n-Amyl Acetate
Isoamyl Acetate

Method no: PV2142

Control no.: T-PV2142-01-0502-CH

Target concentration: 100 ppm (525 mg/m^3) (n-amyl acetate)
100 ppm (525 mg/m^3) (isoamyl acetate)
OSHA PEL: 100 ppm (525 mg/m^3) (n-amyl acetate)
100 ppm (525 mg/m^3) (isoamyl acetate)
ACGIH TLV: 50 ppm (TWA) 100 ppm (STEL) (all isomers of pentyl acetate)

Procedure: Samples are collected by drawing workplace air through glass sampling tubes containing coconut shell charcoal with personal sampling pumps. Samples are extracted with a mixture of 99:1 carbon disulfide:*N,N*-dimethylformamide and analyzed by GC using a flame ionization detector (GC/FID).

Recommended sampling time and sampling rate: 240 min at 0.1 L/min (24 L)

Reliable quantitation limit: 10 ppb (55 µg/m^3) n-amyl acetate
15 ppb (80 µg/m^3) isoamyl acetate

Status of method: Partially validated method. This method has been subjected to established evaluation procedures of the Methods Development Team and is presented for information and trial use.

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1. General Discussion

For assistance with accessibility problems in using figures and illustrations presented in this method, please contact OSHA Salt Lake Technical Center (OSHA SLTC) at (801) 233-4900.

1.1 Background

1.1.1 History

Air samples collected using coconut shell charcoal tubes were received at OSHA SLTC with requested analysis for n-amyl acetate and isoamyl acetate. These two compounds are isomers and often occur together in the same workplace, under the name of amyl acetate. The samples were analyzed following NIOSH methods S45 isoamyl acetate[1] and S51 n-amyl acetate[2]. There were concerns from OSHA industrial hygienists that they could smell n-amyl acetate and isoamyl acetate in the workplace, and yet the results reported from SLTC were below the PEL. The NIOSH methods did not include humid air retention studies or storage stability studies. This work was performed to determine if there was a problem with retention on charcoal sampling tubes and/or with storage. After the storage and retention studies were completed, NIOSH published a revised method that included the additional studies, Method 1450 Esters 1.[3] The breakthrough study using dry air was 34.3 L (871 mg/m^3 atmosphere) for n-amyl acetate and 32.3 L (874 mg/m^3 atmosphere) for isoamyl acetate. The storage study was performed with a loading of 140 µg on dry tubes with storage at 4 °C. Most workplaces have some humidity in the air, and water may affect the collection and storage, so it was decided to complete the work for this method. The retention and storage studies showed good recoveries, so the low results on field samples versus the expectation of high results were due to the strong odor of these compounds. The odor is of bananas or pears, and the odor threshold is 0.051 ppm for n-amyl acetate and 0.0034 ppm for isoamyl acetate.

A special thanks to Gene Brownson who did most of the analyses on the tests performed in this evaluation.

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

n-Amyl acetate and isoamyl acetate are moderately toxic by intraperitoneal administration and are contact irritants affecting the skin, eyes, and respiratory system. Skin contact may cause dermatitis, higher exposure causes necrosis.[4,5] Eye contact with high concentrations may cause temporary impairment of vision and/or transient eye inflammation and ulceration. Inhalation of high concentrations can cause lung irritation, coughing, nausea, central nervous depression resulting in headache and dizziness, slowing of reflexes, fatigue and lack of coordination. Liver and kidney damage may result from massive exposures.[6,7] Isoamyl acetate has been approved by

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[1] *NIOSH Manual of Analytical* Methods, 2nd ed.; Cincinnati, OH, Vol. 2, Method S45.

[2] *NIOSH Manual of Analytical* Methods, 2nd ed.; Cincinnati, OH, Vol. 2, Method S51.

[3] NIOSH Method 1450 Esters 1, www.cdc.gov/niosh, (accessed 5/15/03).

[4] Lewis, R., Ed., *Sax's Dangerous Properties of Industrial Materials,* Tenth ed.; John Wiley & Sons Inc.: New York, 2000, Vol. 3, p 249.

[5] Lewis, R., Ed., *Sax's Dangerous Properties of Industrial Materials,* Tenth ed.; John Wiley & Sons Inc.: New York, 2000, Vol. 3, p 2119.

