Method number:	PV2012
Control no.:	T-PV2012-01-8801-CH
Target concentration:	1.0 mg/m^3
ACGIH TLV-TWA (dust):	5 mg/m^3
ACGIH TLV-TWA (vapor):	5 mg/m^3
Procedure:	Samples are collected by drawing a known volume of air through OSHA versatile sampler (OVS-7) tubes, containing a glass fiber filter and two sections of XAD-7 adsorbent. Samples are extracted with methanol and analyzed by high performance liquid chromatography (HPLC) using an ultraviolet (UV) detector.
Air volume and sampling rate:	100 minutes and 1.0 L/min (100 L)
Detection limit of the overall procedure:	0.007 mg/m^3 based on the recommended air volume
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

This evaluation was undertaken to determine the effectiveness of the OVS-7 sampling tube as a sampling device for caprolactam. It follows the procedure developed for carbaryl. (Ref. 5.1)

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

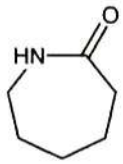
The acute oral LD_{50} for rats is 2140 mg/kg. The inhalation TC Lo for humans is 100 ppm. Caprolactam (dust) has been given a TLV-TWA of 1.0 mg/m^3, ST of 3 mg/m^3 and caprolactam (vapor) has been given a TLV-TWA of 1.0 mg/m^3, ST of 3 mg/m^3 by the ACGIH. (Ref. 5.2)

1.1.3 Potential workplace exposure

No estimate of workplace exposure was found. Caprolactam is used in the manufacture of synthetic fibers, plastics, bristles, film, coatings, synthetic leather, plasticizers, and paint vehicles; cross-linking agent for polyurethanes; synthesis of amino acid lysine. (Ref. 5.3)

1.1.4 Physical Properties: (Ref. 5.2 - 5.3)

CAS #:	105-60-2
Molecular Weight:	113.18
Molecular Formula:	$C_6H_{11}NO$
Melting Point:	68-69 °C
Boiling Point:	180 °C
Appearance:	white flakes
Solubility:	soluble in water, chlorinated solvents, cyclohexene.
Vapor Pressure:	3 mmHg (100 °C) 50 mmHg (180 °C)
Synonyms:	Aminocaproic lactam; 2-oxohexamethylenimine; 2-ketohexamethylenimine; hexahydro-2H-azepin-2-one
Structure:	



1.2 Limit defining parameters

The detection limit of the analytical procedure is 3.4 ng per injection. This is the smallest amount of analyte which will produce a peak 5 times the baseline noise.

2 Sampling procedure

2.1 Apparatus

2.1.1 Samples are collected by using 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 Samples are collected with OVS-7 tubes, which are specially made 13-mm o.d. glass tubes that are tapered to 6-mm o.d. These tubes are packed with a 140-mg, backup section, and a 270-mg sampling section of cleaned XAD-7. The backup section is retained by two foam plugs and the sampling section is between one foam plug and a 13-mm diameter glass fiber filter. The glass fiber filter is held next to the sampling section by a polytetrafluoroethylene (PTFE) retainer. (See Figure 1)

2.2 Reagents

No sampling reagents are required.

2.3 Sampling technique

2.3.1 Attach the small end of the OVS-7 sampling tube to the sampling pump with flexible, plastic tubing such that the large, front section of the sampling tube is exposed directly to the atmosphere. Do not place any tubing in front of the sampler. The sampler should be attached vertically (large end down) in the worker's breathing zone in such a manner that it does not impede work performance.

2.3.2 After sampling for the appropriate time, remove the sampling device and seal the tube with plastic end caps.

2.3.3 Wrap each sample end-to-end with a Form OSHA-21 seal.

2.3.4 With each set of samples, submit at least one blank. The blank should be handled the same as the other samples except that no air is drawn through it.

