

RESEARCH TRIANGLE INSTITUTE

DEVELOPMENT OF ANALYTICAL TECHNIQUES FOR
MEASURING AMBIENT ATMOSPHERIC CARCINOGENIC VAPORS

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INTRODUCTION AND RATIONALE

On October 3, 1975, personnel from the Chemistry and Life Sciences group at RTI attended a meeting held by Dr. A. P. Altschuller at ESRL, Environmental Protection Agency, Research Triangle Park. In response to an OAQPS request, RTI was asked to participate in assessing the nitrosamine problem in the Baltimore, MD area. The effort consisted of obtaining and comparing data on any ambient nitrosamines as measured by gc/ms methods (RTI) and by thermal energy analysis (Thermal Electron Corp.). Samples were to be collected primarily in parallel between these two contractors. The Research Triangle Institute proceeded to undertake a sampling program during the week of October 13-17 and during the period of November 19-25, 1975 in the Baltimore area, specifically near and on the FMC property.

During this sampling period, the Environmental Protection Agency provided a meteorologist to assist the RTI sampling team in obtaining favorable locations with respect to downwind locations from the FMC property.

The primary objectives were to identify unequivocally the presence of N-nitrosodimethylamine in ambient air and estimate the levels in this area.

METHODS OF PROCEDURE

Sampling Methods

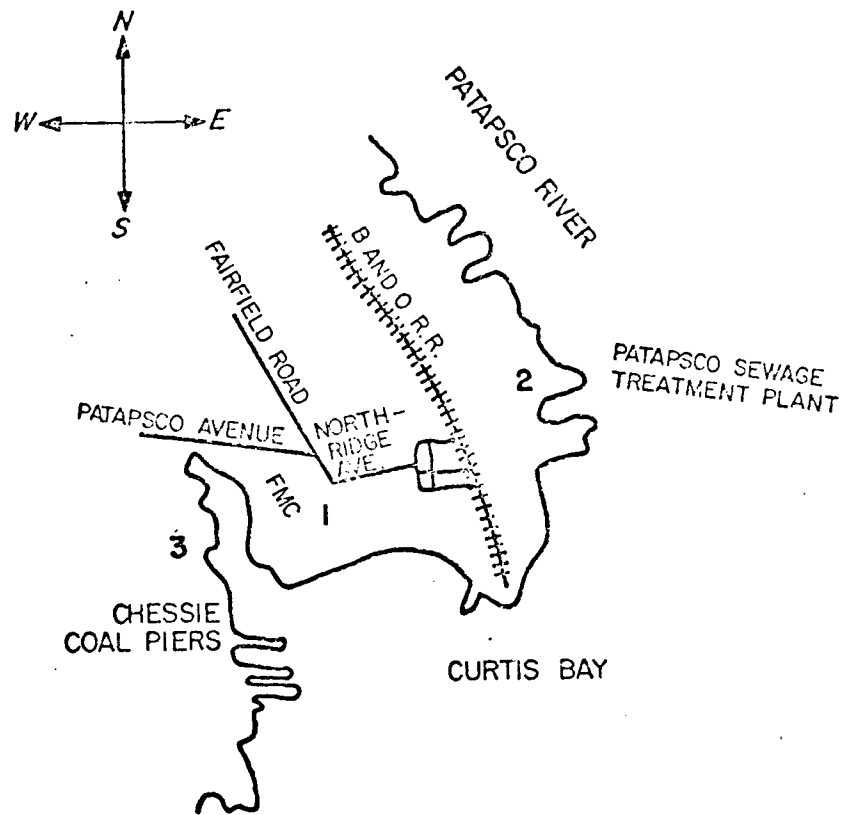
The sampling procedure employed for this study has been previously described (1,2) which consisted of concentrating nitrosamines and other organic vapors on a 1.5 x 6.0 cm bed of Tenax GC (35/60) in a glass cartridge. All sampling cartridges were preconditioned by heating to 275°C for a period of 20 min under a helium purge of 20-30 ml/min. After cooling in precleaned Corex centrifuge tubes, the containers were sealed to prevent contamination of the cartridge. Sampling cartridges prepared in this manner were carried by automobile or air freight to the sampling site; 2-3 cartridges were designated as blanks to determine whether any of the cartridges might have been contaminated by the packing and transportation procedure. Cartridges containing known quantities (100 and 300 ng) of N-nitrosodimethylamine (DMN) were prepared and carried to and from the field, stored and the percent recoveries were determined.

Ambient air samples were collected with Nutech Model 221-A AC/DC portable samplers (2). In general, a sampling rate of 1 l/min/cartridge was used throughout this study.

The sampling locations are shown in Figures 1 and 2 and the sampling protocol used in this study is given in Table 1.

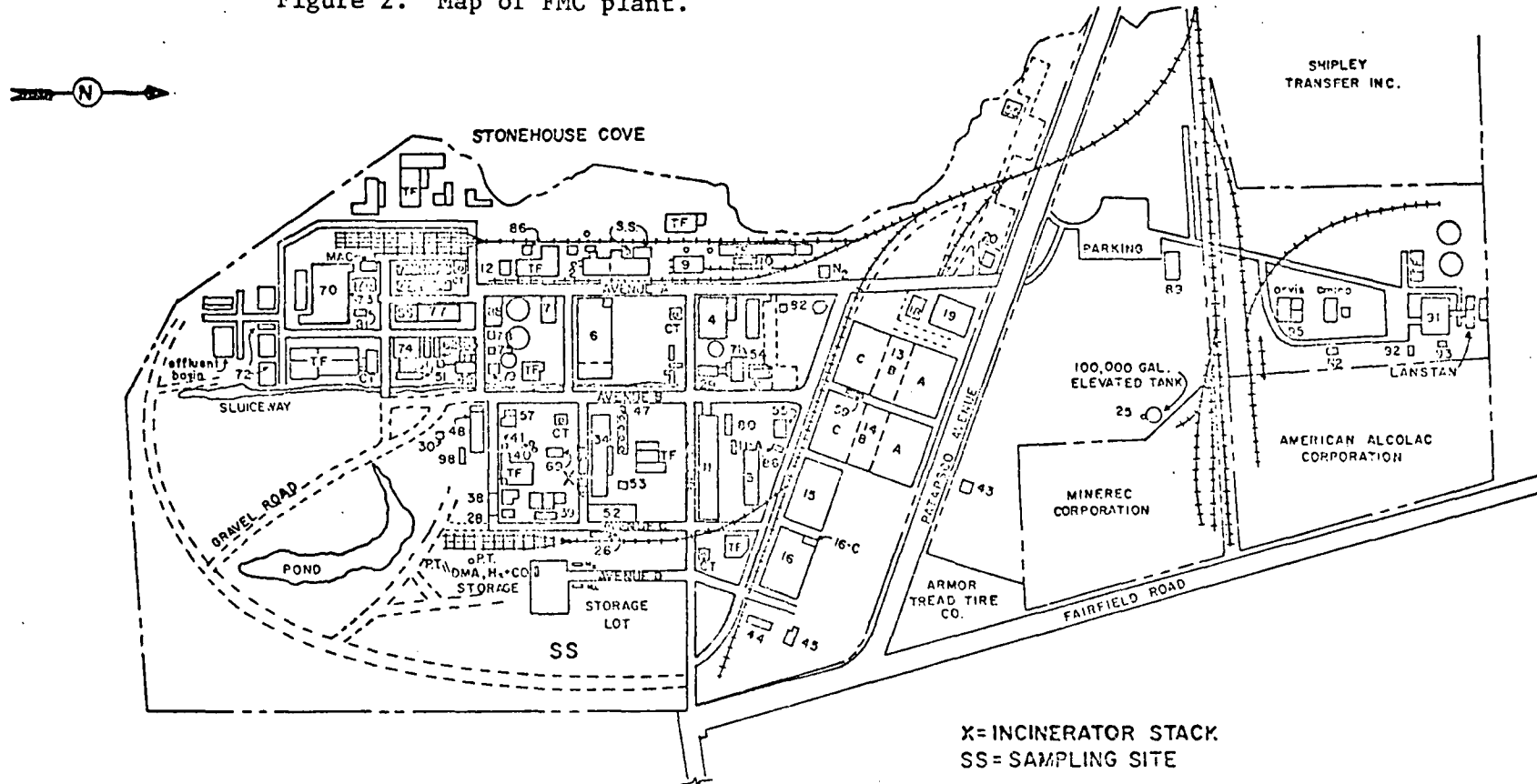
Breakthrough volumes for DMN were determined as previously described (2). This consisted of injecting DMN vapor onto a gas chromatographic column packed with the sorbent Tenax GC and determining the elution volume at a series of decreasing temperatures. By plotting the log of the elution volume vs temperature, and extrapolating to ambient temperatures, (50-90°F) the breakthrough volumes for DMN were determined. These values

Figure 1. EAST BROOKLYN,
BALTIMORE, MARYLAND



SCALE: ONE INCH = 0.5 miles

Figure 2. Map of FMC plant.