[6] Material Safety Data Sheet: n-Amyl acetate, Chemwatch, Victoria, Australia, (accessed 2/17/03).

[7] Material Safety Data Sheet: Isoamyl acetate, Chemwatch, Victoria, Australia, (accessed 2/17/03).

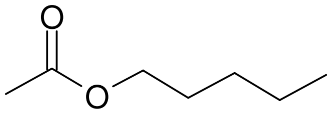
FDA for use as a food additive providing "they are used in the minimum quantity required to produce their intended effect."[8]

1.1.3 Workplace exposure[9,10]

n-Amyl acetate and isoamyl acetate are used in cements and glues, paper coatings, lacquers and paints, leather finishes, flavoring, perfume, nail enamels, plastic wood, textile sizing and finishes, printing compounds, and photographic film. They are used in extraction of penicillin, as a warning odor, as a solvent for nitrocellulose and ethyl cellulose, and as a solvent for phosphors in fluorescent lamps.

1.1.4 Physical properties and other descriptive information

n-amyl acetate[11,12]

CAS number:	628-63-7
synonyms:	acetic acid, amyl ester; amyl acetic ester; birnenoel; pear oil; pent-acetate; 1-pentanol acetate; pentyl acetate; n-pentyl acetate; 1-pentyl acetate; primary amyl acetate
IMIS:[13]	0190
RTECS:	AJ1925000
molecular weight:	130.19
density (g/mL):	0.879 20/20 °C
melting point:	-71 °C
boiling point:	148 °C
appearance:	clear liquid
flash point:	25 °C (77 °F) (closed cup)
odor:	pear- or banana-like
molecular formula:	$C_7H_{14}O_2$
solubility:	very slightly soluble in water, miscible in alcohol and ether
autoignition temperature:	379 °C (714 °F)
lower explosive limit:	1.1%
upper explosive limit:	7.5%
structural formula:	

isoamyl acetate[14,15]

CAS number:	123-92-2
synonyms:	acetic acid, isoamyl ester; acetic acid, isopentyl ester; banana oil; isoamyl acetate; iso-amylacetate; isoamyl alcohol, acetate;

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[8] Title 21, Part 172, Section 172.515, Synthetic Flavorings Substances and Adjuvants, www.fda.gov, (accessed 3/17/03).

[9] Lewis, R., Ed., *Hawley's Condensed Chemical Dictionary,* 14th ed., John Wiley & Sons Inc., New York, 2001, p 73.

[10] Lewis, R., Ed., *Hawley's Condensed Chemical Dictionary,* 14th ed., John Wiley & Sons Inc., New York, 2001, p 649.

[11] Material Safety Data Sheet: n-Amyl acetate, Chemwatch, Victoria, Australia, (accessed 2/17/03).

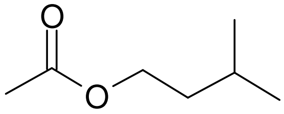
[12] Lewis, R., Ed., *Sax's Dangerous Properties of Industrial Materials,* Tenth ed., Vol. 3, John Wiley & Sons Inc., New York, 2000, p 249.

[13] OSHA Chemical sampling Information, www.osha.gov (accessed 12/17/03).

[14] Material Safety Data Sheet: Isoamyl acetate, Chemwatch, Victoria, Australia, (accessed 2/17/03).

[15] O'Neil, M., Ed., *The Merck Index*, 13th ed., Merck & Co., Inc., Whitehouse Station, NJ, 2001, p 919.

	isopentyl acetate; isopentyl alcohol, acetate;3-methyl-1-butanol acetate; 3-methylbutyl acetate; 3-methyl 1-butylacetate; 3-methylbutyl ethanoate; pear oil; pent-acetate
IMIS:[16]	1530
RTECS:	NS9800000
molecular weight:	130.19
density (g/mL):	0.870 20/20 °C
melting point:	-71 °C
boiling point:	142 °C
lower explosive limit:	1.0%[17]
upper explosive limit:	7.5%[18]
appearance:	clear liquid
flash point:	33 °C (92 °F) (closed cup); 38 °C (100 °F) (open cup);
odor:	banana- or pear like
molecular formula:	$C_7H_{14}O_2$
solubility:	slightly soluble in water (400 parts in water), miscible with alcohol, ether, ethyl acetate, amyl alcohol,
structural formula:	



This method was evaluated according to the OSHA SLTC "Evaluation Guidelines for Air Sampling Methods Utilizing Chromatographic Analysis"[19]. The Guidelines define analytical parameters, specify required laboratory tests, statistical calculations and acceptance criteria. The analyte air concentrations throughout this method are based on the recommended sampling and analytical parameters. Air concentrations in ppm are referenced to 25 °C and 101.3 kPa (760 mmHg).

