1-NITROPROPANE
2-NITROPROPANE

Method no.:	46
Matrix:	Air
Target concentration:	25 ppm (91 mg/m^3) (OSHA PEL for both analytes)
Procedure:	Samples are collected by drawing known volumes of air through commercially available XAD-4 sampling tubes. Samples are desorbed with carbon disulfide and analyzed by gas chromatography using a flame ionization detector.
Recommended air volume and sampling rate:	4 L at 0.1 L/min
Reliable quantitation limit:	25 ppb (91 µg/m^3) for both analytes
Standard errors of estimate at the target concentration: (Section 4.4)	1-Nitropropane, 7.2%; 2-Nitropropane, 6.2%
Special requirements:	After samples are received at the laboratory, they should be stored under refrigeration, until analyzed, to help minimize migration.
Status of method:	Evaluated method. This method has been subjected to the established evaluation procedures of the Organic Methods Evaluation Branch.

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1. General Discussion

1.1. Background

1.1.1. History

In the past there were no validated sampling and analytical procedures for determining 1-nitropropane (1-NP) in air. Samples have been received at the OSHA laboratory that had been collected on charcoal (coconut shell) sampling tubes. Studies were done by the service branch to find a suitable solvent to desorb 1-NP from charcoal. Carbon disulfide, which is used to successfully desorb a wide range of solvents from charcoal, desorbed less than 50% of 1 mg of 1-NP from charcoal. Attempts were made to desorb 1 mg of 1-NP from charcoal with other commonly used desorption solvents. The solvents tried and the desorption efficiencies obtained were: methanol - 55 to 66%, isopropanol - 30 to 33%, 95% methylene chloride/ 5% isopropanol - 71 to 76%, 95% carbon disulfide/ 5% isopropanol - 73%, and methylene chloride - 53%. Due to these low desorption efficiencies, solid sorbents other than charcoal were investigated.

Since there is a validated sampling and analytical procedure for 2-NP that requires collection on Chromosorb 106 and desorption with ethyl acetate (Ref. 5.1.), desorption efficiency studies were done for 1-NP accordingly. Desorption efficiencies appeared to be high (>95%), but diethylbenzene isomers were also desorbed from the samples which proved to be interferences using the GC column recommended in the method. The desorbed diethylbenzene is apparently a contaminant from the Chromosorb 106 tubes used. The isomers were not desorbed from a blank sample, thus for this study, 1-NP had to be present for the isomers to be desorbed with ethyl acetate. The aforementioned validated method for 2-NP required the Chromosorb 106 to be thermally desorbed to clean the resin before collection of air samples. This cleanup procedure would probably rid the resin of the interfering isomers, but it is inconvenient and there is some question of whether thermally desorbed tubes would always be used to collect samples, i.e. the industrial hygienist may use the sampling tubes as received from the supplier. Thus, alternate sampling media were considered for collection of both 1-NP and 2-NP.

Breakthrough studies were performed with XAD-4 and XAD-7 sampling tubes (SKC West, Inc., Fullerton, CA) by sampling an atmosphere of approximately 50 ppm 1-NP (in 80% RH air, 23°C, 656 mm Hg) at 0.2 L/min and monitoring the downstream effluent with a total hydrocarbon analyzer. Only the front sampling section was used in the tubes. The 5% breakthrough volumes were about 5 to 5.5 L for XAD-4 and 3 L for XAD-7, thus XAD-4 was chosen as a possible sampler. Since a flow rate of 0.2 L/min would allow for a maximum sampling period of less than 20 min, breakthroughs were done at a flow of 0.1 L/min. Breakthroughs thus determined for both 1-NP and 2-NP allow for a recommended sample volume of 4 L collected at 0.1 L/min (40 min).

Desorption efficiencies were determined for both 1-NP and 2-NP from XAD-4 using ethyl acetate and carbon disulfide. Both solvents gave high desorptions, but carbon disulfide was preferred since it responds much less than ethyl acetate on a flame ionization detector, thus lower detection limits are obtained when carbon disulfide is used. It was found that a small amount of toluene is desorbed from samples containing 1-NP and/or 2-NP. The desorbed toluene is apparently a contaminant from the XAD-4 tubes used. The toluene is not desorbed from a blank XAD-4 sample. The small amount desorbed from samples proved to be insignificant.

The collection of 1-NP and 2-NP on XAD-4 sampling tubes and desorption with carbon disulfide were successfully evaluated. The only special requirement is samples should be stored (not necessarily shipped) under refrigeration to reduce migration of the analytes to the backup section before analysis. After storing 1-NP samples for 15 days, there was a migration of 0.5 to 2.5% for refrigerated samples and 11 to 15% for ambient samples. Similarly for 2-NP, migrations of 3 to 5% for refrigerated samples and 20 to 25% for ambient samples were found.

1.1.2. Toxic effects. (The section for 1-NP is quoted from "Occupational Health Guidelines for Chemical Hazards", Ref. 5.2. and the section for 2-NP is taken from OSHA Method 15 for 2-NP, Ref. 5.1. These sections are for information only and should not be taken as the basis of OSHA policy.)