- | | | | | |
|---|-------------------------------------|--------------------------|---------------------------------|-----------------------------------|
| 1. Power House | 15. Dapon Manufacturing & Warehouse | 41. Valve House | 69. Hydrogenation | 82. Locker Rm. |
| 2. Stack | 16. Warehouse | 43. Pump House | 70. 7-OH Production | 86. Inert Gas Generator |
| 3. Dapon Bldg. | 18. Lab | 44. Storage | 71. Control Lab | 87. Locker Rm. |
| 4. Refined Chemicals Bldg. | 19. Main Office Bldg. | 44. Storage | 72. Incinerator Area | 88. Calgon Carbon Treatment Bldg. |
| 6. Ethion & Butoxide Bldg. | 20. Gate House | 47. Pump House | 73. Elec. Control Rm. | 89. Shop |
| 7. P ₂ S ₈ Conveyor & Storage Bldg. | 25. Booster Pump House | 48. Chloroformates | 74. 7-OH Warehouse | 91. Resin Semi-Works Plant |
| 9. Diallyl Phthalate Bldg. | 26. Anhydrous Ammonia | 49. Sodium Condensations | 75. Effluent Control Rm. | 92. Pilot Plant |
| 10. Packaging Bldg. | 28. Dimazine Control Rm. | 51. Sodium Storage | 76. Motor Control & Locker Rm. | 93. Elec. Control Rm. |
| 11A. Maintenance Office Trailer | 29. Lab | 52. Tedion Bldg. | 77. 7-OH Shop | 95. Orvis Pilot Plant |
| 12. Fire Pump House | 30. CO Compressor Shed | 53. Lockers | 78. Cl ₂ Control Rm. | 96. Compressor Bldg. |
| 13A. Warehouse | 31. Production Office | 54. Lockers | 79. Fire Pump House | CT. Cooling Tower |
| 13B. Carpenter Shop | 34. Pyrethrum Refining | 55. Cafeteria | 80. Locker Rm. | TF. Tank Farm |
| 13C. Warehouse & Shipping Dept. | 38. Hydrogenators | 59. Paint Storage | 81. Compressor Bldg. | SS. Sub Station |
| 14A. Storage | 39. Nitroso | 61. Maint. & Prod. | | |
| 14B. Storage | 40. Purification & Recovery | 62. Central Eng. | | |
| 14C. Drum Recovery | | 63. J. F. Wilson Trailer | | |

Table 1. SAMPLING PROTOCOL FOR BALTIMORE STUDY

Site	Cartridge Replicates	Time Period	Date
FMC ^a	1 - 2 - 3 - 4	11:00 AM - 2:50 PM	10/14/75
FMC	5 - 6 - 7 - 8	3:00 PM - 6:50 PM	10/14/75
FMC	9 - 10 - 11 - 12	7:00 PM - 10:50 PM	10/14/75
FMC	13 - 14 - 15 - 16	11:00 PM - 2:50 AM	10/14 - 15/75
FMC	17 - 18 - 19 - 20	3:00 AM - 6:50 AM	10/15/75
FMC	1 - 2 - 3 - 4	11:00 AM - 2:50 PM	10/15/75
FMC	5 - 6 - 7 - 8	3:00 PM - 6:50 PM	10/15/75
PS	1 - 2 - 3 - 4	10:00 AM - 1:50 PM	10/16/75
PS	5 - 6 - 7 - 8	2:00 PM - 5:50 PM	10/16/75
CP	1 - 2 - 3 - 4	9:56 AM - 1:46 PM	10/17/75
CP	5 - 6 - 7 - 8	2:10 PM - 6:00 PM	10/17/75
	m/e 74 (Quant.)		
	GC-MS-COMP (Qual.)		
FMC (W of Dimazine Destructor)	1 - 2	2:00 PM - 4:00 PM	11/19/75
Town of Fairfield	1 - 2	3:45 PM - 5:45 PM	11/20/75
Near Memirac Corp.	1 ^b - 2	8:20 PM - 10:20 PM	11/20/75
FMC (SW of Dimazine Destructor)	1 ^b - 2	11:50 AM - 1:50 PM	11/24/75
"	1 ^b - 2	1:55 PM - 3:55 PM	11/24/75
"	1 ^b - 2	6:35 PM - 8:35 PM	11/24/75
"	1 ^b - 2	6:35 PM - 8:35 PM	11/24/75
Northbridge and Convery St.	1 ^b - 2	1:48 PM - 3:48 PM	11/25/75
"	1 ^b - 2	3:59 PM - 5:59 PM	11/25/75
	m/e 74 (Quant.)		

^aParking lot

^bPart of Artifact Experiment

were used for quantitative collection of DMN, if present, during field sampling. The breakthrough volumes are shown in Table 2.

Gas-Liquid Chromatography/Mass Spectrometry Computer Analysis of Ambient Air Samples

The instrumental system (glc-ms-comp) used for the qualitative and quantitative analyses of nitrosamines and other ambient air pollutants and the inlet manifold used for recovering vapors trapped on Tenax GC cartridge samplers were as described elsewhere (1,2). The desorbed vapors were resolved by capillary gas-liquid chromatography and mass cracking patterns were automatically and continuously obtained throughout the glc run with a Varian CH-7 gas chromatograph/mass spectrometer. The cracking patterns and retention time data were accumulated on a magnetic tape deck and further processed by an on-line Varian 620i computer. Computer programs (KOSB) were employed which converted the acquired spectra into a sequential series of mass spectra and were correlated to peak retention time on a total ion current plot. Data output from the 620i computer was provided in two forms: (1) a teletype listing which contained the mass spectrum number, number of peaks in the cracking pattern, total maximum and minimum m/e peak intensity and standard deviation from calibration m/e and (2) an electrostatic plot of total ion current plots and/or normalized mass spectral. Single ion plots (e.g. m/e 74) were obtained as an analog output.

The operating parameters for the glc-ms-comp system for analysis of samples collected on glass cartridges from the Baltimore, MD area are shown in Table 3. Ambient air samples were analyzed on 55, 80 or 100 meter glass SCOT columns coated with either OV-101, OV-225 or DEGS stationary phase, respectively. The desorption of ambient air pollutants

Table 2. BREAKTHROUGH VOL FOR DMN

%F	2.2 g /cartridge (1.5 x 6.0) liters	2.93 g/cartridge (1.5 x 8.0) liters
50	385	513
55	332	493
60	280	373
65	242	322
70	204	272
75	224	244
80	163	217
85	156	207
90	148	197
95	127	170
100	107	143
105	93	123
110	79	103

including nitrosamines from the Tenax cartridge samplers was achieved at 265-270°C. A single stage glass jet separator interfaced the SCOT capillary columns to the mass spectrometer and was maintained at 200°C.

Identification of resolved components was achieved by comparing the mass cracking pattern of the unknown mass spectra to an 8 major peak index of mass spectra (1,2). In several cases the identification was confirmed by comparison with authentic compounds of the mass spectrum and the elution temperature on two different columns. Particular note was made of the relationship of the boiling point of the identified compound to its elution temperature and to its order of elution of constituent in homologous series since the OV-101 SCOT capillary column separates primarily on the basis of boiling point.

For quantitative analysis, the mass spectrometer was set in the single ion plotting mode whereby the selected master charge ratio was recorded on analog output. Quantitation of N-nitrosodimethylamine (by monitoring only m/e 74) was conducted on a 55 meter glass SCOT capillary coated with DEGS.

A standard curve for the response of the mass spectrometer set at m/e 74 vs the concentration of DMN was prepared by introducing known quantities of DMN vapor. Synthetic air-DMN vapor was synthesized and specified quantities collected on Tenax GC cartridges. By thermally desorbing the cartridge and monitoring the m/e 74, responses vs concentration were obtained. Cartridge samplers containing unknown concentrations of DMN were analyzed by monitoring the m/e 74 ion and obtaining the quantity/cartridge from the standard curve. Based on the volume of air sampled and the breakthrough volume for DMN, the concentration of N-nitrosodimethylamine was calculated in $\mu\text{g}/\text{m}^3$ of ambient air.