1.2 Detection limit of the overall procedure (DLOP) and reliable quantitation limit (RQL)

The DLOP is measured as mass per sample and expressed as equivalent air concentration, based on the recommended sampling parameters. Ten samplers were spiked with equal descending increments of analyte, such that the highest sampler loading was 9.64 µg of n-amyl acetate and 9.57 µg of isoamyl acetate. This is the amount spiked on a sampler that would produce a peak at least 10 times the response for a sample blank. These spiked samplers were analyzed with the recommended analytical parameters, and the data obtained used to calculate the required parameters [standard error of estimate (SEE) and slope] for the calculation of the DLOP. The slope was 700 and the SEE was 93.0 for n-amyl acetate. The slope was 776 and the SEE was 149.5 for isoamyl acetate. The DLOP was 0.40 µg for n-amyl acetate, and 0.57 µg for isoamyl acetate.

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[16] OSHA Chemical sampling Information, www.osha.gov (accessed 12/17/03).

[17] Isoamyl acetate, NIOSH Pocket Guide to Chemical Hazards, www.cdc.gov/niosh (accessed 12/17/03).

[18] Isoamyl acetate, NIOSH Pocket Guide to Chemical Hazards, www.cdc.gov/niosh (accessed 12/17/03).

[19] *Evaluation Guidelines For Air Sampling Methods Utilizing Chromatographic Analysis*; www.osha.gov, (accessed 11/15/03).

Table 1.2.1
Detection Limit of the Overall Procedure for
n-Amyl Acetate

mass per sample (µg)	area counts (µV·s)
0.00	0
0.96	715
1.93	1446
2.89	2260
3.85	2962
4.82	3505
5.78	4180
6.75	4788
7.71	5428
8.67	6086
9.64	6902

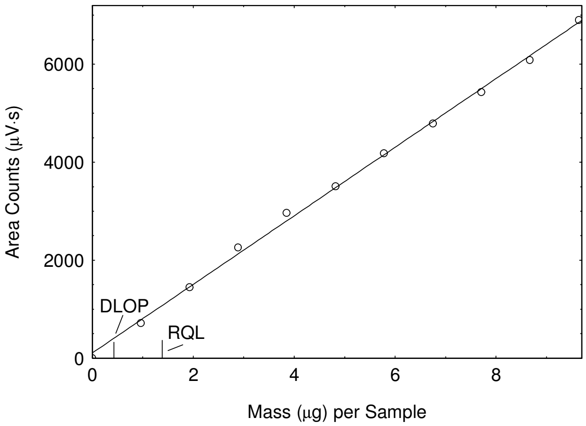

Figure 1.2.1 Plot of data to determine the DLOP/RQL of n-amyl acetate. (y = 700x + 106)

Table 1.2.2
Detection Limit of the Overall Procedure for
Isoamyl Acetate

mass per sample (µg)	area counts (µV-s)
0.00	0
0.96	810
1.91	1517
2.87	2446
3.83	3064
4.79	3636
5.74	4343
6.70	5001
7.66	5894
8.61	6723
9.57	7713

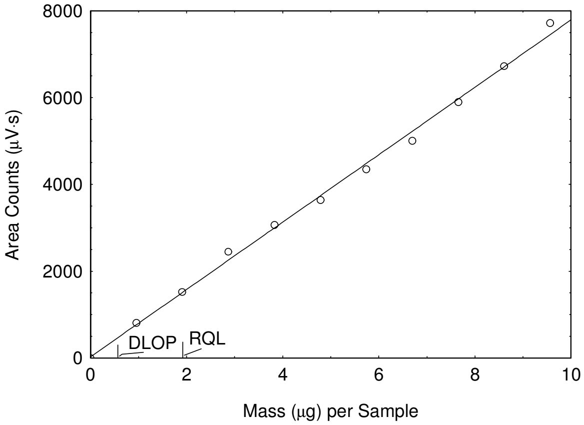

Figure 1.2.2 Plot of data to determine the DLOP/RQL of isoamyl acetate. (y = 776x + 25.6)

The RQL is considered the lower limit for precise quantitative measurements. It is determined from the regression line parameters obtained for the calculation of the DLOP, providing 75% to 125% of the analyte is recovered. The RQL was 1.33 µg/sample (10 ppb, 55 µg/m^3) for n-amyl acetate and 1.91 µg/sample (15 ppb, 80 µg/m^3) for isoamyl acetate. Recovery at this concentration was 99.6% for n-amyl acetate, and 100.5% for isoamyl acetate at this level.

