

State of California
California Environmental Protection Agency
AIR RESOURCES BOARD

APPENDICES

FOR THE

**Report for Air Monitoring
Around a Tarped Drip Irrigation Application
of Metam Sodium in Ventura County
Spring 2002**

Operations Planning and Assessment Section
Quality Management Branch
Monitoring and Laboratory Division

Project No. P-02-001

Date: May 25, 2004

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APPENDIX I
MONITORING PROTOCOL



Air Resources Board



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Agency Secretary

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Chairman

Gray Davis
Governor

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MEMORANDUM

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

FROM:  Jeffrey P. Cook, Chief
Quality Management Branch
Monitoring and Laboratory Division

DATE: April 23, 2002

SUBJECT: FINAL PROTOCOL FOR THE 2002 APPLICATION AIR MONITORING
FOR METAM SODIUM

Attached is the final "Protocol for Air Monitoring Around a Drip Irrigation Application of Metam Sodium During Spring 2002". We received your May 25, 2001, comments on the draft protocol and have made the appropriate changes. We understand that DPR will distribute the protocol to the Metam Sodium Task Force participants.

If you or your staff have questions or need further information, please contact me at 322-3726 or Kevin Mongar of my staff at 322-2249.

Attachment

cc: Randy Segawa, DPR (w/Attachment)
Shifang Fan, DPR (w/Attachment)
Kevin Mongar, MLD (w/Attachment) ✓
Robert Carr, San Luis Obispo Co. APCD (w/Attachment)
Richard Greek, San Luis Obispo Co. Agricultural/Commissioner (w/Attachment)

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State of California
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AIR RESOURCES BOARD

**Protocol for Air Monitoring
Around a Drip Irrigation Application of Metam Sodium
During Spring 2002**

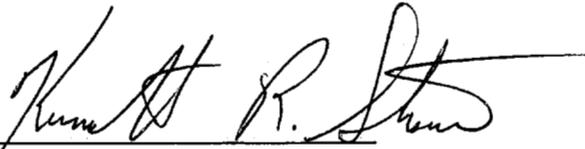
Prepared by
Operations Planning and Assessment Section
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Monitoring and Laboratory Division

Date: April 23, 2002

APPROVED:



Jeffrey R. Cook, Chief
Quality Management Branch
Monitoring and Laboratory Division



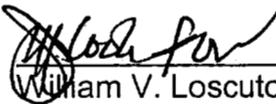
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This protocol has been reviewed by the staff of the California Air Resources Board and is approved for publication. Approval does not signify that the contents necessarily reflect the views and policies of the Air Resources Board, nor does mention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

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- Attachment 2 - Standard Operating Procedure, Sampling, and Analysis of Methyl Isocyanate in Application and Ambient Air Using High Performance Liquid Chromatography With a Fluorescence Detector
- Attachment 3 - Standard Operating Procedure, Sampling, and Analysis of Carbon Disulfide in Silco™ Canisters
- Attachment 4 - Pesticide Canister Sampling Procedures For Application Studies
- Attachment 5 - Pesticide Adsorbent Tube Sampling Procedures For Application Studies
- Attachment 6 - Adsorbent Tube Field Log Sheet Canister Field Log Sheet and Canister Field Data Sheet

**Protocol for Air Monitoring
Around a Drip Irrigation Application of Metam Sodium
During Spring 2002**

I. Introduction

At the request of the California Department of Pesticide Regulation (DPR) (June 28, 2000 Memorandum, Helliker to Lloyd), the Air Resources Board (ARB) staff will conduct application site air monitoring for the pesticide metam sodium (as the breakdown products methyl isothiocyanate, methyl isocyanate, hydrogen sulfide, and carbon disulfide). Monitoring is tentatively scheduled to occur in Spring 2002. This monitoring will be done to fulfill the requirements of AB 1807/3219 (Food and Agricultural Code, Division 7, Chapter 3, Article 1.5) which requires the ARB "to document the level of airborne emissions...of pesticides which may be determined to pose a present or potential hazard..." when requested by the DPR.

The pesticide sampling and analysis will follow the procedures outlined in this protocol, as well as the quality assurance guidelines described in the "Quality Assurance Plan for Pesticide Air Monitoring" (May 11, 1999 version) and the following ARB methods:

"Standard Operating Procedure, Sampling and Analysis of 1,3-dichloropropene (Telone) and Methyl Isothiocyanate (MITC) in Application and Ambient Air using Gas Chromatography/Mass Selective Detector (06/25/01 Version)" (Attachment 1)

"Standard Operating Procedure, Sampling and Analysis of Methyl Isocyanate (MIC) in Application and Ambient Air using High Performance Liquid Chromatography with a Fluorescence Detector (06/25/01 Version)" (Attachment 2).

"Standard Operating Procedure Sampling and Analysis of Carbon Disulfide In Silco™ Canisters (Version 1, March 13, 2002)" (Attachment 3).

II. Sampling

Monitoring for methyl isocyanate (MIC) and methyl isothiocyanate (MITC) will be conducted with sampling tubes. Monitoring for carbon disulfide will be conducted using Silcosteel® canisters. Monitoring for hydrogen sulfide will be conducted using a portable sampler (Jerome sampler).

MIC and MITC Sampling:

The sampling methods for two of the compounds require passing measured quantities of ambient air through adsorbent sampling tubes. For MIC, the tubes are 8 mm x 110 mm, XAD-7, 1-(2-pyridyl)peperazine coated, with 400 mg in the primary section and 200 mg in the secondary (Supelco special order). For MITC, the tubes are 8 mm x 110 mm, coconut shell charcoal with 400 mg in the primary section and 200 mg in the secondary (SKC catalogue #226-09).

Sample collection for MIC is at a flow rate of 75 standard cubic centimeters per minute (sccpm) and MITC is at a flow rate of 2.5 standard liters per minute (sLpm). Immediately after sampling, the tubes are capped, labeled, placed in a culture tube, and stored and transported in an insulated container with dry ice to the ARB laboratory in Sacramento.

Each sample train consists of an adsorbent tube, Teflon fittings and tubing, rain/sun shield, rotameter (or needle valve), train support, and a 12 volt DC vacuum pump. Tubes are prepared for use by breaking off the sealed glass end and immediately inserting the tube into the Teflon fitting. The tubes are oriented in the sample train according to a small arrow printed on the side indicating the direction of flow. A rotameter (or needle valve) with a range of 0-5 Lpm is used to control sample flow for the MITC sampling and a rotameter (or needle valve) with a range of 0-240 ccpm will be used to control the flow for the MIC sampling. The flow rates will be set using calibrated digital mass flow meters (MFM) before the start of each sampling period. An MFM scaled from 0-5 sLpm is used for MITC, and a 0-100 sccpm MFM is used for the MIC samplers. The flow rate is also checked and recorded, using the MFM, at the end of each sampling period. Samplers will be leak checked prior to each sampling period, with the sampling tubes installed. Any change in flow rates will be recorded on the field log sheet. The pesticide sampling procedures for adsorbent tubes are included as Attachment 5.

Caution should be used during field monitoring, transportation, storage, and lab analysis to minimize exposure of samples to sunlight in order to prevent photo-degradation of MITC and MIC.

Carbon Disulfide Sampling:

Integrated ambient air samples will be collected for carbon disulfide using passive air sampling into evacuated six liter, Silcosteel® canisters (from Restec Corporation). The flow rate of 3 sccpm will be set and measured using a 0-10 sccpm mass flow meter. The sampling system will be operated continuously with the exact operating interval recorded in the log-book and on the field data sheets (see Attachment 6). The canister vacuum reading will be recorded at the start and end of each sampling period using the -30 to 0 inHg gauge on the passive sampler. The canister vacuum reading will also be measured using a more accurate gauge in the lab before and after transport to/from the field. The laboratory gauge readings will be used to calculate the sample volume collected.

The critical orifice flow controllers (Silcosteel treated Veriflo SC423XL) will be attached, using a Silcosteel treated swagelock connector, to the valve fitting on the canister (Figure 2). A 6-foot section of 1/8 inch O.D, Silcosteel tubing will be attached to the inlet end of an in-line, 5 micron filter, which will be attached to the inlet end of the flow controller. The inlet end of the tubing will be bent into a U shape (to prevent rain from entering). At the end of each sampling period, the canisters will be placed in shipping containers with a sample identification/chain of custody sheet and will be shipped as soon as reasonably possible to the ARB Monitoring and Laboratory Division laboratory for analysis. The samples will be stored at ambient laboratory temperature prior to analysis.

When using a critical orifice flow restrictor for passive integrated canister sampling, the potential decrease in flow rate as the vacuum in the canister changes must be taken into account. The flow control device used for the study (Veriflo SC423XL, from Restek Corporation) was designed to regulate and maintain a constant flow as the vacuum in the canister decreases. The manufacturer specifications indicate that the controller is capable of maintaining a continuous low flow with vacuum ranges from -29.9 to -5 inHg. The in-line filter helps prevent particles from entering the critical orifice of the flow controller, which could clog the critical orifice and affect the flow through the controller. The manufacturer specifications indicate that the outside temperature can have a slight effect on the flow rate. For example, there could be an approximately 6% flow drop when the temperature changes from 80 °F to 125 °F.

The pesticide ambient sampling procedures for canisters are enclosed as Attachment 4. The canister sampling field log sheet and canister data sheet are enclosed as Attachment 6. These forms will be used to record start and stop times, start and stop vacuum readings, sample identifications, weather conditions, the sampler's initials and any other significant data.

Hydrogen Sulfide Sampling:

Hydrogen Sulfide will be sampled with a portable Jerome 631-X Hydrogen Sulfide Analyzer at each sampling site. A 25-second sample will be taken each time the XAD-4 cartridges are changed out and at approximately mid-way through the application. The Jerome Analyzer samples at 150cc/min and for the expected range of 0.10 to 0.99 ppm must sample for 25 seconds. The detection limit of the Jerome Analyzer is 0.003 ppm and its range is 0.001 to 50 ppm. The DPR's requested quantitation limit was 5 ug/m³ (0.004 ppm). Prior to first use and after each morning and evening recovery, the sampler will be regenerated to assure the sensor is zeroed prior to use. The principle of operation of the instrument is that hydrogen sulfide reacts with a thin gold film by increasing the electrical resistance in proportion to the mass of hydrogen sulfide present. The procedures described in the operational manual supplied by the manufacturer will be followed for field sampling. The sampler will be audited at the Sacramento ARB shops before and after the field test, using an ARB certified standard of H₂S.

Sampling Schedule:

The application sampling schedule consists of samples collected during daylight and overnight periods as shown in Table 1.

Table 1
Application Sampling Schedule

<u>Sample period begins</u>	<u>Sample duration time</u>
Background (pre-application)	24 hours if possible; minimum 12 hours (if <24 hours must meet target EQL)
During application and post –application	Start of application (until 1 hour before sunset)
1 hour before sunset	Overnight (until 1 hour after sunrise)
1 hour after sunrise	Daytime (until 1 hour before sunset)
1 hour before sunset	Overnight (until 1 hour after sunrise)
1 hour after sunrise	Daytime (until 1 hour before sunset)
1 hour before sunset	Overnight (until 1 hour after sunrise)

In the event that application occurs at night, the alternate day-night schedule will be followed, i.e., with six sampling periods following the application sampling period. If the fumigation takes two or more days, samples will be collected during the overnight period separating the applications, and the overnight/daytime schedule will be followed from the last day of application.

The exact location of the application monitoring study has not yet been determined. ARB staff will contact the County Agricultural Commissioner’s offices in the Central Coast area to coordinate the selection of a study site and the test dates. The County Agricultural Commissioner’s staff will make initial contact with, or will at least provide a list of local contacts for growers, applicators, and/or pesticide control advisers that may be willing to cooperate in conducting the study. Monitoring sites are arranged with the voluntary cooperation of growers and applicators. ARB staff will investigate contacts until a cooperative grower is found and an appropriate site is selected. Permission to conduct the study will be obtained from the application plot land-owner and owners of adjacent land where samplers will be positioned.

Candidate fields for application monitoring will be 10 acres or larger. The crop type for the application study was not specified by the DPR. The DPR has recommended that, “monitoring for metam sodium be a drip irrigation application at a site using the highest allowed rates of use (i.e., about 318 pounds AI per treated (raised bed area) acre”).

For the MIC and MITC adsorbent tube samples, 8 samplers will be positioned, one on each side of the field and one at each corner. A 9th replicate sampler will be collocated at one position (downwind). For the carbon disulfide canister samples, 4 samplers will be positioned, 1 on each side of the field. A 5th replicate sampler will be collocated at

one position (downwind) Ideally, samplers should be positioned a minimum of 20 meters from the field; however, site conditions will dictate the exact placement of samplers.

In regard to field data, the monitoring report will include: 1) a record of the positions of the monitoring equipment with respect to the field, and if necessary, how the field was divided to treat over several days; 2) a drawing of the field including precise dimensions, field size, location of the monitoring sites, and showing the precise location of the meteorological equipment, trees, buildings, and other obstacles; 3) meteorological data collected at a minimum of 15-minute intervals, including wind speed and direction, humidity, and air temperature and comments regarding degree of cloud cover; 4) the elevation of each sampling station with respect to the field, and the orientation of the field with respect to North (identified as either true or magnetic North); 5) the start and end time of the application, and 6) the product used and the application rate.

III. Analysis

The ARB method and MITC sampling and analysis is enclosed as Attachment 1. The exposed charcoal tubes are stored in an ice chest or refrigerator until desorbed with 3 ml of dichloromethane. The attached SOP specifies that a gas chromatograph with a mass selective detector is used for analysis. The analyses will be performed by the ARB laboratory in Sacramento. The DPR recommended a target 24-hour estimated quantitation limit (EQL) of 0.5 ug/m³ for MITC. The attached SOP specifies an EQL of 0.18 ug/m³ for MITC for a 24-hour sample collected at 2.5 sLpm.

The draft method for the sampling and analysis of MIC are included as Attachment 2. The MIC method will consist of HPLC analysis with fluorescence detector. The analyses will be performed by the ARB laboratory in Sacramento. The DPR recommended a target 24-hour EQL of 0.1 ug/m³ for MIC. The attached SOP specifies an EQL of 0.42 ug/m³ for MIC for a 24-hour sample collected at 75 sccpm.

The draft method for the sampling and analysis of carbon disulfide are included as Attachment 3. The Standard Operating Procedures (SOP) and method validation results will be included in the laboratory sample report. The procedures are based on EPA Method TO-15 and consist of cryogenic pre-concentration of an aliquot of the whole air sample followed by GC/MS analysis. The canisters arrive from the field at sub-ambient pressure and are pressurized (diluted) in the laboratory before analysis. The analyses will be performed by the ARB laboratory in Sacramento. The DPR recommended a target 24-hour EQL of 15 ug/m³ for carbon disulfide. The attached SOP specifies an EQL of 7.16 ug/m³ for carbon disulfide.

IV. Quality Assurance

Field Quality Control for the application monitoring will include the following for each of the sampling methods:

- 1) Four field spikes will be obtained by sampling ambient air at the application monitoring site for between 12 and 24 hours during the background monitoring (i.e., collocated with a background sample at the same environmental and experimental conditions). The spike levels for MIC, MITC, and carbon disulfide have not yet been determined.
- 2) Four trip spikes will be prepared at the same level as the field spikes.
- 3) Four lab spikes will be prepared at the same level as the field and trip spikes.
- 4) Collocated (replicate) for all sampling periods (except the background period) at one sampling location (downwind).
- 5) A trip blank will be obtained for each type of sample media.

V. Sample Labeling

Samples will be labeled using the following format:

Location-Chemical-Sampling Period-Type of Sample

Where:

Location is designated as north 1, 2, or 3 (N1, N2, N3), west (W), south 1, 2, or 3 (S1, S2, S3), and east (E). These designations can be revised as necessary depending on the configuration of the field.

Chemical is designated as M (for MIC), T (for MITC), CS (for CS₂), H (for H₂S).

Sampling period is designated as B (for background) or 1 through 6.

The type of sample is designated as S (sample), C (collocated), TB (trip blank), TS (trip spike), and FS (field spike).

Examples: S2-T-B-S (South2, MITC, background, sample)
 S2-T-B-FS (South2, MITC, background, field spike)
 S2-T-1-S (South2, MITC, sampling period 1, sample)
 S2-T-1-C (South2, MITC, sampling period 1, collocated)

VI. Personnel

ARB field coordination and sampling personnel will consist of staff from the Source Test Section of MLD/ARB.

