)	25 25; Group II Pesticide 25		5.82
2-Hexanone (methyl n-butyl ketone; MBK; CAS #591-78-6) RTECS MP1400000	CH ₃ (CH ₂) ₃ COCH ₃ (100.16)	liquid; BP 127 °C; d 0.812 g/mL; VP 0.4 kPa (3 mm Hg)	100 1 5		4.09
Methyl isobutyl ketone (MIBK; hexone; isopropyl acetone; 4-methyl- 2-pentanone; CAS #108-10-1) RTECS SA9275000	(CH ₃) ₂ CHCH ₂ COCH ₃ (100.16)	liquid; BP 117 °C; d 0.800 g/mL; VP 2.0 kPa (15 mm Hg)	50; STEL 75 50; STEL 75 50; STEL 75		4.09
2-Pentanone (methyl propyl ketone; CAS #107-87-9) RTECS SA 7875000	CH ₃ CH ₂ CH ₂ COCH ₃ (86.13)	liquid; BP 100 °C; d 0.812 g/mL; VP 3.6 kPa (27 mm Hg)	200; STEL 250 150 200; STEL 250		3.52

* Density @ 25 °C; VP @ 20 °C.