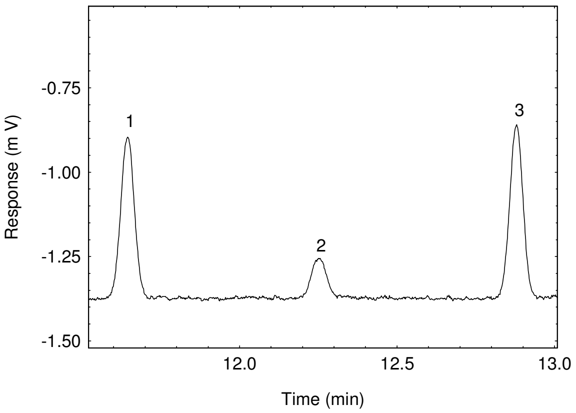

Figure 1.2.2 Chromatogram of the analytes at levels near the RQL amounts. [Key: (1) isoamyl acetate, (2) interferant, and (3) n-amyl acetate]

2. Sampling Procedure

All safety practices that apply to the work area being sampled should be followed. The sampling equipment should be attached to the worker in such a manner that it will not interfere with work performance or safety.

2.1 Apparatus

Samples are collected using a personal sampling pump calibrated, with the sampling device attached, to within ±5% of the recommended flow rate.

Samples are collected with 7-cm × 4-mm i.d. × 7-mm o.d. glass sampling tubes packed with two sections (100/50 mg) of coconut shell charcoal. The sections are held in place with foam plugs and with a glass wool plug at the front. For this evaluation, commercially prepared sampling tubes were purchased from SKC, Inc. (Catalog no. 226-01, lot 2000).

2.2 Reagents

None required

2.3 Technique

Immediately before sampling, break off the ends of the flame-sealed tube to provide an opening approximately half the internal diameter of the tube. Wear eye protection when breaking the tube. Use tube holders to minimize the hazard of broken glass. All tubes should be from the same lot.

The smaller section of the adsorbent tube is used as a back-up and is positioned nearest the sampling pump. Attach the tube holder to the sampling pump so that the adsorbent tube is in an approximately vertical position with the inlet facing down during sampling. Position the sampling pump, tube holder and tubing so they do not impede work performance or safety.

Draw the air to be sampled directly into the inlet of the tube holder. The air being sampled is not to be passed through any hose or tubing before entering the sampling tube.

After sampling for the appropriate time, remove the adsorbent tube and seal it with plastic end caps. Seal each sample end-to-end with an OSHA-21 form as soon as possible.

Submit at least one blank sample with each set of samples. Handle the blank sample in the same manner as the other samples except draw no air through it.

Record sample air volumes (liters), sampling time (minutes), and sampling rate (L/min) for each sample, along with any potential interferences on the OSHA-91A form.

Submit the samples to the laboratory for analysis as soon as possible after sampling. If delay is unavoidable, store the samples at refrigerator temperature. Ship any bulk samples separate from the air samples.

2.4 Extraction efficiency

The extraction efficiency was determined by liquid-spiking charcoal tubes with the analyte at 0.1 to 2 times the target concentration. These samples were stored overnight at ambient temperature, then extracted for 30 minutes using a lab shaker, and analyzed. The mean extraction efficiency over the studied range was 99.5% for n-amyl acetate and 99.4% for isoamyl acetate. An additional test was performed with wet sampling medium by drawing 24 L of humid air (80% relative humidity at 23 °C) through sampling tubes before they were spiked at

1.0 times the target concentration. These samples were also stored overnight at ambient temperature before analysis. The mean recovery from wet sampling medium was 98.9% for n-amyl acetate and 99.0% for isoamyl acetate.

