Dimethazone

Method no.: PV2066

Matrix: Air

Target Concentration: 1 mg/m³ (arbitrary) There is no OSHA permissible exposure level (PEL) or ACGIH threshold limit value (TLV) for dimethazone.

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 toluene and analyzed by gas chromatography (GC) using an electron capture detector (ECD).

Recommended air volume and sampling rate: 60 L at 1.0 L/min

Detection limit of the overall procedure (based on the recommended air volume and the analytical detection limit): 23 µg/m³

Status of method: Stopgap method. This method has been only partially evaluated and is presented for information and trial use.

Date: January 1989 (final draft)

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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 dimethazone. The samples had been collected of OVS-2 tubes. This report describes the analytical method developed.

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

The acute oral LD_{50} is 1369 mg/kg for female rats and 2077 mg/kg for male rats. (Ref. 5.1)

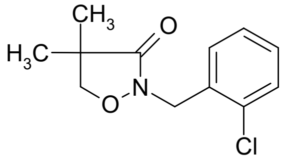
1.1.3 Potential workplace exposure

Dimethazone is a selective herbicide for weed control in soybeans. No estimate of worker exposure to dimethazone could be found. (Ref. 5.1)

1.1.4 Physical properties (Ref. 5.1)

Molecular weight:	239.7 (calculated)
Molecular formula:	$C_{12}H_{14}NO_2Cl$
CAS #:	81777-89-1
IMIS number:	C229
Specific gravity:	1.192 at 20˚C
Flash point:	157-167˚C
Solubility:	soluble in toluene; in water, 1100 ppm
Chemical name:	2-(2-chlorophenyl)methyl-4,4-dimethyl-3-isoxazolidinone
Synonyms:	Command, FMC 57020
Description:	light brown viscous liquid; white solid (observed from EPA standard)

Structure:



1.2 Limit defining parameters

The detection limit of the analytical procedure, including a 5:1 split ratio, is 0.09 ng per injection. This is the amount of analyte which will give a peak whose height is approximately five times the baseline noise. (Figure 1.)

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.

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 15 µL of a 4.12 mg/mL dimethazone standard. These were allowed to dry and then capped with Teflon-lined caps and placed in a drawer over night. The next day the samples were each desorbed with 3 mL of toluene, shaken for 30 min and then analyzed as in Section 3. The results are listed in the Table below.

Table 2.4
Desorption Efficiency

Sample #	Amount Spiked, µg	Amount Found, µg	Recovered, %
Ex1	61.8	61.5	99.5
Ex2	61.8	63.9	103.4
Ex3	61.8	63.7	103.1
Ex4	61.8	62.4	101.0
Ex5	61.8	58.4	94.5
Ex6	61.8	57.9	93.7
		average	99.2

2.5 Retention efficiency

Eighteen OVS-2 tubes were each liquid spiked with 15 µL of a 4.12 mg/mL dimethazone standard on the glass fiber filter. These were allowed to dry and then 240 L of humid air (~80% relative humidity) were drawn through each tube at 1 L/min. Twelve of the tubes were stored in a drawer at ambient temperature and six of the tubes were stored in a freezer. The next day 6 of the tubes from the drawer were each desorbed with 3 mL of toluene, shaken for 30 min and then analyzed as in Section 3. The results are listed in the Table below.

Table 2.5
Retention Efficiency

Sample #	Amount Spiked, µg	Amount Found, µg	Recovered, %

R1	61.8	60.9	98.5
R2	61.8	62.7	101.5
R3	61.8	63.2	102.3
R4	61.8	59.3	96.0
R5	61.8	56.0	90.6
R6	61.8	55.6	90.0
		average	96.5

2.6 Sample storage

After 6 days of storage, 6 tubes, 3 from the ambient storage group and 3 from the freezer storage group, were each desorbed with 3 mL of toluene, shaken for 30 min and then analyzed as in Section 3. After 9 days of storage, the remaining tubes were desorbed and analyzed. The results are given in the Tables below.

Table 2.6.1
Ambient Storage

Sample #	Amount Spiked, µg	Amount Found, µg	Recovered, %
6	61.8	60.7	98.2
6	61.8	62.7	101.5
6	61.8	61.3	99.2
9	61.8	65.7	106.3
9	61.8	59.5	96.3
9	61.8	58.8	95.1
		average of Day 6	99.6
		average of Day 9	99.2

Table 2.6.2
Freezer Storage

Sample #	Amount Spiked, µg	Amount Found, µg	Recovered, %
6	61.8	60.5	97.9
6	61.8	57.7	93.4
6	61.8	54.5	88.2
9	61.8	55.5	89.8
9	61.8	52.6	85.1
9	61.8	51.7	83.7
		average of Day 6	93.2
		average of Day 9	86.2

2.7 Recommended air volume and sampling rate

2.7.1 The recommended air volume is 60 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 collection of dimethazone. Any suspected interference 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 impede 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 A GC equipped with an ECD. A Hewlett Packard (HP) 5890 was used in this evaluation.

3.1.4 A GC column capable of separating dimethazone from any interferences. A 10 m x 0.32 mm i.d. (1.0 µm film) DB-5 capillary column was used in this evaluation.

3.1.5 An electronic integrator, or some other suitable means for measuring detector response. The Hewlett-Packard 3357 Laboratory Data System was used in this evaluation.

3.1.6 Volumetric flasks and pipets.

3.1.7 Vials, 2-mL.

3.2 Reagents

3.2.1 Toluene, reagent grade.

3.2.2 Dimethazone, reagent grade. A standard obtained from EPA (EPA # F815, 99.9% purity) was used in this evaluation.

3.3 Standard preparation

Prepare dimethazone stock standards by weighing 10 to 14 mg of dimethazone. Transfer the dimethazone to separate 10-mL volumetric flasks, and add toluene to the mark. Make working range standards of 0.4 to 50 µg/mL by pipet dilutions of the stock standards with toluene. 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 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 3.0 mL of toluene 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 the samples to 2-mL vials for use on an HP autosampler.

3.5 Analysis

3.5.1 Instrument conditions

Column:	DB-5, 10 m x 0.32 mm i.d., 1.0 µm film
Injector temperature:	275˚C
Column temperature:	145˚C
Detector temperature:	300˚C
Gas flows:	
Column:	8.6 mL/min hydrogen
ECD make up:	42 mL/min nitrogen
Injection volume:	1.0 µL
Split ratio:	5:1
Retention time:	6.7 min

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 GC conditions may be varied to circumvent interferences.

3.6.3 Retention time on a single column is not proof of chemical identity. Analysis by an alternate GC column or 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 dimethazone.

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

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

3.7.4 Determine the air concentration by using the following formula.

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

3.8 Safety precautions (analytical)

3.8.1 Avoid skin contact and air exposure to dimethazone.

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.

5. References

5.1 *Farm Chemicals Handbook*; Berg, Gordon L. Ed.; Meister: Willoughby, Ohio, 1986; p C61.