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California
Environmental
Protection
Agency

Air Resources Board

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MEMORANDUM

TO: John Sanders, Ph.D., Chief
Environmental Monitoring and Pest Management Branch
Department of Pesticide Regulation

FROM: George Lew, Chief
Engineering and Laboratory Branch

DATE: June 4, 1997

SUBJECT: MIC/MITC MONITORING FINAL REPORT

Thank you for your comments on the final draft of the MIC/MITC monitoring report which were provided in your May 5, 1997 memorandum. Don Fitzell of my staff contacted Pam Wofford to discuss your comments and our responses are given below.

a) Pam had commented that it was more appropriate to round off the data after all the calculations have been made. We agree, however, the data presented in the tables also included intermediate values (volume of air sampled). We chose to present the correct number of significant figures in this column; therefore our final numbers differ slightly from Pam's calculation based on using all figures until "rounding" the final result.

b) Pam requested some explanation for the consistent concentration of MIC found in the blank background samples. We did not attempt to explain the presence of the MIC nor MITC found in the background samples because we have no scientific basis to make that judgement. As you may know, some members of the Metam Sodium Task Force contend that MITC is a naturally occurring by-product of nature. While we are skeptical of this assertion, we cannot disprove it either. Therefore to avoid unfounded speculation, we did not state that the MITC (and MIC since it is a breakdown product of MITC) were a result of unobserved applications of metam sodium in the area.

c) Pam noted that the report did not mention collection of meteorological information by datalogger. We have added a sentence on Page 4 to indicate that a

datalogger is used and the met data is provided to your agency on diskette.

The final MIC/MITC report is attached. If you or your staff have questions or need further information, please contact me at 263-1630 or Don Fitzell at 327-0055.

Attachment

cc: Lynn Baker, SSD w/attachment

State of California
California Environmental Protection Agency
AIR RESOURCES BOARD

Ambient Air Monitoring for MIC and MITC after a Soil Injection Application of Metam Sodium in Kern County During August 1995

Engineering and Laboratory Branch
Monitoring and Laboratory Division

Test Report No. C94-046A

Report Date: May 20, 1997

APPROVED:

Don Fitzell, Project Engineer

Cynthia L. Castronovo, Manager,
Testing Section

George Lew, Chief,
Engineering and Laboratory Branch

This report has been reviewed by the staff of the Air Resources Board and approved for publication. Approval does not signify that the contents necessarily reflect the views and policies of the Air Resources Board, nor does mention of trade names or commercial products constitute endorsement or recommendation for use.

Ambient Air Monitoring for MIC and MITC after a Soil Injection Application of Metam Sodium in Kern County during August 1995

This report presents the results of ambient air monitoring after a ground injection application of metam sodium at a selected field in Kern County. Samples were collected before, during and for 72 hours after the start of the application. Samples were analyzed for methyl isothiocyanate (MITC) and methyl isocyanate (MIC). MITC levels ranged from 0.24 ug/m^3 to 250 ug/m^3 while MIC values ranged from 0.2 ug/m^3 to 5.8 ug/m^3. Both compounds were detected throughout the monitoring period, including the 12 ½ hour background sampling prior to the application. The source of this background could not be determined.

Acknowledgments

Jack LaBrue of the Air Resources Board was the Instrument Technician. David German of Wilbur Ellis arranged for a suitable field to monitor. Assistance was also provided by Cara Roderick, Lynn Baker, and Ruth Tomlin of the ARB's Air Quality Measures Branch. Mario Fracchia, Diamon Pon and Paul Larkin of the Environmental Health Laboratory Branch, Department of Health Services analyzed all samples. Mark Lee of the California Department of Food and Agriculture performed the confirmation of the MIC.