Artifact Experiments

The possibility of formation of DMN on the Tenax sampling cartridge was investigated. Since the Tenax cartridge sampler would concentrate the dimethylamine if present in ambient air (breakthrough volume approximately 35 l at 25°C), it is conceivable that in the presence of NO_x , the dimethylamine could become nitrosated to form DMN. Two types of experiments were conducted to either support or refute this possibility. The first set of experiments were conducted under laboratory conditions. Synthetic mixtures of NO, water vapor, and air were prepared at known concentrations of 1, 10 and 250 parts-per-billion (in NO) in 7 l glass bulbs. Tenax cartridges were loaded with approximately 5 parts-per-million of dimethylamine and the NO_x air vapor mixture was drawn through the cartridge. Cartridges were subsequently analyzed by single ion monitoring of the m/e 74. In another set of experiments, NO ($\sim 2 \times 10^{-7}$ mole/min, 1 ppm) was introduced into a stream of laboratory air which was pulled across a permeation tube containing dimethylamine at 1 l/min and into the Tenax cartridge. The sampling period was approximately 30 min. The dimethylamine permeation tube permeated at a rate of $\sim 9 \times 10^{-6}$ mole/min (~ 4.5 ppm). Thus, the amine to NO ratio was $\sim 45:1$.

Experiments were also conducted during field sampling to demonstrate whether an enhancement in the collection of N-nitrosodimethylamine could occur above background when Tenax cartridges were either pre-loaded with dimethylamine or when field air was drawn across a permeation tube containing dimethylamine and into the Tenax cartridge. The permeation tube containing dimethylamine ($\sim 2.4 \times 10^{-6}$ g/min) was placed in front of the glass fiber filter on the Tenax cartridge. Therefore in these experiments, the dimethylamine was introduced prior and after the glass fiber

filter. All experiments were conducted in at least triplicate. A total of ~120 l of ambient air was sampled in all cases.

Further experiments were conducted to also demonstrate whether storage of cartridges which had been exposed to dimethylamine and NO_x might gradually produce DMN. Replicate cartridges were stored at room temperature and at -15°C for periods of 1, 2 and 3 weeks.

RESULTS AND DISCUSSION

As shown in Figure 1, the sampling sites selected were (1) on the FMC property, (2) at the Patapsco Sewage Treatment Plant, and (3) the Chessie Coal Piers. During the ambient air sampling on the FMC property, the samplers were located either in the parking lot area or near the dimazine thermal destructor as shown in Figure 2.

The sampling protocol for the Baltimore study was depicted in Table 1. During the week of October 14, the sampling protocol included the collection of quadruplicate cartridges during each time period. One cartridge from the series of replicates were committed to gas chromatography/mass spectrometer computer for qualitative analyses. Another series of cartridges were designated for quantitation. The remaining cartridges served either as duplicates for qualitative or quantitative analysis. During the period from November 19 to November 25, the sampling protocol was slightly modified to allow only the collection of duplicate cartridges as well as inclusion of the field artifact studies (see Table 1). For quantitative analysis in all cases the calculation of the amount of N-nitrosodimethylamine in ambient air was based on the breakthrough volume and the ambient air temperature at the time of field sampling.

Figure 3 depicts the profile of ambient air pollutants in a sample taken during the day in the FMC parking lot area. The volume of air collected represents 75 ℓ . A 100 meter glass SCOT column containing OV-101 stationary phase was used to effect this separation. The remaining conditions for glc-ms-comp analysis is shown in Table 3. In contrast to Figure 3, Figure 4 represents a profile of ambient air pollutants in a sample taken during the subsequent evening at the same location in the

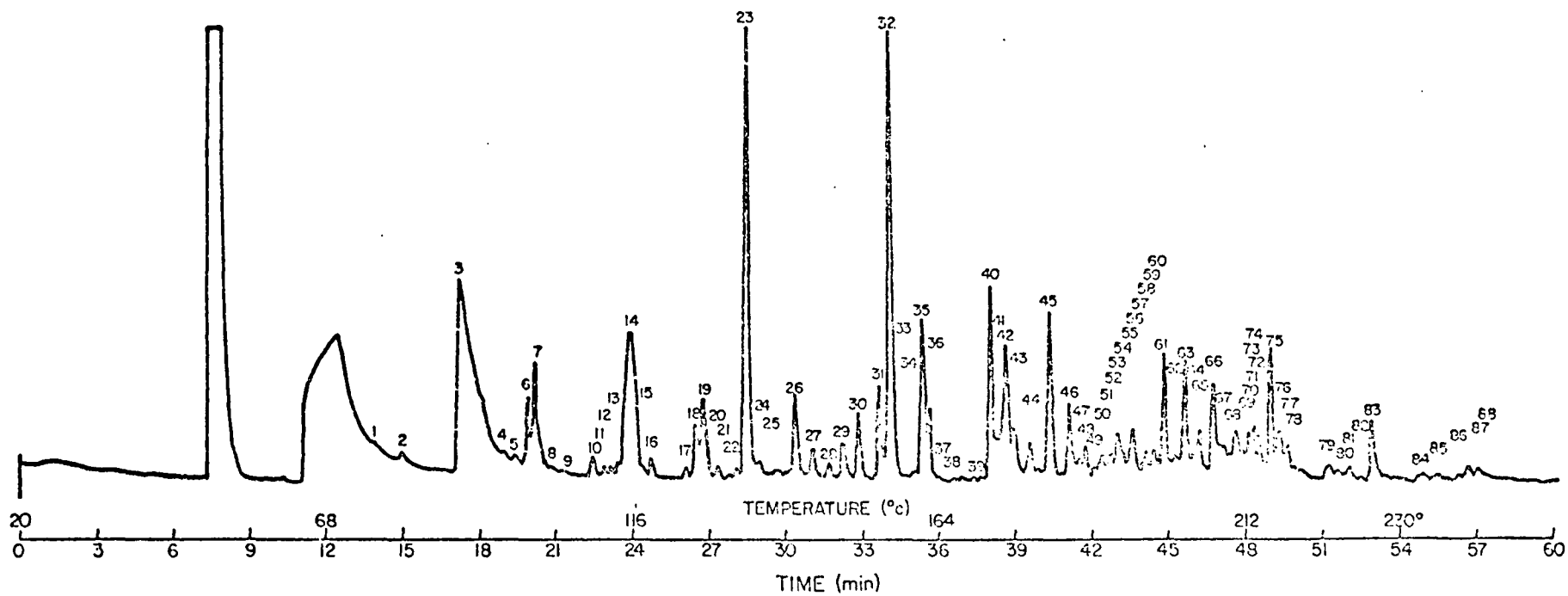


Figure 3. Profile of ambient air pollutants in a sample taken during the day on FMC parking lot. A 100M glass SCOT column containing OV-101 was used, see Table 3 for GLC/MS/COMP conditions.

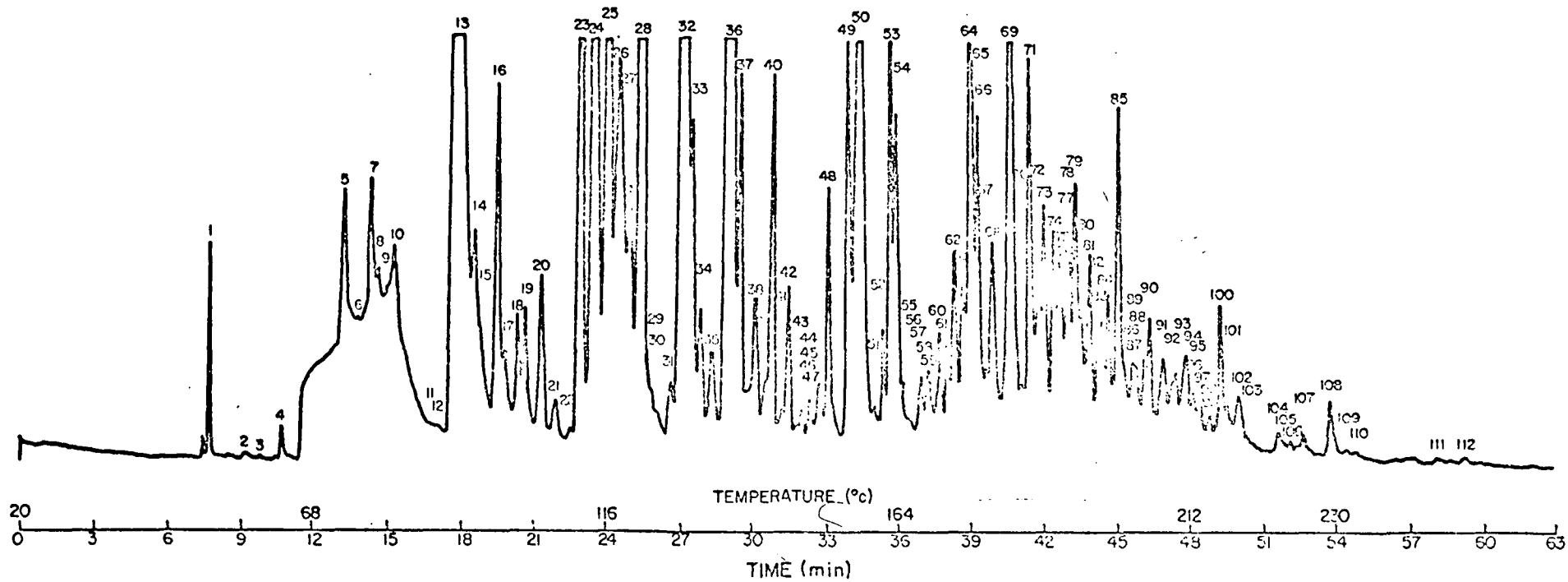


Figure 4. Profile of ambient air pollutants in a sample taken during the night on FMC parking lot. A 100M OV-101 glass SCOT column was used, see Table 3 for GLC/MS/COMP conditions.

