**Bendiocarb (Ficam)**

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|---|---|
| Method number: | PV2008 |
| Matrix: | Air |
| Target Concentration: | 0.5 mg/m$^3$ (arbitrary). There is no OSHA permissible exposure level (PEL) or ACGIH threshold limit value (TLV) for bendiocarb. |
| Procedure: | Samples are collected by drawing known volumes of air through OSHA versatile sampler (OVS-2) tubes, each containing a glass fiber filter and two sections of XAD-2 adsorbent. Samples are desorbed with acetonitrile and analyzed by high performance chromatography (HPLC) using an ultraviolet detector (UV). |
| Recommended air volume and sampling rate: | 240 L at 1.0 L/min |
| Detection limit of the overall procedure (based on the recommended air volume and the analytical detection limit): | 0.01 mg/m$^3$ |
| Status of method: | Stopgap method. This method has been partially evaluated and is presented for information and trial use only. |

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# 1. General Discussion

## 1.1. Background

### 1.1.1. History of procedure

The OSHA Analytical Laboratory received a set of samples requesting the analysis of bendiocarb along with other pesticides. The samples had been collected on OVS-2 tubes. This report describes the analytical method developed for bendiocarb.

### 1.1.2. Toxic effects (This section is for information only and should not be taken as the basis of OSHA policy.)

The oral $LD_{50}$ of bendiocarb is 35-100 mg/kg in mammals. (Ref. 5.3) From another source, the oral $LD_{50}$ for rats is 40-156 mg/kg depending on strain. (Ref. 5.2)

### 1.1.3. Potential workplace exposure

Bendiocarb is used as an insecticide. (Ref. 5.2) There was no information available on the number of workers potentially exposed to bendiocarb.

### 1.1.4. Physical properties (Ref. 5.1 to 5.3)

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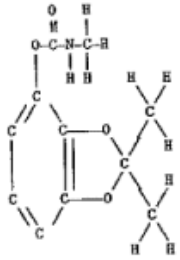
|---|---|
| CAS number: | 22781-23-3 |
| IMIS number: | F108 |
| Molecular weight: | 223.23 |
| Molecular formula: | $C_{11}H_{13}NO_4$ |
| Vapor pressure: | $5.0 \times 10^{-6}$ mm Hg at 25°C |
| Melting point: | 129-130°C |
| Solubility: | Soluble in acetonitrile (observed); Soluble in hexane 350 ppm; soluble in water 40 ppm. |
| Chemical name: | 2,2-Dimethyl-1,3-benzodioxol-4-yl methylcarbamate |
| Synonyms: | Bencarbate; 1,3-bezodioxole; 2,2-dimethyl-4-(N-methylcarbamato)-; Dycarb; Ficam; Ficam D; Ficam ULV; Garvox;. Multamat; Multimet; Niomil; Ratate; Tattoo; Turcam. |
| Description: | white crystalline solid |
| UV Scan: | Figure 1 |

Structure:



1.2. Limit defining parameters

The detection limit of the analytical procedure is 2.4 ng per injection. This is the amount of analyte which will give a peak whose height is approximately five times the baseline noise.

2. Sampling Procedure

2.1. Apparatus

2.1.1. A personal sampling pump that can be calibrated to within ± 5% of the recommended flow rate with the sampling device in line.

2.1.2. OVS-2 tubes, which are specially made 13-mm o.d. glass tubes that are tapered to 6 mm o.d., packed with a 140-mg backup section, a 270-mg sampling section of cleaned XAD-2 adsorbent and a 13-mm diameter glass fiber filter. The backup section is retained by two foam plugs and the sampling section is between one foam plug and the glass fiber filter. The glass fiber filter is held next to the sampling section by a polytetrafluoroethylene (PTFE) retainer. (Figure 2)

2.2. Reagents

No sampling reagents are required.

