Method number:	PV2084
Control number:	T-PV2084-01-8706-CH
Target concentration:	10 ppm - There is no TLV or OSHA PEL for acetamide.
Procedure:	Samples are collected by drawing a known volume of air through a silica gel tube. Samples are desorbed with 1 mL methanol and analyzed by gas chromatography with a nitrogen-phosphorous detector (GC-NPD).
Recommended air volume and sampling rate studied:	100 minutes at 0.1 Lpm (10 L)
Status of method:	Partially validated method. This method has been only partially evaluated and is presented for information and trial use.

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1   General Discussion

1.1   Background

1.1.1   History of procedure

The OSHA Laboratory recently received samples collected on silica gel tubes requesting analysis for acetamide. There is a NIOSH method S254 for the collection and analysis of dimethyl acetamide. This method recommends collection on silica gel tubes and analyzes them by gas chromatography with a flame ionization detector. A nitrogen-phosphorous detector was used since acetamide does not respond as well on a flame ionization detector. The silica gel tubes were found to have adequate desorption, retention, and storage stability.

1.1.2   Potential workplace exposure (Ref. 5.1)

Acetamide is used as a solubilizer, plasticizer, stabilizer, to denature alcohol, and in organic syntheses.

1.1.3   Toxic Effects (This section is for information purposes and should not be taken as the basis for OSHA policy.) (Ref. 5.2)

Acetamide is classified as a mild irritant with a low toxicity; but it has also been classified as a carcinogen by IARC as it tested positive as an animal carcinogen.

1.1.4   Physical properties (Ref. 5.1):

CAS: 60-35-5
IMIS: A625
Molecular weight: 59.07
Melting point: 81 °C
Boiling point: 222 °C
Odor: odorless when pure, but frequently has mousy odor
Color: white crystals
Molecular formula: C_2H_5NO
Compound: CH_3CONH_2

1.2   Limit defining parameters

1.2.1   The detection limit of the analytical procedure is 10 ng. This is the smallest amount that could be detected under normal operating conditions. The injection volume is 1 µL (Figure 1).

1.2.2   The detection limit of the overall procedure is 10 ng per sample (0.4 ppm based on a 10-liter sample). This is the amount of analyte placed on a silica gel tube which corresponds to the detection limit of the analytical procedure.

1.3   Advantages

1.3.1   The sampling procedure is convenient.

1.3.2   The analytical method is reproducible and sensitive.

1.3.3   Reanalysis of samples is possible.

1.3.4　It may be possible to analyze other compounds at the same time.

1.3.5　Interferences may be avoided by proper selection of column and GC parameters.

1.4　Disadvantages

None known

2　Sampling procedure

2.1　Apparatus

2.1.1　A calibrated personal sampling pump, the flow of which can be determined within ±5% at the recommended flow with the sample tube in line.

2.1.2　Silica gel tubes (20/40 mesh) containing a 150-mg adsorbing section with a 75-mg backup section separated by a 2-mm portion of urethane foam, with a silanized glass wool plug before the adsorbing section and a 3-mm plug of urethane foam at the back of the backup section. The ends are flame sealed and the glass tube containing the adsorbent is 7-cm x 6-mm o.d. and 4-mm i.d., SKC tubes or equivalent.

2.2　Sampling technique

2.2.1　The ends of the silica gel tube are opened immediately before sampling.

2.2.2　Connect the silica gel tube to the sampling pump with flexible tubing.

2.2.3　Tube should be placed in a vertical position to minimize channeling, with the smaller section towards the pump.

2.2.4　Air being sampled should not pass through any hose or tubing before entering the silica gel tube.

2.2.5　Seal the silica gel tube with plastic caps immediately after sampling. Seal each sample lengthwise with Form OSHA-21 seal.

2.2.6　With each batch of samples, submit at least one blank tube from the same lot used for samples. This tube should be subjected to exactly the same handling as the samples (break ends, seal, & transport) except that no air is drawn through it.

2.2.7　Transport the samples and corresponding paperwork to the lab for analysis.

2.2.8　Bulks submitted for analysis must be shipped in a separate container from the air samples.

2.3　Desorption efficiency

2.3.1　A loading of 241.8 µg on the tubes corresponds to 10.6 ppm based on a 10-liter air volume and the overall desorption efficiency of 94.4% (Table 1).