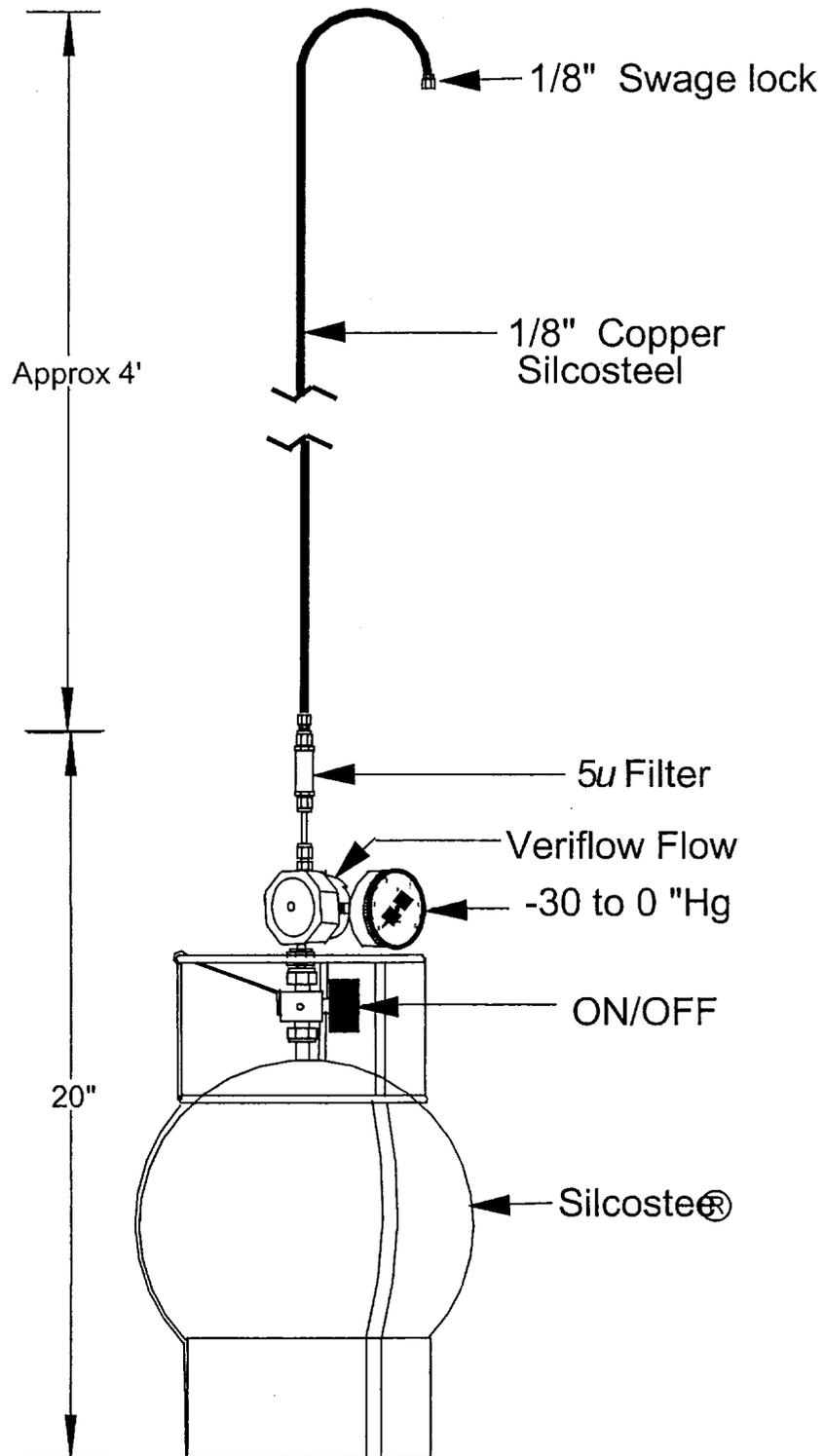
VII. Safety Recommendations

The DPR has revised the standard application study sampling schedule (Table 1) to minimize exposure to sampling personnel and states: "Due to high application rates and high volatility of these pesticides, the potential for exposure is higher than most other pesticides. However, this recommendation should not require any special safety equipment or precautions for sampling personnel."

The DPR's 'Monitoring Recommendations' also include the following safety recommendations.

"The metam sodium product labels warn that metam sodium causes skin damage and may be fatal if absorbed through the skin. Prolonged or frequent contact may cause an allergic reaction. Metam sodium is harmful if inhaled or swallowed and is irritating to eyes, nose, and throat. Monitoring personnel should use proper protective equipment to prevent exposure to the vapors or spray mist and refer to the label of the actual product used for further precautions. According to the product labels, proper protective equipment for applicators making direct contact or for applicators outside an enclosed cab includes coveralls, waterproof gloves, chemical resistant footwear plus socks, face sealing goggles, chemical resistant headgear (for overhead exposure) and apron, and a respirator with an organic-vapor removing cartridge. Concentrations should not exceed 0.5 ppm for any of the sampling intervals at the 60-foot sampling distance from the field."

Figure 1
Passive Canister Sampling



Attachment 1

Standard Operating Procedure
Sampling and Analysis of
1,3-dichloropropene (Telone) and Methyl Isothiocyanate (MITC)
In Application and Ambient Air Using
Gas Chromatography/Mass Selective Detector

California Environmental Protection Agency



Air Resources Board

**Standard Operating Procedure
Sampling and Analysis of 1,3-dichloropropene (Telone)
and Methyl Isothiocyanate (MITC) in Application and
Ambient Air using Gas Chromatography/Mass Selective
Detector**

**Special Analysis Section
Northern Laboratory Branch
Monitoring and Laboratory Division**

06/25/01 version

Approved by:

1. SCOPE

The method uses resin tubes and a gas chromatograph/mass selective detector for the determination of 1,3-dichloropropene (Telone) and methyl isothiocyanate (MITC), one of the breakdown products of Metam-Sodium, for application and ambient air sample analysis. The Department of Pesticide Regulation (DPR) asked the Air Resources Board (ARB) to do ambient and application monitoring of Telone and MITC at a requested quantitation limit of $0.5 \mu\text{g}/\text{m}^3$ for MITC.

2. SUMMARY OF METHOD

Coconut based charcoal tubes are placed on the sampler for 24 hours at 3.0 liters per minute (LPM) flow rate. The samples are stored in an ice chest or refrigerator until extracted with 3 ml of dichloromethane (DCM). The injection volume is $1 \mu\text{l}$. A gas chromatograph with a mass selective detector in the selected ion monitoring (SIM) mode is used for analysis.

3. INTERFERENCES/LIMITATIONS

The primary interference encountered with the previous method was the presence of the MITC near the cis-DCP. The retention time difference is only about 0.05 minutes and even operating in SIM mode, similar ions are detected by the instrument. This makes it difficult to accurately quantitate if both cis-DCP and MITC are present. The installation of a different column than that used in the previous method resolved the issue and easily separates the target compounds. As with any method, additional interferences may be caused by contaminants in solvents, reagents, glassware and other processing apparatus that can lead to discrete artifacts or elevated baselines. Method blanks, both solvent and resin, must be run concurrently with each batch of samples to detect any possible interferences.

4. EQUIPMENT AND CONDITIONS

A. INSTRUMENTATION:

Hewlett-Packard 6890 Series gas chromatograph
Hewlett-Packard 5973 Network mass selective detector
Hewlett-Packard 6890 Enhanced Parameters ALS

MS Transfer line: 280°C

Injector: 210°C , Splitless, Liner 4 mm straight liner with glass wool.

Column: Restek Rtx-200, 60 meter, $320 \mu\text{m}$ i.d., $1.5 \mu\text{m}$ film thickness.

GC Temperature Program: Oven initial 40 °C, hold 4 min. Ramp to 220 °C @ 12 °C/min., hold 1 min., ramp to 240 °C @ 20 °C/min., hold 2.0 min. Retention time: cis-DCP= 11.63 min., trans-DCP= 12.10 min., MITC=12.23 min.

Splitter open @ 1.0 min.

Flows: Column: He, 1.6 ml/min, 9.1psi. (velocity: 32cm/sec)

Splitter: 50 ml/min.

Mass Spectrometer: Electron Ionization

Selective Ion Monitoring: dichloropropene: 75 (quant. ion 100%), 110 (qual. ion 30%); methyl isothiocyanate: 73 (quant. ion 100%), 72 (qual. ion 46%). Tuning: PFTBA on masses 69, 219, 502.

B. Auxiliary Apparatus

1. Precleaned vials, 8 ml capacity with teflon caps.
2. Whatman filters, 0.45 μ m
3. Disposable syringes, 3 ml
4. Sonicator
5. GC vials with septum caps.

C. Reagents

1. Dichloromethane, Pesticide grade or better.
2. 1,3 -Dichloropropene (cis- and trans- mixture), Chem Service PS- 1 52, 99 (+) % or equiv.
3. Methyl Isothiocyanate, Chem Service MET-221A, 99.5%
4. Coconut charcoal sorbent tubes, SKC, Fullerton, CA #226-09.

5. ANALYSIS OF SAMPLES

1. A daily manual tune shall be performed using PFTBA. The instrument is tuned using masses: 69, 219, 502. The criterion for the tune are the peak widths at $\frac{1}{2}$ the peak height, 0.60 ± 0.05 , and the criteria for relative abundance: 69:100%, 219:100-120%, and 502: 7-12%.
2. It is necessary to analyze a solvent blank with each batch of samples. The blank must be free of interferences. A solvent blank must be analyzed after any sample which may result in possible carry-over contamination.
3. A 5-point calibration curve shall be analyzed with each batch of samples. For dichloropropene the analysis is calibrated at 10, 20, 40, 60, 100 ng/ml cis and trans. For methyl isothiocyanate the calibration is at 0.5, 1.0, 2.0, 3.0, 5.0 μ g/ml.

4. With each batch of samples analyzed, a laboratory blank and a laboratory control spike will be run concurrently. A laboratory blank is an unexposed charcoal tube prepared and analyzed the same way the samples are analyzed. A laboratory control spike is a charcoal tube spiked with a known amount of standard. The control sample is prepared and analyzed the same way as the samples. Laboratory check samples should have recoveries that are at least 70% of the theoretical spiked value.
5. A DCP calibration check sample of 10 ng/ml is run after the calibration and every 10 samples and at the end of each sample batch. The calibration check for MITC is 0.75 µg/ml. The value of the check must be within $\pm 3\sigma$ (the standard deviation) or $\pm 10\%$ of the expected value, whichever is greater. If the calibration check is outside the limit, then those samples in the batch after the last calibration check that was within the limit need to be reanalyzed.
6. Score and snap the sample tube, transfer the charcoal into a 8 ml vial. (Save the back-up bed for future analysis if necessary.) Rinse the tube with 3.0 ml of DCM into the extraction vial. Cap and place the vial in the sonicator for 1 hour.
7. Filter the samples using a 3 ml syringe and 0.45 µm filter directly into a GC vial and cap securely.
8. The atmospheric concentration is calculated according to:

$$\text{Conc (ng/m}^3\text{)} = \text{Extract Conc (ng/ml)} \times 3 \text{ ml} / \text{Air Volume Sampled (m}^3\text{)}$$

6. QUALITY ASSURANCE

A. Instrument Reproducibility

The reproducibility of the instrument and analytical method was established by analyzing five(5) 1.0 µl injections of dichloropropene and methyl isothiocyanate standard at three concentrations (low, mid, and high range). The low, mid and high concentrations of dichloropropene were 10, 40 and 100 ng/ml, respectively. The low, mid and high concentrations of methyl isothiocyanate were 0.5, 2.0 and 5.0 µg/ml, respectively.

B. Calibration

The five-point calibration curve is constructed for each compound using linear regression analysis. A curve cannot be used if its correlation coefficient is less than 0.995.

C. Calibration Check

A calibration check control is run after the calibration and every 10 samples and at the end of the sample batch to verify the system is in calibration. The value of the check must be within $\pm 3\sigma$ (the standard deviation) or $\pm 10\%$ of the expected value, whichever is greater. If the calibration check is outside the limit, then those samples preceding the out of limit check need to be reanalyzed.

D. Minimum Detection Limit

Detection limits are based on US EPA MDL calculation. Using the analysis of seven (7) replicates of a low-level matrix spike, the method detection limit (MDL) and the estimated quantitation limit (EQL) for 1,3-dichloropropene is calculated by: $MDL = 3.14 * (\text{std dev values})$, where std dev = the standard deviation of the concentration calculated for the seven replicate spikes. For dichloropropene, the MDL is 2.0 ng/ml for each isomer. EQL, defined as $5 * MDL$, is 10 ng/ml based on a 3 ml extraction volume. For methyl isothiocyanate, the MDL is 0.04 $\mu\text{g/ml}$ with an EQL of 0.22 $\mu\text{g/ml}$. Results above the EQL are reported to 3 significant figures. Results below EQL but above the MDL are reported as DET (detected) and results less than the MDL are ND (nondetect).

E. Collection and Extraction Efficiency (Recovery)

The target compounds at a low and high level are spiked on charcoal tubes (3 at each concentration). The spiked tubes are placed on field samplers with airflows of 3 LPM for 24 hours. The samples are extracted with DCM and prepared as described in section 5, #6-7. The average percent recovery should be $\pm 20\%$ of the expected value. Normal recoveries for DCP were found to be greater than 90%. Normal recoveries for MITC are greater than 85%.

F. Storage Stability

Storage stability studies were completed in the previous analysis and not continued further here. All analyses are to be completed within 4 days of receipt.

G. Breakthrough

No breakthrough analysis was done for DCP. The breakthrough was checked for MITC since the field sampling flow rate was set to 3 LPM. The recovery of charcoal tubes spiked at 5.0 $\mu\text{g/ml}$ was greater than 85% with no MITC detected in the secondary beds.

H. Safety

This procedure does not address all of the safety concerns associated with chemical analysis. It is the responsibility of the analyst to establish appropriate safety and health practices. For hazard information and guidance refer to the material safety data sheets (MSDS) of any chemicals used in this procedure.

Attachment 2

Standard Operating Procedure
Sampling and Analysis of Methyl Isocyanate
in Application and Ambient Air Using
High Performance Liquid Chromatography With a Fluorescence Detector

California Environmental Protection Agency



**Standard Operating Procedure
Sampling and Analysis of Methyl Isocyanate in
Application and Ambient Air using High Performance
Liquid Chromatography with a Fluorescence Detector**

**Special Analysis Section
Northern Laboratory Branch
Monitoring and Laboratory Division**

06/25/01 version

Approved by:

1. SCOPE

The analysis of methyl isocyanate (MIC), a degradation product of the soil fumigant metam-sodium, is based on OSHA Method 54 using a high-performance liquid chromatograph with a fluorescence detector. This method analyzes application and ambient air samples for MIC using XAD-7 resin tubes coated with 1-(2-pyridyl) piperazine, a derivatizing agent. The Department of Pesticide Regulation (DPR) asked the Air Resources Board (ARB) to do ambient monitoring of MIC at a requested quantitation limit of $0.05 \mu\text{g}/\text{m}^3$ and application monitoring at a quantitation limit of $0.1 \mu\text{g}/\text{m}^3$.

2. SUMMARY OF METHOD

Resin tubes, XAD-7 coated with 1-(2-pyridyl)piperazine, are placed on the sampler for 24 hours at a flowrate of 75 milliliters per minute (mLPM). The samples are stored in an ice chest or refrigerator until extracted with 3 ml of acetonitrile (ACN). The injection volume is 0.01 mL. A high performance liquid chromatograph (HPLC) with a fluorescence detector is used for the analysis.

3. INTERFERENCES/LIMITATIONS

Interferences may be caused by contaminants in solvents, reagents, glassware and other processing apparatus that can lead to discrete artifacts or elevated baselines. For this method the derivatizing agent, 1-(2-pyridyl)piperazine, is an additional factor in possible interferences. A method blank, including both solvent and resin, must be analyzed with each batch of samples to detect any possible interferences.

4. EQUIPMENT AND CONDITIONS

A. Instrumentation:

Dionex LC20 Chromatography Module
Dionex GP50 Gradient Pump
Dionex AS40 Autosampler
Dionex RF-2000 Fluorescence Detector: 240 nm excitation, 370 nm emission.
Sensitivity: medium; Gain: 1

Eluant: Acetonitrile (ACN) and 25 mM Ammonium Acetate ($\text{NH}_4 \text{AC}$), pH 6.1.
Gradient: 5% ACN/95% $\text{NH}_4 \text{AC}$ to 30%ACN/70% $\text{NH}_4 \text{AC}$ in 20 minutes.
Flowrate: 1.0 mL/min.

Column: Restek Ultra PFP, 4.6 mm i.d. x 250 mm, 5 μm .

B. Auxiliary Apparatus

1. Precleaned vials, 8 ml capacity with teflon caps.
2. Whatman filters, 0.45 μm
3. Disposable syringes, 3 ml
4. Sonicator
5. Dionex Polyvials with filter caps, 0.5 mL.

C. Reagents

1. Acetonitrile, HPLC/Pesticide grade or better.
2. Ammonium Acetate, 99.99%.
3. Glacial Acetic Acid, HPLC Grade or better.
4. Nanopure Water, Type I
5. 1-(2-Pyridyl)piperazine, 99.5+% or better.
6. Methyl Isocyanate, Chem Service #O-2179, 99+%.
7. XAD-7 resin sorbent tubes, coated with 1-(2-pyridyl)piperazine. Supelco ORBO 657, 80/40 mg, Bellefonte, PA.

5. ANALYSIS OF SAMPLES

1. The instrument is equilibrated for approximately one (1) hour before analysis of samples. Check that the volume in the eluant reservoirs is sufficient for the sample batch.
2. It is necessary to analyze a solvent blank and a resin blank with each batch of samples to ascertain the presence of possible interferences.
3. A 6-point calibration curve is analyzed with each batch of samples. For the ambient and application studies the calibration will be 0.013 to 0.260 $\mu\text{g/mL}$ of the purified MIC derivative. (See section 6.0 B for the preparation of the purified derivative.)
4. A calibration check sample of 0.078 $\mu\text{g/ml}$ is run after the calibration and every 10 samples and at the end of the sample batch. The value of the calibration check must be within $\pm 3\sigma$ (the standard deviation) or $\pm 10\%$ of the expected value, whichever is greater. If the calibration check is outside this limit then those samples in the batch after the last calibration check that was within limits need to be reanalyzed.
5. With each batch of samples analyzed, a laboratory resin blank and a laboratory control spike will be run concurrently. A laboratory blank is XAD-7 extracted and analyzed the same way as the samples. A laboratory control

spike is XAD-7 spiked with a known amount of MIC. The laboratory control sample is extracted and analyzed the same way as the samples.

6. Score and snap the sample resin tube, transfer the resin into an 8 ml vial. (Save the second tube for future analysis if necessary.) Rinse the tube with 3.0 ml of ACN into the extraction vial. Cap and place the vial in the sonicator for 1 hour.
7. Filter the samples using 0.45 μm filter attached to a 3 ml syringe directly into a Dionex sampling vial and cap securely. Cap and refrigerate the remaining solution vial if necessary for further analysis.
8. The atmospheric concentration is calculated according to:

$$\text{Conc } (\mu\text{g}/\text{m}^3) = \text{Extract Conc } (\mu\text{g}/\text{ml}) \times 3 \text{ ml} / \text{Air Volume Sampled } (\text{m}^3)$$

6. QUALITY ASSURANCE

A. Instrument Reproducibility

The reproducibility of the instrument has been established by analyzing five (5) injections of MIC-derivative standard at three concentrations (low, mid, and high). The low, mid, and high concentrations were 0.013, 0.078 and 0.260 $\mu\text{g}/\text{ml}$, respectively.