Table 2.4.1
Extraction Efficiency (%) of n-Amyl Acetate

level		sample number				mean
× target concn	mg per sample	1	2	3	4	
0.1	1.28	99.1	100.1	98.7	99.5	99.4
0.5	6.37	99.5	98.8	99.5	100.2	99.5
1.0	12.8	98.9	99.7	100.2	99.9	99.7
2.0	25.5	99.2	99.8	100.3	98.5	99.5
1.0 (wet)	12.8	99.1	99.6	98.7	98.2	98.9

Table 2.4.2
Extraction Efficiency (%) of Isoamyl Acetate

level		sample number				mean
× target concn	mg per sample	1	2	3	4	
0.1	1.26	99.4	98.7	100.1	99.1	99.3
0.5	6.31	99.8	99.3	99.7	99.1	99.5
1.0	12.6	98.9	98.8	99.5	100.2	99.4
2.0	25.2	100.1	98.8	99.7	99.1	99.4
1.0 (wet)	12.6	98.4	99.2	98.7	99.5	99.0

2.5 Retention efficiency

Six samplers were liquid-spiked with 25.5 mg (199.6 ppm) n-amyl acetate, allowed to equilibrate for 6 h, then they had 24 L of humid air (80% relative humidity at 23 °C) pulled through them at 0.2 L/min. The samples were extracted and analyzed. The mean recovery was 98.9%. There was an average of 2.9% found on the back-up section of the tubes.

Table 2.5.1
Retention Efficiency (%) of n-Amyl Acetate at 0.2 L/min

section	sample number						
	1	2	3	4	5	6	mean
front of spiked tube	96.9	95.7	96.1	95.1	95.5	96.8	96.0
rear of spiked tube	2.5	2.2	3.0	3.5	2.8	3.1	2.9
total	99.4	97.9	99.1	98.6	98.3	99.9	98.9

Because there was some n-amyl acetate found on the back-up portions of the tubes that were sampled at 0.2 L/min, a lower flow of 0.1 L/min was tried on six more samplers. They were liquid-spiked with 25.5 mg (199.6 ppm) n-amyl acetate, allowed to equilibrate for 6 h, then they had 24 L of humid air (80% relative humidity at 23 °C) pulled through them at 0.1 L/min. The samples were extracted and analyzed. The mean recovery was 99.2%. There was no analyte found on the back-up section of any of the tubes indicating this flow rate and air volume should be used for sampling.

Table 2.5.2
Retention Efficiency (%) of n-Amyl Acetate at 0.1 L/min

section	sample number						
	1	2	3	4	5	6	mean
front of spiked tube	98.9	99.9	99.1	98.4	99.8	99.2	99.2
rear of spiked tube	0.0	0.0	0.0	0.0	0.0	0.0	0.0
total	98.9	99.9	99.1	98.4	99.8	99.2	99.2

Six samplers were liquid-spiked with 25.2 mg (197.3 ppm) isoamyl acetate, allowed to equilibrate for 6 h, then they had 24 L of humid air (80% relative humidity at 23℃) pulled through them at 0.2 L/min. The samples were extracted and analyzed. The mean recovery was 98.6%. There was an average of 3.0% analyte found on the back-up section of the tubes.

Table 2.5.3
Retention Efficiency (%) of Isoamyl Acetate at 0.2 L/min

section	sample number						
	1	2	3	4	5	6	mean
front of spiked tube	95.4	94.9	95.1	96.4	95.5	96.1	95.6
rear of spiked tube	3.1	3.4	2.8	3.2	3.3	2.4	3.0
total	98.5	98.3	97.9	99.6	98.8	98.5	98.6

Because there was some isoamyl acetate found on the back-up portions of the tubes that were sampled at 0.2 L/min, a lower flow of 0.1 L/min was tried on six more samplers. Six samplers were liquid-spiked with 25.2 mg (198.3 ppm) isoamyl acetate, allowed to equilibrate for 6 h, then they had 24 L of humid air (80% relative humidity at 23℃) pulled through them at 0.1 L/min. The samples were extracted and analyzed. The mean recovery was 98.9%. There was no analyte found on the back-up section of any of the tubes indicating this flow rate and air volume should be used for sampling.

