

ENVIRONMENTAL MONITORING RESULTS OF BASAMID[®] G (DAZOMET)
APPLICATION IN WATSONVILLE, CALIFORNIA 2006
Study 212

By

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ABSTRACT

Dazomet, the active ingredient of Basamid[®] (99% dazomet), is one of the three soil fumigant pesticides that generate methyl isothiocyanate (MITC). Though dazomet use is smaller relative to other soil fumigants, its use in the State of California is increasing. Department of Pesticide Regulation (DPR) initiated a pilot air monitoring study to gather information on MITC emissions following a dazomet field application. Such information was needed to adapt mitigation measures for human exposure and environmental contamination. Hence DPR scientists proposed at least two more studies in commercial field settings. This study was done in Watsonville, California in October 2006. A total mass of 205.0 kg (451.94 lbs) Basamid[®] G was broadcast applied to 0.4118 ha (1.0175 ac), a field of raised beds prepared for strawberry planting. Both beds and furrows were treated. This was equivalent to a rate of 493 kg/ha (440 lbs/ac) of dazomet or 22.2 g/m² of MITC. Sprinkler irrigation was used following application to activate the dazomet and to hold the soil near field capacity to minimize MITC losses as recommended by the label.

The highest individual concentration of MITC was 1058 µg/m³ recorded 10 hours after start of application. The high concentration may have resulted in part from two inadvertent spills of Basamid[®] G upwind and off the treated field. MITC maximum concentrations nearby up to 70 hours after the start of Basamid[®] G application ranged between 1058 µg/m³ and 192.7 µg/m³. By 274 hours after the start of Basamid[®] G application (end of monitoring), the MITC concentration dropped to 3 µg/m³.

The back-calculation method was used to estimate flux. Only 3 out of the 18 simulated periods showed significant r^2 values ($p < 0.05$). Measured and the modeled concentrations for the 15 non-significant intervals were sorted and reanalyzed. After 274 hours from the beginning of Basamid[®] G application, 32% of applied MITC was emitted from the plot. Ten per cent of applied MITC was released to air by the end of first 24-hr period. Twelve percent was released during the second 24-hr period. Peak emission was 8 % from 28 to 34 hours after the beginning of Basamid[®] G application. In general, emissions during night intervals were higher than the day intervals.

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Cindy Garretson and her staff at soil analytical laboratory in Fresno analyzed the soil samples, and we are thankful for their help. Carissa Ganapathy procured the sampling tubes and coordinated activities with two laboratories involved and with the field staff of this study. Dave Kim helped preparation of sampling equipment. Dr. Bruce Johnson reviewed this manuscript and made valuable comments. We thank all of them.

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INTRODUCTION

In June 2003, the California Department of Pesticide Regulation (DPR) declared methylisothiocyanate (MITC) and all pesticidal sources of MITC, to be toxic air contaminants that may cause or contribute to increases in illness or death (CDPR, 2003). Metam sodium, metam potassium, and dazomet are the three pesticidal sources of MITC. There is a reasonable body of knowledge on the environmental fate of metam sodium. Metam sodium has a linear molecule. Metam potassium has a similar chemical structure to that of metam sodium, and therefore, is expected to have a similar degradation rate. However, little is known about the degradation rates, off-site air concentrations following an application and flux estimates, and other characteristics of dazomet that may affect the public and the environment under field conditions. The chemical name of dazomet is Tetrahydro-3,5, -dimethyl-2H-1,3,5-thiadiazine-2-thione. Dazomet's chemical structure shows a heterocyclic ring containing carbon, nitrogen, sulfur, and hydrogen (Figure 1). Therefore, degradation products of dazomet could be different from those of the relatively better-understood metam sodium.

Dazomet use in agriculture has been minor relative to other soil fumigants, but is increasing (Table 1). This necessitates reliable estimates of MITC losses, off-site movement and flux estimates of dazomet under various field conditions (Wales, 2002; Fan et al., 2008). Fan et al. (2008) studied off-site MITC concentrations and estimated flux following a dazomet application to a field in June 2005, in Manteca, California. The Manteca study was a preliminary one with small field plots. Hence DPR scientists proposed at least two more studies in commercial field settings (Wofford and Johnson, 2006). The objective of this study, therefore, was to collect more information on off-site movement and flux estimates of MITC following an application of dazomet in a commercial field in Watsonville, California, in October 2006.

Table 1. Statewide dazomet use in lbs (Kg). Source: DPR's pesticide use database.

Site	2000	2001	2002	2003	2004	2005
Agriculture related	5,560 (2,522)	4,619 (2,095)	2,769 (1,556)	16,303 (7,394)	18,541 (8,409)	2,736 (1,241)
Landscape maintenance	1,103 (500)	2,384 (1,081)	1,851 (839)	2,963 (1,344)	4,773 (2,165)	25,120 (11,392)
Right of way	2,139 (970)	36,897 (16,733)	38,966 (17,672)	24,880 (11,283)	35,111 (15,923)	18,220 (8,263)
Soil- seedbeds	692 (314)	2 (0.9)	0 (0)	11 (5)	22 (10)	23 (10)
All other	992 (450)	397 (180)	1,434 (650)	1,158 (525)	45 (20)	1,827 (829)
Total	10,486 (4,756)	44,299 (20,090)	45,020 (20,417)	45,315 (20,551)	58,492 (26,527)	47,926 (21,735)

Dazomet is a broad-spectrum pesticide and has at least 18 registered products in California as of November 2006. It is a soil fumigant and used to control fungi, bacteria, nematodes,

weeds, and soil insects. In moist soils, dazomet decomposes rapidly to methyl (methylaminomethyl) dithiocarbamic acid, which further degrades to MITC, formaldehyde, hydrogen sulfide, and methylamine. The commercial pesticide, Basamid[®] G used in this study (EPA Registration number 700051-101, manufactured by CERTIS, Columbia, MD) contained 99.0% dazomet as active ingredient. It is the soil fumigant, labeled “For Use in California only”, and recommended for pre-planting control of most weeds in strawberries and tomatoes. According to the original manufacturer of Basamid[®] G, it is this combination of volatile gases that give the fumigant properties (BASF, 1989). The degradation of dazomet can occur rapidly, in 10-15 minutes from application (Thompson, 1989). The aerobic soil half-life (50% dissipation time) for dazomet was reported to be 18 hours at pH 5.8 in a loamy sand soil (DPR, 1999). Water is the primary factor in dazomet degradation. However, soil temperature, pH, and soil type can affect the rate of degradation (Wales, 2002; Munnecke and Martin, 1964; Sczerzenie, et al., 1987).

In most agricultural applications, Basamid[®] is used as a granular soil fumigant. The treatment could be a surface broadcast or sub-surface application and with or without tarp. Water applied on a schedule to keep the soil near field capacity generates the fumigant efficiently. The same wet soil at this condition is believed to keep the fumigant sealed in the soil, slowing down its release to air. Basamid[®] G is insoluble in water and non-volatile (Table 2). However, its major breakdown product, MITC, has a relatively high vapor pressure of 16.0 mm Hg at 20 °C (Wales, 2002; Greek Ministry of Rural Development and Food-Productive Branches, 2006). This results in a highly volatile fumigant (Wofford et al., 1994; Levine et al., 2005; Fan et al., 2008).

Table 2: Physicochemical properties of dazomet^a and MITC.

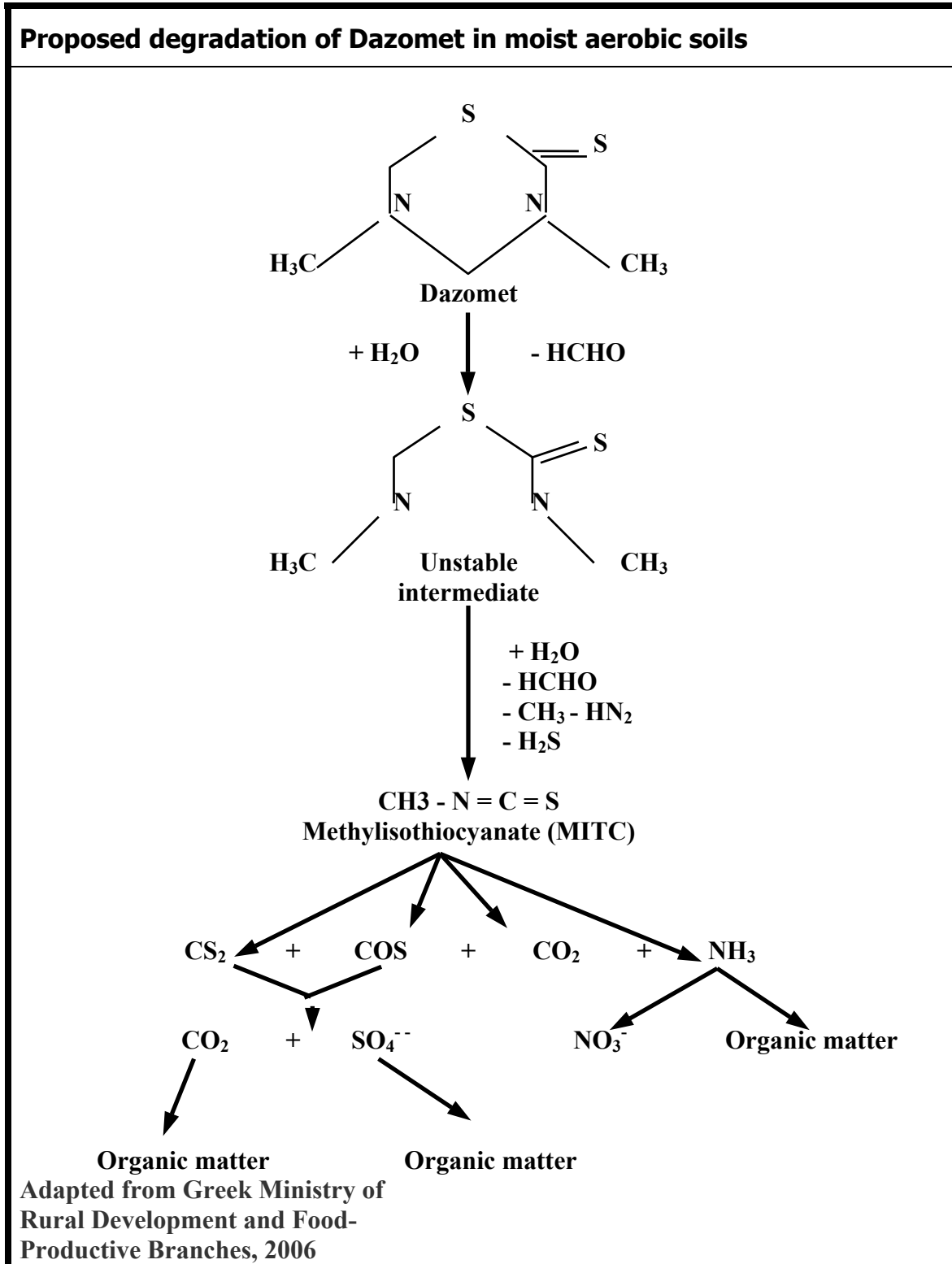
	Dazomet	MITC
Molecular formula	C ₅ H ₁₀ N ₂ S ₂	C ₂ H ₃ NS
Molecular weight (g)	162.3	73.12
Solubility in water (ppm)	3.63 x 10 ⁻³ (20 °C)	8.61 x 10 ³ (25 °C)
Vapor pressure (mmHg)	9.88 x 10 ⁻⁶ (25 °C)	16.0 (25 °C)
Boiling point ^b (760 mm Hg)	104 °C	Na
Henry's law constant (atm·m ³ /mole)	2.57 x 10 ⁻¹⁰ (20 °C)	1.79 x 10 ⁻⁴ (25 °C)
Hydrolysis half-life (days)	0.146 (pH 7, 25 °C)	20.4 (pH 7, 25 °C)
Aerobic soil half-life (days)	0.75 (pH 5.8, loamy and sandy soil)	0.5-50 (25 °C)
Anaerobic soil half-life (days)	14.10	Na

^aAll data are from the DPR's Pesticide Chemical Database (DPR, 1999), unless otherwise indicated.