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Ambient Air Monitoring for MIC and MITC after a Soil Injection Application of Metam Sodium in Kern County during August 1995

I. INTRODUCTION

The Air Resources Board (ARB) Engineering and Laboratory Branch (ELB) staff conducted a three-day source impacted ambient monitoring program for an application of metam sodium to a field in Kern County during August 1995. This monitoring was performed at the request of the California Office of Environmental Health Hazard Assessment (OEHHA). The monitoring occurred from August 23 through August 27, 1995. This monitoring was conducted to determine if methyl isocyanate (MIC) is a breakdown product of metam sodium's primary breakdown product, methyl isothiocyanate (MITC), under conditions which occur after an application of metam sodium to a field. Recent research has indicated that MIC may be an atmospheric breakdown product of MITC (Geddes, J; Miller, G; Taylor, G, Jr., "Gas Phase Photolysis of Methyl Isothiocyanate", Environ. Sci. Technol., **29**, 2590-2594, 1995). Samples were also collected to determine what levels of MITC were present.

The Pesticide Use Report for 1995 indicates metam sodium is most widely used prior to planting carrots (5,308,000 pounds), tomatoes (2,888,000 pounds), potatoes (1,448,000 pounds), and cotton (1,213,000 pounds). These crops accounted for over two thirds of the total 15,274,000 pounds used during that year.

II. DESCRIPTION

Metam sodium (molecular weight 129.18 g/mole) is a soil fumigant used as a fungicide, herbicide, insecticide and nematicide. It has an unpleasant odor, similar to that of carbon disulfide. It is soluble in water (72.2 g/100 mL), moderately soluble in alcohol and sparingly soluble in other solvents. Application is by soil injection or sprinkler irrigation. Metam sodium rapidly breaks down in the presence of water into MITC, which has pesticidal activity. (Merck Index, Twelfth Edition, 1996). As a result of monitoring conducted in 1993, metam sodium is now regulated as a restricted use material under section 6400, Title 3 of the California Code of Regulations.

MITC is a crystalline substance (m.p. 35-36°C, b.p. 119°C) with a molecular weight of 73.12. It is slightly soluble in water and freely soluble in alcohol and ether (Merck Index, Twelfth Edition, 1996).

Lethality values for MITC via inhalation vary greatly. An LC_{50} in rats of 1900 mg/m^3 (655 ppm) for a 1-hour exposure was reported in the most complete study available. There were no lethalities at 600 mg/m^3 (207 ppm). The Reference Exposure Level (REL) to prevent discomfort (based on irritation of the ocular mucosa in cats) is 0.0014 mg/m^3 (0.5 ppb),

after adjusting for a 1-hour exposure. The REL to prevent life-threatening effects is 150 ppb for a 1-hour exposure, based on the highest nonlethal level in mice. (Alexeeff, G., et. al., Risk Analysis, **14**, No. 2, 195 (1994)).

MIC (molecular weight 57.05) is a colorless liquid with a boiling point of 39-40°C. It has a vapor pressure of 300 mm Hg at 20.6°C. The LD_{50} in male rats for a single dose is 140 mg/kg. It has been reported that exposure to 2 ppm for 1 - 5 minutes produces tears and irritation of the nose and throat (G. Kimmerle, A. Eben, Arch. Toxicol. **20**, 235 (1960)).

III. SAMPLING LOCATIONS

A field of about 80 acres (FIGURE I) was selected by David German of Wilbur-Ellis Company and approved by ARB staff for application monitoring. Five samplers were set up (see FIGURE II): one on the eastern perimeter (site E) at a distance of about 12 yards from the field, two about 13 yards from the southern perimeter (site S), one about 13 yards from the northern perimeter (site N) and one approximately 20 yards from the western side of the field (site W). All samplers were located at a height of about 1 ½ meters above ground. A meteorological station with a strip chart recorder was set up adjacent to site W to determine wind speed and direction. A second meteorological station equipped with a data logger was also set up near the southwest corner of the field. Site S took duplicate samples to determine precision of the data. All other sites collected single samples.

The application was by tractor and took about 19 hours. The application began in the northwest corner. The tractor traversed from north to south and from west to east. The application began midday on August 23 and was mostly completed by nightfall the following day. Only "touch up" application occurred for about an hour on the morning of August 25 where small areas near the eastern border needed coverage. The application occurred at a soil depth of 10 to 12 inches. The formulation was Soil Prep by Wilbur-Ellis which contains 33% active ingredient. The application rate was 50 gallons per treated acre (3.1 pounds active ingredient per gallon). A liquid fertilizer and Till-it zinc (chelate) were also applied. The pesticide use recommendation for this application is in APPENDIX I.