Table 2.5.4
Retention Efficiency (%) of Isoamyl Acetate at 0.1 L/min

section	sample number						
	1	2	3	4	5	6	mean
front of spiked tube	99.2	99.9	98.3	97.9	99.4	98.8	98.9
rear of spiked tube	0.0	0.0	0.0	0.0	0.0	0.0	0.0
total	99.2	99.9	98.3	97.9	99.4	98.8	98.9

2.6 Sample storage

Fifteen samplers were each spiked with 12.8 mg (100 ppm) of n-amyl acetate, then they had 24 L at 0.1 L/min of humid air (80% relative humidity at 23℃) drawn through them. Three samples were analyzed immediately, and the rest were sealed. Six were stored at room temperature (23℃), while the other six were stored at refrigerated temperature (4℃). Three samples stored at room temperature and three samples stored at refrigerated temperature were analyzed after 7 days and the remaining six after 14 days. The amounts recovered were not corrected for extraction efficiency indicates good storage stability for the time period studied. No analyte was found on the back-up section of any of the sampling tubes.

Table 2.6.1
Storage Test for n-Amyl Acetate

time (days)	ambient storage recovery (%)			Refrigerated storage recovery (%)		
0	99.3	99.9	98.8			
7	97.8	99.7	98.9	99.9	99.5	98.7
14	99.9	99.4	99.8	99.8	100.8	99.2

Fifteen samplers were each spiked with 12.6 mg (98.6 ppm) of isoamyl acetate, then they had 24 L at 0.1 L/min of humid air (80% relative humidity at 23℃) drawn through them. Three samples were analyzed immediately, and the rest were sealed. Six were stored at room temperature (23℃), while the other six were stored at refrigerated temperature (4℃). Three samples stored at room temperature and three samples stored at refrigerated temperature were analyzed after 7 days and the remaining six after 14 days. The amounts recovered were not corrected for extraction efficiency and indicate good storage stability for the time period studied. There was no analyte found on the back-up section of any of the sampling tubes.

Table 2.6.2
Storage Test for Isoamyl Acetate

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
0	99.8	98.6	99.0			
7	98.8	99.5	98.3	99.2	98.9	99.9
14	97.3	98.7	98.6	99.4	99.7	98.7

2.7 Recommended air volume and sampling rate

Based on the data collected in this evaluation, 24-L air samples should be collected at a sampling rate of 0.1 L/min for 240 minutes.

3. Analytical Procedure

Adhere to the rules set down in your Chemical Hygiene Plan. Avoid skin contact and inhalation of all chemicals and review all appropriate MSDSs.

3.1 Apparatus

A gas chromatograph equipped with an FID detector. For this evaluation, an Agilent 6890 GC was used.

A GC column capable of separating n-amyl acetate and isoamyl acetate from the extraction solvent, internal standard, and any potential interferences. A 60-m × 0.32-mm i.d. capillary column, DB-Wax 0.5-µm df (J&W Scientific, Folsom, CA) was used in this evaluation.

An electronic integrator or some other suitable means of measuring peak areas. A Waters Millennium^{32} Data System and an Agilent 3396 integrator were used in this evaluation.

Glass vials with poly(tetrafluoroethylene)-lined caps. For this evaluation 2-mL vials were used.

A dispenser capable of delivering 1.0 mL of extraction solvent to prepare standards and samples. If a dispenser is not available, a 1.0-mL volumetric pipet may be used.

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

Calibrated 10-µL or 25-µL syringe for preparing standards. A Hamilton 25-µL syringe was used for this evaluation.

A shaker or rotator to agitate samples during extraction. An Eberbach mechanical shaker was used in this evaluation.

3.2 Reagents

n-Amyl acetate, reagent grade. Acros 99% (lot A011333301) was used in this evaluation.

Isoamyl acetate, reagent grade. Aldrich 99% (lot MS00852AR) was used in this evaluation.

Carbon disulfide, reagent grade. EM Science Omni-Solv 99.99% (lot 43279343) was used in this evaluation.

N,N-Dimethylformamide (DMF), anhydrous reagent grade. Aldrich 99% (lot 11703TR) was used in this evaluation.

p-Cymene, reagent grade. Aldrich 99% (lot 11703TR) was used in this evaluation.

The extraction solvent solution was carbon disulfide:*N,N*-dimethylformamide (99:1) with 0.25µL/mL of *p*-cymene as internal standard.