^bMSDS, Basamid[®], Certis U.S.A. LLC, 2005.

Na = Not available.

Figure 1: A proposed degradation of dazomet in moist soil under aerobic conditions.



Gamliel et al., (2001) estimated that 98% of the Basamid[®] applied to moist soil is broken down to MITC rapidly. Other breakdown products in low amounts included CS₂, HCHO,

H₂S and NH₃. According to Munnecke and Martin (1964), warmer soil temperatures increased the decomposition rate of dazomet, although they noted that the same amounts of MITC were eventually produced at all temperatures tested. Rate of decomposition increased with soil moisture, up to approximately 80% of soil saturation. Decomposition was fastest at pH 6.5, and declined at lower or higher pH levels. Soil type has an effect on dazomet degradation. Clays may act as catalysts in the initial breakdown of dazomet to MITC (Sczerzenie et al., 1987). The addition of peat moss to soil decreased breakdown of dazomet to MITC, presumably due to the sorption of dazomet to peat moss (Munnecke and Martin, 1964). Little is known about the fate of dazomet in water. Sczerzenie et al. (1964) summarized several studies on the fate of dazomet in water. This report suggests pH as the key factor affecting the decomposition of dazomet. In aqueous solution at pH levels of 5, 7, and 9, dazomet decomposed with half-lives of 8.6, 2.6, and 1.5 hours respectively. However, no temperatures were given. The half-life of dazomet in aqueous solution at pH 5 under irradiation was 4 hours compared to a dark control, where the half-life was 11 hours. Here too, the temperature was not reported. MITC and carbon disulfide were identified as degradation products (Wales, 2002).

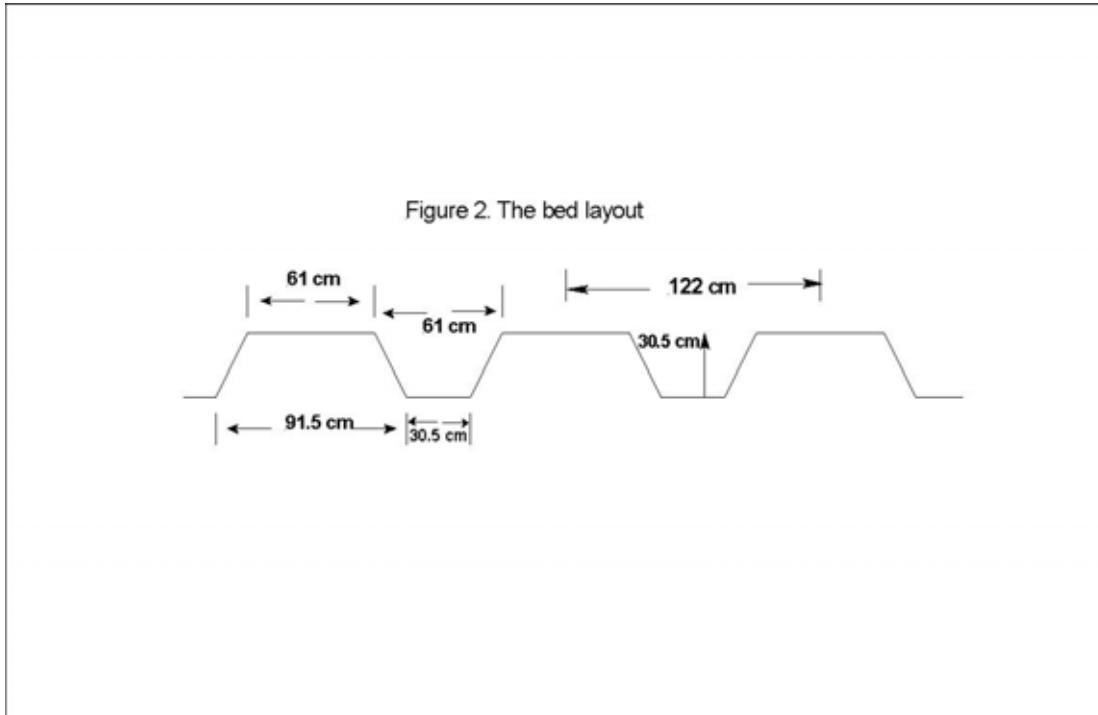
Studying the MITC concentrations from another source, metam sodium, Wofford et al. (1994) found MITC levels that exceeded the Office of Environmental Health Hazard Assessment's 1-hour Reference Exposure Level (REL) for eye irritation of 1.2 µg/m³. Conditions were considered extreme in that study due to high air temperature, low humidity, warm soil temperature, and the fumigant applied at the highest allowable rate of 935.0 l/ha (100 gals/acre). DPR has recognized the potential of MITC to drift into adjacent communities. For this reason, DPR is working to generate data to define buffer zones for mitigation. DPR has established an 8-hour reference concentration of 660 µg/m³ (220 ppb) of MITC for metam sodium, which was identified in the DPR risk assessment as the no-observable effect level (NOEL), (CDPR, 2002).

MATERIALS AND METHODS

All the field measurements have been converted to metric units.

Application Site

A 0.4118 ha (1.0175 acre) plot at a nursery facility in Watsonville, California was the experimental site (Photograph 1). The periphery of the field was fenced on all sides except the west end where the main access was. On both north and south sides of the plot, and beyond the fence, there were raspberry screen houses, approximately 20 meters (65 feet) from the fence and about 7 meters (22 feet) tall. The farm roads ran along the three fences. A temporary fourth road at the west divided the field into experimental and non-experimental areas. The beds were standard raised bare beds usually prepared to plant strawberry. Beds in the experimental area as well as in the non-experimental area were prepared as contiguous beds. (Photograph 2). The soil was dry and there were few weeds on the beds. The beds ran in an east-west direction and were 1.22 meters (48 inches) wide from center of furrow to center of furrow (Figure 2). The top of the bed was 61 cm (24 inches) wide, and the bottom of the bed was 91.5 c.m. (36 inches) wide. The top of the furrow was 61 cm (24 inches). The bottom of the furrow was 30.5 cm (12 inches), and the beds were 30.5 cm (12 inches) tall (Figure 2 and Photograph 2).



Air Sampling

SKC Universal sampler pumps, model 224-PCXR4 and model 224-PCXR8 were used in this study. Sampler pumps were set to a constant flow of 1000 ml/min. In both types of pumps, timer and the flow fault functions were active. At each location (station) two sampler pumps were mounted on a metal stake at about 0.7 meters aboveground (Figure 3 and Photograph 3). In Photograph 3, two pumps were under the black polyethylene rain guard and not visible, but the two tygon tubes connecting the pumps to samples are visible. The second pump was used to mount field spikes and collocated samples, and also was a back up. The air samples were collected using two-stage (200-400 mg) coconut charcoal tubes (SKC 226-09) that were mounted at about 1.5 meters aboveground, pointed towards the plot. The charcoal tubes were protected from sun and moisture by using aluminum foil wraps. Fully charged 12-volt car batteries powered the sampler pumps and were replaced every 48 hours. A flashing strobe light helped to locate the samplers in the night.

From one end to the other, the field was approximately 105 meters in length and 63 meters in width. Inside this field an area, 81.3 meters in length and 50.6 meters in width was treated with Basamid[®] G (Figure 4). This gave a treatment area of 0.4118 hectares (1.0175 acres) having 67 rows of beds. Sampler pumps ringed the field at 12 meter and 18 meter distances (Figure 4). The precise distances are given in Table 3.

Table 3. Distance of samplers from the edge of the field (meters).

Sampler location	1	2	3	4	5	6	7	8
Distance	18.3	12.2	18.1	11.6	18.1	12.2	18.5	12.3

Figure 3. Layout of a sampling unit (station)