2.3.5 Bulks samples should be submitted for analysis in a separate container. Do not ship with the air samples.

2.4 Extraction Efficiency

Five 13-mm glass fiber filters were each liquid spiked with 127.6 µg of caprolactam. The five filters, along with a blank filter, were each placed in separate 4-mL vials which also contained 270 mg of XAD-7 adsorbent. These vials were stored overnight at room temperature, and then extracted with 4 mL of methanol. The average extraction efficiency for these five filters (with the XAD-7 adsorbent present) was 111%.

Table 2.4
Extraction Efficiency

sample #	µg recovered	% recovered
1	162.1	127
2	158.2	124
3	164.6	129
4	135.3	106
5	86.8	68

average = 111%

2.5 Retention efficiency

Three OVS-7 tubes were each spiked with either 134.8 µg or 127.6 µg caprolactam by liquid spiking the 13-mm glass fiber filter. One hundred liters of humid air (>70% RH) were drawn

through each tube. The three tubes were then extracted as in Section (3.4.). No significant breakthrough to the backup section was observed. The average retention efficiency for these two filters was 88.3%.

Table 2.5
Retention Efficiency

sample #	µg spiked	µg recovered	% recovered
1	134.8	127.8	94.8
2	134.8	127.8	94.8
3	127.6	96.2	75.4

average = 88.3%

2.6 Three OVS-7 tubes were spiked with varying amounts of caprolactam as above. One hundred liters of humid air (>70% RH) were drawn through each tube. The tubes were stored for seven days at ambient temperature in a drawer. These were extracted as in Section (3.4). No caprolactam was recovered from the backup section of these tubes. The average recovery after seven days of storage was 97.5%.

Table 2.6
Ambient Storage

sample #	µg spiked	µg recovered	% recovered
1	127.6	112.9	88.5
2	134.8	134.8	100
3	255.2	265.4	104

average = 97.5%

2.7 Recommended air volume and sampling rate

2.7.1 The recommended air volume is 100 L.

2.7.2 The recommended flow rate is 1.0 L/min.

2.8 Interferences

It is not known if any compounds will interfere with the collection of caprolactam. Suspected interferences should be reported to the laboratory with submitted samples.

2.9 Safety precautions

2.9.1 Attach sampling equipment in such a manner that it will not interfere with work performance or 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 high-performance liquid chromatograph equipped with a UV detector, and manual or automatic injector. A Waters M6000A pump, Waters 710B autosampler, and Waters 490 UV variable wavelength detector was used in this evaluation.

3.1.2 An HPLC column capable of separating caprolactam from any interference. A 25 cm × 4.6 mm i.d. LC-18DB (5 µm) Supelco column was used in this evaluation.

3.1.3 An electronic integrator or other suitable means of measuring detector response. A Hewlett-Packard 3357 data system was used in this evaluation.

3.1.4 Vials, 4-mL glass with PTFE-lined septa.

3.1.5 Volumetric flasks, pipettes, and syringes for preparing standards, making dilutions, and performing injections.

3.2 Reagents

3.2.1 HPLC grade methanol.

3.2.2 HPLC grade water. A Millipore Milli-Q system was used to prepare the water for this evaluation.

3.2.3 Caprolactam, reagent grade.

3.3 Standard preparation

Stock standard solutions are prepared by adding methanol to pre-weighed amounts of caprolactam. Working range standard solutions are prepared by diluting stock solutions with methanol. Stock and dilute standards are stored in a freezer.

3.4 Sample preparation

3.4.1 Transfer each section, the 13-mm glass fiber filter, the 270-mg section, and the 140-mg section, into separate vials. Use a small glass funnel to facilitate the transfer of the adsorbent. Discard the first and rear foam plug. Do not discard the glass sampling tube; it can be reused after it has been cleaned with surfactant or suitable solvent.

3.4.2 Add 4.0 mL of methanol to each of the three vials.

3.4.3 Seal the vials with PTFE-lined septa and allow them to extract for one hour. Shake the vials by hand periodically during the one-hour extraction time.