1.1.2.1. 1-Nitropropane

"1-Nitropropane vapor is an eye irritant and in animals causes mild respiratory irritation and severe liver damage. Rabbits died from exposure to 5,000 ppm for 3 h, but 10,000 ppm for 1 h was not lethal. Effects were conjunctival irritation, lacrimation, slow respiration with some rales, muscular incoordination, ataxia, and weakness. Autopsy of animals exposed to lethal concentrations revealed severe fatty infiltration of the liver and moderate kidney damage. Human volunteers exposed to over 100 ppm noted eye irritation. There are no reports of systemic effects in humans."

1.1.2.2. 2-Nitropropane

"Exposure to concentrations of 2-nitropropane on the order of 20 to 45 ppm are reported to cause in humans nausea, vomiting, diarrhea, loss of appetite, and severe headaches and concentrations of 165 to 445 ppm, nausea, dizziness, headaches, and diarrhea. Cats which died within 17 days of exposure to 328 ppm 2-nitropropane sustained severe liver damage. Liver damage attributed to high concentrations of 2-nitropropane in humans has been reported. Liver cancer was found in rats exposed to 207 ppm 2-nitropropane for 6 months. This data raises the question of carcinogenic effects in humans. While additional studies are in progress, NIOSH believes it would be prudent to handle 2-nitropropane as if it were a human carcinogen."

1.1.3. Potential workplace exposure

Following are some common operations where exposure to 1-NP may occur as reported in "Occupational Health Guidelines for Chemical Hazards" (Ref. 5.2.) and also where exposure to 2-NP may occur as reported in OSHA method 15 (Ref. 5.1.).

1.1.3.1. 1-Nitropropane

1-Nitropropane is used:

as a thinner and solvent for cellulose compounds, lacquers, and dopes; in vinyl resins for industrial coatings and printing inks ;in synthetic finish removers; and for oil and spirit-soluble dyes of molded plastics.

as an extraction solvent for purification, separation, recrystallization, and recovery for natural and synthetic resins, tars, coating materials, fats, and oils.

as a reaction medium in polymer technology, as a catalyst, initiator, and solvent.

in organic chemical synthesis for preparation of amines, nitrated alcohols, acids, and chloronitroparaffins.

in manufacture of explosives.

1.1.3.2. 2-Nitropropane

"Solvent systems containing 2-nitropropane are used in coatings (e.g., vinyl, epoxy, nitrocellulose, and chlorinated rubber), printing inks, and adhesives. Occupational exposure to these products may occur in various industries including industrial construction and maintenance, printing (rotogravure and flexographic inks), highway maintenance (traffic markings), shipbuilding and maintenance (marine coatings), furniture, food packaging, and plastic products."

1.1.4. Physical properties (Ref. 5.3. unless otherwise noted)

	1-NP	2-NP
molecular weight:	89.09	89.09
boiling pt., 760 mm Hg:	131.6°C	120.3°C
color:	colorless	colorless
density (25/4°C):	0.99	0.9821
vapor pressure, 20°C:	7.5 mm (Ref. 5.2.)	12.9 (Ref.5.1.)
flash pt., closed cup:	34°C	24°C
odor:	mild, fruity odor (Ref. 5.2.)	
flammable limits in air, % by volume (lower):	2.2 (Ref. 5.2.)	2.6 (Ref. 5.1.)
autoignition temp:	420.6°C (Ref. 5.2.)	
formula:	$CH_3CH_2CH_2NO_2$	$CH_3CH(NO_2)CH_3$
synonyms:	none (Ref. 5.2.)	dimethylnitro-methane; isonitropropane; nitroisopropane and 2-NP (Ref. 5.1.)

1.2. Limit defining parameters (The analyte air concentrations listed throughout this method are based on an air volume of 4 L and solvent desorption volume of 1.0 mL. The ppb and ppm values are referenced to an atmospheric pressure of 760 mm Hg and temperature of 25°C.)

1.2.1. Detection limit of the analytical procedure

The detection limit of the analytical procedure is 0.4 ng for both 1-NP and 2-NP. This is the amount of analytes which will give peaks whose heights are approximately 5 times the height of baseline noise. (Section 4.1.)

1.2.2. The detection limit of the overall procedure is 0.4 µg per sample (25 ppb or 91 µg/m^3) for both 1-NP and 2-NP. This is the amount of analytes spiked on the sampling device which allow recoveries approximately equivalent to the detection limits of the analytical procedure. (Section 4.2.)

1.2.3. The reliable quantitation limit is 0.4 µg per sample (25 ppb or 91 µg/m^3) for both 1-NP and 2-NP. This is the smallest amount of analytes which can be quantitated within the requirements of recoveries of at least 75% and a precision (1.96 SD) of ±25% or better. (Section 4.2.)

The reliable quantitation limit and detection limits reported in the method are based upon optimization of the instrument for the smallest possible amount of analyte. When the target concentration of an analyte is exceptionally higher than these limits, they may not be attainable at the routine operating parameters.