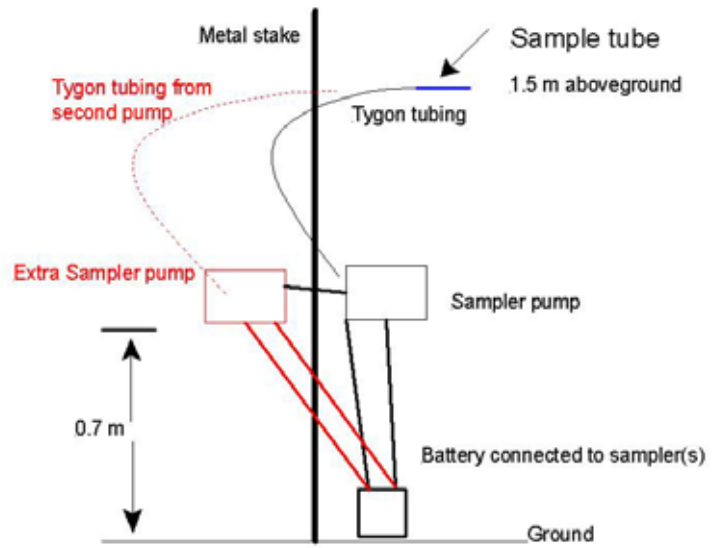
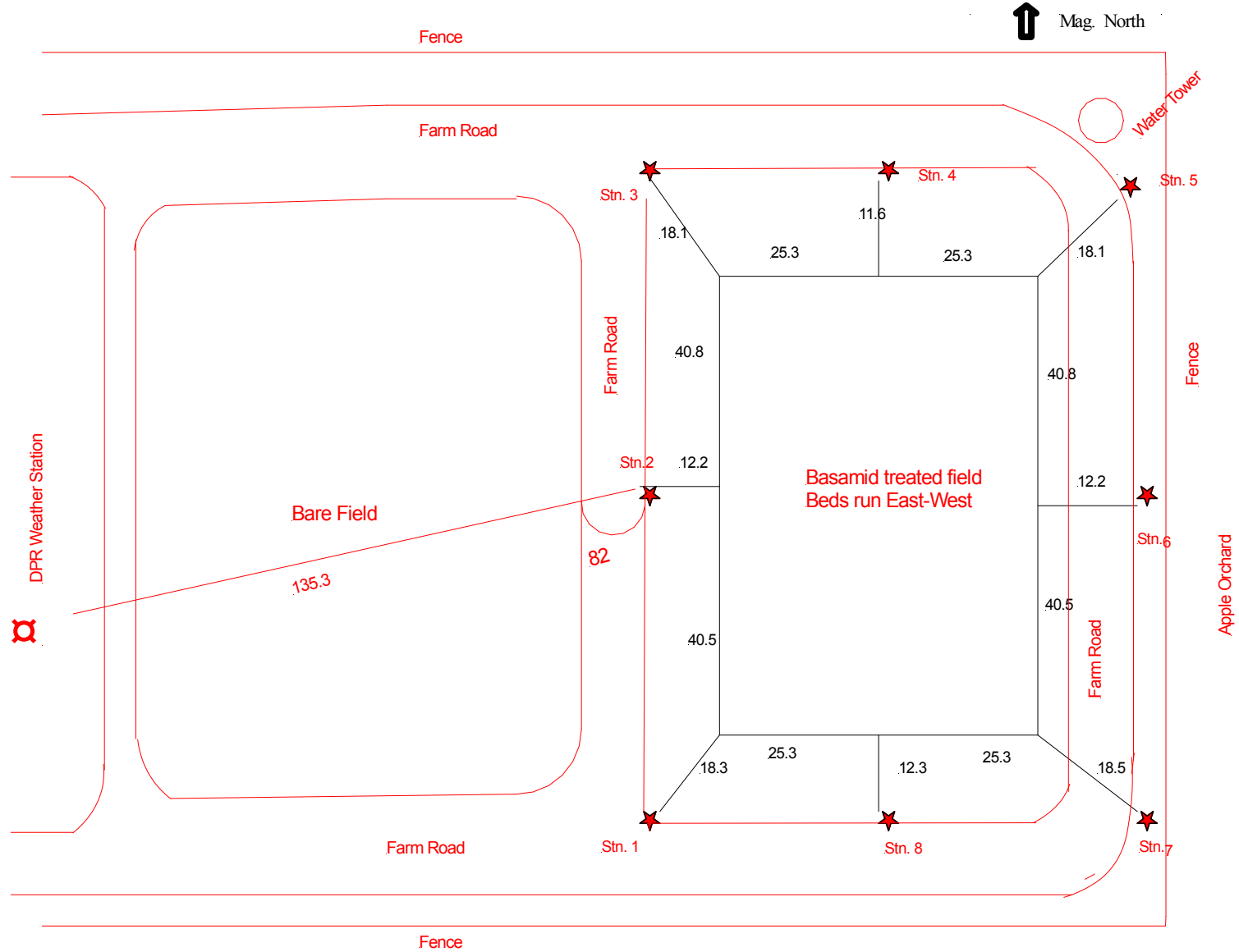


Figure 4: Sketch of the field.



All distances are in meters.

Weather data

A weather station was set up approximately 135 meters west of the application area (Figure 4). The trailer-mounted mast was approximately 10 meters (32.8 feet) high. Wind speed, wind direction, ambient air temperature, solar radiation, and relative humidity were collected for the duration of the study. The Met 1[®] meteorological sensors recorded data onto a Campbell Scientific, Inc. CR 21 X Datalogger. The data were recorded as one-minute averages of one-second readings, except for wind direction, which was collected as an instantaneous reading every minute. In addition to these readings, percent cloud cover was noted at each sample change.

Application of Test Substance

A total of 205.0 kg (451.94 lbs) of Basamid[®] G (EPA Reg.# 70051-101) was applied to the 0.4118 ha (1.0175 ac) plot giving an application rate of 497.81 kg/ha (444.17 lbs/ac) or 49.78 g/m². Since Basamid[®] G contains 99% dazomet, 497.81 x 0.99 = 493 kg/ha (440 lbs/ac) of dazomet was applied. The herbicide was packaged in 22.68 kg (50 lbs) double-layer plastic bags. The product was a fine white granule that flowed easily (Photograph 2).

Each pass treated a single row covering the bed and one half each furrow on either side. Thus the entire field surface was treated. Each pass took approximately 2 minutes to complete. The application was made with a Gandy Hopper, mounted on a John Deer Model 990 tractor, and the granular pesticide fell through a series of orifices perpendicular to the row. The pesticide dropped onto a slanted plate, 10.2- 12.7 c.m. (4-5 inches) above the ground, and was spread evenly upon the soil surface (Photograph 2).

Four background air-monitoring samples were started on October 17 at approximately 1800 hours and ended on October 18, approximately 0700 hours. The Basamid[®] G application commenced at 0845 hours and was completed at 1100 hours on October 18. Sampler pumps were started just prior to the start of the application. A little Basamid[®] G was dropped inadvertently, upwind, and to the north of the station 2 sampler, at about 1000 hours. On a subsequent pass of the tractor, a smaller amount of Basamid[®] G was spilled again, this time, upwind and south of the same sampler.

Irrigation

The sprinklers were started and were at full pressure at 1135 hours. However, irrigation to the beds between samplers 2 and 3 and to the west end was insufficient (Photograph 3). Irrigation was interrupted at 1140 hours, and a new pipe with a sprinkler outlet to cover this area was installed. After several adjustments, steady irrigation started at 1205 hours and continued until 1515 hours. Sprinkler lines were laid in the furrows of the treated beds. During the course of the trial, several irrigations were done (Table 4). The amount of water applied during each irrigation was measured by keeping a one inch (2.54 cm) diameter glass cylinder graduated to measure one tenth of an inch (0.25 cm) in the field. After the end of second irrigation and up to the 6th irrigation, standing water was observed accumulating in the field at the east end.

Table 4. Irrigation schedule.

Irrigation Event	Date	Start Time	Approximate Duration	Approximate Amount of water, inches/(mm)
1	10/18/06	1200	3 hrs 15 minutes (min)	0.75 / 19.1
2	10/18/06	1918	1 hr	0.25 / 6.4
3	10/19/06	1020	15 min	0.10 / 2.5
4	10/19/06	1215	15 min	0.10 / 2.5
5	10/19/06	1415	35 min	0.20 / 5.0
6	10/19/06	1715	35 min	0.20 / 5.0
7	10/20/06	1140	20 min	0.10 / 2.5
8	10/20/06	1315	18 min	0.10 / 2.5
9	10/20/06	1500	40 min	0.20 / 5.0
10	10/20/06	1645	20 min	0.10 / 2.5
11	10/21/06	1108	35 min	0.20 / 5.0
12	10/21/06	1324	36 min	0.20 / 5.0
13	10/22/06	1005	30 min	0.20 / 5.0

Soil Samples

Soil samples were collected prior to treatment, at two locations for bulk density and soil moisture estimates. These samples were collected from a depth of 15.2 – 30.5 cm (6-12 inches) below the surface, and from two beds towards the middle of the field to represent the study area. A composite surface soil sample was collected from several places randomly, over the treatment area for soil texture analysis. These soil samples were analyzed at the Fresno field laboratory of DPR, following the SOP FSSO001.00 and SOP FSSO002.00 (Garretson 1999a and 1999b).

Air Sampling

Table 5: Approximate sampling times for 18 sampling intervals and sample specifics.

Interval	Date	Start	End	Time of day	Hours and minutes sampled	Sample details
BG ¹	17/18	1800	0700	night	13 h 0 min	At 1,2,3 and 6
1	18	0845	1520	day	6 h 35 min	All 8 locations
2	18	1520	1850	day	3 h 30 min	All 8 ² + CL ³ at 2,4,6, and 8
3	18/19	1850	0100	night	6 h 10 min	All 8
4	19	0100	0645	night	5 h 45 min	All 8 + CL ³ at 2,4,6, and 8
5	19	0645	1245	day	6 h 0 min	All 8
6	19	1245	1845	day	6 h 0 min	All 8
7	19/20	1845	0045	night	6 h 0 min	All 8
8	20	0045	0645	night	6 h 0 min	All 8
9	20	0645	1245	day	6 h 0 min	All 8
10	20	1245	1845	day	6 h 0 min	All 8
11	20/21	1845	0645	night	12 h 0 min	All 8
12	21	0645	1845	day	12 h 0 min	All 8
13	21/22	1845	0645	night	12 h 0 min	All 8 + FS ⁴ at 2, 4, and 6
14	22	0645	1845	day	12 h 0 min	All 8
15	22/23	1845	0645	night	12 h 0 min	All 8 + FS ⁴ at 4, 6, and 8
16	23	0645	1845	day	12 h 0 min	All 8
17 ⁵	28/29	1845	0645	night	12 h 0 min	All 8
18	29	0645	1845	day	12 h 0 min	All 8

¹ BG = Background samples before the commencement of treatment.

² Sampler at location 1 malfunctioned, no sample.

³ CL = Collocated samples at locations; 2, 4, 6, and 8.

⁴ FS = Field Spikes at locations 2, 4, and 6, during interval 13 and at 4, 6, and 8, during interval 15.

⁵ 17 = Interval 17 started approximately 5 days after interval 16.

For every sample the start time, start flow rate, end time, and the end flow rate were recorded. At every start, the flow rate was kept within 1000 ml/minute \pm 50, by adjusting the pump when necessary. During the second sampling interval, the sampler pump at location 1 malfunctioned, and no sample was collected.

Sample Handling

Prior to the commencement of the study, over 300, two-stage (200-400 mg) coconut charcoal tubes were purchased from the manufacturer. The study number (212) and sample identification number were attached to each individual tube. A Chain of Custody (COC) and Lab Result Report form was prepared to record sample information, and four samples were logged on each COC (Appendix 2). For convenience, two COCs and 8 sample tubes were placed in a 10-inch zip-lock bag, and 22 such sets were prepared giving 4 extra sets for the required 18 sampling intervals.

The collected samples were packaged and handled according to the procedures in DPR's SOP QAQC004.01 (Jones, 1999). Because of the distance involved, all samples were held on dry ice. The first set was delivered to the labs on October 24th and the second set on October 30th.