2.3. Sampling technique

2.3.1. Immediately before sampling, remove the plastic caps from the OVS-2 tube.

2.3.2. Attach the small end of the tube to the sampling pump with flexible tubing.

2.3.3. Attach the tube vertically in the employee's breathing zone in such a manner that it does not impede work performance.

2.3.4. After sampling for the appropriate time, remove the tube and seal with plastic caps.

2.3.5. Wrap each sample end-to-end with an OSHA seal (Form 21).

2.3.6. Record the air volume for each sample, and list any possible interferences.

2.3.7. Submit at least one blank for each set of samples. Handle the blank in the same manner as the samples, except no air is drawn through it.

2.3.8. Submit bulk samples for analysis in a separate container. Do not ship with air samples

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2.4. Desorption efficiency (glass fiber filter and XAD-2 adsorbent)

Six vials each containing a 13-mm glass fiber filter and 270-mg of XAD-2 adsorbent were each liquid spiked on the glass fiber filter with 50 µL of a 2.16 mg/mL solution of bendiocarb. These samples were allowed to equilibrate overnight in a drawer at ambient temperature. The next day each sample was desorbed with 2.0 mL of acetonitrile, shaken for 30 min and analyzed as in Section 3. The results are listed in Table 2.4.

Table 2.4.
Desorption Efficiency

| Sample # | Amount Spiked, µg | Amount Found, µg | % Recovered |
|---|---|---|---|
| Ex1 | 108 | 99.37 | 92.0 |
| Ex2 | 108 | 100.24 | 92.8 |
| Ex3 | 108 | 94.53 | 87.5 |
| Ex4 | 108 | 96.71 | 89.5 |
| Ex5 | 108 | 97.94 | 90.7 |
| Ex6 | 108 | 95.81 | 88.7 |
| | | Average = | 90.2 |

2.5. Retention efficiency

Eighteen OVS-2 tubes were each liquid spiked with 50 µL of a 2.16 mg/mL solution of bendiocarb by spiking the glass fiber filter. These were allowed to equilibrate overnight in a drawer at ambient temperature. The next day 240 L of humid air (~80% relative humidity) were drawn through each tube at 1 L/min. Six of the tubes were each desorbed with 2.0 mL of acetonitrile, shaken for 30 min and then analyzed as in Section 3. The results are listed in Table 2.5. No bendiocarb was found on the backup sections of these tubes. The remaining samples were stored, six in a drawer at ambient temperature and six in a freezer, for use in the storage study below.

Table 2.5.
Retention Efficiency

| Sample # | Amount Spiked, µg | Amount Found, µg | % Recovered |
|---|---|---|---|
| R1 | 108 | 108.02 | 100.0 |
| R2 | 108 | 103.23 | 95.6 |
| R3 | 108 | 104.13 | 96.4 |
| R4 | 108 | 102.32 | 94.7 |
| R5 | 108 | 105.72 | 97.9 |
| R6 | 108 | 107.41 | 99.5 |
| | | Average = | 97.4 |

2.6. Sample storage

After four days of storage, six tubes were each desorbed with 2.0 mL of acetonitrile, shaken for 30 min and then analyzed as in Section 3. Three of the tubes were from ambient storage and the other three were from the freezer storage samples. The remaining tubes were analyzed after eight days of storage. The results are given in Tables 2.6.1. and 2.6.2.

Table 2.6.1.
Ambient Storage

| Days Stored | Amount Spiked, µg | Amount Found, µg | % Recovered |
|---|---|---|---|
| 4 | 108 | 97.98 | 90.7 |
| 4 | 108 | 104.71 | 97.0 |
| 4 | 108 | 98.56 | 91.3 |
| 8 | 108 | 96.54 | 89.4 |
| 8 | 108 | 102.35 | 94.8 |
| 8 | 108 | 86.04 | 79.7 |
| | | Average of four days = | 93.0 |
| | | Average of eight days = | 88.0 |