1.2.4. Sensitivity

The sensitivities of the analytical procedure over a concentration range representing 0.5 to 2 times the target concentration based on the recommended air volume are 420 area counts per µg/mL for 1-NP and 415 for 2-NP. These were determined by the slopes of the calibration curves. (Section 4.3.) The sensitivity will vary with the particular instrument used in the analysis.

1.2.5. Recovery

The recoveries of 1-NP and 2-NP from samples used in 15 day storage tests remained above 87 and 91% respectively when the samples were stored at ambient temperatures (20-26°C) in a closed drawer. The recovery of analyte from the collection medium after storage must be 75% or greater. (Section 4.4.)

1.2.6. Precision

The pooled coefficients of variation obtained from replicate determinations of analytical standards at 0.5, 1, and 2 times the target concentration are 0.009 for 1-NP and 0.013 for 2-NP. (Section 4.3.)

1.2.7. Precision (overall procedure)

The precisions at the 95% confidence level for 15-day storage tests are ±14.1% for 1-NP and ±12.1% for 2-NP. (Section 4.4.) These limits include an additional ±5% for sampling error. The overall procedure must provide results at the target concentration that are ±25% or better at the 95% confidence level.

1.2.8. Reproducibility

Six samples for each analyte, collected from controlled test atmospheres, and a draft copy of this procedure were given to a chemist unassociated with this evaluation. The samples were analyzed after 57 days of storage at 2°C. The average recoveries were 95.4% and 97.6% with standard deviations of 1.2% and 2.4% for 1-NP and 2-NP respectively. (Section 4.5.)

1.3. Advantages

1.3.1. The solid sorbent tube provides a convenient method for sampling.

1.3.2. The XAD-4 tubes do not have to be treated before sampling.

1.3.3. The analysis is rapid, sensitive, and precise.

1.3.4. The desorption solvent is carbon disulfide, which is a better solvent for use with a flame ionization detector than ethyl acetate.

1.4. Disadvantages

1.4.1. This method has not been field tested.

1.4.2. The amount of sample that can be taken is limited by the total milligrams the XAD-4 tube will adsorb before overloading.

2. Sampling Procedure

2.1. Apparatus

2.1.1. Samples are collected by use of a personal sampling pump that can be calibrated within ±5% of the recommended flow rate with the sampling tube in line.

2.1.2. Samples are collected on solid sorbent sampling tubes containing XAD-4 resin. The tubes contain two sections of XAD-4 separated by urethane foam plugs. The front section contains 80 mg of sorbent and the back section, 40 mg. The sections are held in place with glass wool plugs in a glass tube 70 mm × 4-mm i.d. The glass tube is flame-sealed at both ends. For this evaluation, SKC sorbent tubes (catalog number 226-30-11-04, lot 146) were used.

2.2. Reagents

No sampling reagents are required.

2.3. Sampling technique

2.3.1. Immediately before sampling, break open the ends of the XAD-4 tube. All tubes should be from the same lot.

2.3.2. Connect the sampling tube to the sampling pump with flexible tubing. Position the tube so that sampled air first passes through the 80-mg section.

2.3.3. Air being sampled should not pass through any hose or tubing before entering the sampling tube.

2.3.4. Place the sampling tube vertically (to avoid channeling) in the employee's breathing zone.

2.3.5. After sampling, seal the tubes immediately with plastic caps and wrap lengthwise with OSHA Form 21.

2.3.6. Submit at least one blank sampling tube with each sample set. Blanks should be handled in the same manner as samples, except no air is drawn through them.

2.3.7. Record sample volumes (in liters of air) for each sample, along with any potential interferences.

2.3.8. Ship any bulk sample(s) in a separate container(s) from the air samples.

2.4. Breakthrough

2.4.1. The average 5% breakthrough volume from a test atmosphere (air at approx. 80% relative humidity) containing 52.6 ppm (191.8 mg/m^3) 2-NP was determined to be 4.8 L for three measurements. This corresponds to a sampled amount of 0.92 mg of 2-NP. The sampling rate was approximately 0.1 L/min and the test atmosphere was at 23°C and 654.3 mm Hg. (Section 4.6.)

2.4.2. Since it was found that the breakthrough volume was greater (as expected) for 1-NP than 2-NP at 0.2 L/min, only one determination for breakthrough volume was done for 1-NP at 0.1 L/min. The 5% breakthrough volume from an atmosphere containing 53.3 ppm (194.1 mg/m^3) 1-NP was 6.0 L. This corresponds to a sampled amount of 1.16 mg of 1-NP. The recommended sample volume of 4 L for this combined method is based on the breakthrough volume of 2-NP. (Section 4.6.)

2.5. Desorption efficiency

2.5.1. The average desorption efficiency of 1-NP from spiked samples is 95.4% over the range of 0.5 to 2 times the target concentration. (Section 4.7.)

2.5.2. The average desorption efficiency of 2-NP from spiked samples is 96.4% over the range of 0.5 to 2 times the target concentration. (Section 4.7.)

2.5.3. Desorption efficiencies must by determined for each lot of XAD-4 sampling tubes.

2.6. Recommended air volume and sampling rate

2.6.1. The recommended air volume is 4 L.

2.6.2. The recommended sampling rate is 0.1 L/min.

2.7. Interferences (sampling)

2.7.1. It is not known if any compound will severely interfere with the collection of 1-NP or 2-NP on XAD-4. In general, the presence of any other contaminant vapors in the air will reduce the capacity of XAD-4 to collect 1-NP or 2-NP.

