METHYLENE BISPHENYL ISOCYANATE (MDI)

Method no.	47
Matrix:	Air
Target concentration:	200 µg/m^3 (20 ppb) (OSHA PEL)
Procedure:	Samples are collected by drawing a known volume of air through a glass fiber filter coated with 1.0 mg of 1-(2-pyridyl)piperazine (1-2PP) which is contained in an open-face cassette. Samples are extracted with 90/10 (v/v) acetonitrile/dimethyl sulfoxide (ACN/DMSO) and analyzed by high performance liquid chromatography (HPLC) using an ultraviolet (UV) or fluorescence detector.
Recommended air volume and sampling rate:	15 L at 1 L/min
Detection limit of the overall procedure:	0.8 µg/m^3
Reliable quantitation limit:	0.6 µg/m^3
Standard error of estimate at the target concentration: (Section 4.7)	6.2%
Special requirements:	If the coated glass fiber filters are to be stored for any length of time before sampling, they should be kept in a refrigerator.
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

Two of the earliest procedures to determine atmospheric diisocyanate concentrations were developed by Ranta and Marcali (Ref. 5.1). Both of these procedures are inconvenient as they use a bubbler for sampling and their colorimetric analyses are non-specific. A later sampling procedure used p-nitrobenzyl-N-n-propylamine (nitro reagent) in toluene bubblers (Ref. 5.2). While this method is specific for diisocyanates, it still retains the use of the bubbler. Also nitro reagent is unstable when stored for long periods of time, even if it is kept in a freezer. The past few years have seen several new derivatizing reagents being used: N-methyl-1-naphthalenemethylamine (Ref. 5.3), 9-(N-methylaminomethyl)anthracene (Ref. 5.4), and 1-(2-pyridyl)piperazine (1-2PP) (Refs. 5.5-5.7). The collection procedure of these new studies all involve the use of toluene bubblers. The purpose of this study is to find a collection system that does not use a bubbler, yet retains the sensitivity, precision and accuracy of the nitro reagent method.

1-2PP is a suitable derivatizing reagent, when coated on a glass fiber filter, for several reasons:

1) The high boiling liquid is retained on a glass fiber filter.
2) Aromatic diisocyanates react rapidly and exothermically with 1-2PP (Ref. 5.7). The analysis of field samples has shown that MDI is collected and reacted on the coated glass fiber filters.
3) The derivative has a higher molar absorptivity in the UV region than the one formed with nitro reagent (Ref. 5.5).

Additional work was performed on this procedure as a result of changes made in Title 29 CFR 1910.1000, Table Z-1-A in 1989. Although no change was made to the Ceiling PEL for MDI, the valve for toluene-2,4-diisocyanate (2,4-TDI) became an 8-h TWA-PEL. As with 2,4-TDI, it was found that long term sampling (240 min at 1 L/min) for MDI is feasible. Retention tests indicate that a glass fiber filter coated with 1 mg of 1-(2-pyridyl)piperazine, should retain 12 µg of MDI.

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

MDI vapor is a potent respiratory sensitizer. It also is a strong irritant of the eyes, mucous membranes, and skin and can cause pulmonary edema. Exposure of humans to high concentrations causes cough, dyspnea, increased secretions, and chest pains. MDI and other diisocyanates cause pulmonary sensitization in susceptible individuals; should this occur, further exposure should be avoided, since extremely low levels of exposure may trigger an asthmatic episode; cross sensitization to unrelated materials probably does not occur. The liquid in contact with the eyes may cause irritation (Ref. 5.8).

1.1.3 Operations where exposure may occur

The manufacture of polyurethane foams, coatings and elastomers potentially expose approximately 100,000 workers to diisocyanates. MDI is used in the manufacture of rigid foams, fire retardants, coated fabrics, automobile bumper components, and hundreds of other applications. Over 300 million pounds of MDI were produced in 1975 (Ref. 5.2). Workers using polymethylene polyphenyl isocyanate (PAPI) (which is a polymer of MDI) may also be exposed to MDI because PAPI can contain up to 30% MDI.

1.1.4 Physical properties

CAS no.:	101-68-8
MW:	250.26
bp:	314°C at 760 mm Hg
mp:	38°C
Sp gr:	1.23 at 25°C
vp:	0.000005 mm Hg at 25°C
color:	white to pale yellow
odor:	none

flash point: (closed cup) 200°C

synonyms: 4,4'-methylenediphenyl isocyanate; 1,1'-methylenebis(4-isocyanato)benzene; 4,4'-diisocyanate-diphenylmethane; isocyanic acid, methylene di-p-phenylene ester; MDI

structure: Figure 1.1.4

1.2 Limit defining parameters (The analyte air concentrations listed throughout this method are based on an air volume of 15 L and a solvent extraction volume of 4 mL.)