IV. SAMPLING METHODOLOGY

The samples were collected using the apparatus shown in APPENDIX II, Attachment I. Measured quantities of air were pulled through the sample tubes containing either charcoal for MITC or XAD-7 treated with 1-(2-pyridyl) piperazine for MIC sampling. The charcoal tubes are 8 mm x 110 mm, with 400 mg in the primary section and 200 mg in the secondary (SKC catalog #226-09). The XAD-7 tubes are 8 mm x 110 mm with 175 mg in each tube (SKC catalog #226-97) with two tubes per set. Tube A of each set was designated as the primary, tube B as secondary. The treated XAD-7 tubes were stored either in a freezer or on dry ice until used in the field. Subsequent to sampling, all tubes were stored on dry ice in the field and on blue ice for delivery (approximately 1 ½ hours) to the laboratory. In the laboratory they were stored in a freezer until analysis was complete.

Sampling trains designed to operate continuously were set up at the four sampling sites

identified in FIGURE II. The sampling schedule outlined in the protocol (APPENDIX II) was followed as closely as possible. Each sample train consisted of a charcoal tube and an XAD-7 tube set (tube A and tube B), each with its own cover, Teflon fittings, tubing, rain shield, flow meter with valve, and train support. Ambient air was pulled through both simultaneously using a battery-powered double-headed vacuum pump. The tubes were approximately 1.5 meters above the ground. Each tube was prepared for use by breaking off each sealed glass end and then immediately inserting the tube into a Teflon fitting. The tubes were oriented in the sampling train according to a small arrow printed on the side of each tube indicating the direction of flow. Covers were placed around the tube to protect the adsorbent from exposure to sunlight. For the MIC sampling, the pair of XAD-7 tubes in each set (tube A as the primary, tube B as the secondary) were placed in series since each individual tube did not contain a backup section.

The sample pump was started and the flow through a rotometer was adjusted with a metering valve to an indicated reading of 2.0 liters per minute (L/min) for the charcoal tubes and 70 milliliters per minute (mL/min.) for the XAD-7 tubes. A leak check was performed by blocking off the sample inlet. The sampling train was determined to be leak-free, if the indicated flow dropped to zero. Upon completion of a successful leak check, the indicated flow rate was again reset if necessary. The date, time, and site location were recorded in the field log. Calibration prior to use in the field indicated that an average flow rate of 1.9 L/min. was actually achieved for the charcoal tubes when the rotometers were set to 2.0 L/min. The setting of 70 mL/min. for the XAD-7 tubes actually produced a flowrate of 90 mL/min. These average flow rates were used to calculate all sample volumes.

At the end of each sampling period the final indicated flow rate (if different than the set value), the stop date and time were recorded. The tubes were then removed from the sample train, end caps installed on both ends, and identification labels affixed to each tube. Each tube was then placed in a culture tube with a screw cap and stored with dry ice in a covered chest while in the field. The samples were taken to Sacramento where they were stored in a freezer until delivery to the Environmental Health Laboratory Branch (EHLB) in Berkeley where they were again stored in a freezer until analysis. Transfer from Sacramento to Berkeley was accomplished with the samples stored on blue ice.

V. ANALYTICAL METHODOLOGY

The charcoal tubes recovered from each sampler were analyzed by the EHLB staff of the Department of Health Services, in Berkeley. The charcoal in the primary and secondary section of each sample tube was extracted with carbon disulfide followed by gas chromatography (GC) separation on a DB-5 capillary column and measurement by a nitrogen/phosphorous detector (NPD) (see APPENDIX III). All of the MITC samples were analyzed between September 6 and September 20, 1995.

The XAD-7 tubes for MIC analysis were extracted with 4 mL of acetonitrile followed by high performance liquid chromatography with a fluorescence (240 nm excitation, 370 nm emission) detector. The detailed method can be found in APPENDIX III. All MIC samples were analyzed between August 31 and October 3, 1995.