3.3 Standard preparation

Prepare standards by spiking microliter quantities of n-amyl acetate and isoamyl acetate from a microliter syringe into 2-mL vials, each containing 1 mL of the extraction solution. For example, 14 µL of n-amyl acetate in 1-mL extraction solvent is equivalent to 12.3 mg/mL and 14µL of isoamyl acetate in 1-mL extraction solvent is equivalent to 12.2 mg/mL. Standards at lower concentrations were prepared using microliter injections into volumetric flasks containing the extraction solvent, or pipet dilutions of analytical standards using volumetric flasks and the extraction solvent. For this evaluation, standards in the range of 0.001 to 25.5 mg/mL for n-amyl acetate and 0.001 to 25.2 mg/mL for isoamyl acetate were used. An additional standard from a second source should be prepared to check the calibration.

Bracket sample concentrations with standard concentrations. If, upon analysis, sample concentrations fall outside the range of prepared standards, prepare and analyze additional standards to confirm instrument response, or dilute high samples with extraction solvent and reanalyze the diluted samples.

3.4 Sample preparation

Remove the plastic end caps from the sample tubes and carefully transfer each adsorbent section to separate 2-mL vials. Discard the glass tube, urethane foam plug and glass wool plug.

Add 1.0 mL of extraction solvent to each vial using the same dispenser as used for preparation of standards.

Immediately seal the vials with poly(tetrafluoroethylene)-lined caps, and shake the vials on a shaker for 30 minutes.

3.5 Analysis

Gas chromatographic conditions

GC conditions

Temperatures:
column: initial 50 °C, hold 1 min, ramp at 5 °C/min to 150 °C, hold 5 min
injector: 200 °C
detector: 250 °C

run time: 26 min
column gas flow: 1.2 mL/min (hydrogen)
injection size: 1.0 μL (10:1 split)
column: 60-m × 0.32-mm i.d. capillary DB-Wax (df = 0.5 μm)

FID conditions
hydrogen flow: 30 mL/min
air flow: 400 mL/min
nitrogen makeup flow: 25 mL/min

retention times: 5.6 min (CS_2); 8.1 min (benzene contaminant in CS_2); 11.6 min (isoamyl acetate); 12.9 min (n-amyl acetate); 15.7 min (*p*-cymene): 17.4 min (DMF)

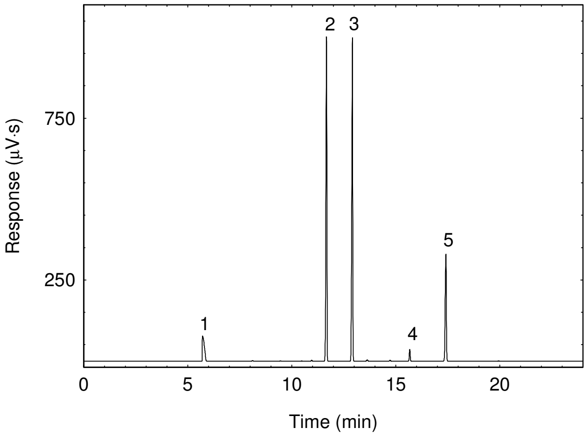

Figure 3.5.1 A chromatogram of 12.6 mg/mL isoamyl acetate and 12.8 mg/mL n-amyl acetate in 99:1 CS_2:DMF with 0.25 μl/mL *p*-cymene as internal standard. [Key: (1) CS_2; (2) isoamyl acetate; (3) n-amyl acetate; (4) *p*-cymene; and (5) DMF]

Peak areas are measured with an integrator or other suitable means.

An internal standard (ISTD) calibration method is used. A calibration curve can be constructed by plotting internal standard corrected response of standard injections versus micrograms of analyte per sample. Bracket the samples with freshly prepared analytical standards over the range of concentrations.

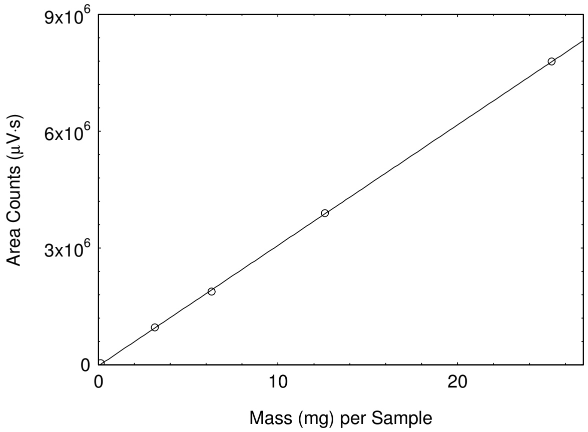

Figure 3.5.2 Calibration curve of n-amyl acetate. (y = 3.05E5x – 6720)