B. Purified Derivative and Calibration

1. The purified MIC derivative is prepared as described in OSHA Method 54, section 3.3.1. A stock standard is prepared by dissolving the MIC derivative into ACN. The derivative is expressed as free MIC by multiplying the amount of MIC urea weighed by the conversion factor 0.2590. (See OSHA Method 54, section 3.3.2)
2. A six (6)-point calibration curve is made at 0.013, 0.026, 0.052, 0.078, 0.134, and 0.260 $\mu\text{g}/\text{ml}$ of the MIC derivative.

C. Calibration Check

A calibration check sample is run after the calibration, after every 10 samples and at the end of the sample batch to verify the system is in calibration. The value of the check must be within $\pm 3\sigma$ (the standard deviation) or $\pm 10\%$ of the expected value, whichever is larger. If the calibration check is outside the limit,

then those samples in the batch after the last calibration check that was within the limit need to be reanalyzed.

D. Minimum Detection Limit

The detection limit is based on US EPA MDL calculation. The method detection limit (MDL) and the estimated quantitation limit (EQL) for methyl isocyanate is calculated by the analysis of seven (7) replicates of a low-level matrix spike. The MDL = 3.14*(std dev values), where std dev = the standard deviation of the concentration calculated for the seven replicate spikes. For MIC the MDL is 0.009 µg/sample (0.003 µg/mL). EQL, defined as 5*MDL, is 0.045 µg/sample (0.015 µg/mL) based on a 3 ml extraction volume. Results above the EQL are reported to 3 significant figures. Results below EQL but above the MDL are reported as DET (detected) and results less than the MDL are reported as ND (nondetect).

E. Collection and Extraction Efficiency (Recovery)

Methyl isocyanate at a low and high level are spiked on XAD-7 tubes. The spiked tubes are placed on field samplers with airflows of 75 mLpm for 24 hours. The samples are extracted with ACN and prepared as described in section 5, #6-7. The recovery of MIC for this method is low, ranging 50% to 70%. At concentrations above 1.0 µg/mL the recovery is greater than 70%.

F. Storage Stability

Storage stability will be run concurrent with analysis of samples.

G. Breakthrough

A low sample flow rate is required for this method and optimization of the bed weights with the derivatizing agent is necessary to capture the MIC and minimize interference.

H. Safety

This procedure does not address all of the safety concerns associated with chemical analysis. It is the responsibility of the analyst to establish appropriate safety and health practices. For hazard information and guidance refer to the material safety data sheets (MSDS) of any chemicals used in this procedure.

Attachment 3

Standard Operating Procedure Sampling and Analysis of Carbon Disulfide In Silco™ Canisters

California Environmental Protection Agency

 **Air Resources Board**

**Special Analysis Section
Northern Laboratory Branch
Monitoring and Laboratory Division**

**Standard Operating Procedure
Sampling and Analysis of Carbon Disulfide
In Silco™ Canisters Using a Varian
Stand Alone Cryogenic Sampler**

**Version 1
March 13, 2002**

Approved by:

**Russell Grace, Manager
Special Analysis Section**

This SOP has been reviewed by staff of the California 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 of commercial products constitute endorsement or recommendation for use.

SCOPE

This method, which follows closely EPA Method TO-15, is for the sampling and analysis of ambient air using six-liter Silco™ canisters for sample collection. The compound, carbon disulfide (CS₂) is analyzed by gas chromatography/mass spectrometry.

2. SUMMARY OF METHOD

Ambient air is collected into evacuated six-liter Silco™ canisters using a sub-atmospheric pressure collection mode. Sample canisters are subsequently pressurized in the laboratory to approximately fifteen pounds per square inch gauge (psig) and analyzed by Gas Chromatography / Mass Spectrometry (GC/MS) using a cryogenic concentrator to prepare the air sample. Samples are analyzed in the Selected Ion Monitoring (SIM) mode using bromomethane-d₃ as the primary internal standard for quantitation, and 1,2-dichloropropane-d₆ as a secondary internal standard for quantitation. 1,2-dichloropropane-d₆ is used only if interference makes bromomethane-d₃ unusable. Estimated quantitation levels for this method range from 9.6 to 62.4 µg/m³.

3. INTERFERENCES/LIMITATIONS

Interference may result from improperly cleaned canisters. Analysis of samples containing high concentrations of early eluting pesticide components may cause significant contamination of the analytical equipment. Co-eluting compounds trapped during sample collection may interfere. Running canister blanks and system blanks should minimize contamination originating in the analytical instrument.

4. EQUIPMENT AND CONDITIONS

A. Instrumentation

Hewlett Packard 5890 Series II gas chromatograph:

Detector: 280° C

Injector: 220° C

Column: Restek Rtx-200, 60 meter, 0.32mm I.D., 1.5 micron film thickness

GC temperature program: initial -10° C, -10 to 80° C @ 10° C/min, 80° to 200° C @ 25° C/min, hold for 1 minute, 200 to 240° C @ 25° C/min

Carrier Gas: Helium, grade 5 or better

Hewlett Packard 5973 mass selective detector:

Acquisition Mode: SIM

Tune File: PFTBA Autotune, maximum sensitivity

Ions monitored: 76 m/z quant, 78 m/z qualifier

Varian Stand Alone cryogenic concentrator:

Valve Oven: 60° C

Autosampler Oven: 60° C

Nafion Dryer: ambient

Sample Line: 150° C

Cryotrap: -180° C to 150° C

Transfer Line: 150° C

Cryofocus: -180° C to 150° C

Sample Size: 100 ml

Internal Standard Loop: 1 ml

B. Auxiliary Apparatus

Compressed helium: Grade 5 or better

Compressed nitrogen: Grade 5 or better

Liquid nitrogen for cryogenic concentrator

Certified standard for carbon disulfide

Restek, 6.0 liter Silcosteel™ canisters with Silcosteel™ valve

Pressure gauge, -30 inches Mercury (Hg) to 30 pounds psig.

Canister cleaning system (Appendix 1)

5. ANALYSIS OF SAMPLES

- 1) Perform a PFTBA autotune and evaluate tune criteria. See Appendix 2. Place a copy of the autotune results in the autotune folder.
- 2) Check and record the pressure in the field sample canisters. Pressurize the field sample canisters to approximately 14.7 psig with Grade 5 or better nitrogen. Record the final pressure.
- 3) Prepare a sample sequence for the GC/MS. The sequence should include a continuing calibration check (CCV), and a blank, for every 10 field samples. A laboratory control sample (LCS) and a duplicate are run once per analytical batch not to exceed 20 samples. Load the sequence into the GC/MS in the remote start mode.
- 4) Prepare a sample sequence for the Varian. The sample sequence should be organized as follows: system blank, CCV, LCS, field samples, duplicate field sample, and CCV. If the CCV is not within $\pm 20\%$ of its assigned value the system must be recalibrated.
- 5) Attach the sample canisters to the Varian autosampler ring as per the sequence. Execute the Varian sequence, which in turn will initiate the GC sequence.
- 6) Sample quantitation reports will print out after each analysis.
- 7) Review and edit the quantitation reports as needed.

8) Calculations will require a correction for the required pressurization performed prior to analysis. Instrument reports will be in units of $\mu\text{g}/\text{m}^3$ and must be corrected for the analysis dilution using the following calculation:

$$(F_p / I_p) \times C_i = C_r$$

I_p = initial canister pressure in mm Hg

F_p = final canister pressure in mm Hg

C_i = concentration from the analysis report in $\mu\text{g}/\text{m}^3$

C_r = reported concentration in $\mu\text{g}/\text{m}^3$

6. QUALITY ASSURANCE

A. Instrument Reproducibility

Establish the reproducibility of the instrument and analytical method as follows. Inject five replicate samples of CS_2 standards at three concentrations ($0.9 \mu\text{g}/\text{m}^3$, $2.55 \mu\text{g}/\text{m}^3$, and $9.49 \mu\text{g}/\text{m}^3$). See Table 1.

B. Linearity

A 5-point calibration is performed. Calibration standards ranging from approximately 1.6 to $10.4 \mu\text{g}/\text{m}^3$ are used to calibrate the method. If ambient samples are diluted times six, the calibrated ambient sample range is approximately 9.6 to $62.4 \mu\text{g}/\text{m}^3$. The results are used to calculate calibration curves using linear or quadratic regression. See Appendix 3

C. Minimum Detection Limit

Detection limits are based on the US EPA MDL calculation. Using the analysis of seven replicates of a low-level analysis, the method detection limits (MDL) and the EQL for the pesticide components are calculated as follows:

$$\text{MDL} = 3.14*s$$

$$\text{EQL} = 5*\text{MDL}$$

Where s equals the standard deviation of the response calculated for the seven replicate spikes. The calculated MDL for CS_2 is $0.23 \mu\text{g}/\text{m}^3$. The respective EQL is $1.15 \mu\text{g}/\text{m}^3$. Assuming a 1 to 6 dilution to pressurize the ambient samples the EQL is $6.9 \mu\text{g}/\text{m}^3$ for CS_2 .

Results are reported to 3 significant figures above the EQL. Results below EQL but above MDL are reported as DET (detected). Results reported as non-detect (ND) and reported values less than the MDL are reported as less than MDL (<MDL).

D. Storage Stability

A CS₂ storage stability study will not be performed. Carbon Disulfide is included in EPA Method T0-15 and as stated in section 1.3 "under conditions of normal usage for sampling ambient air, most VOC's can be recovered from canisters near their original concentrations after storage of up to 30 days." During this program all samples will be analyzed within 21 days of collection.

E: Safety Precautions

This procedure does not address all of the safety concerns associated with chemical analysis. It is the responsibility of the analyst to establish appropriate safety and health practices. For hazard information and guidance refer to the material safety data sheets (MSDS) of any chemicals used in this procedure. All applicable safety precautions must be observed for the use of compressed gas cylinders.

TABLE 1
 REPRODUCIBILITY STUDY
 CARBON DISULFIDE

Target Concentration	$0.92 \mu\text{g}/\text{m}^3$	$2.55 \mu\text{g}/\text{m}^3$	$9.49 \mu\text{g}/\text{m}^3$
Sample Number			
1	0.9	2.76	9.62
2	0.93	2.65	9.54
3	0.89	2.52	9.44
4	1.02	2.46	9.43
5	0.84	2.36	9.42
Average	0.92	2.55	9.49
SD	0.067	0.16	0.09
RSD	7.27	6.18	0.92

Appendices

Appendix 1

CAN CLEANING PROCEDURE

The canister cleaning procedure uses repeated cycling from -30 inches Hg to 30 pounds per square inch gauge with humidified ultra pure nitrogen. The procedure includes four complete cycles each 24 minutes (19 minutes vacuum and 5 minutes pressure) at 60 degrees C.

Canister data should be logged into the canister-cleaning book for each cleaning batch. When the batch is complete one canister is chosen for analysis. The canister is pressurized with ultra pure nitrogen and analyzed by the GCMS method. If target analytes are not less than their MDL the entire batch should be cleaned again.

Procedure:

A. Fill dewar with Liquid Nitrogen (LN2)

1. Remove dewar cover.
2. CAREFULLY place hose from LN2 tank into dewar (orange and silver container behind oven).
3. Open LN2 tank 3 turns
4. Close tank when LN2 can be seen near top of dewar.
5. CAREFULLY remove hose and replace dewar cover.

B. Turn on the vacuum pump.

1. Switch is located on pump to the left of the canister oven.

C. Open Nitrogen (N₂) Tank

1. Open regulator on N₂ tank to the left of the canister oven.

D. Load canisters in oven

1. Attach cans to manifold in oven and tighten.
2. If you are cleaning less than 8 canisters the unused ports must be capped.
3. Open the canister valves

E. Start Timers Located on top left of canister oven

1. Push auto button on top timer and auto light should come on. If the light is off, hit the button again and it should light.

2. Push the run button on the bottom timer. The 1 light should light up briefly then switch to 2.
3. The system should begin to evacuate.
4. Verify the system evacuates all the way by reading the gauge on the back of the oven. The gauge should go to -30 psi.

F. Fill cans and shutdown system.

1. Close all canister valves except the ones you want to fill.
2. On the top timer hit the ADV button until the 2 light comes on.
3. Monitor the pressure of the canisters on the gauge on the back of the oven.
4. Close can valves when filled.
5. Close N₂ Regulator
6. Turn off Vacuum pump.
7. Remove canisters and place plugs on manifold ports.
8. Hit the stop button on both timers.

Appendix 2

Autotune Criteria

A maximum sensitivity autotune should be performed on the detector each day prior to sample analysis. The autotune report should be evaluated for the following:

1. Any unusual change in the EM voltage
2. Peak width for all tune masses should be between 0.4 amu and 0.6 amu.
3. The relative abundance of tune mass 219.0 should be greater than 25% of tune mass 69.0.
4. Isotope abundance ratio for tune mass 70.0 should be between 0.54% and 1.6 %; isotope abundance ratio for tune mass 220.0 should be between 3.2% and 5.4%.
5. Masses 28 and 18 should be evaluated to check for air leaks in the system.

If autotune criteria are not met the system should be evaluated for problems. After the system problems are corrected the detector should be autotuned prior to sample analysis. Autotune reports should be filed in the instrument autotune folder.

Appendix 3

Calibration Standard Preparation for Carbon Disulfide

The certified stock gas used for calibration during this study was purchased from Scott Marrin and has the following specifications:

Cylinder No:	CC72058
Expiration date	May 9, 2002
Carbon Disulfide	0.985 PPM/M

Working analysis standard is prepared by diluting the stock gas using the following procedure.

1. A six liter Silco canister is evacuated to -30 " Hg.
2. 60 ml of carbon disulfide stock is transferred to the canister using a gas tight syringe equipped with a stopcock.
3. 100 ul of reagent grade water is added to the canister using a syringe and syringe adapter.
4. The canister is pressurized to approximately 29.4 psig with grade five or better nitrogen.

The canister will contain CS₂ at the following concentration:

Carbon Disulfide	10.39 $\mu\text{g}/\text{m}^3$
------------------	--------------------------------

The standard sample injection volume is 100 ml. Using the cryogenic sampler to introduce the following volumes of working standard to the GCMS generates a calibration curve.

<u>Volume</u>	<u>Carbon disulfide</u>
100 ml	10.39 $\mu\text{g}/\text{m}^3$
75 ml	7.79 $\mu\text{g}/\text{m}^3$
50 ml	5.20 $\mu\text{g}/\text{m}^3$
25 ml	2.60 $\mu\text{g}/\text{m}^3$
15 ml	1.56 $\mu\text{g}/\text{m}^3$

Attachment 4

Pesticide Canister Sampling Procedures
For Application Studies

Pesticide Canister Sampling Procedures For Application Studies

Overview:

- Collect samples, according to the schedule in Table 1 of this protocol.
- Collect 4 background samples, one from each of the 4 sampling positions.
- Collocate 1 field spike with each of the 4 background samples.
- Collect a collocated sample from the downwind site for all sampling periods (except the background period).
- Submit 1 trip blank.
- With the trip blank, there should be a total of 35 samples collected during the study, plus 4 trip and 4 field spikes.
- The field log sheet is filled out as the sampling is conducted. All QA samples must be logged onto the log sheet.
- The chain of custody (COC) forms are filled out prior to sample transfer; the originals are transferred with the samples. Make and retain copies if desired (not necessary).

Sampling Procedure:

Materials that will be needed to conduct the sampling include:

- Clip board with log sheets
- pencils/pens
- 9/16 inch open end wrench
- allen wrench
- sample cans with data sheets
- 0 to 10 sccpm mass flow meter (MFM) with battery (make sure battery has full charge)

Figure out your route for sampling the sampling locations, and keep this the same throughout the study.

Sample Start:

On the way to the first site, plug the MFMs into the batteries. It takes the MFMs about 10 minutes to warm up before they can be used. Unplug when not in use to minimize drop in battery charge. Recharge the batteries regularly.

- a) check to make sure that the canister valve is closed,
- b) remove the ¼ inch brass cap from the inlet of the can,

- c) securely attach the canister to the passive sampler, tighten the ¼ inch swagelock fitting,
- d) open the canister valve,
- e) record the canister pressure; if the can vacuum is **less than -29 "Hg** (e.g., -25) then replace with a new can (and return the bad one with appropriate comments made on the data sheet). Sometimes the cans will read beyond the scale, e.g., -31 or -32 "Hg; this is OK. When in doubt, use the spare gauge to verify the vacuum reading.

Using the 0 to 10 sccpm MFM, measure the flow rate; it should be 3.0 sccpm. If the reading is **between 2.95 and 3.05**, then record the value on the data sheet. If it is outside of this range, then record the value and adjust the flow back to 3.0 sccpm using an allen wrench. If you have to adjust the flow, then note it on the log sheet.

Fill out the Data Sheet and field log sheet, including log #, start date, time, beginning vacuum reading, any comments, samplers initials, and the general weather conditions (e.g., sunny, cloudy, raining, etc.).

Sample collection and Shipment:

Measure (do not re-set) the flow rates at the end of the sampling period with the MFM; record the end data on the log sheet and data sheet. **Close the can valve! (Do not use excessive force when closing the valve. When the knob stops turning, the valve is closed.)** Detach the can from the sampler and put a ¼ inch brass swagelock cap on the can inlet **and tighten**. Put the can back into a shipping container.

Log-in a trip blank (TB), once per study. Log the TB into the log sheet.