1.2.1 Detection limit of the analytical procedure

The detection limit of the analytical procedure is 0.06 ng per injection of MDI (derivatized) with the fluorescence detector. This is the amount of analyte which will give a peak whose height is about 5 times the height of the baseline noise. (Section 4.1)

1.2.2 Detection limit of the overall procedure

The detection limit of the overall procedure is 12 ng per sample (0.8 µg/m^3). This is the amount of MDI (derivatized) spiked on the sampling device which allows recovery of an amount of analyte equivalent to the detection limit of the analytical procedure. (Section 4.2)

1.2.3 Reliable quantitation limit

The reliable quantitation limit is 39.5 ng per sample (2.6 µg/m^3). This is the smallest amount of MDI (derivatized) which can be quantitated within the requirements of a recovery of at least 75% and a precision (±1.96 SD) of ±25% or better. (Section 4.3)

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 sensitivity of the analytical procedure over a concentration range representing 0.5 to 2 times the target concentration based on the recommended air volume is 168000 area units per µg/mL. This is determined by the slope of the calibration curve. (Section 4.4) The sensitivity will vary with the particular instrument used in the analysis.

1.2.5 Recovery

The recovery of MDI derivative from samples used in a 15-day storage test remained above 94.8% when the samples were stored at 22°C. (Section 4.7) The recovery of analyte from the collection medium during storage must be 75% or greater.

1.2.6 Precision (analytical method only)

The pooled coefficient of variation obtained from replicate determinations of analytical standards at 0.5, 1, and 2 times the target concentration is 0.013. (Section 4.4)

1.2.7 Precision (overall procedure)

The precision at the 95% confidence level for the 15-day storage test is ±12%. (Section 4.7) This includes an additional ±5% for sampling error. The overall procedure must provide results at the target concentrations that are ±25% or better at the 95% confidence level.

1.2.8 Reproducibility

Six samples, spiked by liquid injection, and a draft copy of this procedure were given to a chemist unassociated with this evaluation. The samples were analyzed after three days of storage at 22°C. The average recovery was 106.7% with a standard deviation of 4.5%. (Section 4.8)

1.3 Advantages

1.3.1 The analytical procedure is specific and sensitive for MDI. (Ref. 5.7)

1.3.2 The collection system is less cumbersome than the use of a bubbler.

1.3.3 1-2PP is more stable and less expensive than p-nitrobenzyl-N-n-propylamine (nitro reagent).

1.4 Disadvantage

Glass fiber filters coated with 1-2PP are not commercially available.

2. Sampling Procedure

2.1 Apparatus

2.1.1 Samples are collected by use of 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 A three-piece styrene cassette containing a glass fiber filter coated with 1.0 mg of 1-2PP and a untreated backup pad. (Figure 2.1.2) Coated filters are prepared by applying 0.5 mL of a solution of 2.0 mg/mL 1-2PP in methylene chloride to each glass fiber filter. The wet filters are allowed to air dry before they are placed in a jar and completely dried in a vacuum oven to remove residual methylene chloride. This section has been revised based on data received from Mobay Chemical Corporation. The amount of 1-2PP on the filters has been increased from 0.1 to 1.0 mg. (Section 4.11)

2.1.3 Coated filters should be stored in a closed jar at reduced temperature as a precaution to prevent decomposition of the 1-2PP. Exposure to strong sunlight should be avoided.

2.2 Reagents

No sampling reagents are required.

2.3 Sampling technique

2.3.1 Open-face sampling is performed by removing the top cover from the three-piece cassette and the small plug from the exit port.

2.3.2 Attach the cassette to the sampling pump with flexible tubing and place the cassette in the breathing zone of the employee to be monitored.

2.3.3 The recommended air volume and flow rate is 15 L at 1 L/min. Valid analytical results can be obtained for MDI when it is collected simultaneously with 2,4-TDI. A 240-min sampling time is permissible to accommodate the simultaneous sampling for MDI and 2,4-TDI.

2.3.4 After sampling for the appropriate time, remove the sampling device and replace the small plug and top cover.

2.3.5 Wrap each sample end-to-end with an OSHA Form 21 seal.

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

2.3.7 Bulk samples submitted for analysis must be shipped in sealed vials and in a separate container from air samples.

2.4 Retention efficiency

2.4.1 Experimental design

Due to present laboratory limitations, controlled test atmospheres of MDI cannot effectively be generated. Because MDI has a tendency to polymerize, the derivative of MDI was liquid spiked onto coated filters for this test. An amount of MDI derivative equivalent to 4.3 µg of MDI was spiked onto the filter and 20 L of humid air were pulled through the sampling cassette. Retention efficiency was defined as the percent of the analyte remaining on the coated filter after air had been pulled through the cassette.

2.4.2 Retention results

The retention efficiency of MDI derivative was found to be 97% after 20 L of air at about 80% relative humidity and 22°C had been pulled through the cassette. (Section 4.5)

2.5 Extraction efficiency

The average extraction efficiency for MDI derivative from filters spiked at the target concentration was 96.3%. (Section 4.6)

2.6 Recommended air volume and sampling rate

2.6.1 The recommended air volume is 15 L.

2.6.2 The recommended air sampling rate is 1 L/min.

2.7 Interferences (sampling)

Any compound that could react with the 1-2PP, or compete with it in the reaction to derivatize MDI, should be considered as an interference. Potential interferences include anhydrides, amines, alcohols, and carboxylic acids.

2.8 Safety precautions (sampling)

The sampling equipment should be attached to the worker in such a manner that it will not interfere with work performance or safety.