FMC parking lot. Of particular interest here is the comparison of the overall quantity of ambient air pollutants which are significantly greater during the night sampling period as compared to the day. This phenomenon is frequently observed in our other studies where day and night sampling have been compared in geographical areas such as Los Angeles, CA, Houston, TX, St. Louis, MO, Denver, CO and the Kanawha Valley in W. VA.

Figure 5 depicts a profile of ambient air pollutants in a sample taken during the daylight hours near the Patapsco Sewage Treatment Plant. This sampling location is upwind from the FMC Corporation plant but downwind from the Sewage Treatment plant. The profile depicted in Figures 3 and 4 were taken downwind from the FMC Corporation.

Many of the pollutants resolved in Figures 3-5 have been identified, however only a partial listing will be discussed later.

A profile of ambient air pollutants in a sample taken during the daylight hours on the FMC parking lot which is identical to the samples taken as shown in Figure 3, is given in Figure 6. The chromatographic separation of constituents was achieved in this case on a semi-polar stationary phase, OV-225, coated in a 80M glass SCOT column. Semi-polar and polar phases are not well suited for resolution of non-polar pollutants. However, the resolution of semi-polar and polar constituents are significantly better. Figure 7 depicts the profile of ambient air pollutants in a sample taken again during the daylight in the FMC parking lot. In this case, the chromatographic column consisted of a 55M DEGS glass SCOT column.

The mass cracking pattern for N-nitrosodimethylamine as reported in the literature is shown in Figure 8. Upon electron impact, the

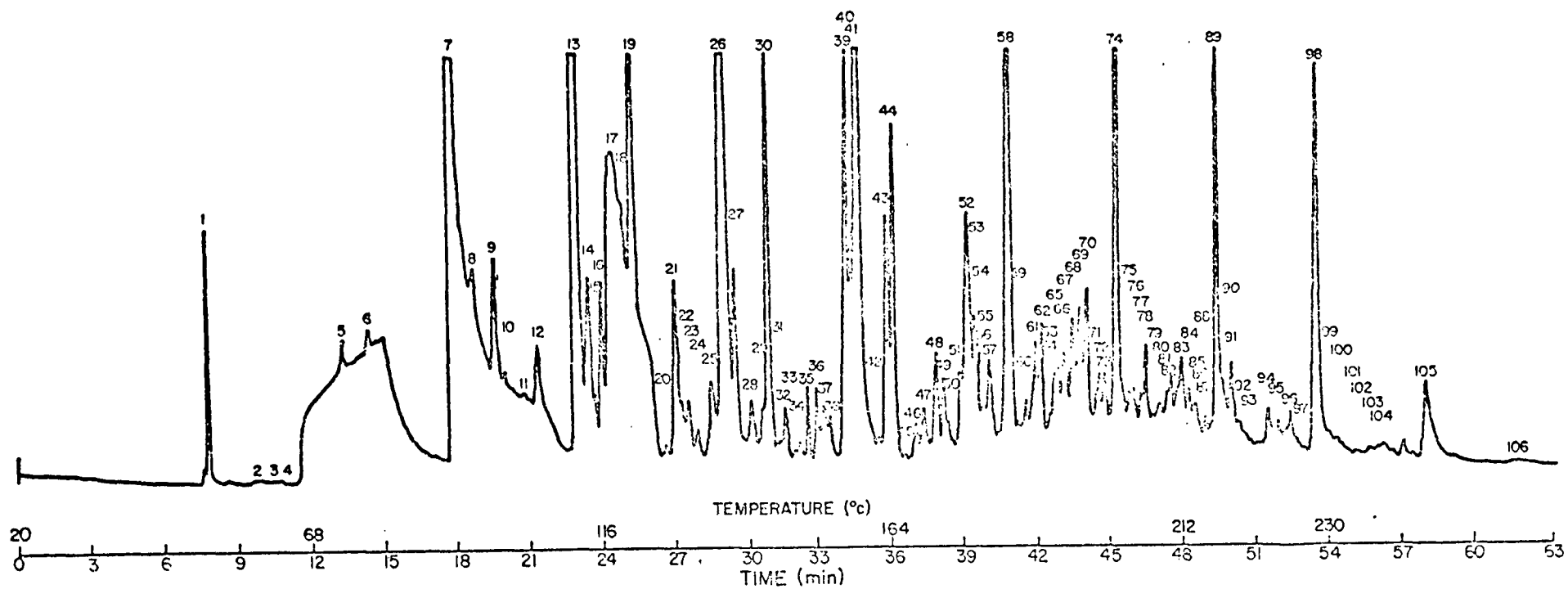


Figure 5. Profile of ambient air pollutants in a sample taken during the day near Patapsco Sewage Plant.

A 100M OV-101 glass SCOT column was used, see Table 3 for GLC/MS/COMP conditions.

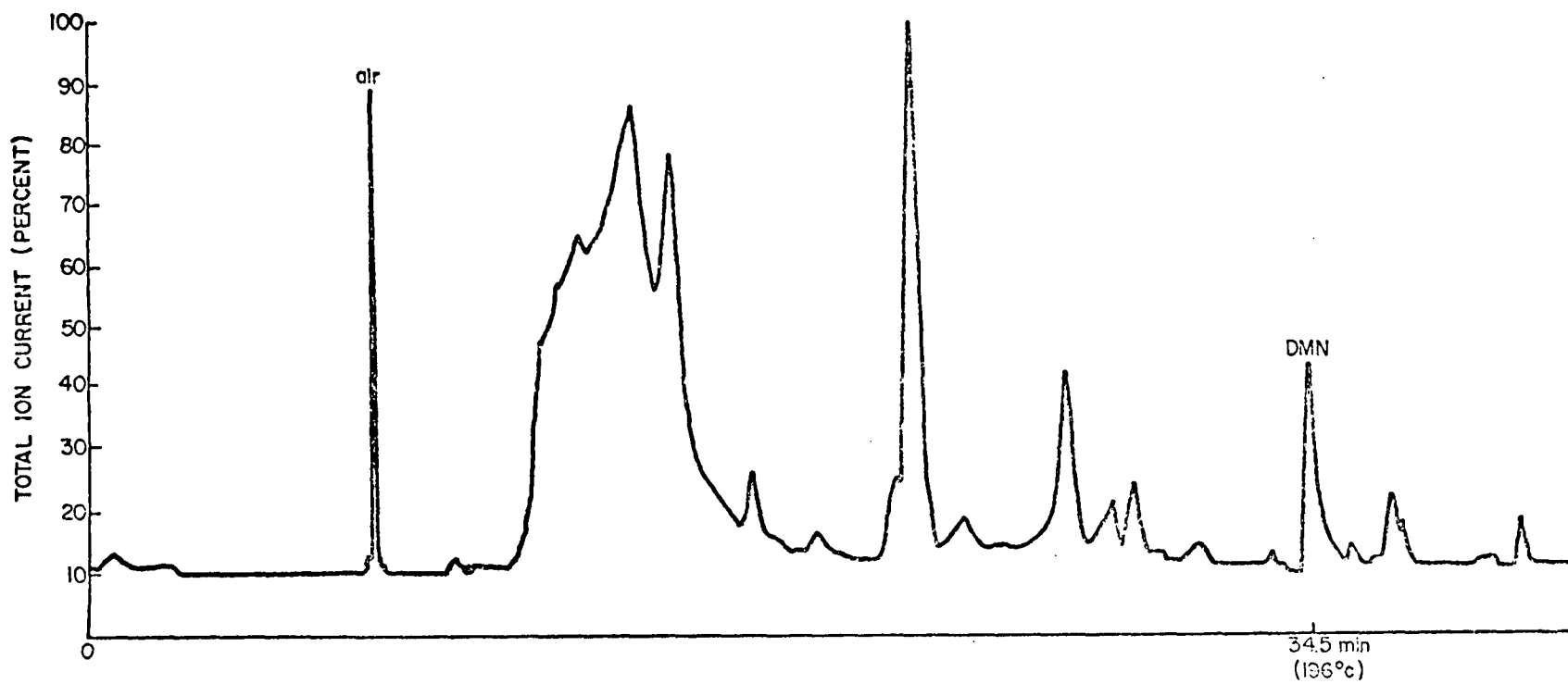


Figure 6. Profile of ambient air pollutants in a sample taken during the day on FHC parking lot. An 80M OV-225 glass SCOT column was used, see Table 3 for GLC/MS/COMP conditions.