3.5 Analysis

3.5.1 Instrument conditions

Column:	25-cm × 4.6-mm i.d. stainless steel column, packed with 5 µm LC-B18
Mobile Phase:	25/75 methanol/water (v/v)
Flow rate:	1.1 mL/min
UV detector:	218 nm
Retention time:	5.5 min
Injection volume:	10 µL

3.5.2 Chromatogram (See Figure 2)

3.6 Interferences (analytical)

3.6.1 Any collected compound that has the same retention time as caprolactam and absorbs at 210 and 218 nm is interference. Generally, chromatographic conditions can be altered to separate interference from the analyte.

3.6.2 Retention time on a single column is not proof of chemical identity. Analysis by an alternate HPLC column, detection at another wavelength, comparison of absorbance response ratios, and confirmation by mass spectrometry are additional means of identification.

3.7 Calculations

3.7.1 A calibration curve is constructed by plotting detector response versus standard concentration.

3.7.2 The concentration of caprolactam in a sample is determined from the calibration curve. If caprolactam is found on the backup section, it is added to the amount found on the front section. Blank corrections for each section should be performed before adding the results together.

3.7.3 The air concentration is then determined by the following formula.

$$mg / m^3 = \frac{(\mu g / mL \text{ in sample})(\text{extraction volume, mL})}{(\text{air volume, L})(\text{extraction efficiency, decimal})}$$

3.8 Safety precautions

3.8.1 Avoid exposure to all standards.

3.8.2 Avoid exposure to all solvents.

3.8.3 Wear safety glasses at all times.

4 Recommendations for Further Study

This method should be fully validated.

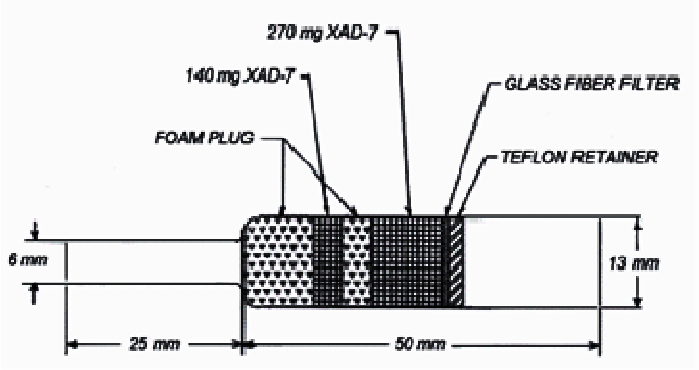


Figure 1. OVS-7 Sampling Device

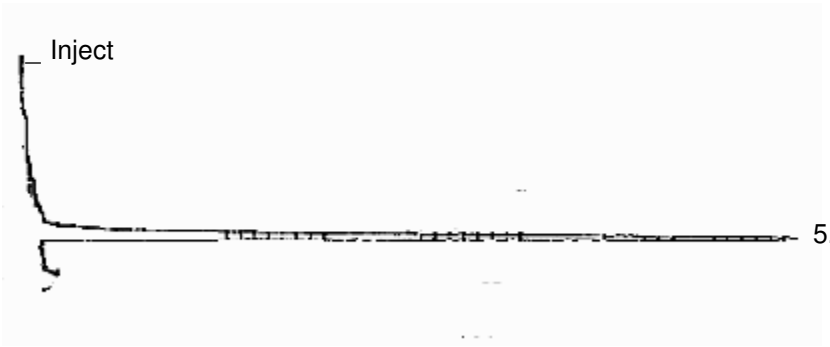


Figure 2. Chromatogram of Caprolactam

5 References

5.1 Burright, D., Method #63, "Carbaryl (Sevin)", OSHA Analytical Laboratory, unpublished, 1987.

5.2 "Registry of Toxic Effects of Chemical Substances," 1983-4, Cumulative Supplement to the 1981-2 Edition; U.S. Department of Health Services, National Institute for Occupational Safety and Health: 1985; volume 1; DHHS (NIOSH) Publ. (U.S.) No. 86-103, p. 356.

5.3 "The Condensed Chemical Dictionary", Tenth ed., Van Nostrand Reinhold Co., p.191, 1983.