Attachment 5

Pesticide Adsorbent Tube Sampling Procedures
For Application Studies

Pesticide Adsorbent Tube Sampling Procedures For Application Studies

Overview:

- Collect samples, according to the schedule in Table 1 of this protocol.
- Collect 4 background samples, one each from the corner sampling positions.
- Collocate 1 field spike with each of the 4 background samples.
- Collect a collocated sample from the downwind site for all sampling periods (except the background period).
- Submit 1 trip blank.
- With the trip blank, there should be a total of 59 samples collected during the study (for each method), plus 4 trip and 4 field spikes.
- All samples are stored after sampling either in an ice-chest on dry ice or in a freezer.
- The field log sheet is filled out as the sampling is conducted. All QA samples must be logged onto the log sheet.
- The chain of custody (COC) forms are filled out prior to sample transfer; the originals are transferred with the samples. Make and retain copies if desired (not necessary).

Sampling Procedure:

Materials that will be needed to conduct the sampling include:

- Clip board with log sheets
- pencils/pens
- sample labels
- sample cartridges
 - end caps
 - plastic test tubes
- zip-lock bags
- 0 to 100 sccpm and 0 to 5 sLpm mass flow meters (MFM) with batteries
- ice chest
- dry ice

Figure out the route for sampling the 8 locations and keep this the same throughout the study.

Preparation and Set-up

On the way to study site, plug the MFM into the battery. It takes the MFMs about

10 minutes to warm up before they can be used. Leave the MFM plugged in until the last sample is taken; unplug when not in use to minimize drop in battery charge. Recharge the batteries regularly.

Securely attach one adsorbent sample cartridge to the sampling tree. **MAKE SURE THE ARROW ON THE CARTRIDGE IS POINTING TOWARDS THE SAMPLE LINE.**

Set the rotameter roughly to the correct setting. Perform the leak check on each sample line by placing a plastic tube cap over the inlet of the cartridge (with the pump on). The rotameter ball should fall to zero. The leak check should be performed before setting the flows with the MFMs.

Using the appropriate MFM, set the flow rate exactly to 75 sccpm for MIC or 2.5 sLpm for MITC.

Make sure that the rain/sun cover is pulled down over the sample tube.

Fill out the log sheet, including log #, start date, time, start counter reading, leak check OK, MFM Serial #, any comments, and the weather conditions.

Sample collection and Shipment

Measure (do not re-set) the flow rates at the end of the sampling period with the MFM; leak check the sample lines. Record the end data on the log sheet.

Remove the sample cartridge and cap the ends. Attach the sample label like a flag on the secondary end of the tube. Make sure that the label does not cover the glass wool separating the primary and secondary beds in the cartridge.

Place the cartridge in the plastic test tube shipping container.

Place all the samples for each period in a zip-lock freezer storage bag and place on dry ice in a cooler.

Collect a trip blank (TB) by breaking the ends off of a tube, capping and labeling as usual, and storing along with the rest of the samples. Log the TB into the log sheet.

Attachment 6

Adsorbent Tube Field Log Sheet
Canister Field Log Sheet and
Canister Field Data Sheet

**CALIFORNIA AIR RESOURCES BOARD
Carbon Di-Sulfide (CS2) Canister Data Sheet
San Luis Obispo County, Project # P-02-001**

Sample Name: _____

Sample Log #: _____

Operator: _____

Canister I.D. #: _____

Sampler I.D. #: _____

Lab I.D. :

	Date	Time (PST)	Vacuum ("Hg)	MFM Reading
Lab-pre*				
Sample Start				
Sample Stop				
Lab-post*				

*Calibrated Gauge Pressure

SAMPLE TYPE: Regular Colocated Spike Blank Other:

SHIPPED TO LABORATORY: Date _____ Time _____ Via _____

SAMPLING CONDITIONS:

- No unusual conditions
- Construction nearby
- Farm operations nearby
- Fire nearby
- Rain
- Wind-blown sand/dust
- Other : _____

FLAGGED SAMPLE? NO or YES

Reason for sample flag (Flow must be between 2.2 & 3.7ccm)

- Canister flow < 2.3ccm
- Canister flow > 3.7ccm
- Sampling equipment inoperative
- Other reasons: _____

FIELD COMMENTS:

FOR LABORATORY USE

Shipped to field by:	Date:	Received in lab by:	Date:	Time:
Custody Seal Intact: Yes _____ No _____ (If No: comment)		LAB COMMENTS:		

APPENDIX II

LABORATORY REPORT FOR MITC AND MIC

California Environmental Protection Agency

 **Air Resources Board**

**Air Sampling Cartridge Analytical Results for Methyl Isothiocyanate (MITC)
and Methyl Isocyanate (MIC) after Application of Metam-Sodium.**

**DATE: May 3, 2004
Revision 1**

Prepared by:

**T.E. Houston, Ph.D.
Special Analysis Section
Northern Laboratory Branch
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Reviewed and Approved by

**Russell Grace, Manager
Special Analysis Section**

Project Number: P-02-001

This report has been reviewed by staff of the California 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 of commercial products constitute endorsement or recommendation for use.

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1.0 INTRODUCTION

The Department of Pesticide Regulation (DPR) requested the Air Resources Board (ARB) to conduct application air monitoring for methyl isothiocyanate (MITC) and methyl isocyanate (MIC), the primary breakdown products of metam-sodium. This report covers the standard operating procedure, analytical, and quality assurance results for the analysis of MITC and MIC following a drip irrigation application of metam-sodium. DPR's target estimated quantitation limits (EQL) for the application are: MITC, 0.5 micrograms per cubic meter ($\mu\text{g}/\text{m}^3$) and MIC, 0.1 $\mu\text{g}/\text{m}^3$.

2.0 STANDARD OPERATING PROCEDURE.

2.1 Overview

The analysis of MITC uses a gas chromatograph/mass selective detector (GC/MSD) operating in the selected ion monitoring (SIM) mode. MITC is collected on coconut charcoal cartridges at a flow rate of 2.5 liters per minute (LPM). The cartridges are extracted with three milliliters (mL) of 0.1% carbon disulfide (CS_2) in ethyl acetate (EA). Methyl isocyanate (MIC) analysis is by high performance liquid chromatography (HPLC) using a fluorescence detector. XAD-7 cartridges coated with a derivatizing agent, 1-(2-pyridyl)piperazine, are placed on a sampler at a flow rate of 0.075 LPM. The XAD-7 cartridges are extracted with 3 mL of acetonitrile (ACN) and analyzed.

2.2 Instrument Reproducibility

Instrumental reproducibility on the GC/MS uses five (5) individual injections of 1 μL of MITC at three concentrations. The instrumental reproducibility for MIC on the HPLC-fluorescence detector uses five injections of 10 μL also at three concentrations. Table 1 shows the results and area responses for both compounds with the average, standard deviation, and relative standard deviation of the determined value.

2.3 Calibration

The calibration for the compounds is described in the respective standard operating procedures (Appendix A and B). A calibration is run before each analytical sample batch. All the calibration curves use a linear curve fit and have a correlation coefficient (r^2) of 0.995 or greater.

2.4. Minimum Detection Limit (MDL) and Estimated Quantitation Limits (EQL)

For the MITC application analysis the target EQL is 0.5 $\mu\text{g}/\text{m}^3$. The target EQL corresponds to 1.5 $\mu\text{g}/\text{sample}$ for a 3 mL extract. The low calibration standard is 0.50 $\mu\text{g}/\text{mL}$, which was selected to accurately quantify samples with concentrations at or above the target EQL. MDL for MITC was determined to be 0.034 $\mu\text{g}/\text{mL}$ and for MIC the MDL was determined to be 0.003 $\mu\text{g}/\text{mL}$.

For MITC application analysis, the method EQL is 0.17 µg/mL. This corresponds to 0.51 µg/sample, which is equivalent to 0.14 µg/m³. For MIC application analysis, the method EQL is 0.015 µg/mL (0.045 µg/sample for a 3 mL extract corresponding to 0.42 µg/m³). A low calibration standard of 0.013 µg/mL for MIC application analysis was selected to ensure accurate quantification at the EQL.

Staff report results above or equal to the low calibration standard to three (3) significant figures. Results below the low calibration standard but above or equal to the EQL are reported to one (1) significant figure. Results below the EQL but greater than or equal to the MDL, are reported as detected (DET). Results less than MDL are reported as <MDL.

2.5. *Collection and Extraction Efficiency (Recovery)*

The change of extraction solvent to 0.1% CS₂/EA for MITC significantly increased the recovery efficiency. The cartridges averaged 80.5% with a standard deviation of 2.5% based on analysis prior to the start of the application. No extraction efficiency was done on MIC for this study. Previous work (2001) during ambient analysis indicated recoveries of greater than 70%.

2.6. *Storage Stability*

No storage stability tests were completed. All samples were analyzed within 7 days of receipt in the lab.

2.7. *Breakthrough*

No breakthrough analysis was done for this study. For breakthrough information see the 2001 MITC/MIC ambient analysis reports.

3.0 APPLICATION AIR MONITORING SAMPLE RESULTS

The laboratory received a total of 66 charcoal cartridges and 66 XAD-7 cartridges for the analysis of MITC and MIC, respectively, including four (4) field spikes and four (4) trip spikes. Table 2 presents the results of the analysis for MITC. Table 3 presents the results for the analysis of MIC.

4.0 ANALYTICAL QUALITY CONTROL SAMPLES

4.1 *Laboratory solvent blanks*

Staff analyzes a laboratory solvent blank with each analytical sample batch. This is to ensure there are no reagent interferences in the analysis. All blanks were less than the MDL.

4.2 *Laboratory spiking solutions*

A spiking solution is analyzed with each analytical batch. Three (3) mLs of 0.1%CS₂/EA is spiked with 24.0 µg of MITC. The spiking solution for MIC is prepared by adding 3 mLs of ACN to the contents of an XAD-7 cartridge in a vial to solubilize the derivative and subsequently adding 0.6 µg of underivatized MIC. The results of the analysis of the spiking solution are in Table 4.

4.3 *Laboratory method blanks*

Each analytical batch includes a laboratory method blank. The method blank is a charcoal cartridge or XAD-7 cartridge prepared and analyzed as described in the respective SOP. Analysis did not detect any of the target compounds above the MDL in these blanks.

4.4 *Laboratory control samples*

Each analytical batch includes a laboratory control sample. These are charcoal or XAD-7 cartridges spiked with the respective target compounds. The control sample is prepared and analyzed as described in the method SOP's. The recovery is based on the spiking solution analyzed for the analytical sample batch. The results are reported in Table 5.

4.5 *Calibration check standards*

Following standard operating procedures, a calibration check standard is run after the initial calibration and every tenth (10) sample in an analytical batch. The calibration check standard must be within $\pm 20\%$ of the target value. If any of the check standards are outside this limit, the associated samples are re-analyzed. All calibration check standards were within range.

5.0 FIELD, TRIP, AND LABORATORY SPIKES

For the application analysis four (4) field spikes, four (4) trip spikes, four (4) laboratory spikes were analyzed during the application testing. The samples were stored in the freezer until analysis.

5.1 *Field spikes*

The field spike results for MITC are in Table 6. The field spike results for MIC are in Table 7. The field spikes are sampled at four (4) locations associated with the designated area for the application. An unspiked collocated sample run at the same time is subtracted from the field spike sample to determine the actual spike recovery values. All of these samples were run prior to the start of the application and constitute a background analysis for the compounds.

5.2 Trip spikes

Table 8 presents the results of the MITC trip spikes. Table 9 contains the MIC trip spike results. Trip spikes are spiked cartridges sent into the field but not placed on samplers.

5.3 Laboratory spikes

Table 10 and 11 present the results of the laboratory spikes for MITC and MIC, respectively. The laboratory spikes are spiked at the same time as the field and trip spikes. These are stored in the refrigerator and analyzed with the field and trip spikes. An additional set of laboratory spikes was run with the analytical sample batches for MITC. These laboratory spikes, just as the samples, were extracted with solvent prepared daily. The results of these spikes showed a greater recovery of the MITC.

6.0 DISCUSSION

No problems occurred during the analysis of the MITC or MIC application samples. The MITC recovery of the field, trip and laboratory spikes in the first analytical sample batch (date analyzed 5/14/02) was low at 55-58%. Prior to the application study, using the mixed solvent of 0.1% CS₂/EA indicated a recovery for spiked field samples at greater than 80%. After the analysis of the first batch, staff looked at recoveries for several samples extracted with the initial solvent, newly prepared solvent, and the previous extraction solvent, dichloromethane. The results indicated that the extraction solvent needed to be prepared fresh for the sample batches to insure adequate recovery. An additional set of laboratory spikes were run with the balance of the samples with a recovery of 80.5±3.0%. The average MITC result is 6.18 µg/mL (18.5 µg/sample), with the maximum of 20.22 µg/mL (60.7 µg/sample) for MIW#19.

The MIC analysis has interferences arising from the derivatizing agent. It is difficult with the current method to separate the MIC derivative baseline to baseline from the interference. The apparent high recoveries of the field, trip and laboratory spikes are a result of this interference. These spikes were prepared a month prior to the field study. The recovery of the MIC does indicate that the derivatized compound is stable up to a month when kept in the freezer. None of the field samples indicated the presence of MIC.

Table 1. Instrument Reproducibility MITC and MIC

MITC			MIC		
Amount	µg/ml	Area Response	Amount	µg/ml	Area Response
0.5 µg/mL	0.5	54838	0.13 µg/mL	0.013	1.838
	0.5	54868		0.012	1.867
	0.5	54744		0.012	1.879
	0.5	54902		0.012	1.854
	0.5	54232		0.012	1.913
	Average	0.50		54717	Average
StandardDeviation	0.00	277	StandardDeviation	0.000	0.028
Relative Std Dev.	NA	0.51	Relative Std Dev.	NA	1.500
3.0 µg/mL	2.62	269124	0.078 µg/mL	0.075	11.912
	2.65	271419		0.075	11.827
	2.62	268944		0.075	11.882
	2.61	267646		0.076	11.967
	2.58	264512		0.075	11.866
	Average	2.62		268329	Average
StandardDeviation	0.03	2530	StandardDeviation	0.000	0.052
Relative Std Dev.	0.96	0.94	Relative Std Dev.	NA	0.440
10.0 µg/mL	9.84	998195	0.260 µg/mL	0.261	41.254
	9.80	993505		0.255	40.253
	9.78	991420		0.256	40.455
	9.75	988308		0.257	40.603
	9.79	992626		0.278	43.821
	Average	9.79		992811	Average
StandardDeviation	0.03	3596	StandardDeviation	0.010	1.471
Relative Std Dev.	0.33	0.36	Relative Std Dev.	3.831	3.56

%Relative Standard Deviation = standard deviation/average x 100

Table 2. Application Monitoring Results of MITC

Log Number	Site Identification	Date Analyzed	Amount $\mu\text{g/mL}$	Amount $\mu\text{g/sample}$
1	MI WBC FS1	5/14/2002	4.56	13.7
2	MI WB	5/14/2002	<MDL	<MDL
3	MI SBC FS2	5/14/2002	4.30	12.9
4	MI SB	5/14/2002	<MDL	<MDL
5	MI EBC FS3	5/14/2002	4.43	13.3
6	MI EB	5/14/2002	<MDL	<MDL
7	MI NBC FS4	5/14/2002	4.37	13.1
8	MI NB	5/14/2002	<MDL	<MDL
9	MI W	5/14/2002	<MDL	<MDL
10	MI S	5/14/2002	3.39	10.2
11	MI EC	5/14/2002	13.0	39.0
12	MI E	5/14/2002	14.8	44.4
13	MI N	5/14/2002	19.8	59.3
14	MI NW	5/14/2002	<MDL	<MDL
15	MI NE	5/14/2002	19.5	58.4
16	MI SE	5/14/2002	3.30	9.90
17	MI SW	5/14/2002	<MDL	<MDL
18	MI NW	5/16/2002	3.52	10.6
19	MI W	5/16/2002	20.2	60.7
20	MI SW	5/16/2002	8.91	26.7
21	MI S	5/16/2002	14.4	43.1
22	MI SE	5/16/2002	5.87	17.6
23	MI E	5/16/2002	6.38	19.1
24	MI EC	5/16/2002	6.06	18.2
25	MI NE	5/16/2002	5.88	17.6
26	MI N	5/16/2002	4.90	14.7
27	MI NW	5/16/2002	<MDL	<MDL
28	MI W	5/16/2002	0.480	1.44
29	MI SW	5/16/2002	<MDL	<MDL
30	MI S	5/16/2002	0.4*	1.2*
31	MI SE	5/16/2002	0.4*	1.1*
32	MI E	5/16/2002	5.43	16.3
33	MI EC	5/16/2002	4.74	14.2
34	MI NE	5/16/2002	7.24	21.7
35	MI N	5/16/2002	8.52	25.6
36	MI NW	5/17/2002	0.500	1.50
37	MI W	5/17/2002	7.83	23.5
38	MI SW	5/17/2002	7.35	22.0
39	MI S	5/17/2002	7.05	21.2

Table 2. Application Monitoring Results of MITC

Log Number	Site Identification	Date Analyzed	Amount $\mu\text{g/mL}$	Amount $\mu\text{g/sample}$
40	MI SE	5/17/2002	0.520	1.56
41	MI E	5/17/2002	1.53	4.59
42	MI EC	5/17/2002	1.65	4.95
43	MI NE	5/17/2002	1.56	4.68
44	MI N	5/17/2002	1.95	5.85
45	MI NW	5/17/2002	<MDL	<MDL
46	MI W	5/17/2002	<MDL	<MDL
47	MI SW	5/17/2002	<MDL	<MDL
48	MI S	5/17/2002	0.620	1.86
49	MI SE	5/17/2002	0.640	1.92
50	MI E	5/17/2002	3.30	9.90
51	MI EC	5/17/2002	2.84	8.52
52	MI NE	5/17/2002	4.13	12.4
53	MI N	5/17/2002	3.70	11.1
54	MI NW	5/20/2002	<MDL	<MDL
55	MI W	5/20/2002	<MDL	<MDL
56	MI SW	5/20/2002	3.53	10.6
57	MI S	5/20/2002	3.73	11.2
58	MI SE	5/20/2002	DET	DET
59	MI E	5/20/2002	0.4*	1.1*
60	MI EC	5/20/2002	0.3*	0.9*
61	MI NE	5/20/2002	0.4*	1.1*
62	MI N	5/20/2002	0.2*	0.7*
63	Trip Spike #1	5/14/2002	4.23	12.7
64	Trip Spike #2	5/14/2002	4.33	13.0
65	Trip Spike #3	5/14/2002	4.32	13.0
66	Trip Spike #4	5/14/2002	4.48	13.4

MDL = 0.034 $\mu\text{g/mL}$

EQL = 0.17 $\mu\text{g/mL}$

Low calibration standard = 0.50 $\mu\text{g/mL}$

FS: field spikes

If the analytical result is \geq MDL and $<$ EQL it is reported in the table as detected (DET). Levels \geq EQL but $<$ low cal. std. are reported to one significant figure. Levels \geq low cal. std. are reported as the actual measured value and are reported to three significant figures.