2.7.2. Suspected interferences should be reported to the laboratory with submitted samples.

2.8. Safety precautions

2.8.1. Attach the sampling equipment to the employee so that it will not interfere with work performance or safety.

2.8.2. Wear eye protection when breaking the ends of the XAD-4 tubes.

2.8.3. Follow all safety procedures that apply to the work area being sampled.

3. Analytical Procedure

3.1. Apparatus

3.1.1. A GC equipped with a flame ionization detector. For this evaluation, a Hewlett-Packard 5840A GC was used with a 7671A Automatic Sampler.

3.1.2. A GC column capable of separating 1-NP and 2-NP from carbon disulfide and any interferences. A 10-ft × 1/8-in stainless steel column packed with 100/120 Supelcoport, coated with 20% SP-2100 and 0.1% CW1500, was used in this evaluation.

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

3.1.4. Small vials with Teflon-lined caps capable of holding 2 mL.

3.1.5. A dispenser capable of delivering 1.0 mL to prepare standards and samples. If a dispenser is not available, a 1-mL pipet may be used.

3.1.6. Syringes, such as 10-µL for preparation of standards and 1-µL for injection of samples and standards into a GC.

3.1.7. Volumetric flasks and pipets to dilute the 1-NP or 2-NP.

3.2. Reagents

3.2.1. Carbon disulfide, reagent grade.

3.2.2. 1-Nitropropane and 2-nitropropane, reagent grade (or redistilled practical grade).

3.2.3. GC grade nitrogen, air, and hydrogen.

3.3. Standard preparation

3.3.1. Analytical standards are prepared in carbon disulfide. Dispense 1.0 mL of carbon disulfide (using the same dispenser or pipet used for samples) into 2-mL vials. Seal the vials immediately with Teflon-lined caps. Using a 10-µL syringe, dispense into the sealed vials a known amount of a 1-NP or 2-NP solution prepared in carbon disulfide. Example: If 3.7 µL of a 1 to 10 dilution of 1-NP in carbon disulfide is injected into a vial containing 1.0 mL carbon disulfide, the vial would contain 367.6 µg (3.7 µL × 993.4 µg/mL × 1/10) of 1-NP per sample. For a 4-L air sample, this would be equivalent to 25.2 ppm or 91.9 mg/m^3, uncorrected for desorption efficiency.

3.3.2. Standard concentrations should bracket sample concentrations. Thus, if samples fall out of the concentration range of prepared standards, additional standards may have to be prepared and analyzed to ascertain linearity of response.

3.4. Sample preparation

3.4.1. Transfer each XAD-4 section of the samples to separate vials. The glass tube and plugs are discarded.

3.4.2. Add 1.0 mL of carbon disulfide to each vial using the same dispenser as used for preparation of standards.

3.4.3. The vials are immediately capped and shaken periodically for 30 min before analysis.

3.5. Analysis

GC conditions

column:	10-ft × 1/8-in SS, 20% SP-2100, 0.1% CW1500 on 100/120 Supelcoport
injection volume:	1 µL
zone temperatures (°C):	75 (column)
	175 (injector)
	250 (FID detector)
gas flows (mL/min):	25 (nitrogen, carrier)
	45 (hydrogen)
	260 (air)
retention times (min):	2.4 (carbon disulfide)
	5.7 (2-NP)
	7.3 (1-NP)
chromatograms:	Section 4.8.

3.6. Interferences (analytical)

3.6.1. Any compound that responds on a flame ionization detector and has the same general retention time of the analyte is a potential interference. Possible interferences should be

reported to the laboratory with submitted samples by the industrial hygienist. These interferences should be considered before samples are desorbed.

3.6.2. GC parameters (i.e. column and column temperature) may be changed to possibly circumvent interferences.

3.6.3. Retention time on a single column is not considered proof of chemical identity. Samples should be confirmed by GC/MS if possible.

3.7. Calculations

The analyte concentration for samples is obtained from the appropriate calibration curve in terms of micrograms per sample, uncorrected for desorption efficiency. The air concentration is calculated using the following formulae. If any analyte is found on the backup section, it is added to the amount found on the front section. This total amount is then corrected by subtracting the total amount (if any) found in the blank.

$$mg/m^3 = \frac{\text{(blank corrected micrograms per sample)}}{\text{(liters of air sampled)(desorption efficiency)}}$$

$$ppm = (mg/m^3)(24.46)/(89.09) = (mg/m^3)(0.2746)$$

where 24.46 = molar volume (liters) at 760 mm Hg, 25°C
89.09 = molecular weight of 1- and 2-nitropropane

3.8. Safety precautions (analytical)

3.8.1. Avoid skin contact and inhalation of all chemicals.

3.8.2. Restrict the use of all chemicals to a fume hood when possible.

3.8.3. Wear safety glasses and a lab coat at all times while in the lab area.

4. Backup Data

4.1. Detection limit of the analytical procedure

The recommended injection size of 1 µL was used to determine the detection limits of the analytical procedure. The detection limit of 0.4 ng for 1-NP and 2-NP was determined by analyzing a analytical standard containing 0.37 ng/µL 1-NP and 0.36 ng/µL 2-NP. Shown in Figure 4.1. is a chromatogram of such an injection made on a Hewlett-Packard 5840A GC equipped with a flame ionization detector set at an attenuation of 2^{-1}. The chart speed was set at 0.2 cm/min.