3. Analytical Procedure

3.1 Apparatus

3.1.1 High performance liquid chromatograph equipped with UV or fluorescence detector, manual or automatic sample injector, and chart recorder.

3.1.2 HPLC column capable of separating MDI from any interferences. The columns employed in this study were a 25-cm × 4.6-mm i.d. Alltech C_8 (10 µm), and a 25-cm × 4.6-mm i.d. DuPont Zorbax CN (6 µm).

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

3.1.4 Vials, 4-mL with Teflon-lined caps.

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

3.1.6 Suitable glassware for preparation of MDI urea derivative.

3.1.7 pH Meter for adjusting the mobile phase.

3.2 Reagents

3.2.1 Methylene chloride, hexane, acetonitrile, and dimethyl sulfoxide, HPLC grade.

3.2.2 Water, HPLC grade. A commercially available filtration system was used to prepare of HPLC grade water.

3.2.3 1-(2-Pyridyl)piperazine (1-2PP), Aldrich.

3.2.4 MDI, Eastman.

3.2.5 Ammonium acetate, HPLC grade.

3.2.6 Glacial acetic acid.

3.3 Standard preparation

3.3.1 A solution containing 0.5 g of recrystalized MDI in 25 mL of methylene chloride is slowly added to a stirred solution of 0.7 g of 1-2PP in 50 mL of methylene chloride. The milky white slurry is stirred for 1 h. Reduce the volume of methylene chloride to about 10 mL with a stream of dry nitrogen. The slurry is added dropwise to 800 mL of stirred hexane, filtered, redissolved in a minimal volume of methylene chloride and reprecipitated. The precipitate is filtered and washed with hexane (approximate yield is 1 g of the derivative after being dried by vacuum). This preparation is a modification of the procedure reported by Goldberg et. al. (Ref. 5.7).

3.3.2 Preparation of working standards

A stock standard solution is prepared by dissolving the MDI derivative into DMSO. To express the derivative as free MDI, the amount of MDI urea weighed is multiplied by the conversion factor 0.4339.

$$\frac{\text{MW MDI}}{\text{MW MDI Urea}} = \frac{250.26}{576.71} = 0.4339$$

All dilutions of the stock solutions are made with ACN to arrive at the working range.

3.4 Sample preparation

3.4.1 The styrene cassette is opened and the glass fiber filter is placed in a 4-mL vial so that the filter is flat against the inside surface of the vial, not folded or crumpled.

3.4.2 Four milliliters of the extracting solution, 90/10 (v/v) ACN/DMSO, are added.

3.4.3 A cap equipped with a Teflon liner is installed.

3.4.4 The vial is shaken to remove large air bubbles from between the filter and the glass. Let the vial sit for 1 h.

3.5 Analysis

3.5.1 Reverse phase HPLC conditions

column:	25-cm × 4.6-mm i.d. stainless steel column packed with 10-µm C_8 by Alltech
mobile phase:	0.01 M ammonium acetate in 50/50 (v/v) ACN/water adjusted to pH 6 with acetic acid
flow rate:	1-1.5 mL/min
UV detector:	254 nm and 313 nm
fluorescence detector:	240 nm excitation 370 nm emission
injection size:	10 - 25 µL
chromatogram:	Figure 3.5.1.

3.5.2 Alternate conditions

column:	25-cm × 4.6-mm i.d. stainless steel column packed with 6-µm DuPont Zorbax CN
mobile phase:	0.01 M ammonium acetate in 37.5/62.5 (v/v) ACN/water adjusted to pH 6 with acetic acid
flow rate:	2.0 mL/min
detectors:	same as above
injection size:	same as above

3.5.3 An external standard procedure is used to prepare a calibration curve using at least two stock solutions from which dilutions are made. The calibration curve is prepared daily. The samples are bracketed with analytical standards.

3.6 Interferences (analytical)

3.6.1 Any compound having the same retention time as MDI derivative is a possible interference. Generally, chromatographic conditions can be altered to separate an interference.

3.6.2 Retention time on a single column is not proof of chemical identity. Analysis by an alternate column system, absorbance response ratioing, and mass spectrometry are additional means of identity. (See UV spectrum for MDI derivative. Figure 4.10.)

3.7 Calculations

The concentration in µg/mL of MDI present in a sample is determined from the area response of the analyte as measured by an electronic integrator or peak heights. Comparison of sample response with a least squares curve fit for standards allows the analyst to determine the concentration of MDI in µg/mL for the sample. Since the sample volume is 4 mL, the results in $\mu g/m^3$ of air are expressed by the following equation:

$$\mu g/m^3 = (\mu g/mL)(4\ mL)/(m^3)(\text{extraction efficiency})$$

3.8 Safety precautions (analytical)

3.8.1 Avoid skin contact with all solvents.

3.8.2 Wear safety glasses at all times.

3.8.3 Avoid exposure to the MDI standards.

4. Backup Data

4.1 Detection limit of the analytical procedure

The detection limit of the analytical procedure was 0.06 ng. This amount produced a peak whose height was about 5 times the height of the baseline noise. The injection size recommended in the analytical procedure (10-25 µL) was used in the determination of the detection limit for the analytical procedure. (Figure 4.1)