2.3.2　A solution of acetamide in methanol was prepared. Aliquots of this solution were liquid spiked onto eighteen tubes, six at each of the following loadings: 483.6, 241.8, and 96.72 µg. These levels correspond to 20, 10, and 4 ppm based on a 10-liter air volume. The tubes were allowed to equilibrate in the refrigerator overnight. They were then opened, each section placed into a separate 2-mL vial, desorbed with 1 mL methanol,

and allowed to desorb for 30 minutes with occasional shaking. They were analyzed by GC-NPD. The overall average desorption was 94.4% (Table 1).

Table 1
Desorption Efficiency

tube #	% desorption		
	483.6 µg	241.8 µg	96.72 µg
1	90.3	92.5	95.7
2	91.6	94.8	92.4
3	92.7	97.1	94.1
4	95.3	96.5	94.1
5	95.3	95.3	94.1
6	96.1	96.0	94.1
average	93.6	95.4	94.1

overall average = 94.4%
standard deviation = ±1.83

2.4   Retention efficiency

A solution of acetamide in methanol was prepared, and aliquots were liquid spiked onto seven silica gel tubes, for a loading of 483.6 µg acetamide. These tubes had 10 liters of humid air (83% RH) drawn through them. They were desorbed and analyzed by GC-NPD. There was no acetamide detected in the backup portions of the tubes. The average amount recovered was 83.5% (Table 2).

Table 2
Retention Efficiency

tube #	% recovered		total
	'A'	'B'	
1	82.5	0.0	83.5
2	84.3	0.0	84.3
3	85.2	0.0	85.2
4	83.4	0.0	83.2
5	83.9	0.0	83.9
6	83.0	0.0	83.0
7	82.1	0.0	82.1

Average = 83.5%
Average (desorption corrected) = 88.4%

2.5   Storage

A solution of acetamide in methanol was prepared, and aliquots were spiked onto three tubes for a loading of 483.6 µg acetamide. These were stored at room temperature (24 °C) for seven days. The amount recovered averaged 94.1% (Table 3).

Table 3
Storage Stability

tube #	% recovered
1	91.8
2	94.7
3	96.2
average	94.1

average (desorption corrected) = 99.8%

2.6 Air volume and sampling rate studied

2.6.1 The air volume studied was 10 liters.

2.6.2 The sampling rate studied was 0.1 liter per minute.

` 2.7 Interferences

Suspected interferences should be listed on sample data sheets.

2.8 Safety precautions

2.8.1 Sampling equipment should be placed on an employee in a manner that does not interfere with work performance or safety.

2.8.2 Safety glasses should be worn at all times.

2.8.3 Follow all safety practices that apply to the workplace being sampled.

3 Analytical method

3.1 Apparatus

3.1.1 Gas chromatograph equipped with a nitrogen-phosphorus detector in the nitrogen mode.

3.1.2 GC column capable of separating the analyte from any interference. A 6-ft 10% UCON 50-HB-5100 on 80/100 Chrom WAW coated with 2% KOH was used.

3.1.3 An electronic integrator or some other suitable method of measuring peak areas.

3.1.4 Two milliliter vials with PTFE-lined caps for sample desorption.

3.1.5 A 1-µL syringe or other convenient size for sample injection.

3.1.6 Pipettes for dispensing the methanol.

3.1.7 Volumetric flasks, 10-mL, and other convenient sizes for preparing standards.

3.1.8 A balance capable of weighing milligram quantities.

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3.2   Reagents

   3.2.1   Purified GC grade nitrogen, hydrogen, and air.
   3.2.2   Reagent grade methanol.

   3.2.3   Reagent grade acetamide.

3.3   Sample preparation

   3.3.1   Sample tubes were opened and the front and back section of each tube was placed in separate 2-mL vials.

   3.3.2   Each section is desorbed with 1 mL methanol.

   3.3.3   The vials are immediately sealed with PTFE lined caps and allowed to desorb for 30 minutes with occasional shaking.