4.2. Detection limit of the overall procedure and reliable quantitation limit data

Samples were prepared by injecting 368 ng of 1-NP on six XAD-4 tubes and 363 ng of 2-NP on another six XAD-4 tubes. The samples were later analyzed to determine the amount recovered. Since the amounts recovered were high and approximately equal to the detection limits of the analytical procedure, the detection limits of the overall procedure and the reliable quantitation limits are taken to be 0.4 µg per sample (25 ppb or 91 $\mu g/m^3$) for both 1-NP and 2-NP. The results of this study are given in Table 4.2.

Table 4.2.
Detection Limits of the Overall
Procedure and Reliable Quantitation Limits Data

sample no.	1-NP mass spiked, ng	2-NP mass spiked, ng	1-NP % recovery	2-NP % recovery
1	368	363	86.4	92.5
2	368	363	87.4	94.1
3	368	363	102.4	98.8
4	368	363	93.7	98.0
5	368	363	95.1	98.0
6	368	363	97.9	96.5
$\bar{X}$			93.8	96.4
SD			6.1	2.6
1.96 SD			12.0	5.1

4.3. Sensitivity and Precision (analytical)

The sensitivity and precision of the analytical procedure were determined from multiple injections of analytical standards. These data are given in Table 4.3. and shown graghically Figure 4.3.1. and 4.3.2.

Table 4.3.
Sensitivity and Precision Data

	1-NP			2-NP		
× target conc.	0.5×	1×	2×	0.5×	1×	2×
µg/mL	183.8	367.6	735.1	181.7	363.4	726.8
area counts	76240	152300	306300	73180	148800	298600
	74520	153500	312000	75080	151800	305000
	75100	152700	306100	74120	147600	298800
	74820	154400	312900	75280	152100	305600
	75280	152600	306000	73480	147900	299100
	75300	154900	313900	75380	152300	306100
$\bar{X}$	75210	153400	309533	74420	150083	302200
SD	585.5	1058	3774	960	2214	3708
CV(%)	0.78	0.69	1.22	1.29	1.48	1.23
$\overline{CV}$		0.009			0.013	

4.4. Recovery data (storage)

Storage samples were generated from test atmospheres (air at about 80% relative humidity) containing 1-NP or 2-NP near the target concentration of 25 ppm. The samples were generated at ambient temperatures (20 to 26°C) and pressures (655 to 660 mm Hg) by drawing about 4 L of the test atmospheres through the sampling tubes at about 0.1 L/min for 40 min. For the two sets of 36 samples each (one set per analyte), six samples were analyzed immediately after generation, fifteen were stored in a refrigerator at 2°C and fifteen were stored in a closed drawer at ambient temperatures. The results of recovery versus storage time are given below in Tables 4.4.1. and 4.4.2., and shown graphically in Figures 4.4.1. and 4.4.2.

Table 4.4.1.
Storage Tests for 1-Nitropropane

storage time (days)	% recovery (refrigerated)			(ambient)		
0	97.7	96.6	97.1	97.7	96.6	97.1
0	96.7	96.8	96.3	96.7	96.8	96.3
3	99.0	99.2	99.0	98.6	98.9	99.2
5	89.3	87.8	87.9	84.8	88.1	80.7
8	89.0	92.1	83.2	91.5	90.4	90.3
10	98.0	98.2	98.7	97.1	97.7	97.6
15	90.6	90.6	90.6	87.1	87.7	81.0

Table 4.4.2.
Storage Tests for 2-Nitropropane

storage time (days)	% recovery (refrigerated)			(ambient)		
0	98.0	97.4	97.4	98.0	97.4	97.7
0	100.5	97.7	98.3	100.5	97.7	98.3
3	100.0	99.5	98.2	99.2	98.4	100.1
5	94.2	92.2	92.7	93.6	93.8	86.4
8	92.8	84.0	86.5	95.7	93.9	92.9
10	99.2	99.4	100.4	99.6	98.6	98.1
15	96.0	93.9	93.1	91.6	94.0	85.3

4.5. Reproducibility

Six samples for each analyte, collected from controlled test atmospheres (80% RH, 25°C, 656 mm Hg) containing the analyte near the target concentration, were analyzed by another chemist unassociated with this evaluation. The samples were generated by drawing the test atmosphere through the sampling tubes for 40 min at approximately 0.1 L/min. The samples were stored for 57 days at 2°C before being analyzed. The results are given in Table 4.5.