4.2 Detection limit of the overall procedure

The detection limit of the overall procedure was extrapolated to be 11.6 ng/sample for MDI or 27 ng/sample for MDI derivative. The equivalent air concentration was 0.8 $\mu g/m^3$ for MDI. The injection size recommended in the analytical procedure (10-25 µL) was used to determine the detection limit of the overall procedure. (Figure 4.2)

Table 4.2
MDI Recoveries Near the Detection Limit

ng spiked	ng recovered
7.9	4.6
15.8	15.7
23.7	25.4
31.6	33.6
39.5	40.8
47.2	47.9

4.3 Reliable quantitation limit

The reliable quantitation limit was determined by liquid spiking nine coated glass fiber filters with 39.5 ng of MDI (91 ng of MDI derivative). The samples had 15 L of humid air pulled through them and were extracted in 4 mL of extracting solution. The injection size recommended in the analytical procedure (10-25 µL) was used for the determination of the reliable quantitation limit.

Table 4.3
Extraction Efficiency at the Reliable Quantitation Limit

% recovered	statistics
104.2	
103.5	$\overline{X}$ = 104.6
102.1	SD = 2.5
100.7	1.96 SD = 4.9
102.8	
106.9	
106.3	
107.6	
106.9	

4.4 Sensitivity and precision (analytical method only)

The following data were obtained from multiple injection of analytical standards. The data are also presented graphically in Figure 4.4. The pooled coefficient of variation for MDI was 0.0127. The sensitivity for MDI was 168000 area counts per µg/mL.

Table 4.4
MDI Derivative Sensitivity and Precision Data

× target concn µg/mL	0.5× 0.94	1.0× 1.88	2.0× 3.76
area counts	164699	322969	631877
	165739	314200	633893
	163344	313084	644331
	162985	318490	644743
	164890	317277	627083
	164337	318009	642001
$\overline{X}$	164332.7	319004.8	637321.3
SD	1021.4	5624.6	7380.1
CV	0.0062	0.0176	0.0116

4.5 Retention efficiency

4.5.1 Retention efficiency for a ceiling sample

Retention efficiency samples were generated by liquid spiking 9.9 µg of MDI derivative on each of six coated glass fiber filters. The filters then had 20 L of air (at approximately 80% relative humidity) pulled through them.

Table 4.5.1
Retention of MDI Derivative

% recovered	statistics
95.7	
96.8	$\overline{X}$ = 97.4
97.2	SD = 1.1
98.4	
98.8	
97.5	

4.5.2 Retention efficiency for a TWA sample

The following data are presented to show that 12.3 µg of MDI derivative, liquid spiked, onto the glass fiber filter is retained after 240 L of humid air is drawn through the sampler at 1 L/min.

Table 4.5.2
Retention of MDI Derivative

% recovered	statistics
104.8	
103.9	$\overline{X}$ = 103.8
104.3	SD = 0.7
103.7	
102.9	
103.3	

4.6 Extraction efficiency

The following data represent the analysis of 14 coated glass fiber filters liquid spiked with MDI derivative.

Table 4.6
Extraction Efficiency of MDI derivative

× target concn µg/sample	0.05× 0.38	1× 7.05
% recovered	98.8	96.5
	100.3	98.9
	95.9	92.5
	95.9	98.2
	97.4	94.7
	95.9	96.2
	91.5	100.7
$\bar{X}$	96.5	96.3

4.7 Storage data

Storage samples were generated by liquid spiking 6.94 µg of MDI derivative on coated glass fiber filters. The filters then had 15 L of air (at approximately 80% relative humidity) pulled through them. For the set of 33 samples, three samples were analyzed immediately after generation, fifteen were stored in a freezer at -20°C and fifteen were stored in a closed drawer at ambient temperature. The results of recovery versus storage time are given below and shown graphically in Figures 4.7.1 and 4.7.2.

Table 4.7
Storage Test of EA

time (days)	percent recovery (ambient, 22 °C)			percent recovery (refrigerated, -20 °C)		
0	94.5	95.6	95.1	94.5	95.6	95.1
3	95.4	94.7	94.5	93.4	95.1	94.8
6	95.2	104.9	97.1	95.4	97.3	94.1
9	91.4	94.8	95.3	95.1	95.8	94.4
12	97.9	86.1	97.7	97.0		
15	93.5	96.7	94.5	98.6	97.6	94.8

4.8. Reproducibility data

Six samples, liquid spiked with 6.78 µg of MDI derivative, had 15 L of humid air drawn through them. The cassettes were given to a chemist unassociated with this work. The samples were analyzed after 3 days storage at ambient temperature. The results are corrected for extraction efficiency.

Table 4.8
Reproducibility Results

% recovered	statistics
105.4	
106.5	mean = 106.7
107.6	SD = 4.5
112.9	
108.6	
99.1	

4.9 Thermostability

The data presented in this section were collected to test the ability of the derivative of MDI to withstand thermal decomposition. Glass fiber filters were spiked with 6.94 µg of MDI derivative and then heated for 7.5 to 8 h, cooled to room temperature and analyzed. The MDI derivative is not significantly affected by temperatures below 105°C.