Calculation of Air Concentration

The sample MITC concentrations were calculated by moving a known volume of MITC laden air through charcoal trapping medium and recovering it through a chemical extraction process. The laboratory analytical results were reported in µg/sample. The air concentrations were converted from µg/sample to µg/m³ using the following relationship.

$$\frac{\text{Sample mass } (\mu\text{g/sample}) * 1000 \text{ L/m}^3}{\text{Flow rate (L/min)} * \text{time (min)}} = \text{Air Concentration } (\mu\text{g/m}^3)$$

Concentrations can be converted from µg/m³ to parts per billion (ppb):

At 25 °C (298 °K) and 1 atmosphere, 1 µg/m³ = 24.45/ molecular weight (MW) in ppb

Therefore, 1 µg/m³ MITC = 24.45 / 73.12 ppb MITC = 0.3344 ppb MITC

Chemical Analysis of Air Samples.

All air samples were analyzed for MITC. Air samples from 18 intervals (regular samples), four background samples, one set of field spikes (3 samples), one set of trip spikes (3 samples), and one trip blank were sent to Morse laboratory, a private laboratory. The MITC analytical procedures followed by the Morse laboratory are given in Appendix 3. The other sets of field spikes (3 samples), trip spikes (3 samples), one trip blank and eight collocated samples were analyzed by the California Department of Food and Agriculture (CDFA) Center for Analytical Chemistry, commencing on October 25, 2006. The details for analytical procedure of CDFA laboratory are given in Appendix 4.

Prior to the commencement of Watsonville study, CDFA conducted a method verification study for MITC using their procedure. The MITC was spiked at three levels, and desorbed from the charcoal in 5 ml of a 0.1% carbon disulfide in ethyl acetate solvent by occasionally agitating for 30 minutes. The extracts were analyzed on a gas chromatograph equipped with a Thermal Spray Detector. Method verification study results are reported in Table 6.

Table 6: Method verification data for MITC in air, CDFA laboratory.

Spike Level µg/sample	Recovery (% of spike)			Mean	SD	UCL	UWL	LWL	LCL
	Rep #1	Rep #2	Rep #3						
0.4	82.5	87.5	87.5	85.83	2.89				
3.0	97.3	107.0	103.0	102.43	4.87				
8.0	97.8	98.5	92.3	96.20	3.40				
				94.82	7.98	119.0	111.0	78.9	70.9

SD = Standard deviation UCL = Upper control limit UWL = Upper warning limit LWL = Lower warning limit LCL = Lower control limit

The Reporting Limit (RL) for CDFA laboratory when the Gas Chromatograph/Nitrogen Phosphorus Detector (GC/NPD) was used was 0.2 µg/sample. When Gas Chromatograph/Thermal Spray Detector (GC/TSD) was used, it improved to 0.05 µg/sample. The RL for the results reported in Table 6 was 0.2 µg/sample. From the recovery % values for three levels of spikes, near 95% recovery and small standard deviations (2.9 to 8.0) show the reliability of MITC recovery by the CDFA laboratory. The data were within the control limits defined. The efficacy of Morse laboratory analytical procedure is shown in Tables 7 and 8 of this report. The chemical analysis by Morse laboratory commenced on October 27, 2006.

QUALITY CONTROL

Field Spikes, Trip Spikes, and Trip Blanks

A spiked sample is a sample that is spiked with a known amount of MITC. For both field spikes and trip spikes, four sets of three different amounts of MITC were spiked on charcoal sampling tubes. Four charcoal tubes were spiked at a level of 0.2 µg/sample, four at 20.0 µg/sample and four at 200.0 µg/sample. All these samples were made on the morning of October 17 and were held on dry ice. In theory a spiked sample when exposed, should contain more MITC than the regular (unspiked) sample exposed at the same location and time. Since the spiked amount is known, the difference in MITC between spiked sample and regular sample provides an estimate of the reliability of field sampling procedures. One sample tube from each spike level was set on the additional pump along with the regular sample during interval 13 (Oct 21) and interval 15 (Oct 22) as field spikes. At the end of intervals 13 and 15, a matching set of trip spikes (the other half of the three level spiked set, but not exposed to field conditions) was added to the samples for that interval (i.e. 13 and 15). Trip spikes provide an estimate of the integrity of the sample storage and transport conditions. One trip blank (unused charcoal tube) per interval was also included for analysis. Trip blanks were handled the same way as other samples, and provided information of any contamination during handling. Therefore, at intervals 13 and 15, there were seven additional samples (3 field spikes, 3 trip spikes, and one trip blank). The extra samples from interval 13 were analyzed at Morse laboratory and the CDFA laboratory analyzed the interval 15 samples. Since all the regular samples were analyzed by Morse laboratory, the percent recovery results of seven extra samples analyzed by CDFA laboratory have an additional source of variability due to two different laboratory analytical procedures. These results are reported in Table 7.

Table 7: Results of field spikes, trip spikes and trip blanks.

Sample	Sample Type	Spiked amount µg/tube	Recovered amount from spiked sample µg/tube	Recovered amount from regular (unspiked) sample µg/tube	Difference (spiked – regular) µg/tube	% Recovery
Reporting Laboratory						
CDFA	Field Spike	0.20	16.60	17.64	-1.04	-520.00*
	Field Spike	20.00	28.10	14.70	13.40	67.00
	Field Spike	200.00	134.00	5.76	128.24	64.12
	Trip Spike	0.20	0.18	NA	NA	90.00
	Trip Spike	20.00	16.50	NA	NA	82.50
	Trip Spike	200.00	152.00	NA	NA	76.00
	Trip Blank	0.00	ND	NA	NA	NA
Morse Lab	Field Spike	0.20	11.50	10.58	0.92	460.00*
	Field Spike	20.00	89.40	72.32	17.08	85.40
	Field Spike	200.00	175.40	18.74	156.70	78.30
	Trip Spike	0.20	0.20	NA	NA	100.00
	Trip Spike	20.00	18.20	NA	NA	91.00
	Trip Spike	200.00	175.00	NA	NA	87.50
	Trip Blank	0.00	ND	NA	NA	NA

* These unusual recovery values may be associated with the experimental error relative to small spiked amounts. ND = not detected, NA=not applicable.

Except for the conflicting results for field spikes at 0.2 µg/tube concentration from two laboratories, the rest of the results show that the percent recovery values are reasonable. This gives confidence in the handling of samples in the field, in transit, and analysis methodologies of the two laboratories.

Continuing Quality Control

To assure that the Morse laboratory analyses were reliable, a set of fortified control samples was included when each batch of field samples were analyzed.

Table 8: Recovery of MITC from fortified samples at Morse laboratory.

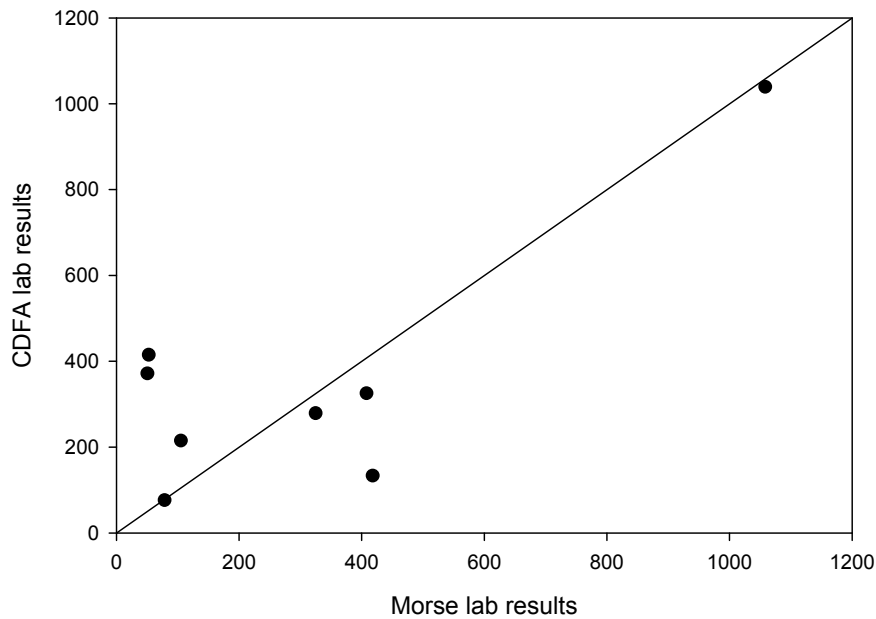
Date	Fortified Level µg/tube	Recovery Level µg/tube	% Recovery
10/30/06	0.1	0.108	108
10/30/06	10.0	8.77	88
10/31/06	0.5	0.478	96
10/31/06	10.0	10.3	103
10/31/06	0.5	0.460	92
11/01/06	0.5	0.483	97
11/01/06	50.0	50.1	100
11/01/06	0.5	0.448	90
11/01/06	50.0	45.4	91
11/02/06	0.5	0.464	93
11/02/06	100.0	89.5	90
11/02/06	0.5	0.417	83
11/02/06	100.0	80.9	81
11/06/06	0.5	0.506	101
11/06/06	100.0	96.5	97
11/07/06	0.5	0.496	99
11/07/06	200.0	171	86
11/07/06	0.5	0.482	96
11/07/06	500.0	456	91
Mean % Recovery			93.79

Table 8 shows the recovery of MITC from fortified samples during the analysis of field samples. In this analysis, the mean percent recovery was 93.79 and the standard deviation was 6.90. The standard error was 1.58, and the 95 % confidence level for percent recovery ranged from 90.46 to 97.12. Hence, the reliability of MITC recovery from the samples by the Morse laboratory analytical method was high.

Background and Collocated Samples

To assess whether any MITC was present in the ambient air prior to the experiment, four background air samples were collected from stations, 1, 2, 3 and 6 from October 17, 1800 hours to October 18, 0700 hours. The County Agricultural Commissioner's Office for Santa Cruz County was contacted to check whether any MITC generating pesticide(s) were used within a radius of one mile from the study location during the month of October 2006. They confirmed that no such use was reported. The four background samples analyzed at Morse laboratory did not detect any MITC.

Figure 5: The relationship of MITC concentration ($\mu\text{g}/\text{m}^3$) of collocated samples between Morse lab and CDFA lab



The collocated samples provide an estimate of field sampling variation. A collocated sample duplicates the regular field sample at the same location, and hence both can be compared, if no other variability was introduced during chemical analysis. It is desirable to do this assessment when the concentrations are near their peak. Therefore, at interval 2 (October 18, 1520-1850 hours), and at interval 4 (October 19, 0100-0645 hours), four extra samples per interval were collected at stations 2, 4, 6, and 8 respectively, giving 8 collocated and 8 regular samples. The regular samples were analyzed by Morse lab and the CDFA lab analyzed the collocated samples.

Table 9: The MITC concentrations ($\mu\text{g}/\text{m}^3$) of collocated samples by two laboratories.