Table 4.5.
Reproducibility

	1-NP			2-NP		
sample	µg found	µg possible	% found	µg found	µg possible	% found
1	333.3	342.8	97.2	340.7	358.2	95.1
2	394.3	412.3	95.6	422.8	423.8	99.8
3	350.1	372.8	93.9	379.0	385.8	98.2
4	363.6	380.3	95.6	384.1	394.1	97.5
5	402.3	423.7	94.9	410.7	434.1	94.6
6	(sample lost)			432.4	429.3	100.7
$\bar{X}$			95.4			97.6
SD			1.2			2.4

4.6. Breakthrough

The average 5% breakthrough volume of 4.8 L for 2-NP was determined by sampling at 0.1 L/min from a test atmosphere (80% RH, 23°C, 654 mm Hg) containing 52.6 ppm (191.8 mg/m^3) of 2-NP. The sampling tubes contained only the front section of adsorbent. Five-percent breakthrough volumes of 4.70, 5.00, and 4.69 L were obtained from three separate determinations to give an average of 4.8 L. This corresponds to a sampled mass of 0.92 mg. A graphical representation of one of the tests is shown in Figure 4.6. The atmosphere downstream from the sampling tubes was monitored by a total hydrocarbon analyzer. A similar determination was made for 1-NP at 53.3 ppm (194.1 mg/m^3). The 5% breakthrough volume was determined to be 6.0 L.

4.7. Desorption efficiency

The desorption efficiency for each analyte was determined by injecting known amounts of 1-NP and 2-NP standards onto the front sections of XAD-4 sampling tubes. The samples were analyzed the next day after storing at room temperature in a closed drawer.

Table 4.7.
Desorption Efficiency Data

	1-NP			2-NP		
µg	183.8	367.6	735.1	181.7	363.4	726.8
ppm	12.6	25.2	50.4	12.5	24.9	49.9
%	95.1	95.0	95.3	99.6	98.3	93.8
desorption	95.3	95.7	94.7	98.3	96.7	95.1
	95.4	95.6	95.6	98.0	96.3	95.3
	94.5	95.0	96.7	98.0	95.7	95.0
	95.9	96.1	96.2	94.0	96.7	94.6
	94.5	94.1	96.0	98.1	96.2	95.5
$\bar{X}$	95.1	95.2	95.8	97.7	96.7	94.9
$\bar{\bar{X}}$		95.4			96.4	

4.8. Chromatogram

A chromatogram of 1-NP and 2-NP is shown in Figure 4.8. The chromatogram is from a 1-µL injection of a standard containing 365 µg/mL each of 1-NP and 2-NP. This concentration is approximately equal to 25 ppm each for a 4-L air sample.

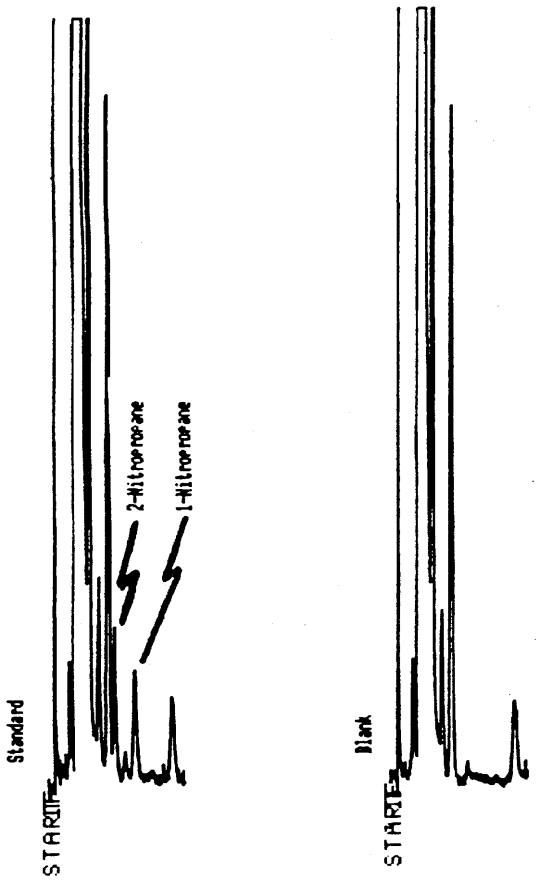

Figure 4.1. Detection limit chromatograms.