VI. RESULTS

The monitoring results for MITC are shown in TABLE I. The monitoring results for MIC are shown in TABLE II. A summary of the on-site meteorological data is presented in TABLE III. A combined summary of the monitoring and meteorological data for MITC and MIC are presented in TABLE IV and TABLE V, respectively. The meteorological data presented in these tables are from the strip chart recorder of the mechanical monitor, not the station with the data logger. The data logger information was provided to DPR in disk format for modeling purposes. Additional detailed meteorological data from the California Irrigation Management Information System (CIMIS) station, located in Seeley, is presented in APPENDIX IV. None of the results presented in this report have been corrected for percentage recovery. The MIC results, however, have been corrected for the blank media background (APPENDIX V).

TABLES I and II present the concentrations as both ug/m^3 and ppbv (parts per billion, volume to volume). The equations used for the conversions are:

$$\text{MITC} \quad \text{ppbv} = \text{ug/m}^3 \times \frac{(8.21 \times 10^{-2}\ \text{L-Atm/mole-°K})(298\text{°K})}{(73.12\ \text{g/mole})(1\ \text{Atm})}$$

$$\text{MIC} \quad \text{ppbv} = \text{ug/m}^3 \times \frac{(8.21 \times 10^{-2}\ \text{L-Atm/mole-°K})(298\text{°K})}{(57.05\ \text{g/mole})(1\ \text{Atm})}$$

The values reported as "less than" (<) by the laboratory for blanks or secondary sections of the sample tube indicate the limit of detection (LOD) for that sample. The LOD represents the concentration calculated for the upper limit of the 99% confidence interval for the intercept for the applicable calibration curve. This is the upper limit of the response that could be observed when there is no MITC (or MIC) present.

All of the MIC results were corrected for blank background. The average background (0.05 ug/tube) for blank tubes was subtracted from any tube which had a measured level greater than the background. If the detected level was less than the average background, it was not included in the total. The MIC levels ranged from 0.2 ug/m^3 to 5.8 ug/m^3.

The levels of MIC detected were so low that in order to confirm by liquid chromatography/mass spectroscopy (LC/MS) several of the samples had to be combined. The complete report on the confirmation of MIC in the samples is presented in APPENDIX VII.

MITC levels ranged from 0.24 ug/m^3 to 250 ug/m^3 while MIC values ranged from 0.2 ug/m^3 to 5.8 ug/m^3. It should be noted that low levels of both compounds were detected during the 12 ½ hour background sampling period. No nearby fields appeared to have been prepared for planting or soil injection of metam sodium. It is unknown if these low levels detected during the background sampling were due to an unknown application of metam sodium in the area or possibly an interferant collected from the ambient air. Earlier studies indicate mixed results from background monitoring. In a report by DPR, "Air Monitoring for

Methyl Isothiocyanate During a Sprinkler Application of Metam Sodium" (June, 1994) it was reported no MITC was detected during a 12 hour background sampling period preceding the application. A 2 ½ hour background sampling by the ARB ("Ambient Air Monitoring in Contra Costa County during March 1993 after an Application of Metam Sodium to a Field") also resulted in no MITC detected. However, monitoring for 2 1/4 hours prior to an application in Kern County ("Ambient Air Monitoring in Kern County During Summer 1993 after a Ground Injection Application of Metam Sodium to a Field") resulted in low (2-4 ug/m^3) levels being found.

VII. QUALITY ASSURANCE

Reproducibility, linearity, collection and extraction efficiency, minimum detection limit and storage stability are described in the Analytical S.O.P.s for each compound found in APPENDIX III. Although the storage stability data for MIC in its SOP is only for 18 days, the laboratory spikes prepared prior to sampling and analyzed throughout the analyses of the field samples resulted in an overall recovery of 95.4% (APPENDIX V, page 7). This indicates the samples were stable for the full time required for the analyses. The procedures outlined in the Quality Assurance Plan (APPENDIX II, Attachment II) were followed as closely as possible. In addition, a flow rate audit, a systems audit and an analytical performance audit were performed by ARB's Quality Management Operations and Support Branch (QMOSB) (see APPENDIX VI).

All of the spikes prepared by the laboratory conducting the analysis (TABLE IV, Trip Spikes and Blanks, In-House Laboratory Spikes) resulted in good recovery levels. However, spikes prepared by the QMOSB (APPENDIX VI) resulted in low recoveries for MIC spikes, and reasonable recoveries (66% for three spikes if the two spiked at the detection limit are discarded) for MITC spikes.