Sample Period	Station	Morse lab results ($\mu\text{g}/\text{m}^3$)	CDFA lab results ($\mu\text{g}/\text{m}^3$)
2	2	1058.32	1039.43
2	4	78.61	76.19
2	6	52.78	415.11
2	8	105.34	215.28
4	2	324.79	278.96
4	4	417.94	133.34
4	6	50.42	371.63
4	8	407.81	325.26

There are at least two factors contributing to the variability; field variability, and laboratory analytical variability. The large variation shown by two laboratories for some of the collocated samples has no apparent explanation.

The plot of all 8 observations from two laboratories is given in Figure 5. The solid line represents a perfect correlation between the two laboratories. Regression analysis between the two labs gave a

statistically significant correlation when all 8 points were used ($p < 0.02$, $r^2 = 0.61$, $n = 8$). However, the relationship was dominated by a single high point. When the large valued data point was removed, the relationship vanished ($p > 0.68$, $r^2 = 0.04$, $n = 7$). Inadvertent spills may have led to this high value point. Collocated samples were one of several Quality Control Quality Assurance (QAQC) samples included in this study.

Storage Stability

MITC samples kept on dry ice were stable for two weeks (Wofford et al., 2003; Leung, 1982). All samples from this study were analyzed within this period.

RESULTS AND DISCUSSION

Air Concentrations of MITC in Relation to Wind Speed and Direction

The sampler symbol and location number, MITC concentration, wind rose plot and their details for the 18 sampling intervals are given in Figure 6, as a series. Figure 6A gives the common notation and wind rose information. Figure 6B gives MITC values and wind data for the 18 sampling intervals. Other sampling interval details are given in Table 5. During interval 1 MITC production started. The station 2 during interval 2 (from 6.5 to 10.0 hours after beginning of Basamid[®] G application) recorded the largest MITC concentration of $1058.32 \mu\text{g}/\text{m}^3$. This reading was corroborated by the collocated sample value of $1039.43 \mu\text{g}/\text{m}^3$. Two small dazomet spills, west of this sampler and upwind may have affected the values recorded. Hence the measurements at this station were not used for the flux calculations.

Figure 6A: The notations and wind-rose information common to all 18 interval plots.

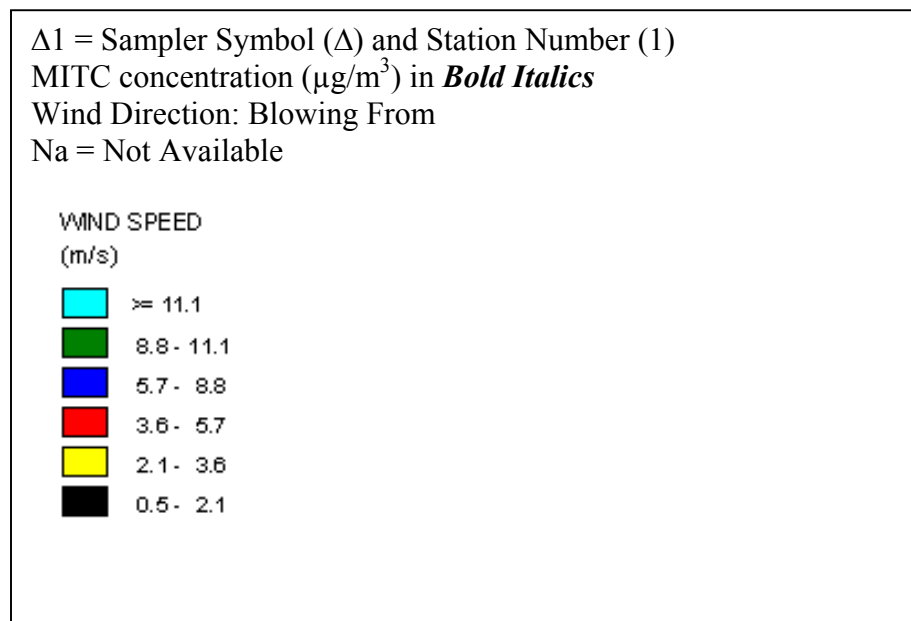
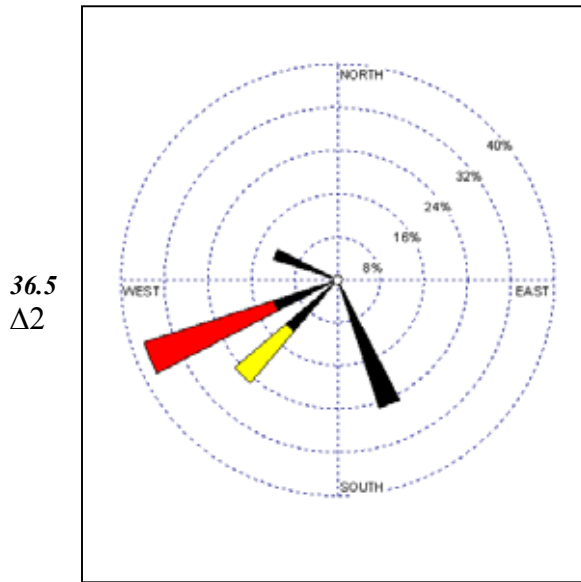


Figure 6B: MITC concentrations ($\mu\text{g}/\text{m}^3$) and wind data.

Interval 1

$\Delta 3$ 31.1 $\Delta 4$ 29.9 $\Delta 5$ 179.1



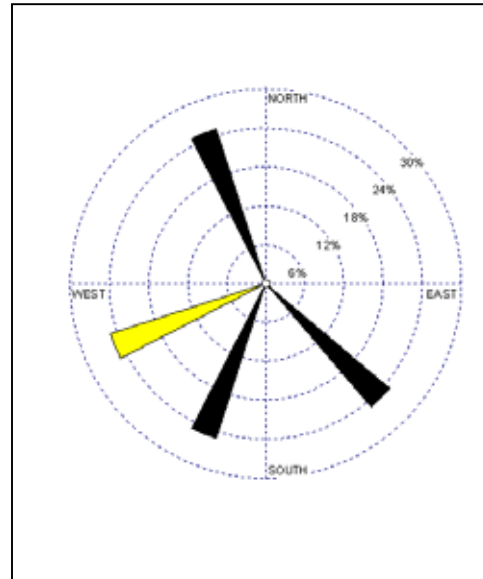
36.5
 $\Delta 2$

$\Delta 1$ 8.2 $\Delta 8$ 12.3 $\Delta 7$ 7.0

Interval 2

▲ Magnetic north

$\Delta 3$ 156.7 $\Delta 4$ 78.6 $\Delta 5$ 94.0



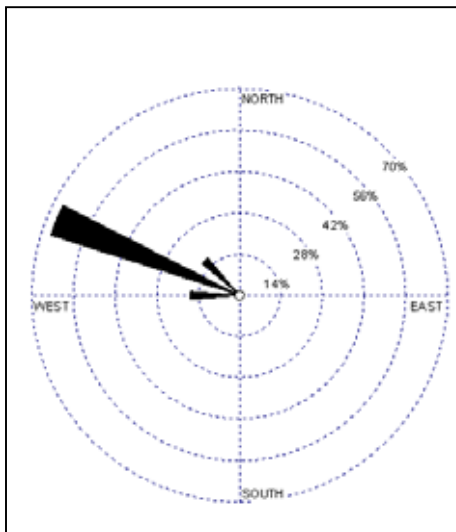
76.4 1058.3
 $\Delta 6$ $\Delta 2$

52.8
 $\Delta 6$

$\Delta 1$ Na $\Delta 8$ 105.3 $\Delta 7$ 5.8

Interval 3

$\Delta 3$ 54.5 $\Delta 4$ 55.1 $\Delta 5$ 66.6



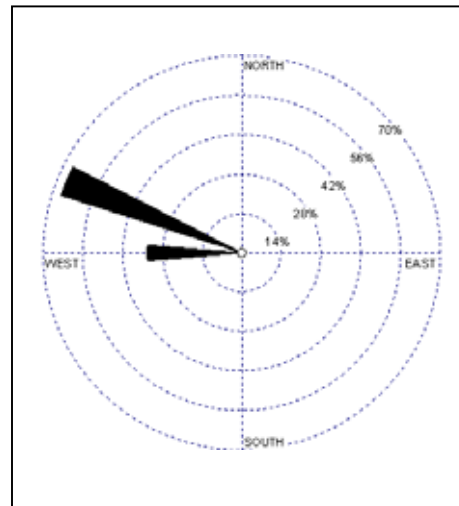
482.9
 $\Delta 2$

451.4 324.8
 $\Delta 6$ $\Delta 2$

$\Delta 1$ 304.2 $\Delta 8$ 559.9 $\Delta 7$ 382.4

Interval 4

$\Delta 3$ 60.6 $\Delta 4$ 417.9 $\Delta 5$ 140.1



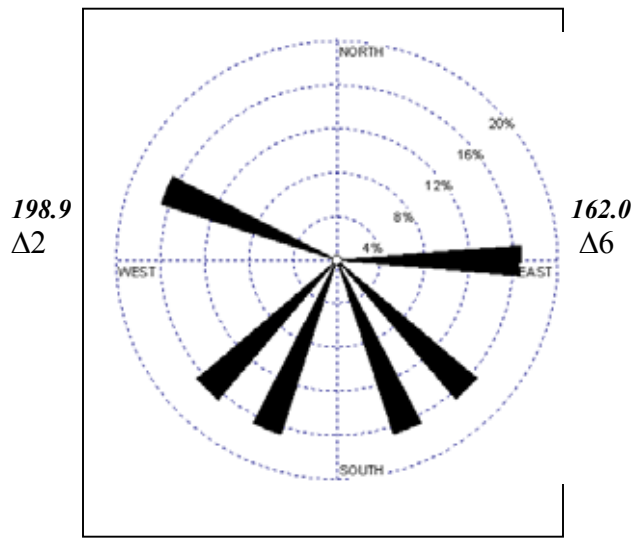
50.4
 $\Delta 6$

$\Delta 1$ 76.0 $\Delta 8$ 407.8 $\Delta 7$ 331.9

Figure 6B MITC concentrations ($\mu\text{g}/\text{m}^3$) and wind data continued.