Table 4.9
Thermostability

°C	°F	% recovery		
40	104	93.4	92.8	94.2
63	145	93.3	95.1	93.8
90	194	83.9	90.4	86.9
105	221	92.3	91.3	92.3
125	257	55.8	81.7	65.5

4.10 UV Spectra

Figure 4.10 is the UV Spectra of the 1-2PP derivative of MDI used in this study.

CAS Number Name

72375-24-7 N,N'-(Methylenediphenylene)bis-4-(2-pyridinyl)-1-piperazine carboxamide

4.11 Revision of filter coating procedure

The procedure for coating the glass fiber filters with 1-2PP has been revised based on a study performed by Mobay Chemical Corporation. (Ref. 5.9) Their study showed that a filter coated with 1.0 mg of 1-2PP collected the MDI vapors more effectively then a coating of 0.1 mg when compared to a glass fiber filter coated with Nitro Reagent (N.R.) and a glass fiber filter coated with sulfuric acid

(S.A.). (Table 4.11) This change in the procedure is being made based on Mobay's data because the OSHA Laboratory did not have the apparatus to safely produce an atmosphere of MDI vapors. Figure 4.11 is a computer representation of a chromatogram of a 20-µL injection of a sample containing a filter coated with 1.1 mg of 1-2PP that had been liquid spiked with 0.344 µg of MDI derivative. This was extracted with 4 mL of extraction solution and represents 1/20 of the PEL (10 µg/m^3 or 1 ppb). The chromatogram shows that increasing the amount of reagent on the filters does not affect the separation of 1-2PP and MDI derivative and that low amounts of MDI are still detectable.

Table 4.11
Comparison of Sampling and Analysis Methods for MDI Vapors in Air[1]

	0.1 mg 1-2PP		1.0 mg 1-2PP		2.5 mg N.R.		7.4 mg S.A.	
sample no.	flow L/min	ppb MDI	flow L/min	ppb MDI	flow L/min	ppb MDI	flow L/min	ppb MDI
1	1.0	6	0.85	17.6	2.0	13.7	1.9	20.5
2	0.85	10	0.85	15.3	2.0	19.3	1.9	17.9
3	0.87	4.2	1.1	7.6	2.0	7.7	2.2	7.3
4	2.2	0.4	2.0	4.0	0.87	4.9	1.1	5.9
5	0.87	1.7	2.2	1.7	2.0	1.6	1.1	3.2

[1] Data supplied by Moby Chemical Corporation

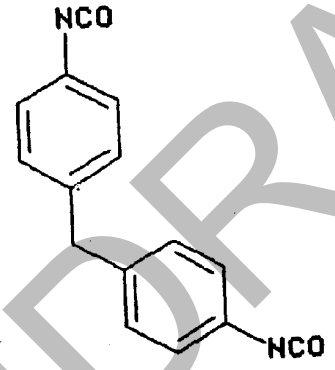

Figure 1.1.4. Structure of MDI.

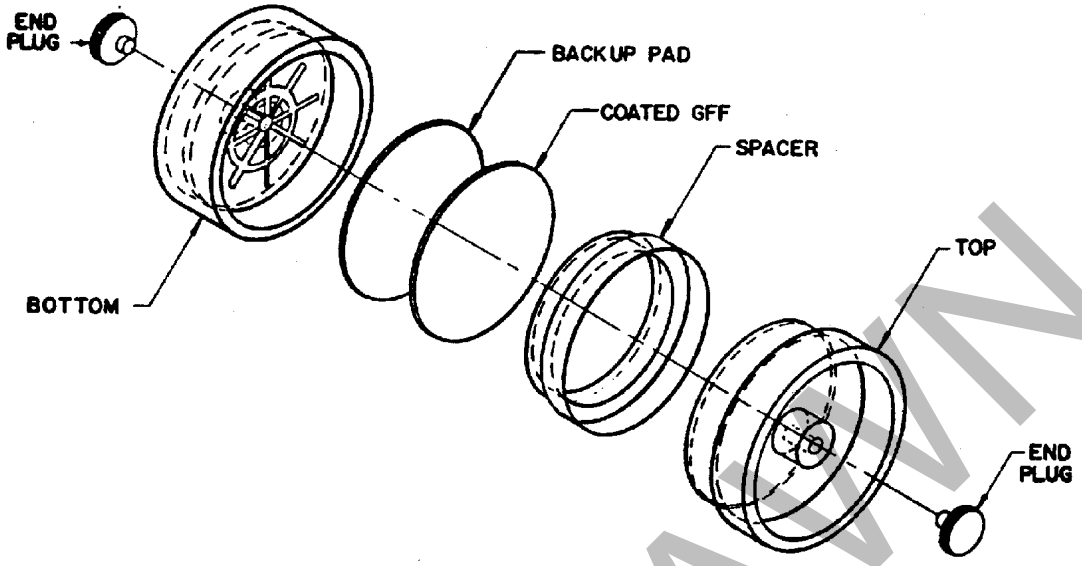

Figure 2.1.2. A drawing of a sample cassette.

