ACETOIN 2558

$C_4H_8O_2$ MW: 88.11 CAS: 513-86-0 RTECS: EL8790000

METHOD: 2558, Issue 1 **EVALUATION: PARTIAL** **Issue 1: 15 March 2003**

OSHA : None
NIOSH: None
ACGIH: None

PROPERTIES: liquid, solid (dimer); d= 0.9972 g/mL @ 20 °C; BP= 148 °C; FP = 50°C; MP= 15°C; Miscible in water and alcohol

SYNONYMS: 3-Hydroxy-2-butanone, 2,3-butanolone, acetyl methyl carbinol, dimethylketol

SAMPLING

SAMPLER: Anasorb CMS Solid Sorbent Tubes (150/75 mg)

FLOW RATE: 0.01 to 0.2 L/min

VOL-MIN: 1 L
-MAX : 10 L

SHIPMENT: Ship cold (5°C) and store in dark

SAMPLE STABILITY: 30 days @ 5°C

BLANKS: 10% of field samples

ACCURACY

RANGE STUDIED: Not Determined

BIAS: Not Determined

OVERALL PRECISION ($\hat{S}_{r,T}$): Not Determined

ACCURACY: Not Determined

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

ANALYTE: Acetoin

DESORPTION: 1 mL of acetone/methanol (95:5) on rotary mixer for 1.5 hours

INJECTION VOLUME: 1µL

TEMPERATURE
-INJECTION: 225°C
-DETECTOR: 250°C
-COLUMN: 35°C (hold 3 min) to 200°C (6°C/min)

CARRIER GAS: He (2.8 mL/min)

COLUMN: Capillary, fused silica, 30 m x 0.32-mm ID; 1-µm film, Stabilwax-DA or equivalent

CALIBRATION: Standard solutions in acetoin in acetone/methanol (95:5) solvent

RANGE: 3 to 378 µg

ESTIMATED LOD: 1.0 µg

PRECISION ($\hat{S}_r$): 0.008

APPLICABILITY: The working range for acetoin was 0.6 to 75.6 mg/m^3 for a 5 L air sample.

INTERFERENCES: Any compounds with similar retention times to acetoin.

OTHER METHODS: None determined.

REAGENTS:

1. Acetoin, chromatographic grade.*
2. Acetone, pesticide grade.*
3. Methanol, pesticide grade.*
4. Helium, pre-purified and filtered.
5. Hydrogen, pre-purified and filtered.
6. Air, compressed, purified, and filtered.
7. Calibration stock solution: Add known amounts of acetoin to solvent in 10-mL volumetric flask.

* See SPECIAL PRECAUTIONS

EQUIPMENT:

1. Sampler: Anasorb CMS solid sorbent tube (SKC, Inc. # 226-121).
2. Personal sampling pump, 0.01 to 0.2 L/min, connected with flexible tubing.
3. Gas chromatograph equipped with FID, data collection system, and capillary column with a deactivated glass inlet liner(page 2558-1).
4. Autosampler vials, 2-mL, glass, with PTFE-lined crimp caps.
5. Syringes, 10-µL, 25-µL, and 1-mL.
6. Pipettes, 3-mL and 5-mL.
7. Volumetric flasks, 10-mL.

SPECIAL PRECAUTIONS: Acetoin is a flammable and toxic compound. Acetone and methanol are flammable and pose a fire hazard. Work with all chemicals in a well ventilated laboratory safety hood.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break the ends of the sampling tube immediately before sampling. Attach sampling tube 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 10 L.
4. Cap the samplers with plastic caps, pack in a manner to insure that the samplers are kept in the dark, and are shipped cold (5°C).

SAMPLE PREPARATION:

5. Place the front and back sorbent sections in separate amber vials. Place the glass wool plug preceding the front section into the vial containing the front sorbent section. Discard the urethane foam plugs.
6. Add 1.0 mL of the acetone/methanol (95:5) solvent into each vial. Securely attach crimp caps to each vial.
7. Place each vial on a rotary mixer for 1.5 hours.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards from below the LOD to 10 times the LOQ. If necessary, additional standards may be added to extend the calibration curve.
   a. Weigh out a known amount of acetoin in a 10-mL volumetric flask and dilute to the mark with solvent. Prepare additional standards by serial dilution in additional 10-mL volumetric flasks.
   b. Analyze together with samples and blanks (steps 11 and 12).
   c. Prepare calibration graph (peak area vs µg acetoin).
9. Determine the desorption efficiency (DE) at least once for each lot of Anasorb CMS used for sampling in the calibration ranges (step 8).
   a. Prepare three tubes at each of five levels plus three media blanks.
   b. Inject a known amount (5 to 20 µL) of DE stock solution directly onto the front section of each Anasorb CMS with a microliter syringe.
   c. Allow the tubes to air equilibrate for several minutes, then cap the ends of each tube and store overnight at ambient temperature.
   d. Desorb (steps 5-7) and analyze together with standards and blanks (steps 11 and 12).
   e. Prepare a graph of DE vs µg acetoin recovered.

10. Analyze a minimum of three quality control blind spikes and three analyst spikes to ensure that the calibration graph and DE graph are in control.

MEASUREMENT:

11. Set gas chromatograph according to manufacturer's recommendations and to the conditions listed on page 2558-1. Inject a 1-µL sample aliquot manually using the solvent flush technique or with an autosampler.

    Note: If peak area is above the linear range of the working standards, dilute with solvent, reanalyze and apply the appropriate dilution factor in the calculations.
12. Measure peak areas.

CALCULATIONS:

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

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

EVALUATION OF METHOD:

This method, like NMAM 2557 (Diacetyl), was developed in response to a request to identify and quantitate possible hazardous chemical causes of workplace lung disease occurring at a microwave popcorn packaging facility. Acetoin, an inhalation irritant and chemically reactive compound, was identified as a possible suspect chemical in the facility.

Due to the reactivity of acetoin (under certain environmental conditions acetoin can be converted to diacetyl and vice versa [2]), several special handling requirements were included in this method development. A deactivated glass inlet liner was used to retard the oxidation of acetoin to diacetyl and to prevent analyte decomposition in the injection port. The Stabilwax-DA capillary was used to reduce peak tailing and decomposition of oxygenated compounds during analysis. The reactivity of acetoin was further retarded by using amber glassware and storage of stock and samples at 5°C. Application of these analytical conditions resulted in an average DE recovery of 94.9% over a range of 57 to 378 µg. The results of the storage stability study indicated that acetoin was stable for 7 days at 5°C with a recovery of 93.5%. A slow increase in the amount of diacetyl formed during the study was noted, with a maximum of 16-18% recorded at 30 days.

It should also be noted that the acetoin monomer (liquid) will convert to a dimer (white solid) upon standing. However, when dissolved, acetoin will convert back to the monomer in solution. Because of the limited solubility of acetoin some sonication may be required to fully dissolve the required amounts in the solvent.

REFERENCES:

[1] Pendergrass SM [2001]. Acetoin Backup Data Report, October.
[2] Fix, GJ[1993]. Diacetyl: Formation, Reduction, and Control, Brewing Tech., July/August.

METHOD WRITTEN (REVISED) BY: Stephanie M. Pendergrass, NIOSH/DART