Interval 5

$\Delta 3$ 80.9 $\Delta 4$ 152.8 $\Delta 5$ 63.8



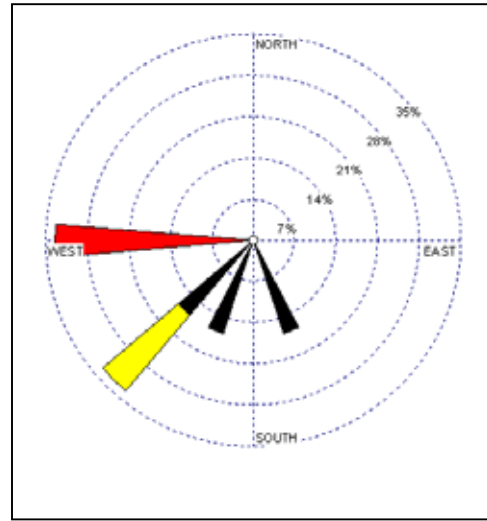
198.9
 $\Delta 2$

162.0
 $\Delta 6$

$\Delta 1$ 85.6 $\Delta 8$ 153.6 $\Delta 7$ 108.9

Interval 6

$\Delta 3$ 44.7 $\Delta 4$ 45.7 $\Delta 5$ 98.8



40.3
 $\Delta 2$

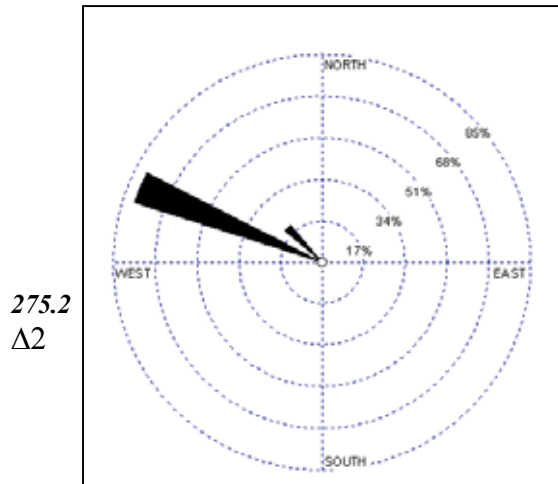
213.4
 $\Delta 6$

$\Delta 1$ 5.0 $\Delta 8$ 7.3 $\Delta 7$ 13.8

▲ Magnetic north

Interval 7

$\Delta 3$ 92.9 $\Delta 4$ 128.3 $\Delta 5$ 97.6



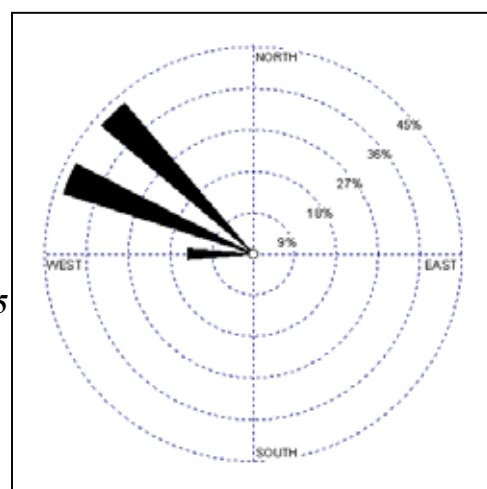
275.2
 $\Delta 2$

199.4
 $\Delta 6$

$\Delta 1$ 155.2 $\Delta 8$ 224.9 $\Delta 7$ 151.1

Interval 8

$\Delta 3$ 62.6 $\Delta 4$ 86.1 $\Delta 5$ 71.2



184.5
 $\Delta 2$

166.4
 $\Delta 6$

$\Delta 1$ 80.0 $\Delta 8$ 209.2 $\Delta 7$ 184.8

Figure 6B MITC concentrations ($\mu\text{g}/\text{m}^3$) and wind data continued.

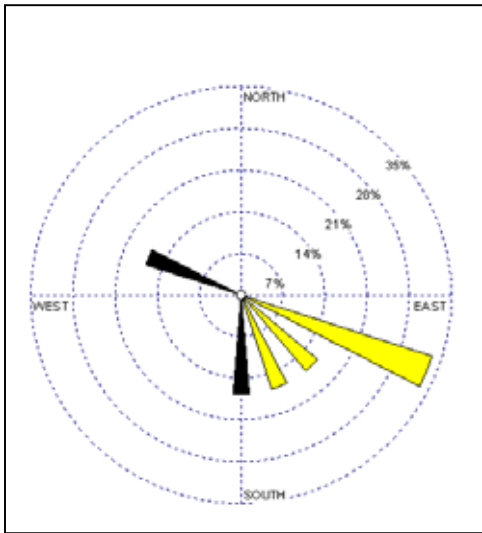
Interval 9

$\Delta 3$ 67.9

$\Delta 4$ 78.8

$\Delta 5$ 20.9

136.1
 $\Delta 2$



90.3 22.3
 $\Delta 6$ $\Delta 2$

$\Delta 1$ 30.6

$\Delta 8$ 87.0

$\Delta 7$ 72.3

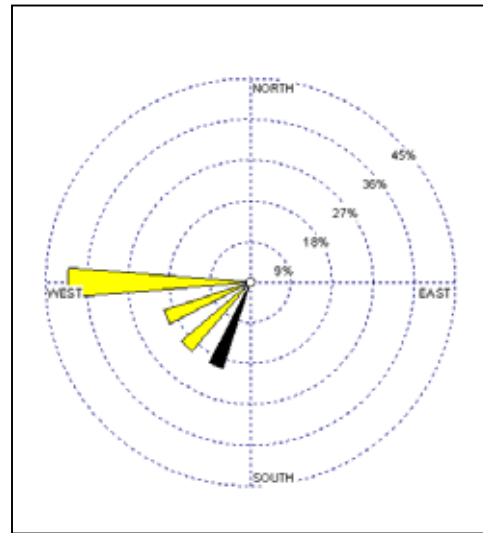
Interval 10

$\Delta 3$ 11.1

$\Delta 4$ 21.5

$\Delta 5$ 39.6

99.8
 $\Delta 6$



$\Delta 1$ 2.8

$\Delta 8$ 7.8

$\Delta 7$ 5.1

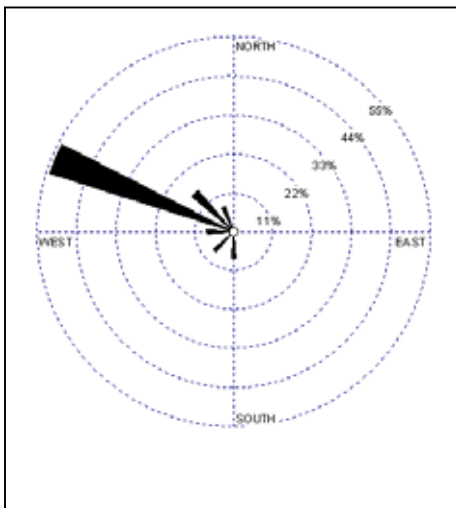
Interval 11

$\Delta 3$ 41.7

$\Delta 4$ 59.6

$\Delta 5$ 44.1

137.9
 $\Delta 2$



102.3 35.0
 $\Delta 6$ $\Delta 2$

$\Delta 1$ 92.4

$\Delta 8$ 192.7

$\Delta 7$ 97.4

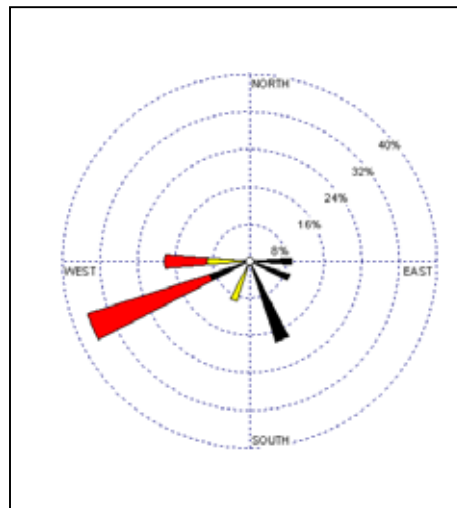
Interval 12

$\Delta 3$ 9.7

$\Delta 4$ 18.7

$\Delta 5$ 27.1

56.2
 $\Delta 6$



$\Delta 1$ 11.2

$\Delta 8$ 21.3

$\Delta 7$ 10.0

Figure 6B MITC concentrations ($\mu\text{g}/\text{m}^3$) and wind data continued.

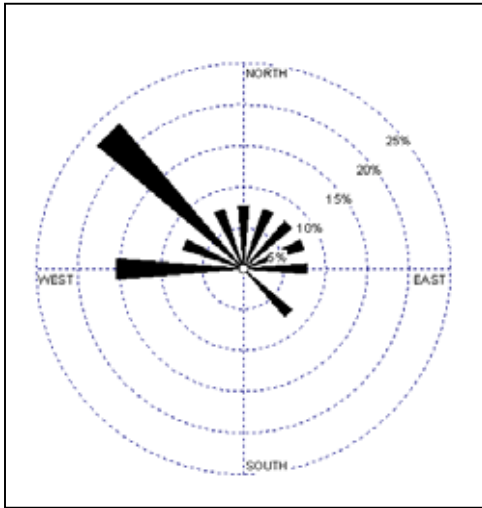
Interval 13

$\Delta 3$ 11.8

$\Delta 4$ 14.4

$\Delta 5$ 9.9

95.8
 $\Delta 2$



26.1
 $\Delta 6$

$\Delta 1$ 77.2

$\Delta 8$ 63.6

$\Delta 7$ 26.4

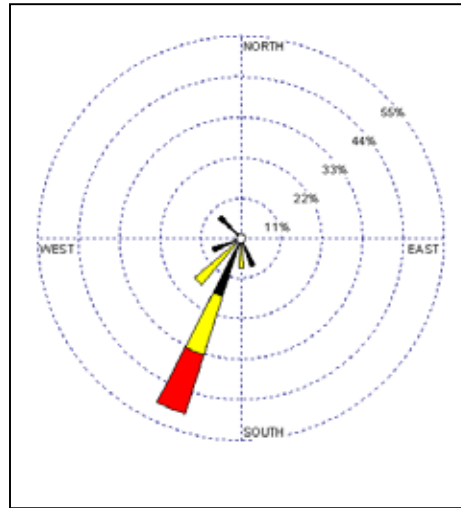
Interval 14

$\Delta 3$ 4.6

$\Delta 4$ 37.6

$\Delta 5$ 23.6

7.5
 $\Delta 2$



21.6
 $\Delta 6$

$\Delta 1$ 1.7

$\Delta 8$ 4.8

$\Delta 7$ 8.5

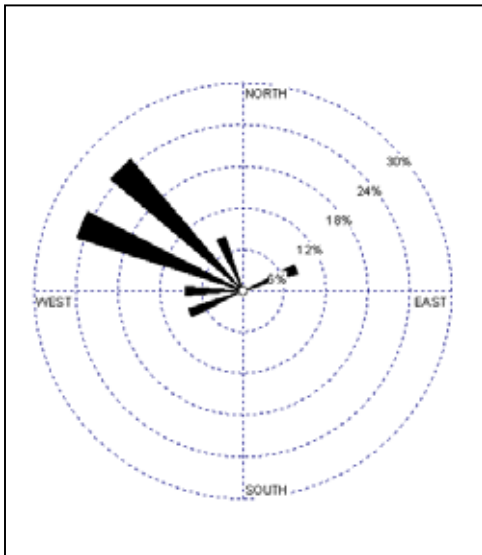
Interval 15

$\Delta 3$ 6.2

$\Delta 4$ 8.1

$\Delta 5$ 5.9

26.9
 $\Delta 2$



20.7
 $\Delta 6$

14.2
 $\Delta 2$

$\Delta 1$ 24.1

$\Delta 8$ 25.8

$\Delta 7$ 15.0

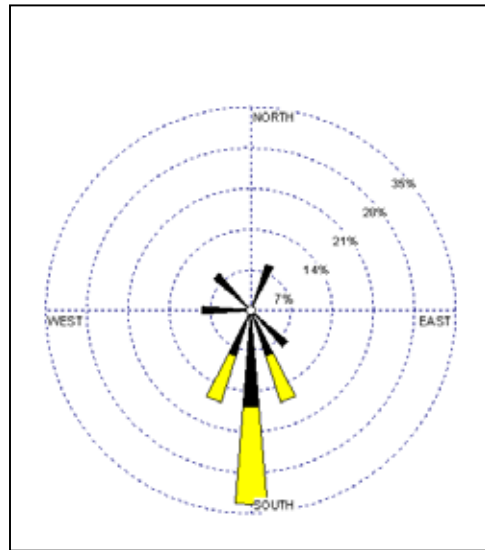
Interval 16

$\Delta 3$ 14.0

$\Delta 4$ 24.9

$\Delta 5$ 8.4

12.9
 $\Delta 6$



$\Delta 1$ 1.8

$\Delta 8$ 3.6

$\Delta 7$ 3.0

Figure 6B MITC concentrations ($\mu\text{g}/\text{m}^3$) and wind data continued.