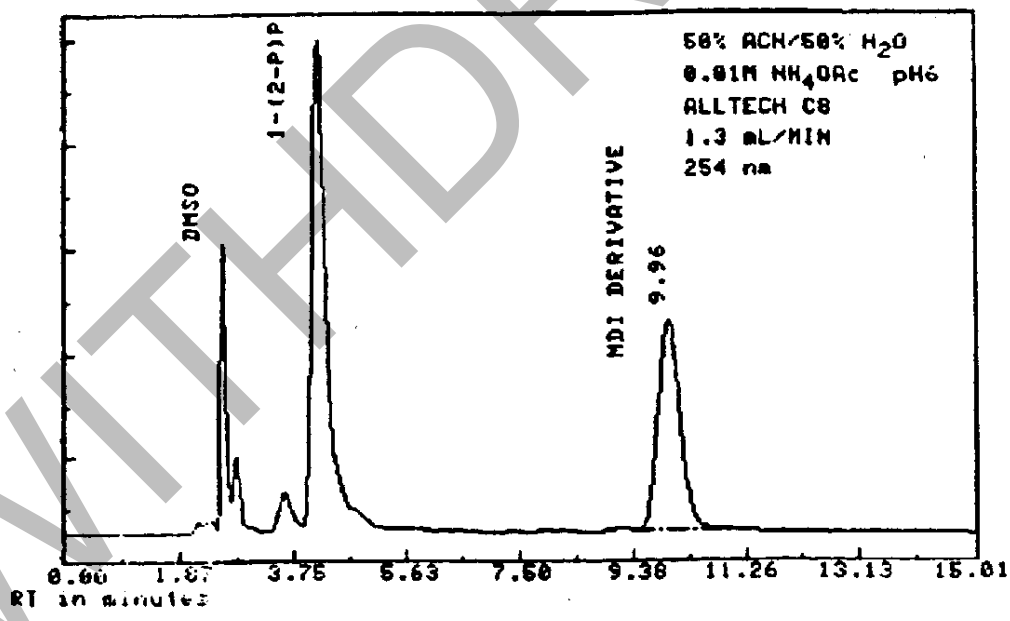

Figure 3.5.1. A chromatogram of a sample of MDI.

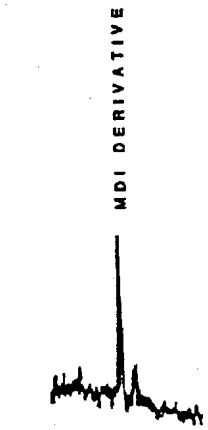

Figure 4.1. Analytical detection limit of MDI, 0.06 ng/injection.

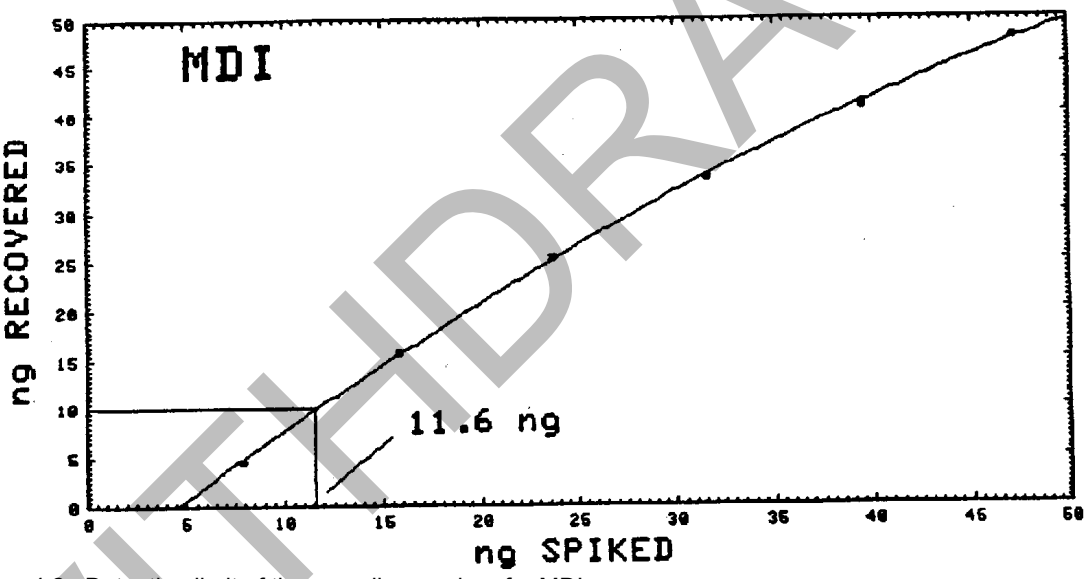

Figure 4.2. Detection limit of the overall procedure for MDI.