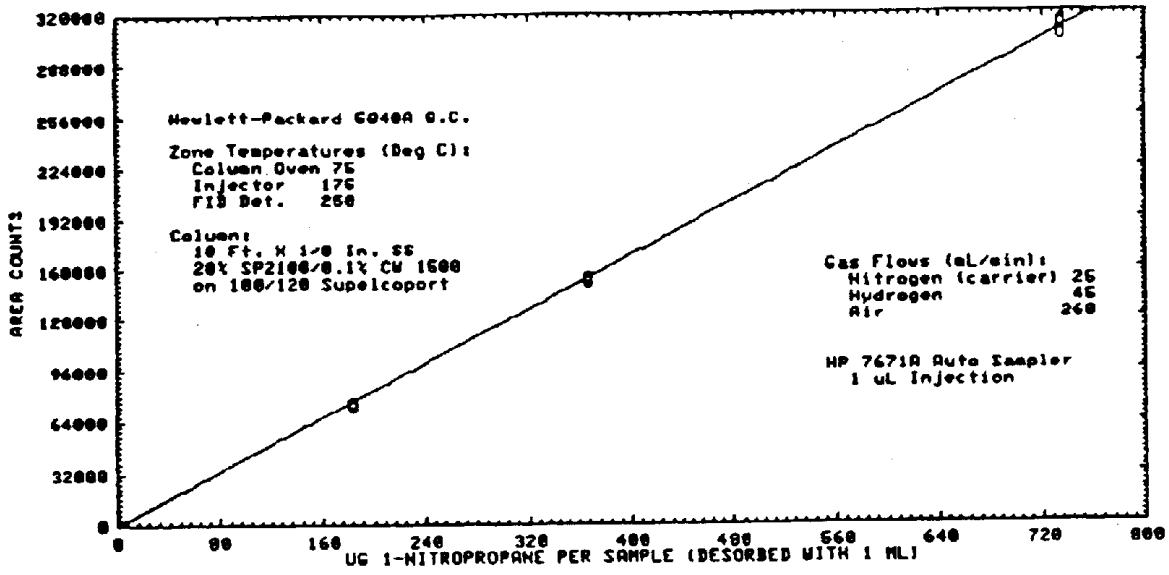

Figure 4.3.1. 1-Nitropropane calibration curve.

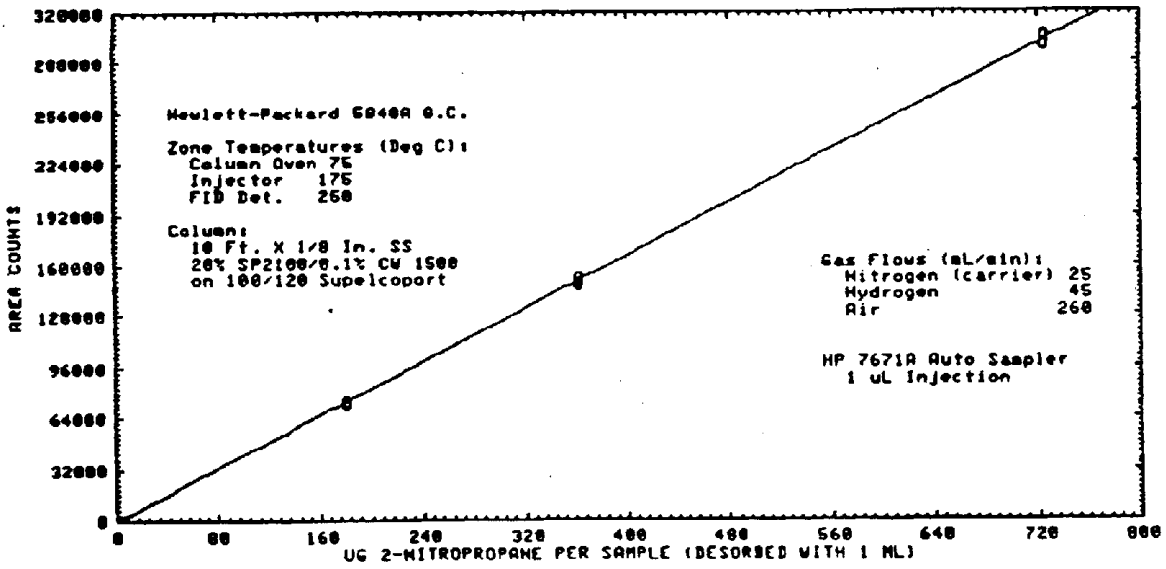

Figure 4.3.2. 2-Nitropropane calibration curve.

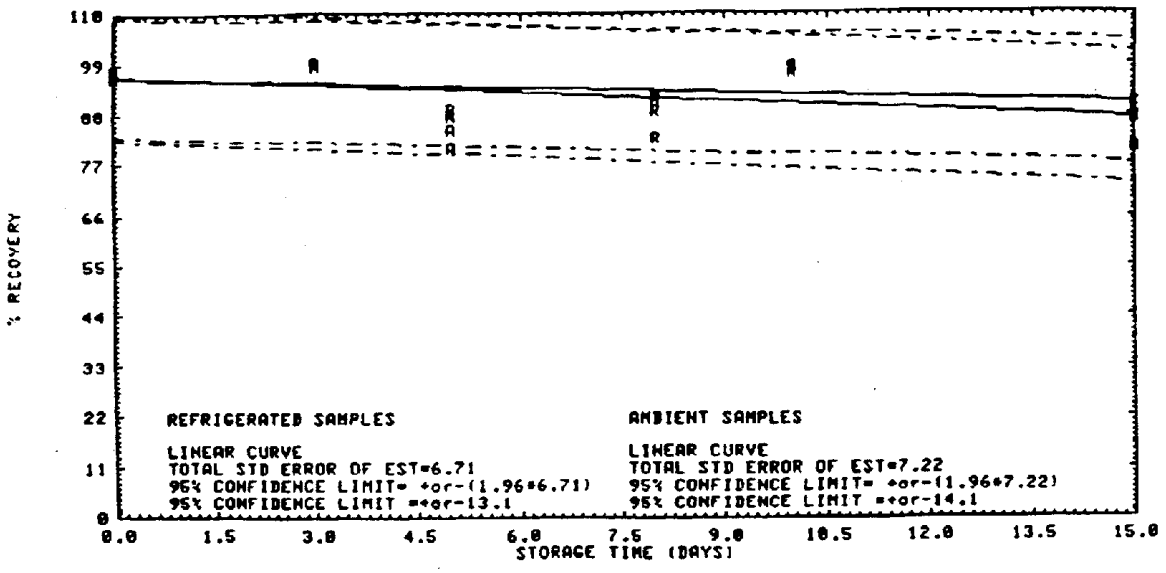

Figure 4.4.1. 1-Nitropropane storage samples.