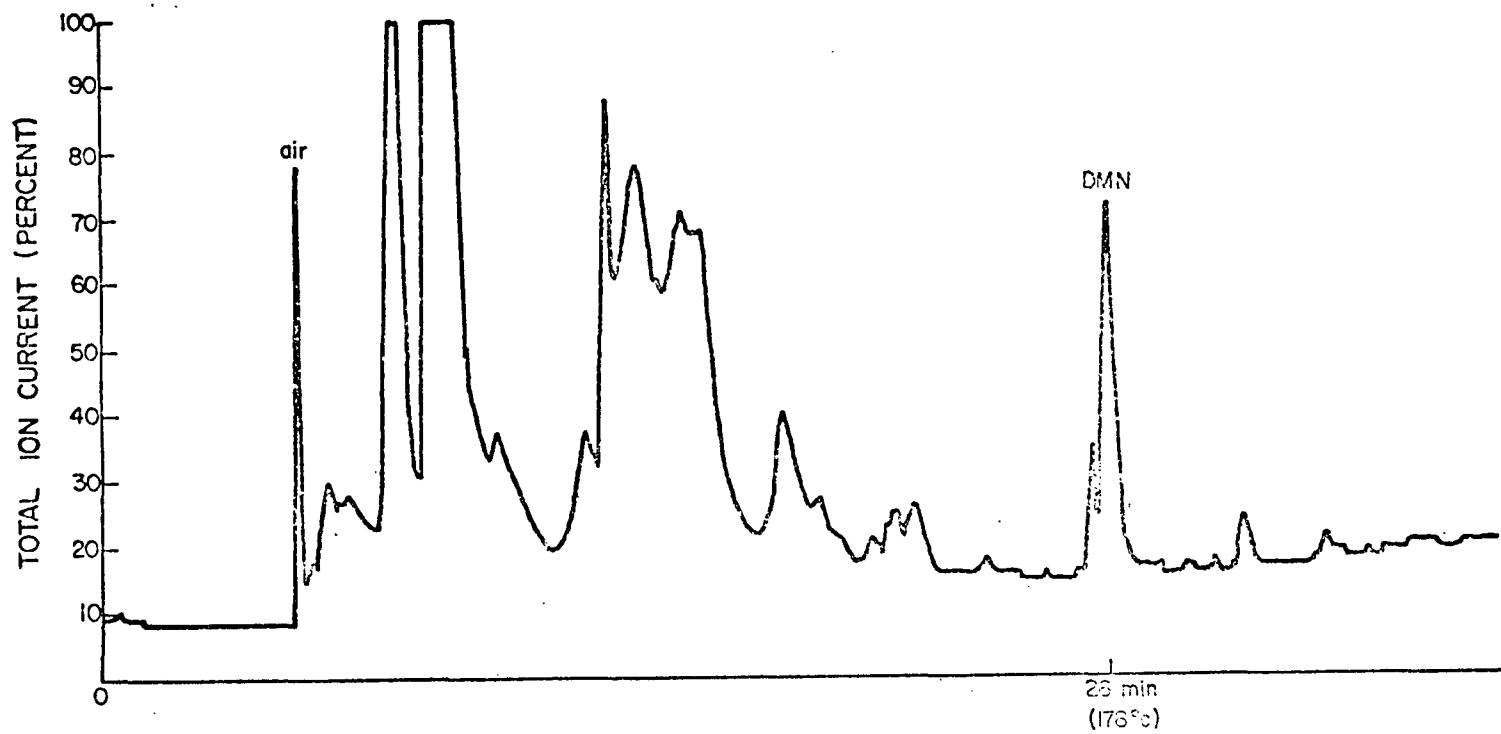
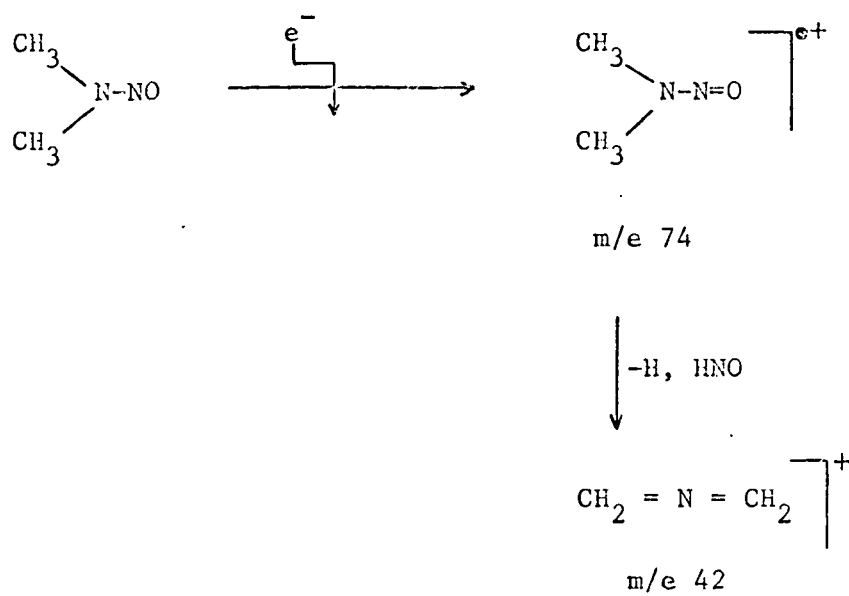


Figure 7. Profile of ambient air pollutants in a sample taken during the day on FMC parking lot. A 55M DEGS glass SCOT column was used, see Table 3 for GLC/MS/COMP conditions.

Figure 8. Mass cracking pattern for N-nitrosodimethylamine



N-nitrosodimethylamine forms a radical ion with an m/e of 74. This ion represents the most intense ion in the mass spectrum. Furthermore, the formation of the ion m/e of 42 is observed which represents ~68% of the base peak.

Figure 9 depicts the mass spectra of an authentic sample of N-nitrosodimethylamine which was chromatographed on the 100M glass SCOT coated with OV-225. Also shown in Figure 9 is an ambient air sample taken at the FMC parking lot which represents a mass spectrum from Figure 6. Comparison of this mass spectrum with the one for the authentic N-nitrosodimethylamine indicates that the unknown peak in Figure 6 is indeed N-nitrosodimethylamine. The mass spectra are essentially superimposable. The retention time of the authentic N-nitrosodimethylamine was identical to that of the unknown peak in Figure 6.

Likewise, Figure 10 depicts the mass spectrum of a peak in Figure 7 which subsequently has been identified as N-nitrosodimethylamine. This mass spectra was obtained on the 55M glass SCOT DEGS column. Furthermore, the retention times of the authentic N-nitrosodimethylamine standard was identical to that of the unknown peak in Figure 7.