Interval 17

$\Delta 3$ 3.1

$\Delta 4$ 4.3

$\Delta 5$ 3.1

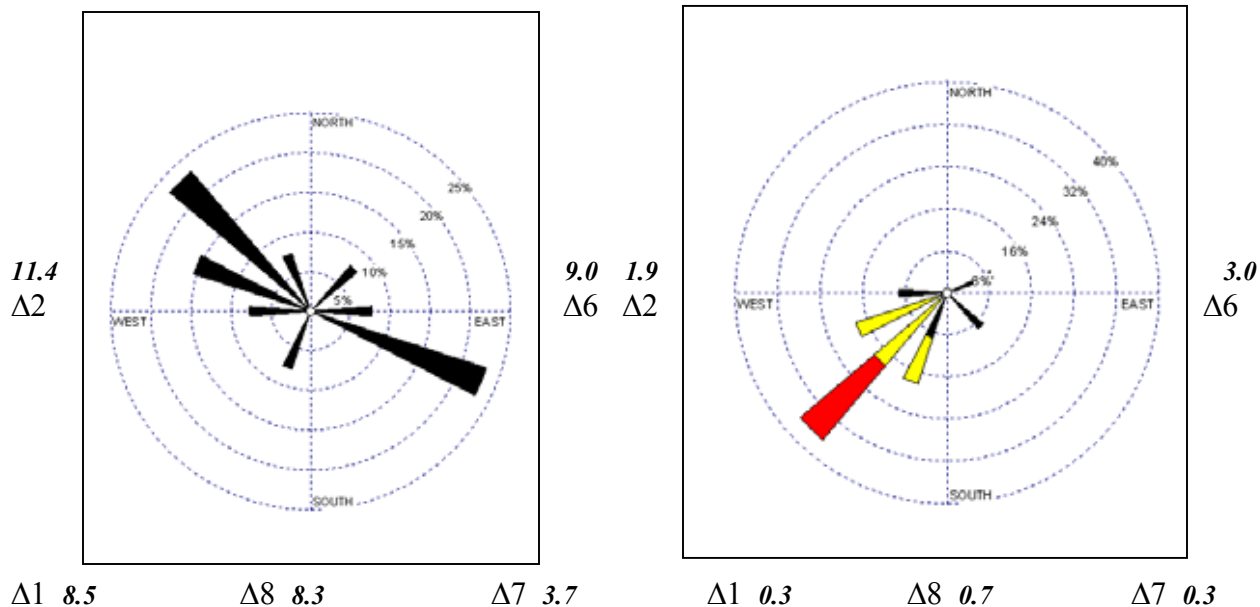
Interval 18

$\Delta 3$ 0.8

$\Delta 4$ 1.9

$\Delta 5$ 2.6

▲ Magnetic north



The predominant wind direction (over 40% of the time) during interval 1 (0 to 6.5 hours after the start of treatment) was from southwest to northeast. The downwind sampler, at station 5 recorded highest concentration, $179 \mu\text{g}/\text{m}^3$ of MITC for this period. Interval two was only 3 hours and 30 minutes long. If the high reading from station 2 is ignored, the MITC recorded by other stations are comparable to interval 1. The intervals 3 (10 - 16.2 hours after start of treatment) and 4 (16.2 - 22 hours after start of treatment) had higher concentrations of MITC ($560 \mu\text{g}/\text{m}^3$ and $418 \mu\text{g}/\text{m}^3$ maximum values respectively) relative to intervals 1 and 2 ($179 \mu\text{g}/\text{m}^3$ and $157 \mu\text{g}/\text{m}^3$ maximum values). The most frequent wind direction, over 65 % of the time for intervals 3 and 4 was from the west and north/west, and most of the high recording stations were down wind. Interval 5 (22 - 28 hours after start of treatment) showed a variation of MITC concentrations from $63.8 \mu\text{g}/\text{m}^3$ to $198.9 \mu\text{g}/\text{m}^3$, and the wind direction was variable too. During interval 6, 28 - 34 hours after start of treatment wind was mostly blowing from southwest and west to northeast and east over 70 % of the time, and down wind stations recorded more MITC than the upwind stations. This interval had wind speeds between 3.8 and 5.7 m/s, approximately 35% of the time. During the intervals 7-9 (34.0 - 52.0 hours after start of treatment), the MITC concentrations fluctuated between 20.9 and $275.2 \mu\text{g}/\text{m}^3$, but higher concentrations were frequently reported at down wind stations. From interval 10 to interval 16 (52 to 130 hours after start of treatment), the MITC concentrations gradually declined and ranged from 192.7 to $2.8 \mu\text{g}/\text{m}^3$. The wind speeds during interval 15 recorded the only calm conditions of the study (i.e. $< 0.5 \text{ m/s}$), 16.7 percent of the time. With the completion of interval 16, collection of MITC samples was suspended. Past experience has suggested that by this duration the losses of MITC to air would be negligible (Fan et al., 2008). To test this concept, interval 17 commenced 250 hours after the start of application, which followed a 5-day suspension of sampling. This suspension is reflected as a “break” in X-axis of Figures 7 and 8. During interval 17 (250 – 262 hours after start of treatment) some MITC were detected ($3.1 - 11.4 \mu\text{g}/\text{m}^3$). The last interval, i.e. interval 18 (262 – 274 hours after start of application) gave small, but detectable MITC, and in general an order of magnitude lower than that of interval 17. Table 10 gives the data used in Figures 7 and 8.

Table 10: Details of maximum wind speed (m/s), maximum concentrations ($\mu\text{g}/\text{m}^3$) of MITC, average wind speed (m/s) and average concentrations ($\mu\text{g}/\text{m}^3$) of MITC values for 18 sampling intervals.

Interval	Date	Time of day	Hours after start of application	Maximum wind speed m/s	Maximum concentration detected ($\mu\text{g}/\text{m}^3$)	Average wind speed m/s	Average concentration detected ($\mu\text{g}/\text{m}^3$)
BG*	17/18	night	NA	NA	ND	NA	ND
1	18	day	6.5	5.2	179.1	2.1	47.6
2	18	day	10	5.3	156.7**	2.5	82.2
3	18/19	night	16.2	2.5	559.9	0.9	294.6
4	19	night	22	2.3	417.9	1.7	226.2
5	19	day	28	2.5	198.9	1.0	125.8
6	19	day	34	5.1	213.4	2.8	58.6
7	19/20	night	40	2.8	275.2	1.0	165.6
8	20	night	46	2.2	209.2	1.2	130.6
9	20	day	52	4.4	136.1	1.7	73.0
10	20	day	58	3.9	99.8	2.4	26.2
11	20/21	night	70	3.0	192.7	1.2	96.0
12	21	day	82	6.2	56.2	2.7	23.7
13	21/22	night	94	2.1	95.8	1.0	40.6
14	22	day	106	5.1	37.6	2.3	13.7
15	22/23	night	118	2.1	26.9	0.6	16.6
16	23	day	130	4.7	24.9	1.8	10.3
17	28/29	night	262	2.8	11.4	1.1	6.4
18	29	day	274	4.9	3.0	2.3	1.4

NA=Not applicable ND= Not detected.

*BG = Background samples

**Note: The high concentration value of $1058.32 \mu\text{g}/\text{m}^3$ MITC was not used in the Figures 7 and 8, and also omitted from flux and average concentration calculations.

Figures 7 and 8 and Table 10 show that high MITC concentrations coincided with low wind speeds. This is true for both maximum (Figure 7) and average (Figure 8) wind speeds and MITC concentration values.