3.4   Standard preparation

   3.4.1   Standards are prepared by diluting a known quantity of acetamide with methanol.

   3.4.2   At least two separate stock standards should be made. Dilutions of these stocks are made at five different levels covering the range of the samples. The standards used ranged from 9.67 to 483.6 µg/mL.

3.5   Analysis

   3.5.1   Gas chromatograph conditions.

Flow rates	**(mL/min)**	**Temperature**	**(°C)**
Nitrogen (carrier):	24	Injector:	200
Hydrogen (detector):	3	Detector:	220
Air (detector):	50	Column:	150

Injection size:	1 µL
Elution time:	2.23 min
Attenuation:	6
Chromatogram:	(See Figure 2)

   3.5.2   Peak areas are measured by an integrator or other suitable means.

3.6   Interferences (analytical)

   3.6.1   Any compound having the general retention time of the analyte used is interference. Possible interferences should be listed on the sample data sheet. GC parameters should be adjusted, if necessary, so these interferences will pose no problems.

   3.6.2   Retention time data on a single column is not considered proof of chemical identity. Samples over the target concentration should be confirmed by GC/Mass Spec or other suitable means.

3.7   Calculations

3.7.1   To calculate the concentration of analyte in the air sample the following formulas are used:

$$mass\ of\ analyte,\ \mu g = \frac{(\mu g\,/\,mL)(desorption\ volume,\ mL)}{(desorption\ efficiency,\ decimal)}$$

$$moles\ of\ analyte = \frac{(mass\ of\ analyte,\ \mu g)(1\,g)}{(molecular\ weight)(10^6\,\mu g)}$$

$$volume\ of\ analyte = (moles\ of\ analyte)(molar\ volume)$$

$$ppm = \frac{(volume\ of\ analyte)(10^6)^*}{(air\ volume,\ L)}$$

* All units must cancel.

3.7.2   The above formulas can be combined to give the formula below. To calculate the ppm of analyte in the sample based on a 10-liter air sample, and a 1 mL desorbing solution:

$$ppm = \frac{(\mu g\,/\,mL)(DV)(24.46)}{(L)(DE)(MW)}$$

Where:

µg/mL = concentration of analyte in sample
24.46 = Molar volume (liters/mole) at 25 °C and 760 mmHg
MW   = Molecular weight (g/mole)
DV   = Desorption volume, mL
10 L   = Air volume, L
DE   = Desorption efficiency, decimal

3.7.3   This calculation is done for each section of the sampling tube and the results added together.

3.8   Safety precautions

3.8.1   All handling of solvents should be done in a hood.

3.8.2   Avoid skin contact with all solvents.

3.8.3.   Wear safety glasses, gloves, and lab coats at all times.

4   Recommendations for further study

Collection efficiencies need to be performed to investigate the apparent loss indicated by the retention study. Longer storage tests should also be performed.

5 References

5.1 Windholz, M., The Merck Index. Tenth Edition, Merck & Co., Rahway N.J., 1983, p. 6.

5.2 Sax, N. Dangerous Properties of Industrial Materials, Fifth Edition, Van Nostrand Reinhold Co., New York, 1979, p. 322.
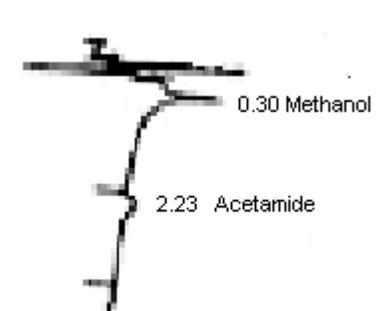
Figure 1. Detection limit standard of 10 µg/mL acetamide in methanol

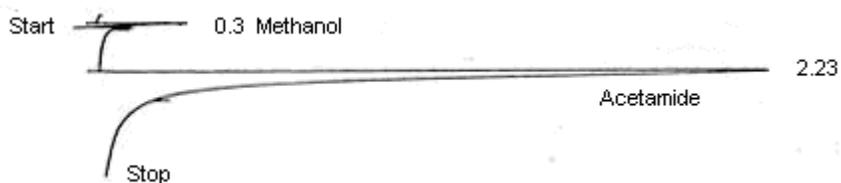Figure 2. Standard of acetamide in methanol at a concentration of 483.6 µg/mL.