A partial listing of the ambient air pollutants which have been tentatively identified in samples of ambient air taken in the FMC parking lot is given in Table 4. As it can be readily seen, many chlorinated hydrocarbons have been detected. The compounds which have been marked with an asterick represents air pollutants which have been identified for the first time. The remaining constituents have been observed fequently in other ambient air samples taken in areas such as Los Angeles, CA, Houston, TX, St. Louis, MO, Denver, CO and the Kanawha Valley, W VA. Of particular interest in this listing are the dimethylvinylchloride. The

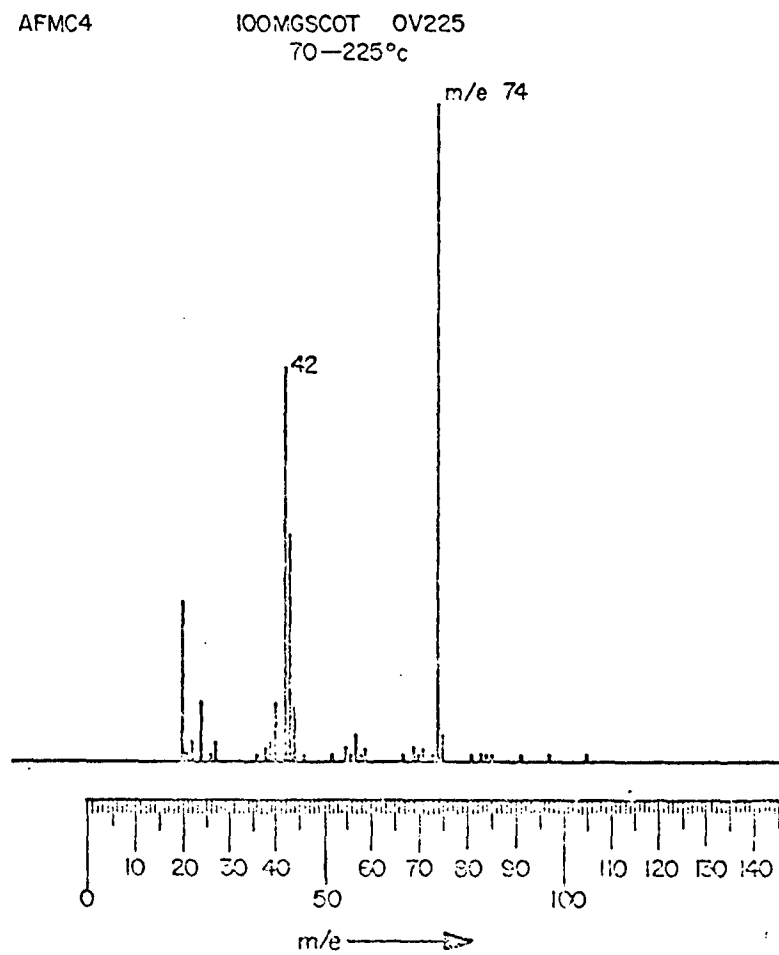
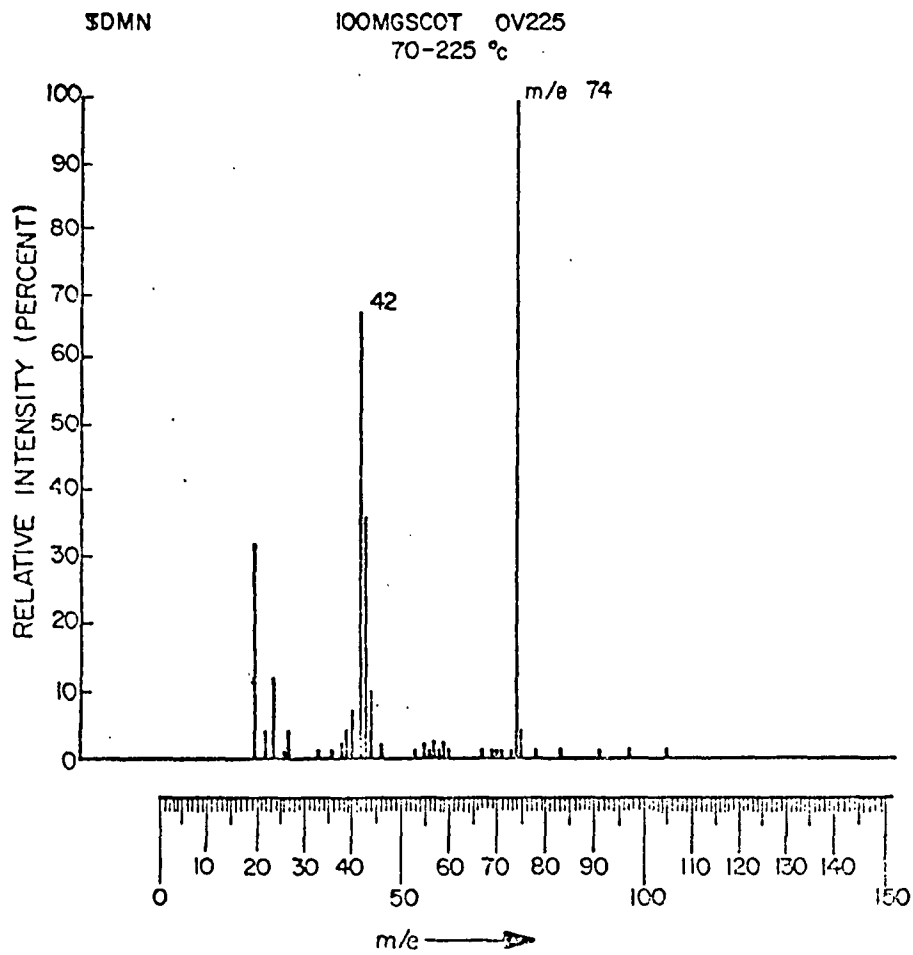


Figure 9. Mass spectra of N-nitrosodimethylamine. AFMC4 represents mass spectra from Figure 6.

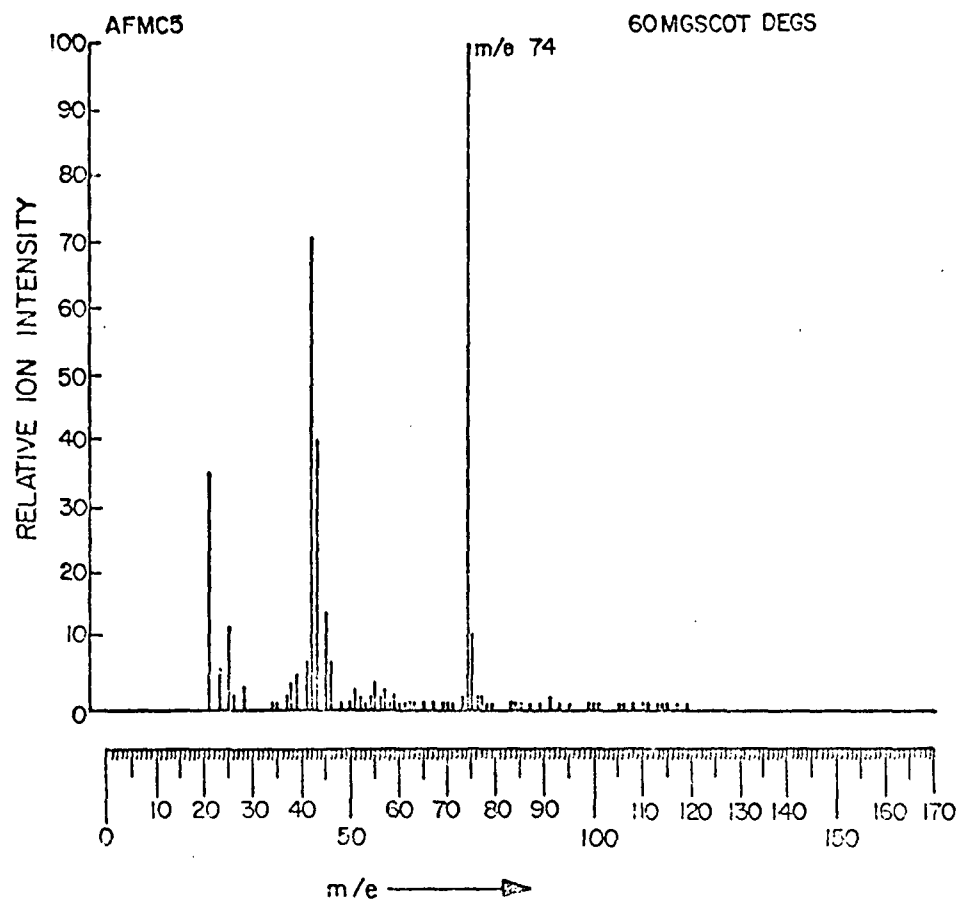


Figure 10. Mass spectrum of N-nitrosodimethylamine, see Figure 7 for chromatographic profile.

Table 4. PARTIAL LISTING OF AMBIENT AIR POLLUTANTS
TENTATIVELY IDENTIFIED IN SAMPLES
FROM FMC PARKING LOT

Compound	Chromatographic Peak No. ^a
*methylene chloride	2
*chloroform	5
1-chloro-2-methylpropene	6
3-chloro-2-methylpropene	7
*1,1,1-trichloroethane	9
*carbon tetrachloride	11
methylene dibromide	15
*trichloroethylene	15B
1-chloro-2-bromopropane	19
2,3-dichlorobutane	21
chlorobutene isomer	22
*tetrachloroethylene	27
dichlorobutene isomer	28
2-chloro-1,3-butadiene	29
*chlorobenzene	30
dibromochlorofluoromethane	33
1,1-dichloropropene + trichlorobutane	40
* <u>m</u> -dichlorobenzene	46
* <u>o</u> -dichlorobenzene	52

^aSee Fig. 3 for corresponding profile.