* Assumes the response curve is linear below the low calibration standard of 0.50 $\mu\text{g/mL}$.

Table 3. Application Monitoring Results of MIC

Log Number	Site Identification	Date Analyzed	Amount $\mu\text{g}/\text{mL}$	Amount $\mu\text{g}/\text{sample}$
1	MS WBC FS	5/13/2002	0.185	0.555
2	MS WB	5/13/2002	<MDL	<MDL
3	MS SBC FS	5/13/2002	ND	ND
4	MS SB	5/13/2002	<MDL	<MDL
5	MS EBC FS	5/13/2002	0.240	0.720
6	MS EB	5/13/2002	<MDL	<MDL
7	MS NBC FS	5/13/2002	0.219	0.657
8	MS NB	5/13/2002	<MDL	<MDL
9	MS W	5/14/2002	<MDL	<MDL
10	MS S	5/14/2002	<MDL	<MDL
11	MS EC	5/14/2002	<MDL	<MDL
12	MS E	5/14/2002	<MDL	<MDL
13	MS N	5/14/2002	<MDL	<MDL
14	MS NW	5/14/2002	<MDL	<MDL
15	MS NE	5/14/2002	<MDL	<MDL
16	MS SE	5/14/2002	<MDL	<MDL
17	MS SW	5/14/2002	<MDL	<MDL
18	MS NW	5/14/2002	<MDL	<MDL
19	MS W	5/14/2002	<MDL	<MDL
20	MS SW	5/14/2002	<MDL	<MDL
21	MS S	5/14/2002	<MDL	<MDL
22	MS SE	5/14/2002	<MDL	<MDL
23	MS E	5/14/2002	<MDL	<MDL
24	MS EC	5/14/2002	<MDL	<MDL
25	MS NE	5/14/2002	<MDL	<MDL
26	MS N	5/14/2002	<MDL	<MDL
27	MS NW	5/15/2002	<MDL	<MDL
28	MS W	5/15/2002	<MDL	<MDL
29	MS SW	5/15/2002	<MDL	<MDL
30	MS S	5/15/2002	<MDL	<MDL
31	MS SE	5/15/2002	<MDL	<MDL
32	MS E	5/15/2002	<MDL	<MDL
33	MS EC	5/15/2002	<MDL	<MDL
34	MS NE	5/15/2002	<MDL	<MDL
35	MS N	5/15/2002	<MDL	<MDL
36	MS NW	5/15/2002	<MDL	<MDL
37	MS W	5/15/2002	<MDL	<MDL
38	MS SW	5/15/2002	<MDL	<MDL
39	MS S	5/15/2002	<MDL	<MDL

Table 3. Application Monitoring Results of MIC

Log Number	Site Identification	Date Analyzed	Amount $\mu\text{g/mL}$	Amount $\mu\text{g/sample}$
40	MS SE	5/15/2002	<MDL	<MDL
41	MS E	5/15/2002	<MDL	<MDL
42	MS EC	5/15/2002	<MDL	<MDL
43	MS NE	5/15/2002	<MDL	<MDL
44	MS N	5/15/2002	<MDL	<MDL
45	MS NW	5/16/2002	<MDL	<MDL
46	MS W	5/16/2002	<MDL	<MDL
47	MS SW	5/16/2002	<MDL	<MDL
48	MS S	5/16/2002	<MDL	<MDL
49	MS SE	5/16/2002	<MDL	<MDL
50	MS E	5/16/2002	<MDL	<MDL
51	MS EC	5/16/2002	<MDL	<MDL
52	MS NE	5/16/2002	<MDL	<MDL
53	MS N	5/16/2002	<MDL	<MDL
54	MS NW	5/16/2002	<MDL	<MDL
55	MS W	5/16/2002	<MDL	<MDL
56	MS SW	5/16/2002	<MDL	<MDL
57	MS S	5/16/2002	<MDL	<MDL
58	MS SE	5/16/2002	<MDL	<MDL
59	MS E	5/16/2002	<MDL	<MDL
60	MS EC	5/16/2002	<MDL	<MDL
61	MS NE	5/16/2002	<MDL	<MDL
62	MS N	5/16/2002	<MDL	<MDL
63	Trip Spike #1	5/13/2002	0.234	0.702
64	Trip Spike #2	5/13/2002	0.237	0.711
65	Trip Spike #3	5/13/2002	0.240	0.720
66	Trip Spike #4	5/13/2002	0.185	0.555

MDL = 0.003 $\mu\text{g/mL}$

EQL = 0.015 $\mu\text{g/mL}$

FS: field spikes

If the analytical result is \geq MDL and $<$ EQL it is reported in the table as detected (DET). Levels \geq EQL are reported as the actual measured value and are reported to three significant figures.

Table 4: Laboratory Spiking Solution Results

MITC Spikes: 8.0 µg/mL		MIC Spikes: 0.20 µg/mL	
Date: 5/14	Amount: 7.94	Date: 5/13	Amount: 0.236
5/16	8.13	5/14	0.140
5/17	8.07	5/15	0.112
5/20	8.02	5/16	0.083

Table 5: Laboratory Control Sample Results

Date	MITC Amount, µg/mL	MITC *** %Recovery	Date	MIC Amount µg/mL	MIC *** %Recovery
5/14	4.21**	53.02	5/13	NA	NA
5/16	6.49	79.83	5/14	0.121	86.43
5/17	6.55	81.16	5/15	0.095	84.82
5/20	6.66	83.06	5/16	0.075	90.36

** This sample batch was extracted with an older solution of the CS2/EA mix. All subsequent analyzes were with CS2/EA prepared daily.

***All recoveries are based on the spiking solution analyzed for any given day.

Table 6: Field Spike Results: MITC

Log Number	Sample Identification	Amount, $\mu\text{g/mL}$	%Recovery	Background Identification
1	MIWBCFS1	4.56**	57.43	MIWB#2
3	MISBCFS2	4.30	54.16	MISB#4
5	MIEBCFS3	4.43	55.79	MIEB#6
7	MINBCFS4	4.37	55.04	MINB#8
Average		4.42	55.60	
Standard Deviation		0.11	1.39	

Field spike results are corrected for the unspiked collocated sample.

** Extracts were made with solvent prepared prior to 5/14.

Table 7: Field Spike Results: MIC

Log Number	Sample Identification	Amount, $\mu\text{g/mL}$	%Recovery	Background Identification
1	MSWBCFS1	0.185	78.39	MSWB#2
3	MSSBCFS2	ND	ND	MSSB#4
5	MSEBCFS3	0.248	105.08	MSEB#6
7	MSNBCFS4	0.219	92.80	MSNB#8
Average		0.217	92.09	
Standard Deviation		0.032	13.36	

Field spike results are corrected for the unspiked collocated sample.

Table 8: Trip Spike Results: MITC

Sample Identification	Amount, $\mu\text{g/mL}$	%Recovery
Trip Spike #63	4.23**	53.27
Trip Spike #64	4.33	54.53
Trip Spike #65	4.32	54.41
Trip Spike #66	4.48	56.42
Average	4.34	54.66
Standard Deviation	0.1	1.30

**Extract made with solvent prepared prior to 5/14.

Table 9: Trip Spike Results: MIC

Sample Identification	Amount, $\mu\text{g/mL}$	%Recovery
Trip Spike #63	0.234	99.15
Trip Spike #64	0.237	100.42
Trip Spike #65	0.240	101.69
Trip Spike #66	0.185	78.39
Average	0.224	94.92
Standard Deviation	0.026	11.07

Table 10: Laboratory Spike Results: MITC

Sample Identification	Amount, µg/mL	%Recovery
Laboratory Spike #1	4.77**	60.08
Laboratory Spike #2	4.74	59.70
Laboratory Spike #3	4.65	58.56
Laboratory Spike #4	4.42	55.67
Average	4.65	58.50
Standard Deviation	0.16	2.00
Laboratory Spike #5	6.36	78.81
Laboratory Spike #6	6.24	77.32
Laboratory Spike #7	6.55	81.67
Laboratory Spike #8	6.74	84.04
Average	6.42	80.46
Standard Deviation	0.22	2.99

**Extract made with solvent prepared prior to 5/14.

Table 11: Laboratory Spike Results: MIC

Sample Identification	Amount, µg/mL	%Recovery
Laboratory Spike #1	0.229	97.03
Laboratory Spike #2	0.243	102.97
Laboratory Spike #3	0.238	100.85
Laboratory Spike #4	0.235	99.58
Average	0.236	100.11
Standard Deviation	0.006	2.48

Appendix A:
Standard Operating Procedure for MITC Analysis

California Environmental Protection Agency



Air Resources Board

**Standard Operating Procedure
Sampling and Analysis of Methyl Isothiocyanate (MITC)
in Application Air Monitoring using Gas
Chromatography/Mass Selective Detector**

**Special Analysis Section
Northern Laboratory Branch
Monitoring and Laboratory Division**

05/03/04 version

Approved by:

**Russell Grace, Manager
Special Analysis Section**

1. SCOPE

The method uses resin tubes and a gas chromatograph/mass selective detector (GC/MSD) for the determination of methyl isothiocyanate (MITC), one of the breakdown products of Metam-Sodium, for application air sample analysis. The Department of Pesticide Regulation (DPR) asked the Air Resources Board (ARB) to do application monitoring of MITC at a target quantitation limit of 0.5 micrograms per cubic meter ($\mu\text{g}/\text{m}^3$).

2. SUMMARY OF METHOD

Coconut based charcoal tubes are placed on the sampler with the flow set at 2.5 liters per minute (LPM). The samples are stored in an ice chest or refrigerator until extracted with 3 milliliters (mL) of 0.1% carbon disulfide (CS_2) in ethyl acetate (EA). The injection volume is 1 microliter (μL). A gas chromatograph with a mass selective detector in the selected ion monitoring (SIM) mode is used for analysis.

3. INTERFERENCES/LIMITATIONS

In the previous standard operating procedure (ARB, 2001) for ambient and application analysis of both 1,3-dichloropropene and MITC the extraction solvent used, dichloromethane, did not have a good recovery rate for MITC. To optimize recovery of the MITC in the current application study the extraction solvent is 0.1% carbon disulfide (CS_2) in ethyl acetate (0.1% CS_2 /EA). As with any method, additional interferences may be caused by contaminants in solvents, reagents, glassware and other processing apparatus that can lead to discrete artifacts or elevated baselines. Method blanks, both solvent and resin, must be run concurrently with each batch of samples to detect any possible interferences.

4. EQUIPMENT AND CONDITIONS

A. INSTRUMENTATION:

Hewlett-Packard 6890 Series gas chromatograph
Hewlett-Packard 5973 Network mass selective detector
Hewlett-Packard 6890 Enhanced Parameters ALS

MS Transfer line: 280°C

Injector: 210 °C, Splitless, Liner 4 mm straight liner with glass wool.

Column: Restek Rtx-200, 60 meter, 320 μm i.d., 1.5 μm film thickness.

GC Temperature Program: Oven initial 40 °C, hold 4 min. Ramp to 220 °C @ 12 °C/min., hold 1 min., ramp to 240 °C @ 20 °C/min., hold 2.0 min. Retention time: MITC=12.23 min.

Splitter open @ 1.0 min.

Flows: Column: He, 1.6 mL/min, 9.1psi. (velocity: 32cm/sec)

Splitter: 50 mL/min.

Mass Spectrometer: Electron Ionization

Selective Ion Monitoring: methyl isothiocyanate: 73 (quant. ion 100%), 72 (qual. ion 46%). Tuning: PFTBA on masses 69, 219, 502.

B. Auxiliary Apparatus

1. Precleaned vials, 8 mL capacity with teflon caps.
2. Whatman filters, 0.45 μm
3. Disposable syringes, 3 mL
4. Sonicator
5. GC vials with septum caps.

C. Reagents

1. Ethyl Acetate, Pesticide grade or better.
2. Carbon Disulfide, 99.9+%, <100 ppb benzene, Aldrich, #42464-1.
3. Methyl Isothiocyanate, Chem Service MET-221A, 99.5%
4. Coconut charcoal sorbent tubes, SKC, Fullerton, CA #226-09.

5. ANALYSIS OF SAMPLES

1. A daily manual tune shall be performed using PFTBA. The instrument is tuned using masses: 69, 219, 502. The criterion for the tune are the peak widths at $\frac{1}{2}$ the peak height, 0.60 ± 0.05 , and the criteria for relative abundance: 69:100%, 219:100-120%, and 502: 7-12%.
2. A solvent blank is analyzed with each batch of samples. The blank must be free of interferences. A solvent blank must be analyzed after any sample which may result in possible carry-over contamination.
3. A six-point calibration curve shall be analyzed with each batch of samples. For methyl isothiocyanate the calibration is at 0.5, 1.0, 2.0, 3.0, 5.0, and 10.0 $\mu\text{g/mL}$.
4. With each batch of samples analyzed, a solvent spike solution, a laboratory resin blank and a laboratory resin control spike will be run concurrently. The solvent spike solution is the amount of standard added just to the designated volume of solvent extract. A laboratory blank is an unexposed charcoal tube prepared and analyzed the same way the samples are analyzed. A laboratory control spike is a charcoal tube spiked with a known amount of standard. The control sample is prepared and analyzed the same way as the

samples. Laboratory check samples should have recoveries that are at least 70% of the theoretical spiked value.

5. A calibration check for MITC of 7.0 µg/mL is run after the calibration and every ten samples and at the end of each sample batch. The value of the check must be within $\pm 3\sigma$ (the standard deviation) or $\pm 20\%$ of the expected value, whichever is greater. If the calibration check is outside the limit, then those samples in the batch after the last calibration check that was within the limit need to be reanalyzed.
6. Score and snap the sample tube, transfer the charcoal into an 8 mL vial. (Save the back-up bed for future analysis if necessary.) Rinse the tube with 3.0 mL of 0.1%CS₂/EA into the extraction vial. Cap and place the vial in the sonicator for 1 hour.
7. Filter the samples using a 3 mL syringe and 0.45 µm filter directly into a GC vial and cap securely.
8. The atmospheric concentration is calculated according to:

$$\text{Conc } (\mu\text{g}/\text{m}^3) = \text{Extract Conc } (\mu\text{g}/\text{ml}) \times 3 \text{ mL} / \text{Air Volume Sampled } (\text{m}^3)$$

6. QUALITY ASSURANCE

A. Instrument Reproducibility

The reproducibility of the instrument and analytical method was established by analyzing five(5) 1.0 µL injections of methyl isothiocyanate standard at three concentrations (low, mid, and high range). The low, mid and high concentrations were 0.5, 3.0 and 10.0 µg/mL.

B. Calibration

The six-point calibration curve is constructed using linear regression analysis. A curve cannot be used if its correlation coefficient is less than 0.995.

C. Calibration Check

A calibration check control is run after the calibration and every ten samples and at the end of the sample batch to verify the system is in calibration. The value of the check must be within $\pm 3\sigma$ (the standard deviation) or $\pm 20\%$ of the expected value, whichever is greater. If the calibration check is outside the limit, then those samples preceding the out of limit check need to be reanalyzed.

D. Minimum Detection Limit

Detection limits are based on US EPA MDL calculation. Using the analysis of seven (7) replicates of a low-level matrix spike, the method detection limit (MDL) and the estimated quantitation limit (EQL) for MITC is calculated by: $MDL = 3.14 * (\text{std dev values})$, where std dev = the standard deviation of the concentration calculated for the seven replicate spikes. For MITC, the MDL is 0.034 $\mu\text{g/mL}$ with an EQL of 0.17 $\mu\text{g/mL}$. The analytical results are reported to the low calibration standard (corresponding to DPR's target EQL of 0.50 $\mu\text{g/m}^3$) to three significant figures. Data equal to or above the EQL but below the low calibration standard are reported to one significant figure. Results below the EQL but equal to or above the MDL are reported as DET (detected) and results less than the MDL are ND (nondetect).

E. Collection and Extraction Efficiency (Recovery)

The MITC at a low and high level are spiked on charcoal tubes (4 at each concentration). The spiked tubes are placed on field samplers with airflows of 2.5 LPM for 24 hours. The samples are extracted with 0.1% CS_2/EA and prepared as described in section 5, #6-7. The average percent recovery should be $\pm 20\%$ of the expected value. Recoveries for MITC are between 80-85% of the expected value.

F. Storage Stability

No storage stability studies were made in this study.

G. Breakthrough

No breakthrough analysis was done for MITC in this study.

H. Safety

This procedure does not address all of the safety concerns associated with chemical analysis. It is the responsibility of the analyst to establish appropriate safety and health practices. For hazard information and guidance refer to the material safety data sheets (MSDS) of any chemicals used in this procedure.

Appendix B:
Standard Operating Procedure for MIC Analysis

California Environmental Protection Agency



**Standard Operating Procedure
Sampling and Analysis of Methyl Isocyanate in
Application Air using High Performance Liquid
Chromatography with a Fluorescence Detector**

**Special Analysis Section
Northern Laboratory Branch
Monitoring and Laboratory Division**

05/03/04 version

Approved by:

**Russell Grace, Manager
Special Analysis Section**

1. SCOPE

The analysis of methyl isocyanate (MIC), a degradation product of the soil fumigant metam-sodium, is based on OSHA Method 54 using a high-performance liquid chromatograph with a fluorescence detector. This method analyzes application air samples for MIC using XAD-7 resin tubes coated with 1-(2-pyridyl) piperazine, a derivatizing agent. The Department of Pesticide Regulation (DPR) asked the Air Resources Board (ARB) to do the monitoring of MIC at a target quantitation limit of 0.1 $\mu\text{g}/\text{m}^3$.