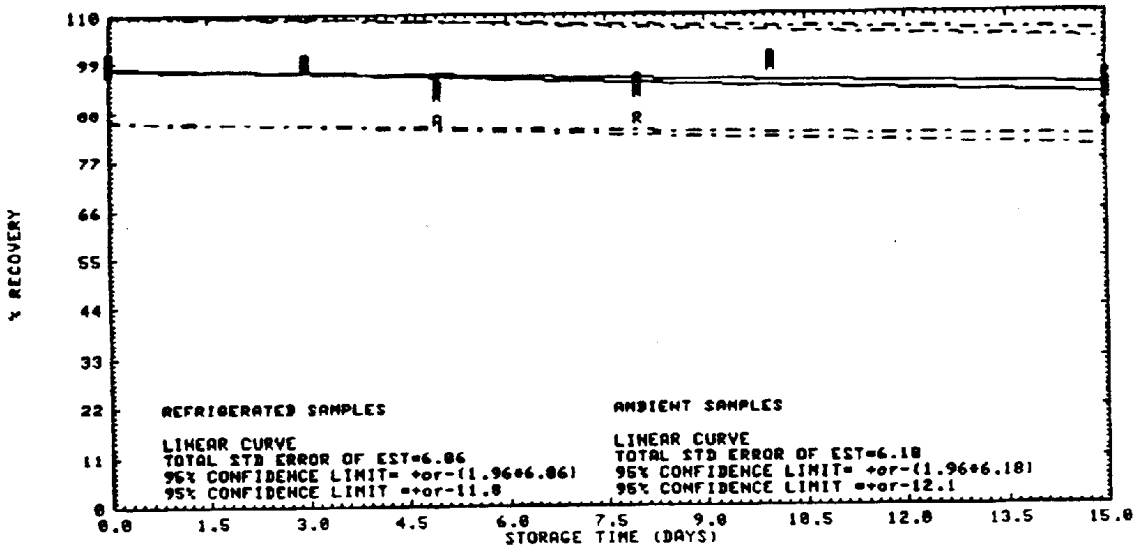

Figure 4.4.2. 2-Nitropropane storage samples.

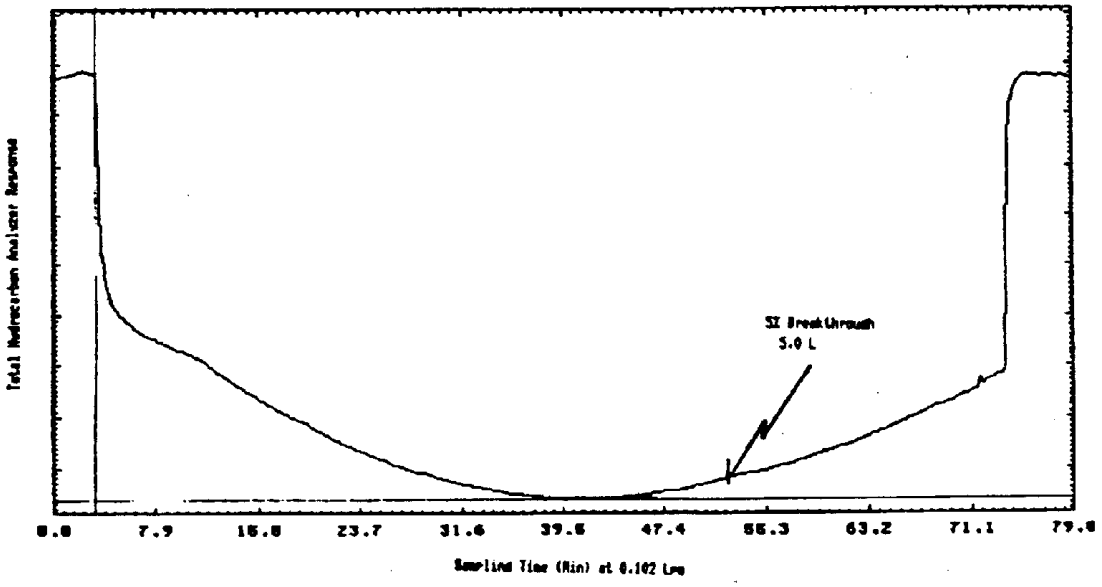

Figure 4.6. 2-Nitropropane breakthrough curve.

Figure 4.8. Chromatogram of a standard.

5. References

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