Table 2.6.2.
Freezer Storage

| Days Stored | Amount Spiked, µg | Amount Found, µg | % Recovered |
|---|---|---|---|
| 4 | 108 | 94.55 | 87.5 |
| 4 | 108 | 100.29 | 92.9 |
| 4 | 108 | 99.73 | 92.3 |
| 8 | 108 | 102.0 | 94.4 |
| 8 | 108 | 103.46 | 95.8 |
| 8 | 108 | 111.83 | 103.5 |
| | | Average of four days = | 90.9 |
| | | Average of eight days = | 97.9 |

2.7. Recommended air volume and sampling rate

2.7.1. The recommended air volume is 240 L.

2.7.2. The recommended flow rate is 1.0 L/min.

2.8. Interferences (sampling)

It is not known if any compounds will interfere with the collec tion of bendiocarb. Any suspected interferences should be reported to the laboratory.

2.9. Safety precautions (sampling)

2.9.1. Attach the sampling equipment in such a manner that it will not interfere with work performance or employee safety.

2.9.2. Follow all safety practices that apply to the work area being sampled.

3. Analytical Procedure

3.1. Apparatus

3.1.1. A balance capable of weighing to the nearest tenth of a milligram. A Mettler HL52 balance was used in this evaluation.

3.1.2. A mechanical shaker.

3.1.3. An HPLC equipped with a W detector. A Hewlett Packard (HP) 1090M equipped with an autosampler and diode array detector was used in this evaluation.

3.1.4. An HPLC column capable of separating bendiocarb from any interferences. A 100 mm × 2.1 mm i.d. Hypersil ODS liquid chromatography column was used in this evaluation.

3.1.5. An electronic integrator, or some other suitable means for measuring detector response. The Hewlett-Packard 1090M Data System was used in this evaluation.

3.1.6. Volumetric flasks and pipets.

3.1.7. Vials, 2-mL and 4-mL.

3.2. Reagents

3.2.1. Acetonitrile, HPLC grade. This was obtained from Burdick and Jackson for this evaluation.

3.2.2. Bendiocarb, reagent grade. A standard obtained from EPA (EPA # 0472, 99.9% purity) was used in this evaluation.

3.2.3. Water, HPLC grade, Milli-Q filtered water, Millipore Inc.

3.3. Standard preparation

Prepare bendiocarb stock standards by weighing 10 to 15 mg of bendiocarb. Transfer the bendiocarb to separate lo-mL volumetric flasks, and add acetonitrile to the mark. Make working range standards of 1.2 to 100 µg/mL by pipet dilutions of the stock standards with acetonitrile. Store stock and dilute standards in a freezer.

3.4. Sample preparation

3.4.1. Transfer the 13-mm glass fiber filter and the 270-mg sampling section of the tube to a 4-mL vial. Place the first foam plug and the 140-mg section in a separate 4-mL vial. A small glass funnel can be used to facilitate the transfer of the adsorbent. Discard the rear foam plug. Do not discard the glass sampling tube; it can be reused.

3.4.2. Add 2.0 mL of acetonitrile to each vial and seal with a Teflon-lined cap.

3.4.3. Shake the vials for 30 minutes on a mechanical shaker.

3.4.4. Transfer, if necessary, the samples to 2-mL vials for use on an HP autosampler.

3.5. Analysis

3.5.1. Instrument conditions

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|---|---|
| Column: | 100 mm × 2.1 mm Hypersil ODS |
| Mobile phase: | 30% acetonitrile 70% water |
| Flow rate: | 0.25 mL/min |
| Wavelength: | 205 nm |
| Retention time: | 6.2 min |
| Injection volume: | 2.0 µL |

3.5.2. Chromatogram (Figure 3)

3.6. Interferences (analytical)

3.6.1. Any collected compound having a similar retention time to that of the analyte is a potential interference.

3.6.2. HPLC conditions may generally be varied to circumvent interferences.

3.6.3. Retention time on a single column is not proof of chemical identity. Analysis on an alternate HPLC column and confirmation by mass spectrometry are additional means of identification.