Based on the additional QA work performed by EHLB, it appears the MIC spiking solution prepared by Chem Service was probably lower in concentration than indicated, resulting in poor or no recovery. Confirmation of this can be found by the favorable comparison of EHLB's standard with the one provided by OSHA in Salt Lake City. Because of the problem with the spiking solution, QMOSB invalidated the audit sample results for MIC.

QMOSB compared its MITC spiking solution with the MITC standards used by EHLB. Unfortunately this was not done until late in the study, after the expiration date of the QMOSB standard provided by Chem Service. The result was 2.32 times the value certified by Chem Service. This is best explained by evaporation of the solvent (carbon disulfide) from the QMOSB spiking solution. The complete results of the QMOSB audit are presented in APPENDIX VI.

FIGURE I. MIC/MITC Monitoring Area

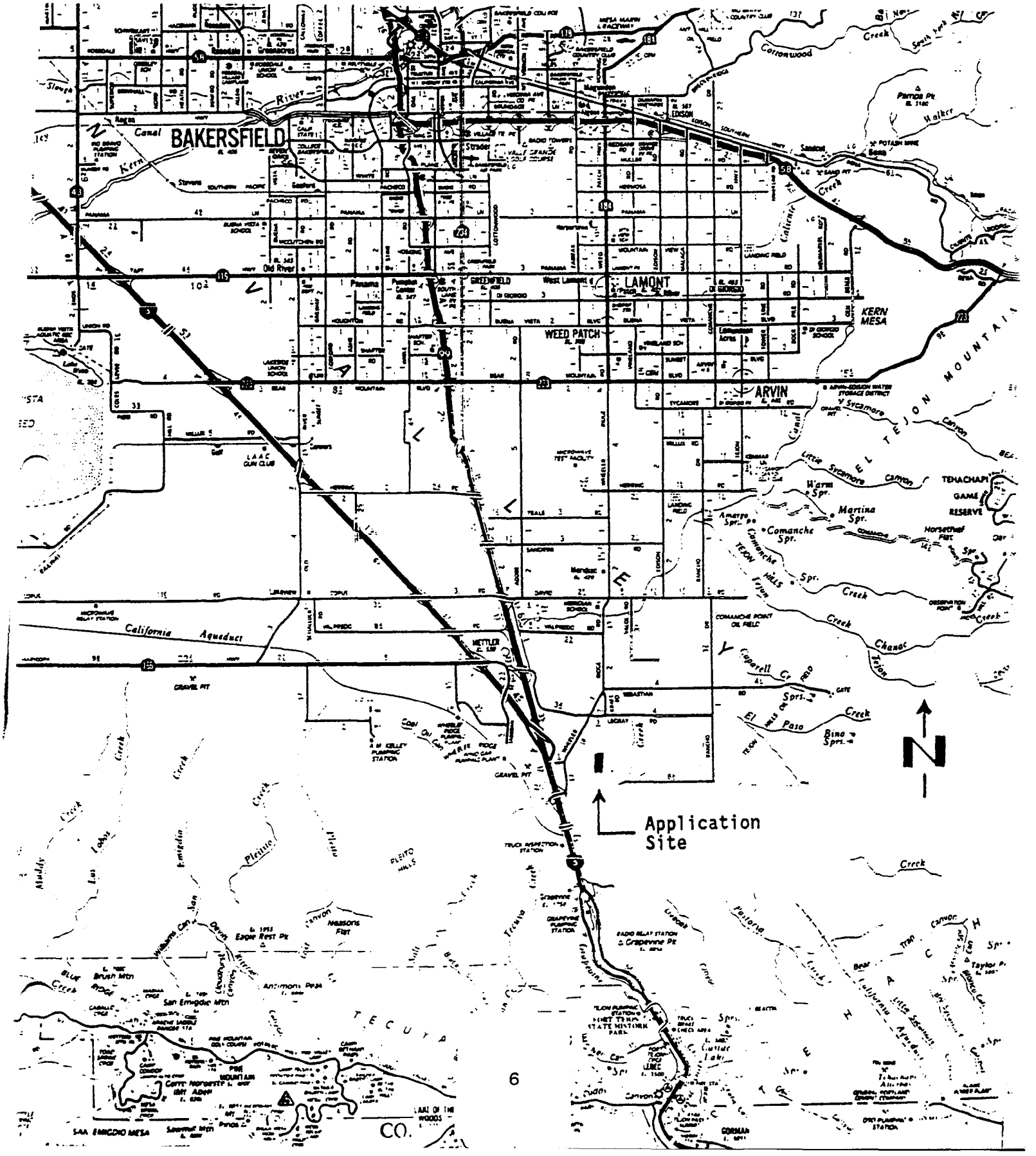


FIGURE II. MIC/MITC Monitoring Sites

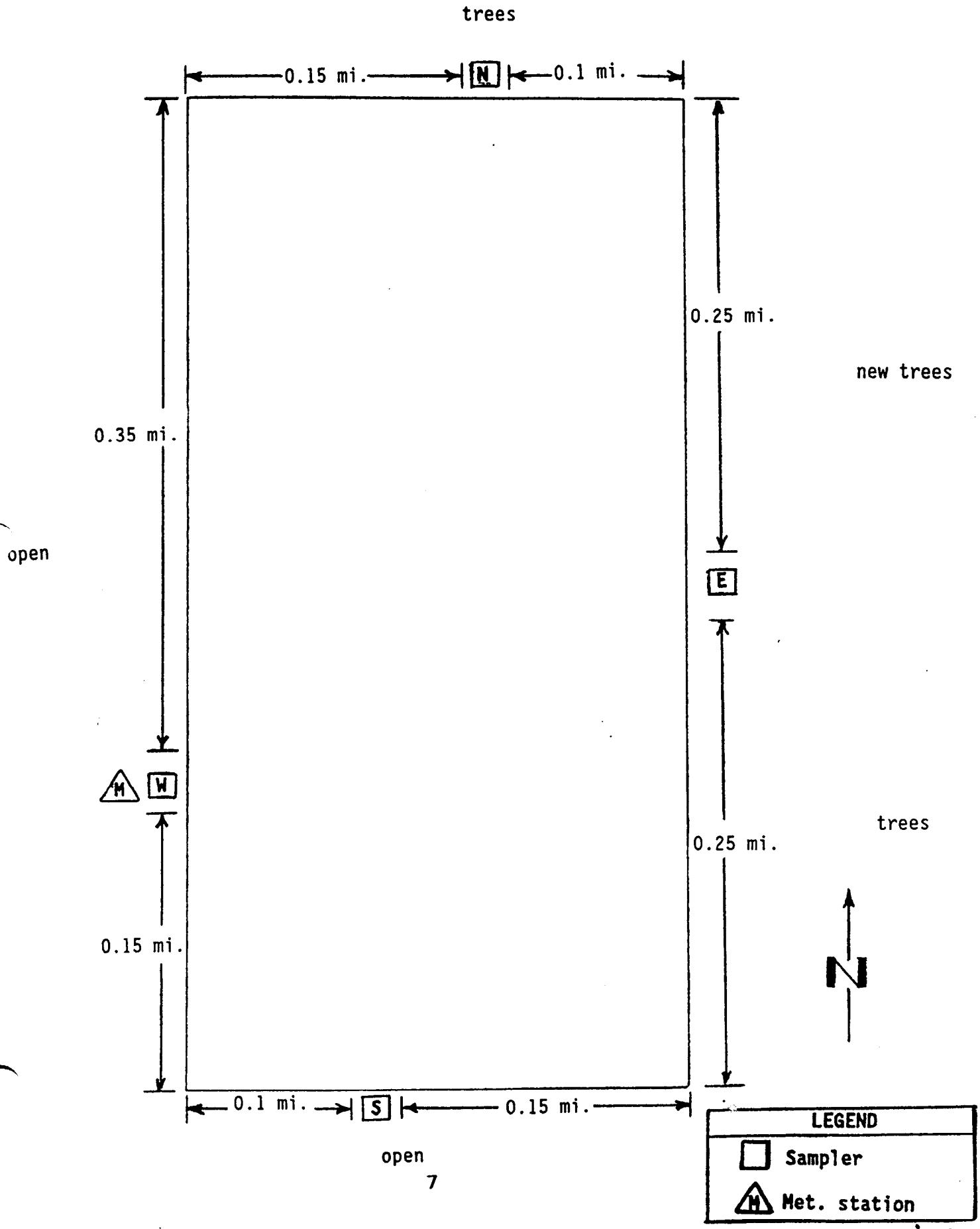


TABLE I. MITC Application Monitoring Data

Sample ID	Time (min.)	Volume (m^3)	Total (ug)	Concentration (ug/m^3)	Concentration (ppbv)	Collection Time (Approx.)
0WC	715	1.4	0.743	0.53	0.18	
0SC-1	715	1.4	0.342	0.24	0.080	(Background)
0SC-2	715	1.4	0.623	0.44	0.15	
0EC	725	1.4	0.644	0.46	0.15	8/23-24/95
0NC	730	1.4	0.736	0.53	0.18	(1900-0730)
1WC	345	0.66	0.553	0.84	0.28	
1SC-1	355	0.67	12.45	19.	6.4	(Application)
1SC-2	355	0.67	17.15	26.	8.7	
1EC	360	0.68	4.840	7.1	2.4	8/24/95
1NC	370	0.70	0.445	0.64	0.21	(1230-1830)
2WC	760	1.4	99.70	71.	24.	
2SC-1	Lost sample					(Application)
2SC-2	Lost sample					
2EC	765	1.5	57.95	39.	13.	