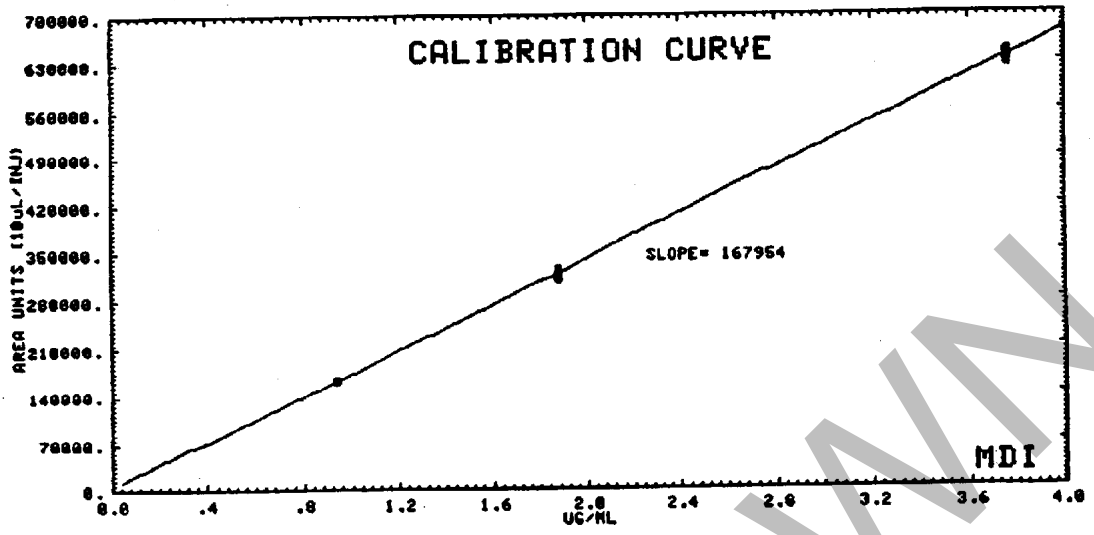

Figure 4.4. Calibration curve for MDI.

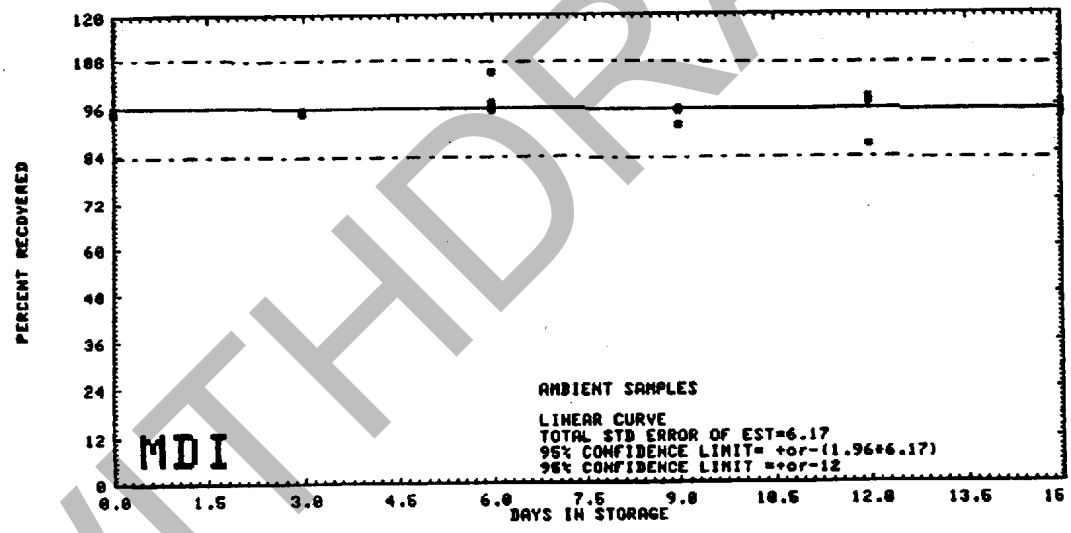

Figure 4.7.1. Ambient storage test for MDI.

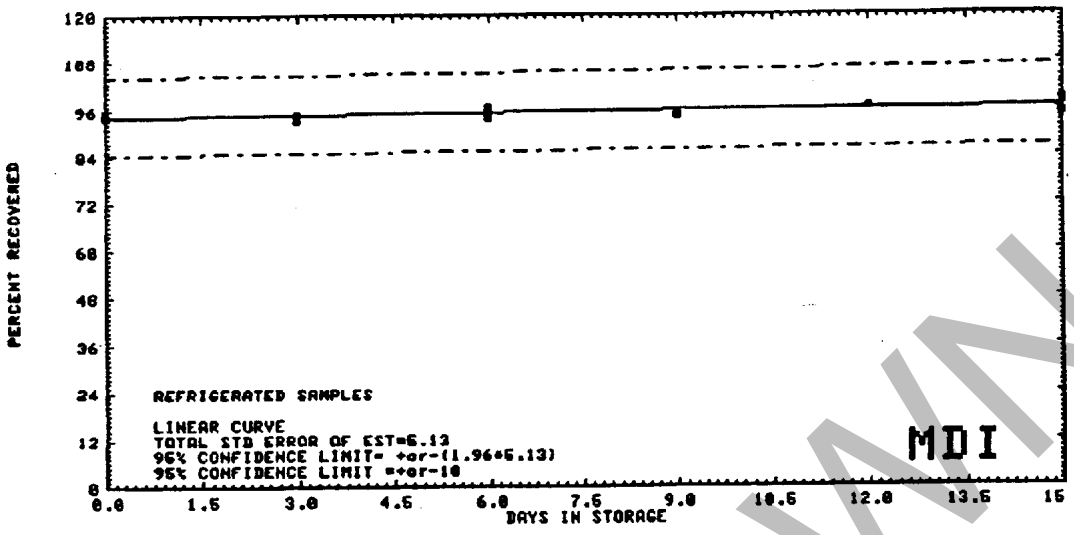

Figure 4.7.2. Refrigerated storage test for MDI.