2. SUMMARY OF METHOD

Resin tubes, XAD-7 coated with 1-(2-pyridyl)piperazine, are placed on the sampler for 24 hours at a flowrate of 75 milliliters per minute (mLPM). The samples are stored in an ice chest or refrigerator until extracted with 3 ml of acetonitrile (ACN). The injection volume is 0.01 mL. A high performance liquid chromatograph (HPLC) with a fluorescence detector is used for the analysis.

3. INTERFERENCES/LIMITATIONS

Interferences may be caused by contaminants in solvents, reagents, glassware and other processing apparatus that can lead to discrete artifacts or elevated baselines. For this method the derivatizing agent, 1-(2-pyridyl)piperazine, is an additional factor in interferences. In the 2001 ambient air analysis of MIC, it was difficult to separate the derivatized MIC from a slightly later eluting peak attributed to the derivatizing agent. A method blank, including both solvent and resin, must be analyzed with each batch of samples to detect any possible interferences.

4. EQUIPMENT AND CONDITIONS

A. Instrumentation:

Dionex LC20 Chromatography Module
Dionex GP50 Gradient Pump
Dionex AS40 Autosampler
Dionex RF-2000 Fluorescence Detector: 240 nm excitation, 370 nm emission.
Sensitivity: medium; Gain: 1

Eluant: Acetonitrile (ACN) and 10 mM Ammonium Acetate (NH_4AC), pH 6.1.
Gradient: 5% ACN/95% NH_4AC to 30%ACN/70% NH_4AC in 20 minutes.
Flowrate: 1.0 mL/min.

Column: Restek Ultra PFP, 4.6 mm i.d. x 250 mm, 5 μm .

B. Auxiliary Apparatus

1. Precleaned vials, 8 mL capacity with teflon caps.
2. Sonicator
3. Dionex Polyvials with filter caps, 0.5 mL.

C. Reagents

1. Acetonitrile, HPLC/Pesticide grade or better.
2. Ammonium Acetate, 99.99%.
3. Glacial Acetic Acid, HPLC Grade or better.
4. Nanopure Water, Type I
5. 1-(2-Pyridyl)piperazine, 99.5+% or better.
6. Methyl Isocyanate, Chem Service #O-2179, 99+%.
7. XAD-7 resin sorbent tubes, coated with 1-(2-pyridyl)piperazine. Supelco ORBO 657, 80/40 mg, Bellefonte, PA.

5. ANALYSIS OF SAMPLES

1. The instrument is equilibrated for approximately one (1) hour before analysis of samples. Check that the volume in the eluant reservoirs is sufficient for the sample batch.
2. It is necessary to analyze a solvent blank and a cartridge blank with each batch of samples to ascertain the presence of possible interferences.
3. A six-point calibration curve is analyzed with each batch of samples. For the application studies the calibration will be 0.013 to 0.260 $\mu\text{g/mL}$ of the purified MIC derivative. (See section 6.0 B for the preparation of the purified derivative.)
4. A calibration check sample of 0.130 $\mu\text{g/mL}$ is run after the calibration and every ten samples and at the end of the sample batch. The value of the calibration check must be within $\pm 3\sigma$ (the standard deviation) or $\pm 20\%$ of the expected value, whichever is greater. If the calibration check is outside this limit then those samples in the batch after the last calibration check that was within limits need to be reanalyzed.
5. With each batch of samples analyzed, a laboratory cartridge blank and a laboratory control spike will be run concurrently. A laboratory blank is XAD-7 extracted and analyzed the same way as the samples. A laboratory control spike is XAD-7 spiked with a known amount of MIC. The laboratory control sample is extracted and analyzed the same way as the samples.

6. Score and snap the sample resin tube, transfer the resin into an 8 mL vial. (Save the second tube for future analysis if necessary.) Rinse the tube with 3.0 mL of ACN into the extraction vial. Cap and place the vial in the sonicator for one hour.
7. Transfer an aliquot of the extract into a Dionex sampling vial and cap securely. Cap and refrigerate the remaining solution vial if necessary for further analysis.
8. The atmospheric concentration is calculated according to:

$$\text{Conc } (\mu\text{g}/\text{m}^3) = \text{Extract Conc } (\mu\text{g}/\text{mL}) \times 3 \text{ mL} / \text{Air Volume Sampled } (\text{m}^3)$$

6. **QUALITY ASSURANCE**

A. Instrument Reproducibility

The reproducibility of the instrument is established by analyzing five (5) injections of MIC-derivatived standard at three concentrations (low, mid, and high). The low, mid, and high concentrations were 0.013, 0.078 and 0.260 $\mu\text{g}/\text{mL}$, respectively.

B. Purified Derivative and Calibration

1. The purified MIC derivative is prepared as described in OSHA Method 54, section 3.3.1. A stock standard is prepared by dissolving the MIC derivative into ACN. The derivative is expressed as free MIC by multiplying the amount of MIC-derivative weighed by the conversion factor 0.2590. (See OSHA Method 54, section 3.3.2)
2. A six (6)-point calibration curve is made at 0.013, 0.026, 0.052, 0.078, 0.134, and 0.260 $\mu\text{g}/\text{mL}$ of the MIC derivative.

C. Calibration Check

A calibration check sample is run after the calibration, after every ten samples and at the end of the sample batch to verify the system is in calibration. The value of the check must be within $\pm 3\sigma$ (the standard deviation) or $\pm 20\%$ of the expected value, whichever is larger. If the calibration check is outside the limit, then those samples in the batch after the last calibration check that was within the limit need to be reanalyzed.

D. Minimum Detection Limit

The detection limit is based on US EPA MDL calculation. The method detection limit (MDL) for methyl isocyanate is calculated by the analysis of seven (7) replicates of a low-level matrix spike. The MDL = 3.14*(std dev values), where std dev is the standard deviation of the concentration calculated for the seven replicate spikes. For MIC the analytical MDL is 0.003 µg/mL. The EQL, defined as 5*MDL, is 0.015 µg/mL based on a 3 mL extraction volume. The corresponding EQL based on a 24 hours collection is 0.42 µg/m³. A low calibration standard of 0.013 µg/mL is selected to ensure accurate quantitation at the EQL. Results equal to or above the EQL are reported to three significant figures. Results below the EQL but equal to or above the MDL are reported as DET (detected) and results less than the MDL are reported as ND (nondetect).

E. Collection and Extraction Efficiency (Recovery)

Methyl isocyanate at a low and high level are spiked on XAD-7 tubes. The spiked tubes are placed on field samplers with airflows of 0.075 LPM for 24 hours. The samples are extracted with ACN and prepared as described in section 5, #6-7. The recovery of MIC for this method is low, ranging 50% to 70%. At concentrations above 1.0 µg/mL the recovery is greater than 70%.

F. Storage Stability

No storage stability analysis has been made with this compound.

H. Breakthrough

No breakthrough studies were done for this application.

H. Safety

This procedure does not address all of the safety concerns associated with chemical analysis. It is the responsibility of the analyst to establish appropriate safety and health practices. For hazard information and guidance refer to the material safety data sheets (MSDS) of any chemicals used in this procedure.

APPENDIX III

LABORATORY REPORT FOR CARBON DISULFIDE



Winston H. Hickox
Agency Secretary

Air Resources Board

Alan C. Lloyd, Ph.D.
Chairman

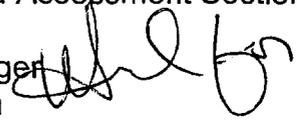
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Gray Davis
Governor

MEMORANDUM

TO: Webster Tasat, Manager
Operations Planning and Assessment Section

FROM: Russell D. Grace, Manager 
Special Analysis Section

DATE: June 27, 2002

SUBJECT: CARBON DISULFIDE CANISTER ANALYTICAL REPORT FOR 2002
METAM-SODIUM APPLICATION MONITORING

The Special Analysis Section (SAS) provided laboratory support for the Carbon Disulfide pesticide monitoring program conducted in 2002. The SAS laboratory performed analyses on samples collected during a four day period at a drip irrigation application site in Ventura County. These samples were collected in six-liter Silco™ canisters from May 7, 2002 to May 11, 2002.

The laboratory analytical results are presented in the attached report, titled "Air Sample Analytical Results for Carbon Disulfide (CS₂) Collected in Silco® Canisters after Application of Metam-Sodium."

If you have any questions or comments, please contact Mr. Jim Omand of my staff at 324-1969 or me at 322-8959.

Attachment

cc: Michael Poore
Kevin Mongar
T. E. Houston
Jim Omand
Michael Orbanosky

The energy challenge facing California is real. Every Californian needs to take immediate action to reduce energy consumption. For a list of simple ways you can reduce demand and cut your energy costs, see our Website: <http://www.arb.ca.gov>.

California Environmental Protection Agency

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 **Air Resources Board**

**Air Sample Analytical Results for Carbon Disulfide (CS₂) Collected in Silco®
Canisters after Application of Metam-Sodium.**

DATE: June 27, 2002

Prepared by:

**James Omand, Air Pollution Specialist
Special Analysis Section, Northern Laboratory Branch
Monitoring and Laboratory Division**

Reviewed and Approved by

**Russell Grace, Manager
Special Analysis Section**

Project Number: P-02-001

This report has been reviewed by staff of the California 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 of commercial products constitute endorsement or recommendation for use.

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1.0 INTRODUCTION

The Department of Pesticide Regulation (DPR) requested the Air Resources Board (ARB) to conduct application air monitoring for carbon disulfide (CS₂), a breakdown product of metam-sodium. This report covers the standard operating procedure, analytical results and quality assurance results for the analysis of CS₂ following a drip irrigation application of metam-sodium. DPR's requested estimated quantitation limit (EQL) for the application is 15 micrograms per cubic meter (µg/m³.)

2.0 STANDARD OPERATING PROCEDURE.

2.1 Overview

The method follows United States Environmental Protection Agency (USEPA) Compendium Method TO-15. Each field sample is pressurized to approximately 15 pounds per square inch gauge (psig) before sample analysis. The canister samples are concentrated using a cryogenic autosampler/concentrator (cryosampler) and then loaded onto a capillary column. The cryosampler contains a Nafion dryer, which reduces water vapor in the sample stream. A gas chromatograph equipped with a high-resolution capillary column separates the sample components. A linear quadrupole mass spectrometer operated in selected ion monitoring (SIM) mode detects the sample components. The results of the analysis are calculated using an internal standard (IS) calibration method. Appendix A contains the standard operating procedure for carbon disulfide.

2.2 Instrument Reproducibility

In order to establish the reproducibility of this method, lab staff prepared canisters spiked with carbon disulfide at three different levels. Lab staff analyzed each standard concentration five times using a 100-milliliter (ml) sample size. Table 1 shows the instrument reproducibility results for carbon disulfide.

2.3 Calibration

The calibration procedure is described in Appendix D. All the calibration curves use a linear fit and must have a correlation coefficient (r²) of 0.995 or greater.

2.4 Minimum Detection Limit (MDL) and Estimated Quantitation Limits (EQL)

This method follows standard USEPA procedures to calculate the MDL. Using the analysis of seven low-level matrix spikes, the MDL and EQL for a 100-ml sample are calculated as follows:

$$\begin{aligned} \text{MDL} &= 3.14 \times s \\ \text{EQL} &= \text{MDL} \times 5 \end{aligned}$$

Where s = the standard deviation of seven replicate spikes. For CS_2 , $s = 0.073 \mu\text{g}/\text{m}^3$:

$$\begin{aligned}\text{MDL} &= 3.14 \times 0.073 \mu\text{g}/\text{m}^3 = 0.23 \mu\text{g}/\text{m}^3 \\ \text{EQL} &= 0.23 \mu\text{g}/\text{m}^3 \times 5 = 1.15 \mu\text{g}/\text{m}^3\end{aligned}$$

For the current project the MDL and EQL are calculated for each individual field sample. This is necessary because of the large variation in sample size, which results in large variation in dilution factor. Individual sample MDL and EQL values are listed in Table 3, and range from 0.5 to 1.5 $\mu\text{g}/\text{m}^3$ and from 2.7 to 7.3 $\mu\text{g}/\text{m}^3$ for MDL and EQL, respectively.

Staff report results above the calculated EQL to three (3) significant figures. Results below the calculated EQL but greater than or equal to the MDL are reported as detected (DET). Results less than MDL are reported as <MDL.

2.5. *Collection and Extraction Efficiency (Recovery)*

Carbon disulfide is listed in USEPA Compendium Method TO-15. Staff did not evaluate CS_2 for collection efficiency.

2.6. *Storage Stability*

Carbon disulfide is listed in USEPA Compendium Method TO-15. Compounds listed for this method are considered stable in the collection canister for 30 days. Method spikes (trip, field and lab) were prepared in excess of 30 days prior to analysis and all method spikes showed excellent recovery (see tables 4,5,6). All field samples were analyzed within 15 days of their collection.

2.7. *Canister Cleaning*

Canisters are cleaned in batches of eight (8). Lab staff chooses one of the cleaned canisters for GC/MS analysis. Normally, lab staff analyzes the canister with the highest level of target compound. If results for the target compounds are not below method MDL's the entire cleaning batch is recleaned and rechecked. Appendix B contains the standard operating procedure for canister cleaning.

3.0 APPLICATION AIR MONITORING SAMPLE RESULTS

The laboratory received a total of 37 canister field samples, including four (4) field spikes. Table 3 contains the results of the analysis for CS_2 field samples.

4.0 ANALYTICAL QUALITY CONTROL SAMPLES

4.1 *Laboratory system blanks*

The laboratory system blank checks the analytical system for contamination. A laboratory system blank, which is 100 ml of ultrapure nitrogen, is run before the start of an analytical batch, after every tenth sample and at the end of the analysis sequence. Lab staff defines an analytical batch as the samples in an automated GC/MS analysis sequence. System blank results are reported in Table 2.

4.2 *Method calibration*

The analytical method uses a certified gas standard for calibration. Before analysis the detector is autotuned and the results are evaluated using the criteria listed in Appendix C. The certified standard used for instrument calibration was obtained from Scott - Marrin Inc. and has the following specifications: 0.986 ppm +/- 2%, cylinder # CC72058, certified 2/27/02. Lab staff prepares working calibration gas by diluting the certified stock standard to produce the calibration concentrations detailed in Appendix D.

Internal standard is used to quantitate both standards and samples. The stock internal standard used for the current project has the following specifications: bromomethane-d₃, 102.6 +/- 7.2 ppb, Scott-Marrin Inc. Working internal standard is prepared by adding the stock bromomethane-d₃ standard to a six-liter canister without dilution. The cryosampler adds internal standard to the analytical system using a fixed volume sample loop. Calibration of the analytical system occurs with each new batch of working internal standard.

4.3 *Laboratory control samples*

Each analytical batch includes a laboratory control sample (LCS.) The LCS is prepared from a stock solution other than the stock solution used to prepare method calibrators. The stock solution used to prepare the LCS for the current project has the following specifications: carbon disulfide 0.984 ppm +/- 5%, Scott-Marrin Inc. The LCS target value is 3.4 µg/m³. The LCS results are: mean, 3.71 µg/m³, standard deviation 0.18 µg/m³, coefficient of variation 4.9%. LCS results are listed in Table 2.

4.4 *Continuing calibration verification*

Lab staff includes a continuing calibration verification standard (CCV) at the beginning of each analytical batch, after every tenth sample in an analytical sequence and at the end of each analytical batch. The CCV must be within ± 20% of the expected value. If the CCV is outside the ±20% limit, lab staff takes appropriate corrective action, and then reanalyzes the CCV and the affected samples. Calibration of the entire system occurs if the reanalysis of the CCV is outside of acceptance limits. For the current project, all CCV results were within limits.

4.5 *Laboratory duplicate samples*

Normally 10% of the field samples are reanalyzed to evaluate the analysis precision for field samples. For the current project none of the field samples had results greater than the EQL. Duplicate analysis were not done for the current project.

5.0 **FIELD, TRIP, AND LABORATORY SPIKES**

For the application analysis four (4) field spikes, four (4) trip spikes, and four (4) laboratory spikes were prepared and analyzed. Each set of four spikes was prepared with two each at high and low concentrations. The low set were spiked at a level that would place them within the method calibration range using routine handling. The high concentration spikes were prepared at a level which required additional dilution to place them within calibration range. Staff diluted the high spikes by injecting a smaller sample volume.

5.1 *Field spikes*

The field spike results for CS₂ are in Table 4. The low-level field spikes were sampled before the metam-sodium application. The high-level field spikes were sampled at the end of the monitoring period. Because none of the colocated field spike samples had values above EQL, the field spikes were not corrected for colocated levels.

5.2 *Trip spikes*

Table 5 presents the results of the CS₂ trip spikes. Trip spikes are spiked canisters sent into the field but not placed on samplers. They are returned to the laboratory along with the field samples.

5.3 *Laboratory spikes*

Table 6 presents the results of the laboratory spikes for CS₂. The laboratory spikes are spiked at the same time as the field and trip spikes. They are stored in the laboratory and analyzed with the field and trip spikes.

6.0 **DISCUSSION**

Some of the system blanks showed detectable amounts of CS₂. Lab staff suspects that this CS₂ contamination comes from the grade five nitrogen purge gas used for the sample concentrator. Several different tanks of nitrogen and ultra pure air were tested as purge gas. Small amounts of CS₂ contamination were detected in each case. In no case was the amount of contamination greater than two times MDL. Because the level of contamination is low compared to the requested EQL (0.2 µg/m³ vs. 15 µg/m³), lab staff did not take further action to eliminate the contamination.