3.7. Calculations

3.7.1. Construct a calibration curve (Figure 4) by plotting detector response versus concentration (µg/mL) of bendiocarb.

3.7.2. Determine the µg/mL of bendiocarb in both sections of each sample and blank from the calibration curve.

3.7.3. Blank correct each sample section by subtracting the µg/mL found in the blank section from the µg/mL found in the sample section and then add the sample sections together.

3.7.4. Determine the air concentration by using the following formula.

$$mg/m^3 = \frac{(\mu g/mL, \text{ blank corrected}) \times (\text{desorption volume, mL})}{(\text{air volume, L}) \times (\text{desorption efficiency, decimal})}$$

3.8. Safety precautions (analytical)

3.8.1. Avoid skin contact and air exposure to bendiocarb.

3.8.2. Avoid skin contact with all solvents.

3.8.3. Wear safety glasses at all times.

4. Recommendation for Further Study

This method should be fully validated.

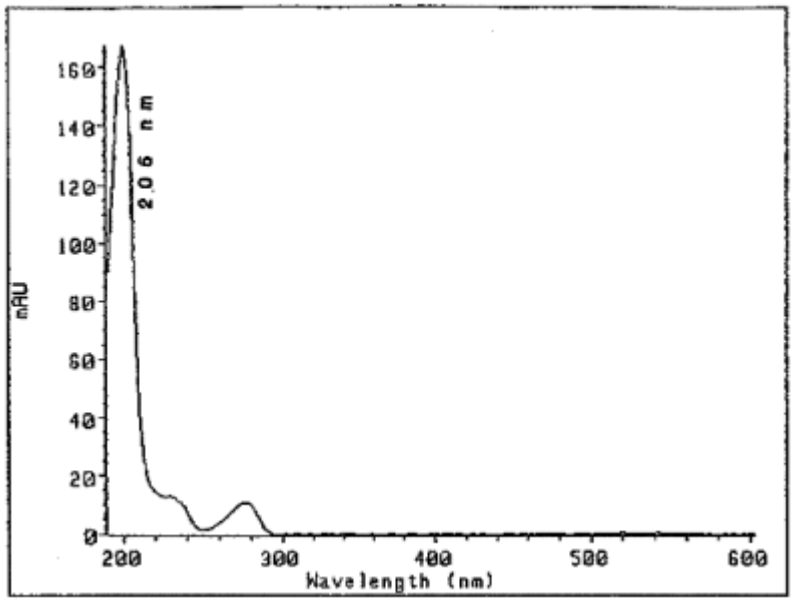


**Figure 1.**
**UV Scan of Bendiocarb in Mobile Phase**

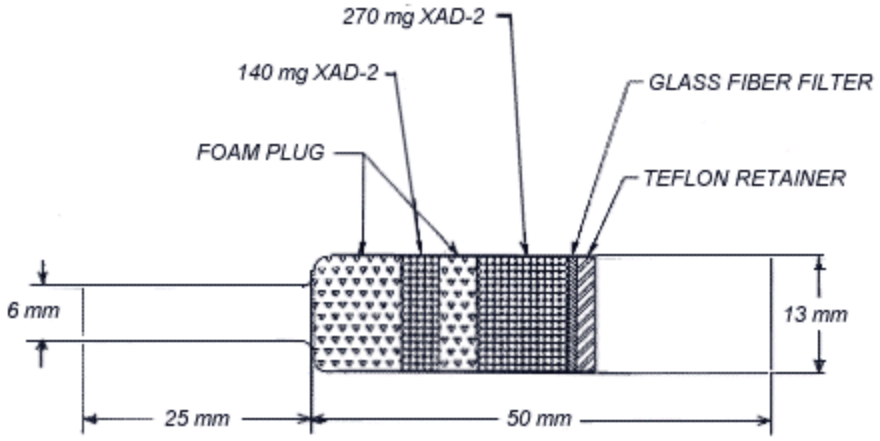


**Figure 2.**
**OVS-2 Sampling Tube**

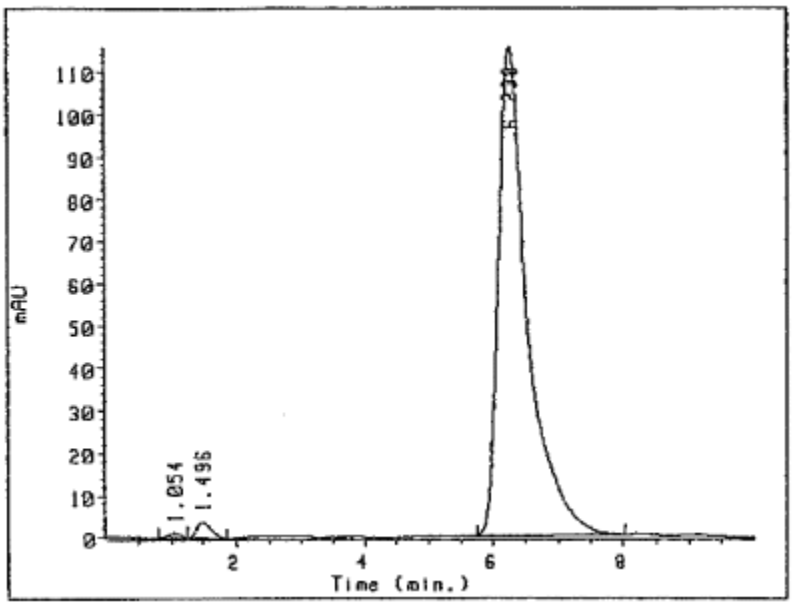


**Figure 3.**
**Chromatogram of Bendiocarb**

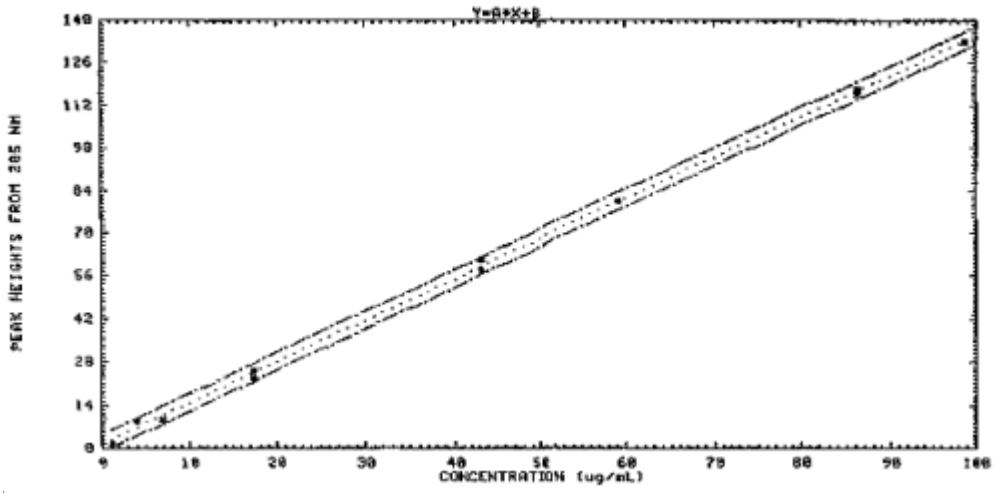


**Figure 4.**
**Calibration Curve**

5. References


5.1. **Registry of Toxic Effects of Chemical Substances 1985-86 Edition**; DHHS(NIOSH)Publication No F87-114, U.S. Department of Health and Human Services: Cincinnati, OH, 1987; p 1423.

5.2. **Farm Chemicals Handbook**; Berg, Gordon L. Ed.; Meister: Willoughby, OH, 1986; p C39.

5.3. **Merck Index**, 10th ed.; Windholz, Martha Ed.; Merck: Rahway, NJ,983; p 146-147.