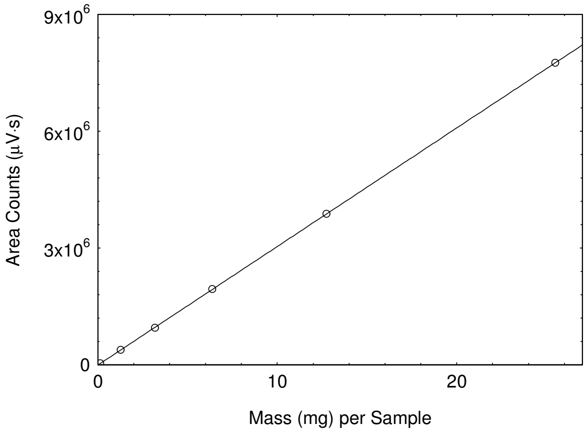

Figure 3.5.3 Calibration curve of isoamyl acetate. (y = 3.09E5x – 2.57E4)

3.6 Interferences (analytical)

Any compound that produces a GC response and has a similar retention time as the analyte is a potential interference. If any potential interferences were reported, they should be considered before samples are extracted. Generally, chromatographic conditions can be altered to separate an interference from the analyte.

When necessary, the identity or purity of an analyte peak may be confirmed by additional analytical data. The mass spectra in Figures 3.6.1 and 3.6.2 were obtained from the NIST spectral library.

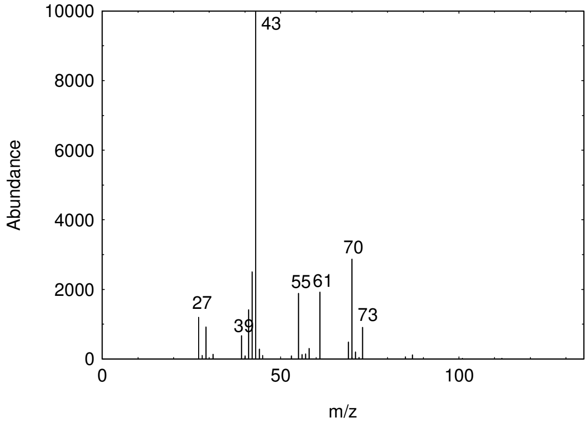

Figure 3.6.1 Mass spectrum of n-amyl acetate.

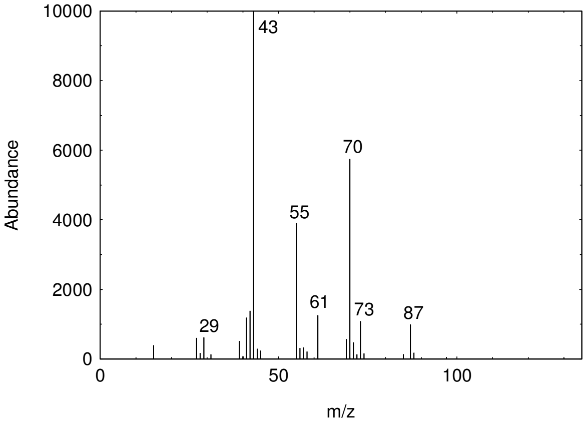

Figure 3.6.2 Mass spectrum of isoamyl acetate.

3.7 Calculations

The amount of analyte per sampler is obtained from the appropriate calibration curve in terms of micrograms per sample, uncorrected for extraction efficiency. This total amount is then corrected by subtracting the total amount (if any) found on the blank. The air concentration is calculated using the following formulas.

$$C_M = \frac{M}{VE_E}$$

where: C_M is concentration by weight
M is micrograms per sample
V is liters of air sampled
E_E is extraction efficiency, in decimal form

$$C_V = \frac{V_M C_M}{M_r}$$

where: C_V is concentration by volume (ppm)
V_M is 24.46 (molar volume at NTP)
C_M is concentration by weight
M_r is molecular weight = 130.19

4. Recommendations for Further Study

A sampler capacity test should be performed with a test atmosphere to better determine sampler capacity.