Figure 7: Maximum MITC concentration and maximum wind speed over time

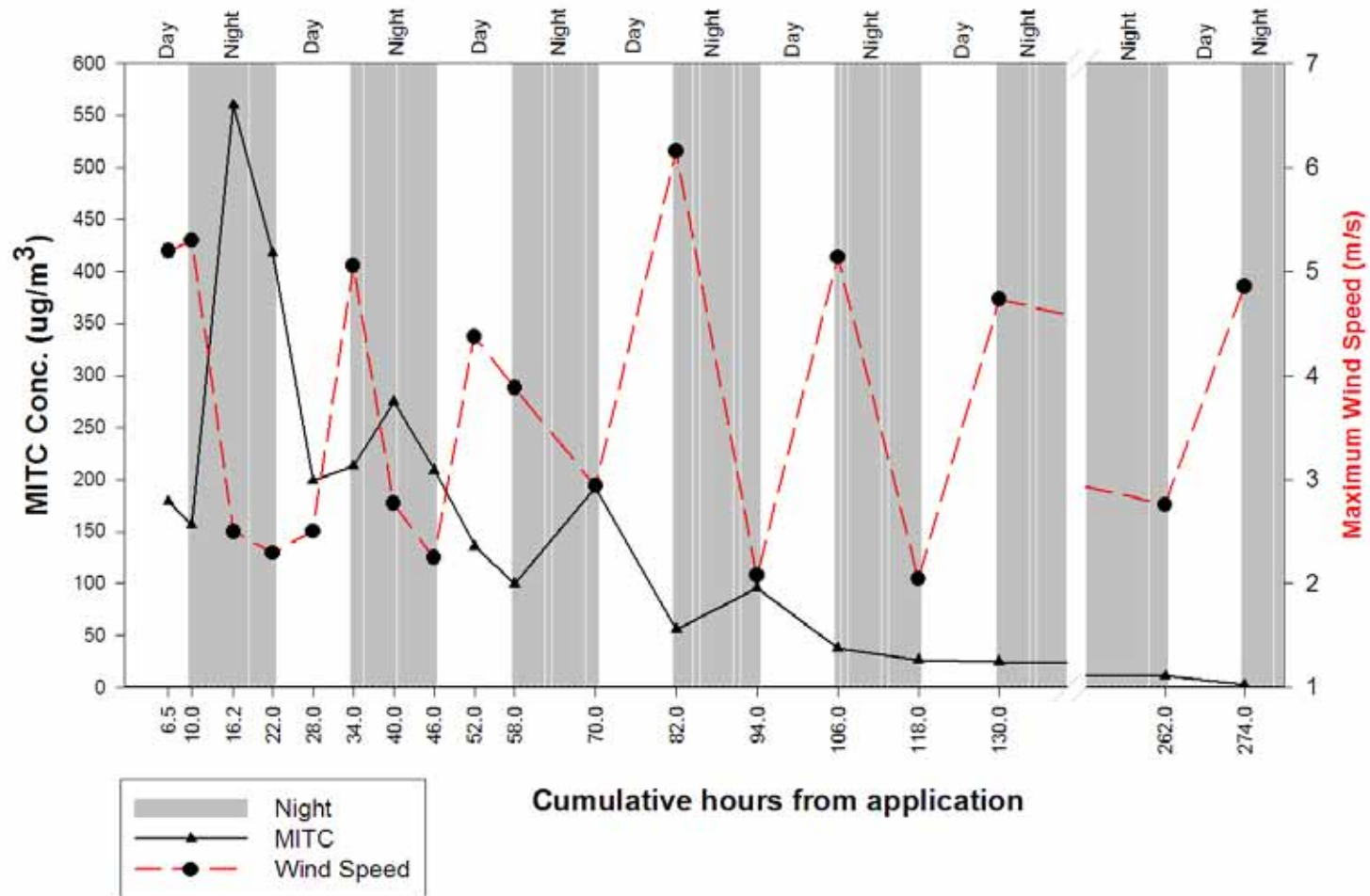
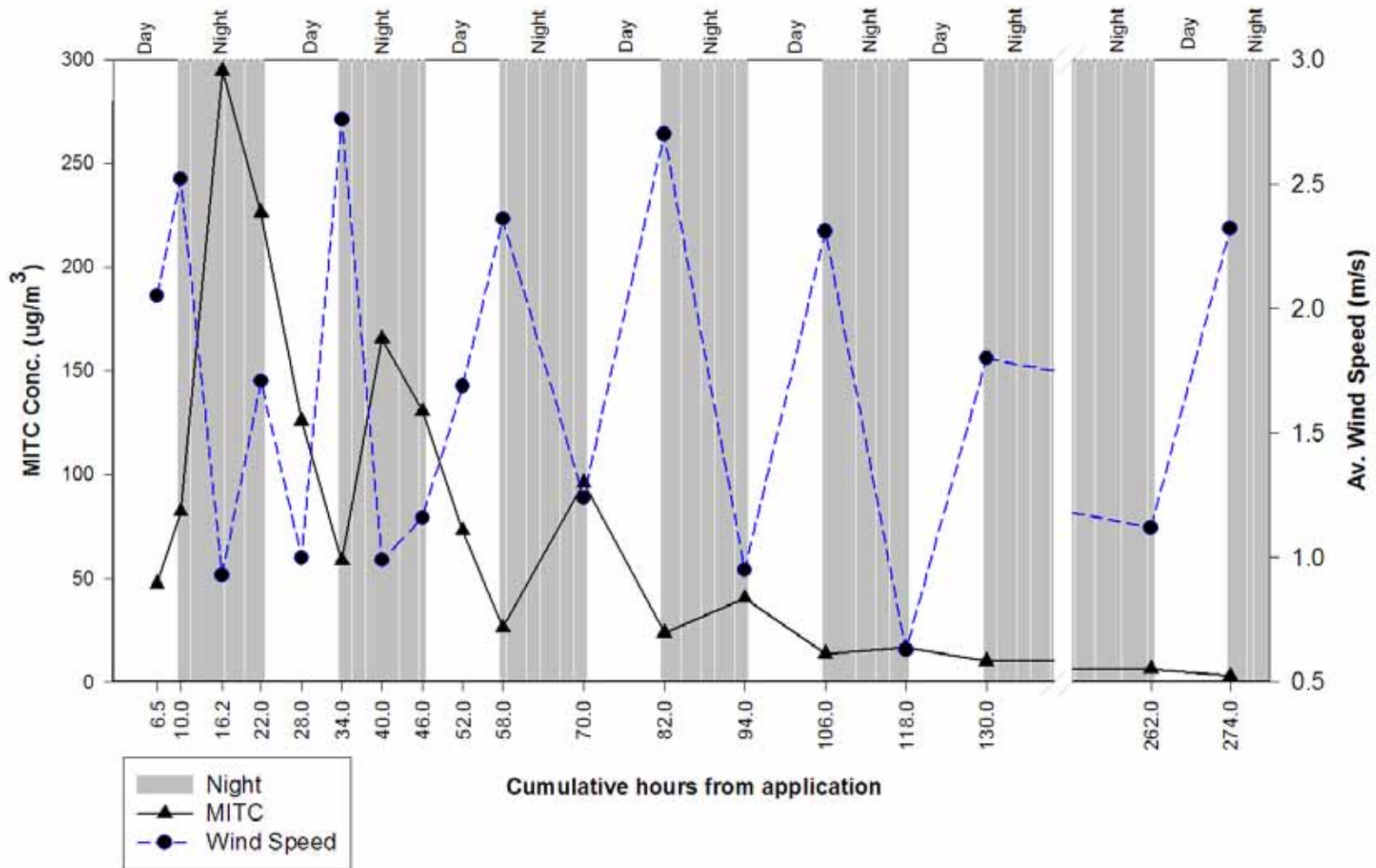


Figure 8: Average MITC concentration and average wind speed over time



Photograph 1: Google Earth hybrid map of the research field.



Photograph 2: Application of Basamid[®] G.



Photograph 3: View of the trial field from location (station) 1, looking to the north. The sampler assembly pumps are covered by black polyethylene to protect from water.

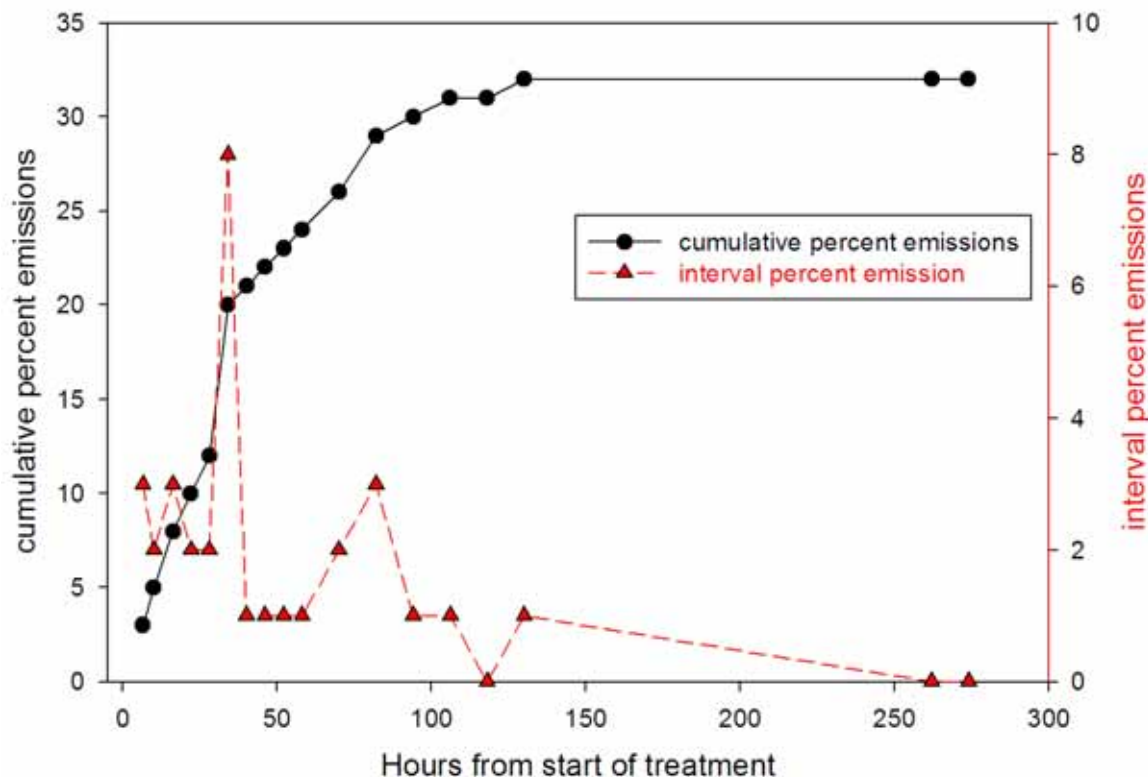


RESULTS OF MODELING

Flux Estimation and emission of MITC:

Appendix 5 presents modeling and back-calculation estimates of flux. By the end of the study (sampling interval 18, and 274 hours after the start of Basamid[®] G application), 32% of applied equivalent of MITC was emitted from the plot. Fan et al. 2008 estimated a loss of 43 % of the applied MITC in 5-days. In this study, 31 % of the applied MITC was emitted during a similar period (end of interval 14). In conducting regression analysis to estimate flux, only 3 out of the 18 simulated periods (intervals) showed significant r^2 values at $p = 0.05$. When the measured and modeled concentrations for each of the 15 non-significant intervals were sorted and reanalyzed, r^2 values improved. Figure 9 shows the cumulative percent and interval percent emissions of MITC. From the cumulative percent emission curve it is clear that the rapid losses took place from about 10 hours up to about 70 hours after the start of Basamid[®] G application. As for individual interval percent emission, the first five intervals reported losses between 2 and 3 % per interval, and the 6th interval (34 hours after the start of application) lost the highest amount of 8 % (Figure 9, right-handed axis). Ten percent of applied MITC equivalent (9,140g) was emitted to air by the end of first 24-hr period. Twelve percent or 10,969g of MITC equivalent was emitted during the second 24-hr period (Appendix 5). By the end of interval 6, the cumulative percent emission of MITC was 20 and by the end of interval 13, this value was 30. During interval 16, and 130 hours after start of application, 1% of the applied MITC was emitted from the plot, and became negligible during last two intervals.

Figure 9: MITC cumulative and interval emissions calculated from flux estimates as a percentage of applied MITC equivalent



(Adapted from Appendix 5: Flux memo).

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