Table 1. Instrument Reproducibility Carbon Disulfide

Sample Number	low range ($\mu\text{g}/\text{m}^3$)	mid range ($\mu\text{g}/\text{m}^3$)	high range ($\mu\text{g}/\text{m}^3$)
1	0.9	2.76	9.62
2	0.93	2.65	9.54
3	0.89	2.52	9.44
4	1.02	2.46	9.43
5	0.84	2.36	9.42
Average	0.92	2.55	9.49
Std. Dev.	0.067	0.16	0.09
c.v.*	7.27	6.18	0.92

* c.v. (coefficient of variation)= (1std. dev./average) x 100

Table 2. Quality Control Data Carbon Disulfide

Date	Blanks ($\mu\text{g}/\text{m}^3$)	Continuing Calibration ($\mu\text{g}/\text{m}^3$)	Lab Control ($\mu\text{g}/\text{m}^3$)
5/14/2002	<MDL	2.74	3.81
	Det	2.92	
5/15/2002	<MDL	2.82	3.80
	<MDL	2.66	
5/20/2002	Det	2.89	3.53
	Det	2.65	
5/21/2002	Det	2.71	3.64
	Det	2.57	
5/22/2002	Det	2.81	3.77
	Det	2.67	
5/28/2002	<MDL	2.54	3.97
	<MDL	2.50	

Table 3. Application Monitoring Results of CS2

Log Number	Laboratory ID	Sample Identification	Canister Number	Date Received	Date Analyzed	CS2 $\mu\text{g}/\text{m}^3$	Injection Volume (ml)	dilution factor	Dilution MDL $\mu\text{g}/\text{m}^3$	Dilution EQL $\mu\text{g}/\text{m}^3$
1	CS2001	MSCWB-FSL-1	DPR 1152	5/13/2002	5/14/2002	15.91	100	2.37	0.5	2.7
2	CS2002	MSCWB-1	DPR 1173	5/13/2002	5/15/2002	Det	100	2.93	0.7	3.4
3	CS2003	MSCSB-1	DPR 1149	5/13/2002	5/15/2002	Det	100	2.60	0.6	3.0
4	CS2004	MSCEBC-FSL-1	DPR 1098	5/13/2002	5/14/2002	17.83	100	2.63	0.6	3.0
5	CS2005	MSCEB-1	DPR 1086	5/13/2002	5/15/2002	Det	100	2.66	0.6	3.1
6	CS2006	MSCNB-1	DPR 1087	5/13/2002	5/15/2002	Det	100	2.54	0.6	2.9
7	CS2007	MSCW-2	DPR 1065	5/13/2002	5/28/2002	<MDL	100	4.66	1.1	5.4
8	CS2008	MSCS-2	DPR 1134	5/13/2002	5/28/2002	Det	100	2.57	0.6	3.0
9	CS2009	MSCEC-2	DPR 1182	5/13/2002	5/28/2002	<MDL	100	6.02	1.4	6.9
10	CS2010	MSCE-2	DPR 1140	5/13/2002	5/28/2002	<MDL	100	4.40	1.0	5.1
11	CS2011	MSCN-2	DPR 1142	5/13/2002	5/28/2002	<MDL	100	5.20	1.2	6.0
12	CS2012	MSCW-3	DPR 1154	5/13/2002	5/22/2002	Det	100	4.46	1.0	5.1
13	CS2013	MSCS-3	DPR 1181	5/13/2002	5/22/2002	Det	100	5.66	1.3	6.5
14	CS2014	MSCEC-3	DPR 1132	5/13/2002	5/22/2002	Det	100	5.51	1.3	6.3
15	CS2015	MSCE-3	DPR 1169	5/13/2002	5/22/2002	Det	100	5.71	1.3	6.6
16	CS2016	MSCN-3	DPR 1133	5/13/2002	5/22/2002	Det	100	6.24	1.4	7.2
17	CS2017	MSCW-4	DPR 1166	5/13/2002	5/22/2002	Det	100	4.65	1.1	5.3
18	CS2018	MSCS-4	DPR 1128	5/13/2002	5/22/2002	Det	100	6.33	1.5	7.3
19	CS2019	MSCEC-4	DPR 1085	5/13/2002	5/22/2002	Det	100	5.57	1.3	6.4
20	CS2020	MSCE-4	DPR 1809	5/13/2002	5/21/2002	Det	100	5.98	1.4	6.9
21	CS2021	MSCN-4	DPR 1147	5/13/2002	5/21/2002	Det	100	5.25	1.2	6.0
22	CS2022	MSCW-5	DPR 1070	5/13/2002	5/21/2002	Det	100	4.32	1.0	5.0
23	CS2023	MSCS-5	DPR 1107	5/13/2002	5/21/2002	Det	100	4.82	1.1	5.5
24	CS2024	MSCEC-5	DPR 1093	5/13/2002	5/21/2002	Det	100	5.18	1.2	6.0
25	CS2025	MSCE-5	DPR 1054	5/13/2002	5/21/2002	Det	100	4.81	1.1	5.5
26	CS2026	MSCN-5	DPR 1092	5/13/2002	5/21/2002	Det	100	5.66	1.3	6.5
27	CS2027	MSCW-6	DPR 1057	5/13/2002	5/21/2002	Det	100	4.73	1.1	5.4
28	CS2028	MSCS-6	DPR 1163	5/13/2002	5/20/2002	Det	100	5.78	1.3	6.6
29	CS2029	MSCEC-6	DPR 1123	5/13/2002	5/20/2002	Det	100	5.19	1.2	6.0
30	CS2030	MSCE-6	DPR 1099	5/13/2002	5/20/2002	Det	100	3.72	0.9	4.3
31	CS2031	MSCN-6	DPR 1102	5/13/2002	5/20/2002	Det	100	5.23	1.2	6.0
32	CS2032	MSCW-7	DPR 1074	5/13/2002	5/20/2002	Det	100	4.05	0.9	4.7
33	CS2033	MSCS-7	DPR 1103	5/13/2002	5/20/2002	Det	100	4.89	1.1	5.6
34	CS2034	MSCEC-7	DPR 1179	5/13/2002	5/14/2002	171.20	15	4.30	1.0	4.9
35	CS2035	MSCE-7	DPR 1063	5/13/2002	5/20/2002	Det	100	4.95	1.1	5.7
36	CS2036	MSCN-7	DPR 1141	5/13/2002	5/20/2002	Det	100	4.76	1.1	5.5
37	CS2037	MSCWHS-7	DPR 1172	5/13/2002	5/14/2002	170.10	15	4.49	1.0	5.2

Table 4: Field Spike Results: CS₂

Instrument	Canister Number	Spike Level	Target Value (µg/m ³)	Actual Value (µg/m ³)	Recovery %
MSD-4	DPR 1098	Low	18.3	17.8	98
MSD-4	DPR 1152	Low	15.7	15.9	101
MSD-4	DPR 1172	High	166	170	102
MSD-4	DPR 1179	High	166	171	103

Table 5: Trip Spike Results: CS₂

Instrument	Canister Number	Spike Level	Target Value (µg/m ³)	Actual Value (µg/m ³)	Recovery %
MSD-4	DPR 1136	Low	7.65	7.48	98
MSD-4	DPR 1165	Low	7.65	7.54	99
MSD-4	DPR 1061	High	38.1	39.6	104
MSD-4	DPR 1094	High	38.1	39.6	104

Table 6: Laboratory Spike Results: CS₂

Instrument	Canister Number	Spike Level	Target Value (µg/m ³)	Actual Value (µg/m ³)	Recovery %
MSD-4	DPR 1066	Low	7.59	7.25	96
MSD-4	DPR 1112	Low	7.59	7.44	98
MSD-4	DPR 1072	High	38.5	38.4	100
MSD-4	DPR 1104	High	38.4	40.0	104

APPENDIX A:

STANDARD OPERATING PROCEDURE FOR CS₂ ANALYSIS

California Environmental Protection Agency



Air Resources Board

**Special Analysis Section
Northern Laboratory Branch
Monitoring and Laboratory Division**

**Standard Operating Procedure
Sampling and Analysis of Carbon Disulfide
In Silco™ Canisters Using a Varian
Stand Alone Cryogenic Sampler**

Version 2
April 23, 2002

Approved by:

Russell Grace, Manager
Special Analysis Section

This SOP has been reviewed by staff of the California 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 of commercial products constitute endorsement or recommendation for use.

1. SCOPE

This method, which follows closely USEPA Method TO-15, is for the sampling and analysis of ambient air using six-liter Silco™ canisters for sample collection. The compound, carbon disulfide (CS₂) is analyzed by gas chromatography/mass spectrometry.

2. SUMMARY OF METHOD

Ambient air is collected into evacuated six-liter Silco™ canisters using a sub-atmospheric pressure collection mode. Sample canisters are subsequently pressurized in the laboratory to approximately fifteen pounds per square inch gauge (psig) and analyzed by Gas Chromatography / Mass Spectrometry (GC/MS) using a cryogenic concentrator to prepare the air sample. Samples are analyzed in the selected ion monitoring (SIM) mode using bromomethane-d₃ as the primary internal standard for quantitation, and 1,2-dichloropropane-d₆ as a secondary internal standard. 1,2-dichloropropane-d₆ is used to evaluate bromomethane-d₃ for interference.

3. INTERFERENCES/LIMITATIONS

Interference may result from improperly cleaned canisters. Analysis of samples containing high concentrations of early eluting pesticide components may cause significant contamination of the analytical equipment. Co-eluting compounds trapped during sample collection may interfere. Running canister blanks and system blanks should minimize contamination originating in the analytical instrument.

4. EQUIPMENT AND CONDITIONS

A. Instrumentation

Hewlett Packard 5890 Series II gas chromatograph:

Detector: 280° C

Injector: 220° C

Column: Restek Rtx-200, 60 meter, 0.32mm I.D., 1.5 micron film thickness

GC temperature program: initial -10° C, -10 to 80° C @ 10° C/min, 80° to 200° C @ 25° C/min, hold for 1 minute, 200 to 240° C @ 25° C/min

Carrier Gas: Helium, grade 5 or better

Hewlett Packard 5973 mass selective detector:

Acquisition Mode: SIM

Tune File: PFTBA Autotune, maximum sensitivity

Ions monitored: 76 m/z quant, 78 m/z qualifier

Varian Stand Alone cryogenic concentrator:

Valve Oven: 60° C

Autosampler Oven: 60° C

Nafion Dryer: ambient

Sample Line: 150° C

Cryotrap: -180° C to 150° C

Transfer Line: 150° C

Cryofocus: -180° C to 150° C

Sample Size: 100 ml

Internal Standard Loop: 1 ml

B. Auxiliary Apparatus

Compressed helium: Grade 5 or better

Compressed nitrogen: Grade 5 or better

Liquid nitrogen for cryogenic concentrator

Certified standard for carbon disulfide

Restek, 6.0 liter Silcosteel™ canisters with Silcosteel™ valve

Pressure gauge, -30 inches Mercury (Hg) to 30 pounds psig.

Canister cleaning system (Appendix B)

5. ANALYSIS OF SAMPLES

- 1) Perform a PFTBA autotune and evaluate tune criteria. See Appendix C. Place a copy of the autotune results in the autotune folder.
- 2) Check and record the pressure in the field sample canisters. Pressurize the field sample canisters to approximately 14.7 psig with Grade 5 or better nitrogen. Record the final pressure.
- 3) Prepare a sample sequence for the GC/MS. The sequence should include a continuing calibration check (CCV), and a blank, for every 10 field samples. A laboratory control sample (LCS) and a duplicate are run once per analytical batch not to exceed 20 samples. Load the sequence into the GC/MS in the remote start mode.
- 4) Prepare a sample sequence for the Varian concentrator. The sample sequence should be organized as follows: system blank, CCV, LCS, field samples, duplicate field sample, and CCV. If the CCV is not within $\pm 20\%$ of it's assigned value the system must be recalibrated.
- 5) Attach the sample canisters to the Varian autosampler ring as per the sequence. Execute the Varian sequence, which in turn will initiate the GC sequence.
- 6) Sample quantitation reports will print out after each analysis.
- 7) Review and edit the quantitation reports as needed.

- 8) Calculations will require a correction for the required pressurization performed prior to analysis. Instrument reports will be in units of $\mu\text{g}/\text{m}^3$ and must be corrected for the analysis dilution using the following calculation:

$$(F_p / I_p) \times C_i = C_r$$

I_p = initial canister pressure in mm Hg

F_p = final canister pressure in mm Hg

C_i = concentration from the analysis report in $\mu\text{g}/\text{m}^3$

C_r = reported concentration in $\mu\text{g}/\text{m}^3$

6. QUALITY ASSURANCE

A. Instrument Reproducibility

Establish the reproducibility of the instrument and analytical method as follows. Inject five replicate samples of CS_2 standards at three concentrations (low range, mid range, and high range).

B. Linearity

A 5-point calibration is performed. Calibration standards ranging from approximately 1.6 to 10.4 $\mu\text{g}/\text{m}^3$ are used to calibrate the method. If ambient samples are diluted six fold, the calibrated ambient sample range is approximately 9.6 to 62.4 $\mu\text{g}/\text{m}^3$. The results are used to calculate calibration curves using linear regression.

C. Minimum Detection Limit

Detection limits are based on the USEPA MDL calculation. Using the analysis of seven replicates of a low-level analysis, the method detection limits (MDL) and the EQL for the pesticide components are calculated as follows:

$$\text{MDL} = 3.14*s$$

$$\text{EQL} = 5*\text{MDL}$$

Where s equals the standard deviation of the response calculated for the seven replicate spikes. The calculated MDL for CS_2 is 0.23 $\mu\text{g}/\text{m}^3$. The respective EQL is 1.15 $\mu\text{g}/\text{m}^3$. Assuming a 1 to 6 dilution to pressurize the ambient samples the EQL is 6.9 $\mu\text{g}/\text{m}^3$ for CS_2 .

Results are reported to 3 significant figures above the EQL. Results below EQL but above MDL are reported as DET (detected). Results reported as non-detect

(ND) and reported values less than the MDL are reported as less than MDL (<MDL).

D. Storage Stability

A CS₂ storage stability study will not be performed. Carbon Disulfide is included in USEPA Method T0-15 and as stated in section 1.3 "under conditions of normal usage for sampling ambient air, most VOC's can be recovered from canisters near their original concentrations after storage of up to 30 days." During this program all samples will be analyzed within 21 days of collection.

E. Safety Precautions

This procedure does not address all of the safety concerns associated with chemical analysis. It is the responsibility of the analyst to establish appropriate safety and health practices. For hazard information and guidance refer to the material safety data sheets (MSDS) of any chemicals used in this procedure. All applicable safety precautions must be observed for the use of compressed gas cylinders.

APPENDIX B:

STANDARD OPERATING PROCEDURE FOR CANISTER CLEANING

CANNISTER CLEANING PROCEDURE

The canister cleaning procedure uses repeated cycling from -30 inches Hg to 30 pounds per square inch gauge with humidified ultra pure nitrogen. The procedure includes four complete cycles each 24 minutes (19 minutes vacuum and 5 minutes pressure) at 60 degrees C.

Canister data should be logged into the canister-cleaning book for each cleaning batch. When the batch is complete one canister is chosen for analysis. The canister is pressurized with ultra pure nitrogen and analyzed by the GCMS method. If target analytes are not less than their MDL the entire batch should be cleaned again.

Procedure:

A. Fill dewar with Liquid Nitrogen (LN2)

1. Remove dewar cover.
2. CAREFULLY place hose from LN2 tank into dewar (orange and silver container behind oven).
3. Open LN2 tank 3 turns
4. Close tank when LN2 can be seen near top of dewar.
5. CAREFULLY remove hose and replace dewar cover.

B. Turn on the vacuum pump.

1. Switch is located on pump to the left of the canister oven.

C. Open Nitrogen (N₂) Tank

1. Open regulator on N₂ tank to the left of the canister oven.

D. Load canisters in oven

1. Attach cans to manifold in oven and tighten.
2. If you are cleaning less than 8 canisters the unused ports must be capped.
3. Open the canister valves

E. Start Timers Located on top left of canister oven

1. Push auto button on top timer and auto light should come on. If the light is off, hit the button again and it should light.
2. Push the run button on the bottom timer. The 1 light should light up briefly then switch to 2.
3. The system should begin to evacuate.

4. Verify the system evacuates all the way by reading the gauge on the back of the oven. The gauge should go to -30 psi.

F. Fill cans and shutdown system.

1. Close all canister valves except the ones you want to fill.
2. On the top timer hit the ADV button until the 2 light comes on.
3. Monitor the pressure of the canisters on the gauge on the back of the oven.
4. Close can valves when filled.
5. Close N_2 Regulator
6. Turn off Vacuum pump.
7. Remove canisters and place plugs on manifold ports.
8. Hit the stop button on both timers.

APPENDIX C:

STANDARD OPERATING PROCEDURE FOR MSD AUTOTUNE

Autotune Criteria

A maximum sensitivity autotune should be performed on the detector each day prior to sample analysis. The autotune report should be evaluated for the following:

1. Any unusual change in the EM voltage
2. Peak width for all tune masses should be between 0.4 amu and 0.6 amu.
3. The relative abundance of tune mass 219.0 should be greater than 25% of tune mass 69.0.
4. Isotope abundance ratio for tune mass 70.0 should be between 0.54% and 1.6 %; isotope abundance ratio for tune mass 220.0 should be between 3.2% and 5.4%.
5. Masses 28 and 18 should be evaluated to check for air leaks in the system.

If autotune criteria are not met the system should be evaluated for problems. After the system problems are corrected the detector should be autotuned prior to sample analysis. Autotune reports should be filed in the instrument autotune folder.

APPENDIX D:

CALIBRATION STANDARD PREPARATION FOR CS₂

Calibration Standard Preparation for Carbon Disulfide

The certified stock gas used for calibration during this study was purchased from Scott Marrin and has the following specifications:

Cylinder No: CC72058
Expiration date May 9, 2002
Carbon Disulfide 0.985 PPM/M

Working analysis standard is prepared by diluting the stock gas using the following procedure.