2NC	760	1.4	350.4	250	84.	8/24-25/95
2BC	BLANK	--	< 0.12	--	--	(1830-0700)
3WC	680	1.3	1.354	1.0	0.33	
3SC-1	675	1.3	13.84	11.	0.37	(Application)
3SC-2	675	1.3	22.33	17.	5.7	
3EC	675	1.3	26.48	20.	6.7	8/25/95
3NC	675	1.3	10.49	8.1	2.7	(0700-1830)
4WC	765	1.5	1.506	1.0	0.33	
4SC-1	765	1.5	2.827	1.9	0.64	
4SC-2	765	1.5	3.686	2.5	0.84	
4EC	765	1.5	303.5	200	67.	8/25-26/95
4NC	770	1.5	252.0	170	57.	(1830-0730)
5WC	610	1.2	1.123	0.94	0.31	
5SC-1	610	1.2	2.805	2.3	0.77	
5SC-2	610	1.2	2.504	2.1	0.70	
5EC	605	1.1	5.377	4.9	1.6	8/26/95
5NC	600	1.1	3.568	3.2	1.1	(0730-1730)
6WC	815	1.5	3.335	2.2	0.74	
6SC-1	790	1.5	1.793	1.2	0.40	
6SC-2	790	1.5	1.681	1.1	0.37	
6EC	795	1.5	11.27	7.5	2.5	8/26-27/95
6NC	795	1.5	29.43	20.	6.7	(1730-0700)

All flows at 1.9 liters per minute (see SAMPLING METHODOLOGY).
No values corrected for percentage of recovery.