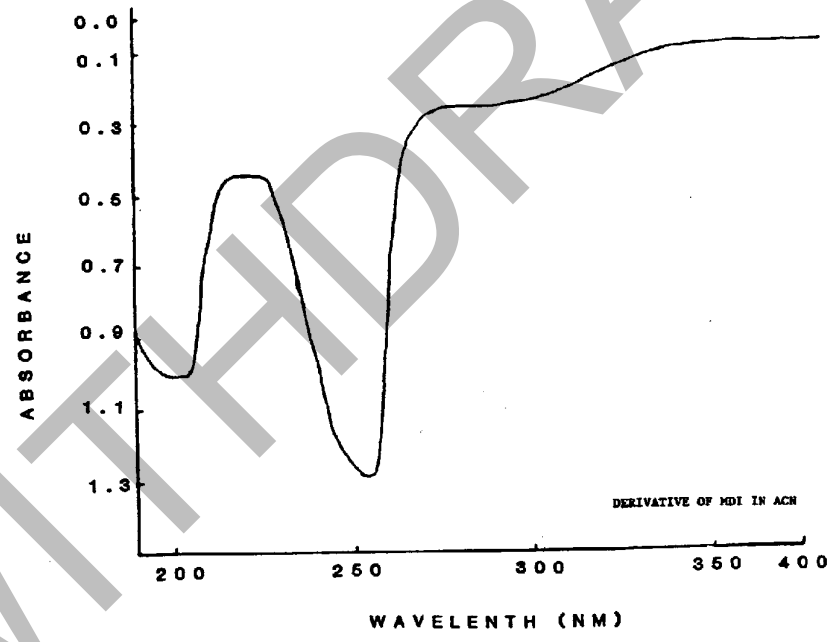

Figure 4.10. UV spectrum of MDI derivative in acetonitrile.

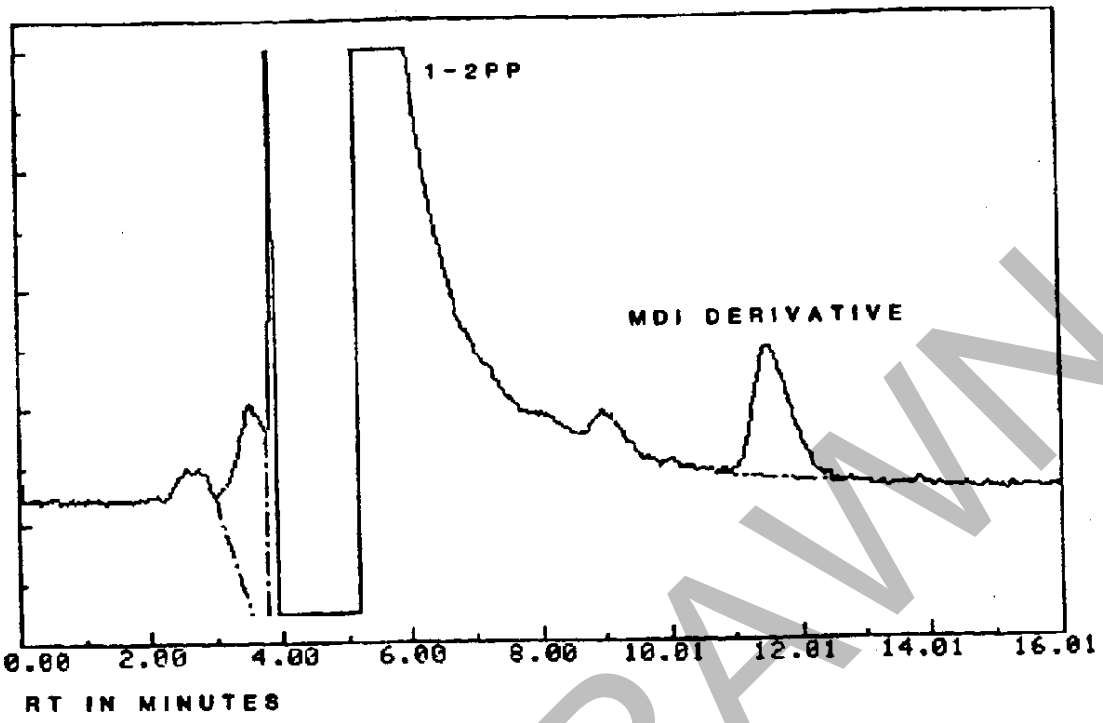

Figure 4.11. Chromatogram of 1.1 mg of 1-2PP on a glass fiber filter and 1/20 times the PEL of MDI.

5. References

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