* These pollutants have been also found in many other areas of the country.

unequivocal identification of many of these compounds awaits the comparison of the mass spectra and the retention time of authentic compounds to those listed in Table 4.

The standard curve for N-nitrosodimethylamine generated on the gas chromatograph/mass spectrometer during single ion monitoring of m/e 74 is given in Figure 11. Because of the concentrations anticipated as a result of the qualitative analysis, the standard curve was constructed from 10-3,000 ng. This would represent the linear concentration range which is present on a cartridge sampler. The detection limit observed for the m/e 74 ion is ~.5 parts-per-trillion when the ambient air temperature during field sampling is 85°F. Thus, the minimum amount which can be quantitated is 5 parts-per-trillion.

Single ion plots for N-nitrosodimethylamine of replicate cartridges are shown in Figure 12 and 13. It is evident from these two figures that the replicate cartridge sampling technique is highly reproducible. All single ion monitoring for quantitative analysis was conducted on the 55M glass SCOT capillary coated with DEGS.

Using the described procedures for quantiting N-nitrosodimethylamine, the concentration of the nitrosodimethylamine in ambient air was determined in the immediate area of the FMC plant in Baltimore, MD. Table 5 depicts these results. During the week of October 14, the highest values observed were ~13.4 $\mu\text{g}/\text{m}^3$ in the late afternoon. Only trace quantities were observed upwind from the FMC plant at the Patapsco Sewage Treatment Plant. Significant quantities were also measured at ~1/2 mile downwind from the FMC Corporation at the Chessie Coal Piers.

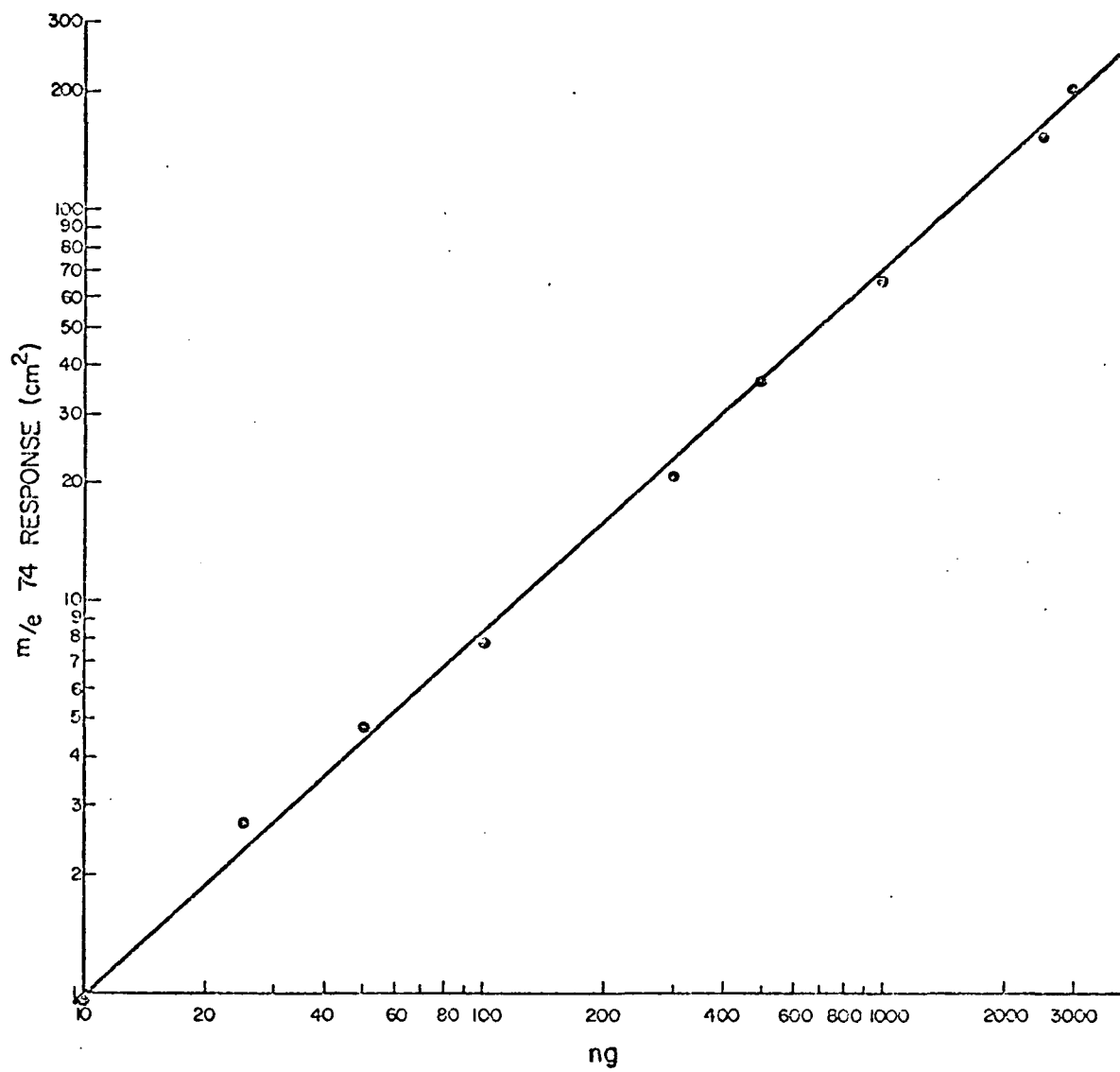


Figure 11. Standard curve for N-nitrosodimethylamine

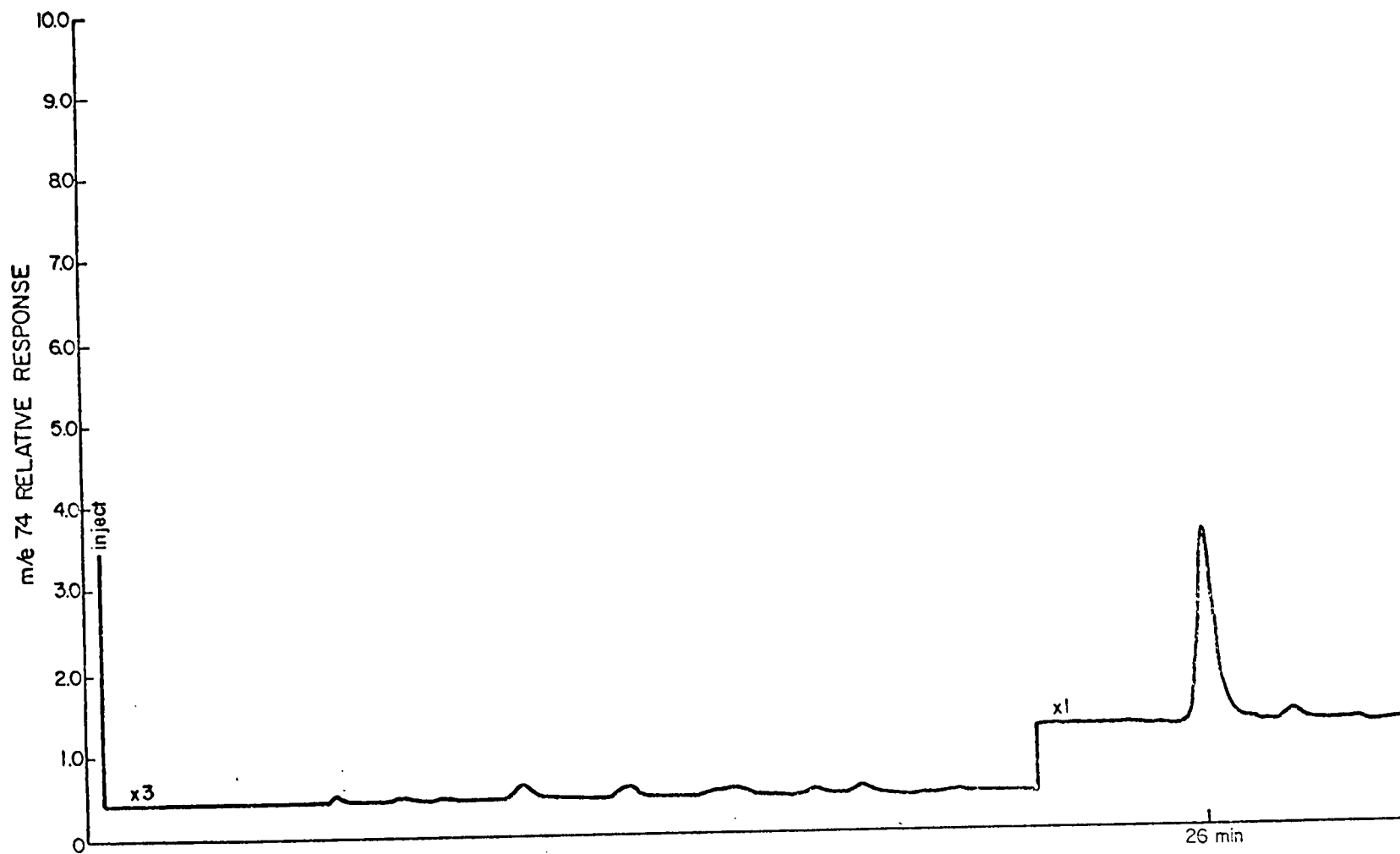


Figure 12. Single ion plot (m/e 74) for N-nitrosodimethylamine, see Table 3 for GLC/MS/COMP conditions.