TABLE II. MIC Application Monitoring Data

Sample ID	Time (min.)	Volume (m^3)	Total (ug)	Concentration (ug/m^3)	Concentration (ppbv)	Collection Time (Approx.)
0WX	715	0.064	0.01	0.2	0.09	
0SX-1	715	0.064	0.03	0.5	0.2	(Background)
0SX-2	715	0.064	0.02	0.3	0.1	
0EX	725	0.065	0.03	0.5	0.2	8/23-24/95
0NX	730	0.066	0.03	0.4	0.2	(1900-0730)
1WX	345	0.031	0.07	2.	0.9	
1SX-1	355	0.032	0.02	0.6	0.3	(Application)
1SX-2	355	0.032	0.07	2.	0.9	
1EX	360	0.032	0.07	2.	0.9	8/24/95
1NX	370	0.033	0.06	2.	0.9	(1230-1830)
2WX	760	0.068	0.09	1.	0.6	
2SX-1	750	0.068	0.12	1.8	0.77	(Application)
2SX-2	750	0.068	0.15	2.2	0.94	
2EX	765	0.069	0.14	2.	0.86	
2NX	760	0.068	0.17	2.5	1.1	8/24-25/95
2BX	BLANK	--	<0.02	--	--	(1830-0700)
3WX	680	0.061	0.05	0.8	0.3	
3SX-1	675	0.061	0.09	1.	0.4	
3SX-2	675	0.061	0.14	2.3	0.99	(Application)
3EX	675	0.061	0.09	1.	0.4	8/25/95
3NX	675	0.061	0.09	1.	0.4	(0700-1830)
4WX	765	0.069	0.08	1.	0.4	
4SX-1	765	0.069	0.09	1.	0.4	
4SX-2	765	0.069	0.10	1.4	0.60	
4EX	765	0.069	0.40	5.8	2.5	8/25-26/95
4NX	770	0.069	0.28	4.1	1.8	(1830-0730)
5WX	610	0.055	0.12	2.2	0.94	
5SX-1	610	0.055	0.10	1.8	0.77	
5SX-2	610	0.055	0.12	2.2	0.94	
5EX	605	0.054	0.16	3.0	1.3	8/26/95
5NX	600	0.054	0.11	2.0	0.86	(0730-1730)
6WX	815	0.073	0.10	1.4	0.60	
6SX-1	790	0.071	0.04	0.6	0.3	
6SX-2	790	0.071	0.13	1.8	0.77	
6EX	795	0.072	0.12	1.7	0.73	8/26-27/95
6NX	795	0.072	0.08	1.	0.4	(1730-0700)

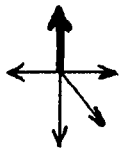
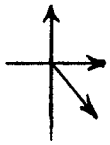
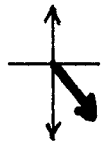
All flows at 0.090 liters per minute (see SAMPLING METHODOLOGY).
No values corrected for percentage of recovery.
All values corrected for blank media background.

TABLE III. MIC/MITC Meteorological Data

Sampling Period	Wind Direction	Wind Speed (mph)	Cloud Cover
0	**S**/NW/W/E/N	3	K
1	**NW**/N	5	K
2	S/NW/W	3	K
3	**NW**/N	4	K
4	**SW**/W/S/NW	4	K
5	**NW**/N/S	4	K
6	**SW**/S/NW	3	K

BOLD indicates predominant wind direction, if any.
Indicates direction wind blows from.
K = clear, PC = partly cloudy, O = overcast

TABLE IV. Summary of MITC Data (ug/m^3)

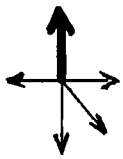
(0) [N] 0.53 [W] 0.53  0.46 [E] [S] 0.34	(1) [N] 0.64 [W] 0.84 7.1 [E] [S] 22.
(2) [N] 250 [W] 71.  39. [E] [S] --	(3) [N] 8.1 [W] 1.0 20. [E] [S] 14.
(4) [N] 170 [W] 1.0 200 [E] [S] 2.2	(5) [N] 3.2 [W] 0.94  4.9 [E] [S] 2.2
(6) [N] 20. [W] 2.2 7.5 [E] [S] 1.2	

( ) indicates the sampling period. [ ] indicates sampling site represented.
Arrow indicates direction wind is blowing toward.
Bold arrow indicates predominant wind direction, if any.
Site S is the average of duplicate samples.

TABLE V. Summary of MIC Data (ug/m^3)

(0)

[N] 0.4

[W] 0.2  0.5 [E]

[S] 0.4

(1)

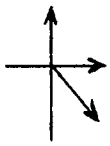
[N] 2.

[W] 2. 2. [E]

[S] 1.3

(2)

[N] 2.5

[W] 1.  2. [E]

[S] 2.

(3)

[N] 1.

[W] 0.8 1. [E]

[S] 1.6

(4)

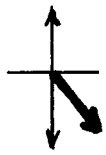
[N] 4.1

[W] 1. 5.8 [E]

[S] 1.2

(5)

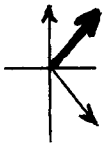
[N] 2.0

[W] 2.2  3.0 [E]

[S] 2.0

(6)

[N] 1.

[W] 1.4  1.7 [E]

[S] 1.2

( ) indicates the sampling period. [ ] indicates sampling site represented.
Arrow indicates direction wind is blowing toward. **Bold** arrow indicates predominant wind direction, if any.
Site S is the average of duplicate samples.