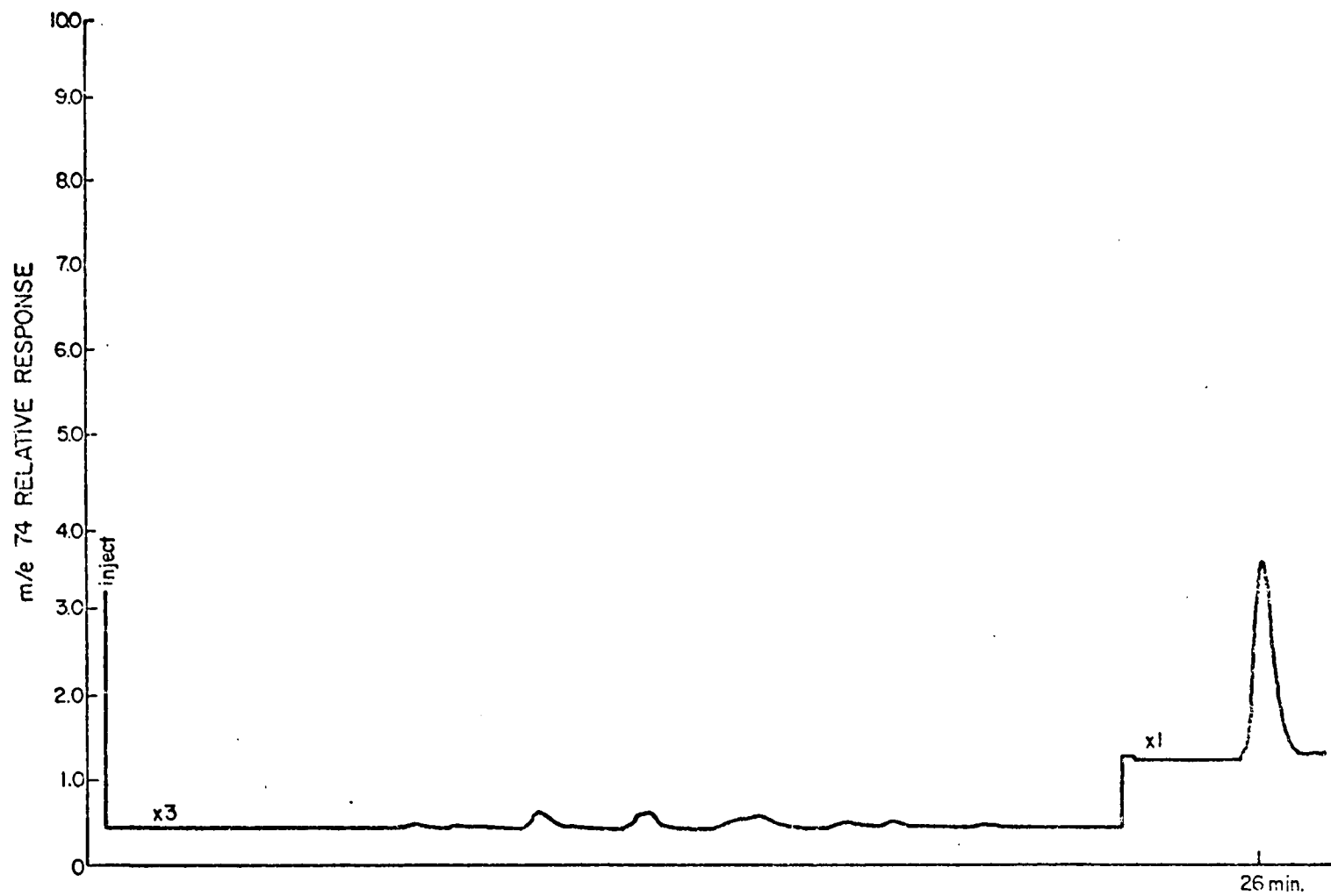


Figure 13. Single ion plot (m/e 74) for N-nitrosodimethylamine (replicate cartridge of Fig. 12), see Table 3 for GLC/MS/COMP conditions.

Table 5. SAMPLING CONDITIONS AND CONCENTRATION OF N-NITROSODIMETHYLAMINE IN AMBIENT AIR

Date	Time (EDT)	Location	Temperature (°F)	RH (%)	Wind		Molecular Verification		DMN Concentration (ng/m ³)
					Direction	Speed (KTS)	DEGS	OV-225	
10/14	11-2:50 PM	FMC (P. Lot)	83	40-50	WNW	10	x		2,133
	3-6:50 PM	FMC (P. Lot)	85	45-50	WNW	7	x		10,500 ± 1,167 (±11%)
	7-10:50 PM	FMC (P. Lot)	74	65-85	WSW	7	x	x	1,375 ± 125 (±9%)
10/15	11-2:50 AM	FMC (P. Lot)	65	90-97	Calm	-			416
	3-6:50 AM	FMC (P. Lot)	61	97	S				571
10/15	11-2:50 PM	FMC (P. Lot)	85	40-50	WSW	8-10	x		3,200
	3-6:50 PM	FMC (P. Lot)	83	40-50	WSW	5-8	x	x	13,437 ± 937 (±7%)
10/16	10-1:50 PM	Sewage Plant	72	45-57	NNW	9-11	-		trace
	2-5:50 PM	Sewage Plant	72	45-50	NNW	5-12	-		trace
10/17	9:56-1:46 PM	Chessie Pier	55	88-94	ENE	7-9	x		909
	2:10-6:00 PM	Chessie Pier	55	94-100	E	10-14	x	x	84

Table 6. SAMPLING CONDITIONS AND CONCENTRATIONS OF N-NITROSODIMETHYLAMINE

Date	Time	Wind/Temperature	Location	$\mu\text{g}/\text{m}^3$
11/19/75	2:00 PM-4:00 PM	E-SE	West-Northwest of dimazine thermal destructor on FMC property.	32 ± 1.5
11/20/75	3:45 PM-5:45 PM	6KN-SW, 3KN-SE, 63°	Town of Fairfield, 200 yd from residential area, Conoco parking lot.	1.95 -
11/20/75	8:20 PM-10:20 PM	6KN-S, 57°	Memirac Corp., north of FMC plant.	1.36 ± 0.51
11/24/75	11:50 PM-1:50 PM		FMC lot, SW of dimazine plant	20 ± 4
11/24/75	1:55 PM-3:55 PM		"	14 ± 0.2
11/24/75	6:35 PM-8:35 PM		"	26 ± 0.5
11/25/75	1:48 PM-3:48 PM		Northbridge & Cannery St, downwind of FMC	7.6

Approximately one month later during the period of November 19-25, additional values for N-nitrosodimethylamine were obtained. Again on the FMC property, high values of this nitrosamine were detected. However, the sampling locations were much closer to the general area of the dimazine thermal destructor. The concentrations ranged from 14-32 $\mu\text{g}/\text{m}^3$.

Experiments on artifact formation, i.e. production of DMN on the cartridge sampler during field sampling could not be detected when the air stream was deliberately spiked with dimethylamine from a permeation tube. Laboratory experiments indicated that 10 or more ppm of NO/NO₂ spiked into an air stream containing DMA can produce DMN; 1 ppm or less yielded undetectable quantities. We found it necessary to purify our DMA by subambient vacuum distillation prior to any field experiments since it contained 1 part in 10⁵ DMN.