1. A six liter Silco canister is evacuated to -30 " Hg.
2. 60 ml of carbon disulfide stock is transferred to the canister using a gas tight syringe equipped with a stopcock.
3. 100 ul of reagent grade water is added to the canister using a syringe and syringe adapter.
4. The canister is pressurized to approximately 29.4 psig with grade five or better nitrogen.

The canister will contain CS₂ at the following concentration:

Carbon Disulfide 10.39 $\mu\text{g}/\text{m}^3$

The standard sample injection volume is 100 ml. Using the cryogenic sampler to introduce the following volumes of working standard to the GCMS generates a calibration curve.

<u>Volume</u>	<u>Carbon disulfide</u>
100 ml	10.39 $\mu\text{g}/\text{m}^3$
75 ml	7.79 $\mu\text{g}/\text{m}^3$
50 ml	5.20 $\mu\text{g}/\text{m}^3$
25 ml	2.60 $\mu\text{g}/\text{m}^3$
15 ml	1.56 $\mu\text{g}/\text{m}^3$

APPENDIX IV

NOTICE OF INTENT TO APPLY
RESTRICTED MATERIALS



The Best People
The Best Products
The Best Service

RECOMMENDATION-N.O.I./WORK ORDER

WORK ORDER 212930-0
ORDER DATE 5/06/02
APPLY DATE 5/08/02

FARM SERVICE

1015 E WOODLEY RD /4075 DUFAU 93016
OXNARD, CA 93030
(805)487-4961

CUSTOMER-SUN WORLD INTERNATIONAL
ACCOUNT 60-20953

PERMITTEE	LOCATION	COMMODITY	RESTRICTIONS
MIN NISHIMORI FARMS	I EAST BELL S 2	I 5013 PEPPERS, BELL	I HARVEST DAYS- RE-ENTRY HOURS- 48 I
771 MOUNTAIN VIEW AVE	I SITE-2	I PLANTING-	I PLANT BACK-NO PASTURE RESTR- NO I
OXNARD CA. 93030	I SEC-19 TWA-02N RNS-20W	I TREATED ACRES- 10.00 I	I POSTING REQ-YES EARLY ENTRY I
PERMIT- 56-02-56-X0014-R	I BLK-3	I PLANTED ACRES- 27.00 I	I PPE REQ-NO PPE REQUIRED- NO I
CUST PO#-	I MAPID-MNIS002	I ICROP USE-	I BEES-NO FEED WASTE- NO I

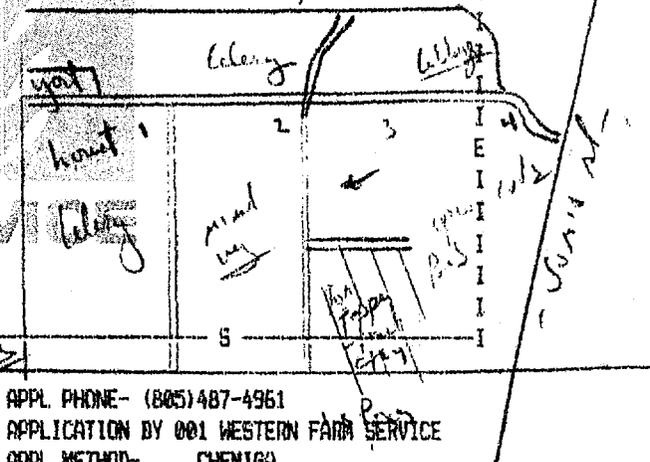
SEE ADDITIONAL INFO BELOW

SURROUNDING HAZARDS / CROPS		"X" IF TOXIC TO		"X" IF APPLICABLE	
W-	N-	I FISH	I	I REQUIRES CLOSED	I
E-	E-	I BIRDS	I	I SYSTEM	I
W-	S-	I WILDLIFE	I	I AVOID RUNOFF, KEEP	I
E-	W-	I BEE, NOTIFY	I	I OUT OF WATERWAYS	I
		I BEEKEEPER	I	I AVOID DRIFT	I

APPLIC INSTRUCTIONS- 50 GAL PER ACRE VOLUME ROW SETTING 68-00
DELIVERY INSTRUCTIONS-

PRODUCT#	PRODUCT/SERVICE DESCRIPTION	EPA REGISTRATION	RATE/ACRE	TOT QTY	CODE/PEST	GROWER MATL
1-40650	CHEMIGATION	I	I	I 10.00 ACI		I
19-79510	SECTAGON 42 BULK	I 61842-00006	I 50.00 GAL	I 500.00 GAL	F WEEDS	I *G

A-MINOR DAMAGE; INCREASING; NO PREDATORS PRESENT. E-IPM EFFECTIVE PESTICIDE INDICATED I
 B-POYENTL CROP LOSS UNLESS CONTROLLED NOW. F-PREVENTIVE RESIDUAL I
 C-HEAVY EGG POP; RESIDUAL PESTICIDE NEEDED. G-SEE REMARKS I
 D-NEMATODE PROTECTION; PRE-PLANT "G" DENOTES GROWER MATERIAL I



ADVISOR TOM NAGEL PCA# 3141

I CERTIFY THAT I HAVE CONSIDERED ALTERNATIVES AND MITIGATION MEASURES THAT
 WOULD SUBSTANTIALLY LESSEN ANY SIGNIFICANT IMPACT ON THE CROP OR ENVIRONMENT,
 AND HAVE ADOPTED THOSE FOUND FEASIBLE.

ADDITIONAL INFORMATION, PLEASE READ
 7979510/SECTAGON 42 BULK REG# 61842-00006

APPL PHONE- (805)487-4961
 APPLICATION BY 001 WESTERN FARM SERVICE
 APPL METHOD- CHEMIGA

Injected into drip lines.
 Sol. l granules will be in field

RIG	HOURS	DRIVER
I	I	I
I	I	I
I	I	I

[] APPROVED [] DENIED

APPENDIX V

METAM SODIUM APPLICATION METEOROLOGICAL DATA

Export Filename : C:\MICROMET\CARBAP\EXPORT\02050711.TXT

Export data for station : Metam Sodium App

Printing Date : 2002/05/16

		RWS	RWD	AT	RH	BP	Sigma
		(mph)	(Deg)	(Deg C)	(%)	(mmHg)	(Deg)
05/07/2002	9:00	None	None	None	None	None	None
05/07/2002	9:15	2.6	268	-6.3	58.3	754	69.8
05/07/2002	9:30	4.9	249	14.4	77.2	755	25.4
05/07/2002	9:45	5.4	248	14.7	75.3	755	11.1
05/07/2002	10:00	5.7	250	15.0	74.4	755	22.8
05/07/2002	10:15	6.6	241	15.2	73.4	755	25.7
05/07/2002	10:30	7.8	248	15.6	72.2	755	14.1
05/07/2002	10:45	9.4	235	15.7	71.7	755	13.4
05/07/2002	11:00	9.2	228	15.8	70.7	755	23.1
05/07/2002	11:15	8.8	240	16.0	69.2	755	14.8
05/07/2002	11:30	9	244	16.3	67.9	755	18.7
05/07/2002	11:45	11.9	247	16.4	68.7	755	12.2
05/07/2002	12:00	12.7	242	16.1	69.3	755	10.3
05/07/2002	12:15	13.4	250	16.0	70.1	755	11.2
05/07/2002	12:30	11	239	15.7	70.4	755	8.9
05/07/2002	12:45	11.2	249	15.7	69.8	755	11.7
05/07/2002	13:00	12.3	253	16.0	68.8	755	10.6
05/07/2002	13:15	12.7	252	16.3	67.6	755	9.6
05/07/2002	13:30	13.4	242	16.3	67.5	755	15.2
05/07/2002	13:45	13.5	241	16.6	66.9	755	12.6
05/07/2002	14:00	15.2	239	16.3	66.8	755	10.1
05/07/2002	14:15	13.7	246	16.2	67.3	754	19.1
05/07/2002	14:30	14	246	16.2	66.7	754	12.7
05/07/2002	14:45	11.7	243	16.3	65.4	754	15
05/07/2002	15:00	11.3	252	16.1	65.6	754	16.2
05/07/2002	15:15	11.1	247	16.1	65.1	754	15.4
05/07/2002	15:30	11.8	250	16.2	65.1	754	11.9
05/07/2002	15:45	12.1	253	16.0	65.4	754	8.1
05/07/2002	16:00	10.5	242	16.2	64.6	753	17.9
05/07/2002	16:15	12.1	250	16.2	65.3	753	9.3
05/07/2002	16:30	11.6	243	15.9	65.7	753	15.1
05/07/2002	16:45	11.1	227	15.8	65.0	753	12.1
05/07/2002	17:00	9.9	232	15.8	65.2	753	12.5
05/07/2002	17:15	9.1	235	15.7	65.2	753	12.8
05/07/2002	17:30	10.3	234	15.5	65.6	753	12.3
05/07/2002	17:45	10.3	233	15.4	65.8	753	12.1
05/07/2002	18:00	9.8	235	15.2	66.3	753	12.7
05/07/2002	18:15	9.3	231	15.1	66.7	753	11.6
05/07/2002	18:30	8.9	231	14.9	67.4	753	13.3
05/07/2002	18:45	9.2	235	14.5	68.2	753	8.2
05/07/2002	19:00	9.1	245	14.2	68.5	753	6.5
05/07/2002	19:15	7.5	227	13.9	71.0	753	21.5
05/07/2002	19:30	6.9	228	13.7	72.6	753	12.7
05/07/2002	19:45	7	220	13.2	74.4	753	10.3
05/07/2002	20:00	7.1	229	12.9	76.7	753	6.9
05/07/2002	20:15	5.7	218	12.7	77.9	753	18.5
05/07/2002	20:30	5.1	208	12.4	79.7	753	8.6

Export Filename : C:\MICROMET\CARBAPI\EXPORT\02050711.TXT

Export data for station : Metam Sodium App

Printing Date : 2002/05/16

		RWS (mph)	RWD (Deg)	AT (Deg C)	RH (%)	BP (mmHg)	Sigma (Deg)
05/07/2002	20:45	3.7	207	12.0	80.2	753	7.9
05/07/2002	21:00	3.6	199	11.8	81.1	753	14
05/07/2002	21:15	4.1	184	11.4	82.5	753	10.6
05/07/2002	21:30	4.3	197	11.3	82.7	753	10
05/07/2002	21:45	2.2	175	11.0	83.1	753	17.2
05/07/2002	22:00	1.2	358	10.8	84.1	753	35.3
05/07/2002	22:15	0.6	13	10.5	84.2	753	25.3
05/07/2002	22:30	0.8	34	10.5	84.6	753	23.9
05/07/2002	22:45	1.8	351	10.6	84.4	753	4.6
05/07/2002	23:00	1	74	10.5	84.1	753	27.8
05/07/2002	23:15	0.5	140	10.5	85.1	753	20.4
05/07/2002	23:30	0.5	116	10.6	84.9	753	29.3
05/07/2002	23:45	0.6	96	10.6	85.0	753	28.7
05/07/2002	24:00:00	1.1	97	10.6	85.2	753	12
05/08/2002	0:15	0.7	70	10.4	84.8	753	51.3
05/08/2002	0:30	0.7	54	10.0	84.4	752	16.8
05/08/2002	0:45	0.2	18	9.6	85.1	752	21.2
05/08/2002	1:00	0.5	15	9.5	87.8	752	17.5
05/08/2002	1:15	1.1	80	9.1	87.0	752	19.3
05/08/2002	1:30	1	68	9.0	86.5	752	21.1
05/08/2002	1:45	2.6	56	8.9	87.0	752	10
05/08/2002	2:00	2.1	77	8.6	87.3	752	16.6
05/08/2002	2:15	3.4	62	8.4	87.0	752	14.6
05/08/2002	2:30	3.1	41	8.3	88.1	752	18.7
05/08/2002	2:45	3	79	8.1	87.3	752	11.3
05/08/2002	3:00	2.8	87	8.1	86.8	752	7.3
05/08/2002	3:15	2.5	92	8.4	86.9	752	10.1
05/08/2002	3:30	2.8	87	8.7	87.3	752	12
05/08/2002	3:45	1.3	32	9.3	85.5	753	41.6
05/08/2002	4:00	0.7	46	9.5	85.6	753	57.3
05/08/2002	4:15	0.7	92	9.5	85.0	753	38.6
05/08/2002	4:30	0.4	109	9.8	83.7	753	38.3
05/08/2002	4:45	0.7	91	9.9	83.7	753	64.8
05/08/2002	5:00	1	17	10.1	83.7	753	15.2
05/08/2002	5:15	1.6	48	10.0	84.8	753	22.5
05/08/2002	5:30	0.9	40	9.8	85.6	753	34.4
05/08/2002	5:45	1.5	67	9.9	85.9	753	47.9
05/08/2002	6:00	1.3	50	10.3	84.5	753	36.1
05/08/2002	6:15	1.1	32	10.7	84.2	753	39.7
05/08/2002	6:30	1.6	94	11.2	83.1	754	52.6
05/08/2002	6:45	2.1	106	11.2	83.4	754	31.6
05/08/2002	7:00	1.5	24	11.6	82.5	754	50.6
05/08/2002	7:15	1.5	313	12.2	80.1	754	53
05/08/2002	7:30	3.4	259	12.2	79.8	754	30.1
05/08/2002	7:45	4.2	222	12.6	77.5	755	27.8
05/08/2002	8:00	4.3	246	13.0	75.7	755	23.6
05/08/2002	8:15	5.6	249	13.7	74.5	755	25.8

Export Filename : C:\MICROMET\CARBAP\EXPORT\02050711.TXT
 Export data for station : Metam Sodium App
 Printing Date : 2002/05/16

		RWS	RWD	AT	RH	BP	Sigma
		(mph)	(Deg)	(Deg C)	(%)	(mmHg)	(Deg)
05/08/2002	8:30	6.7	212	14.0	72.6	755	17.1
05/08/2002	8:45	8.6	225	14.5	70.5	755	15.4
05/08/2002	9:00	8.8	221	15.0	69.1	756	17.2
05/08/2002	9:15	8.8	233	15.2	68.8	756	28.3
05/08/2002	9:30	8.2	235	15.7	67.1	756	26.3
05/08/2002	9:45	8.6	246	16.1	65.6	756	22.6
05/08/2002	10:00	9.3	238	16.6	64.3	756	19
05/08/2002	10:15	9.5	226	16.9	62.9	756	17.6
05/08/2002	10:30	12.2	233	17.2	62.1	756	14.7
05/08/2002	10:45	12.8	239	17.4	61.1	756	13.4
05/08/2002	11:00	14.6	232	17.5	60.6	756	13.3
05/08/2002	11:15	14	244	17.5	61.3	756	15.7
05/08/2002	11:30	14.8	239	17.5	62.0	756	15.6
05/08/2002	11:45	15.2	231	17.5	61.9	756	13.6
05/08/2002	12:00	15.6	243	17.5	62.1	756	11.1
05/08/2002	12:15	15.4	234	17.5	62.2	756	17.3
05/08/2002	12:30	15.3	220	17.8	61.6	756	15.1
05/08/2002	12:45	16.1	229	17.5	62.3	756	13.4
05/08/2002	13:00	16.2	230	17.6	62.4	756	13.9
05/08/2002	13:15	15	232	17.8	61.9	756	15
05/08/2002	13:30	16.9	226	18.0	61.3	756	14.7
05/08/2002	13:45	17.4	242	17.6	61.5	756	11.5
05/08/2002	14:00	16.5	239	17.9	60.7	756	14.2
05/08/2002	14:15	15.4	241	17.5	61.7	756	13.4
05/08/2002	14:30	15.3	234	17.8	60.9	755	14.7
05/08/2002	14:45	15.7	236	17.5	61.0	755	13.6
05/08/2002	15:00	14.6	235	17.6	60.6	755	15.5
05/08/2002	15:15	14.8	239	17.6	59.9	755	15.5
05/08/2002	15:30	14.5	239	17.7	59.8	755	14.4
05/08/2002	15:45	13.4	227	17.6	59.8	755	14.9
05/08/2002	16:00	13.6	234	17.5	60.2	755	12
05/08/2002	16:15	14	241	17.3	60.4	755	11.6
05/08/2002	16:30	13.7	224	17.2	60.0	755	10.9
05/08/2002	16:45	13.8	237	17.0	61.0	755	14.6
05/08/2002	17:00	13	241	16.5	62.4	755	14.9
05/08/2002	17:15	11.5	249	16.0	63.2	755	11.7
05/08/2002	17:30	9.3	243	15.7	64.1	755	12.9
05/08/2002	17:45	8.1	235	15.7	64.4	755	11.5
05/08/2002	18:00	9	230	15.6	64.5	755	10.6
05/08/2002	18:15	9.2	227	15.2	66.3	755	8.5
05/08/2002	18:30	7.4	236	14.8	68.7	755	6.9
05/08/2002	18:45	6.4	232	14.5	69.2	755	5.9
05/08/2002	19:00	5.5	234	14.3	69.6	755	5.4
05/08/2002	19:15	4.6	237	14.1	70.4	755	10.8
05/08/2002	19:30	1.7	248	13.6	72.1	755	24.9
05/08/2002	19:45	1.1	291	13.2	73.3	755	27.1
05/08/2002	20:00	0.9	335	13.1	73.4	754	35.2

Export Filename : C:\MICROMET\CARBAPI\EXPORT\02050711.TXT

Export data for station : Metam Sodium App

Printing Date : 2002/05/16

		RWS (mph)	RWD (Deg)	AT (Deg C)	RH (%)	BP (mmHg)	Sigma (Deg)
05/08/2002	20:15	0.4	19	12.8	73.1	754	9.8
05/08/2002	20:30	1.4	6	12.2	73.6	754	20.8
05/08/2002	20:45	3.1	7	11.8	76.7	754	13.1
05/08/2002	21:00	3.4	54	11.6	77.9	754	17.9
05/08/2002	21:15	4.5	39	11.4	79.4	755	6.1
05/08/2002	21:30	3.5	52	11.1	80.0	755	18.7
05/08/2002	21:45	1.3	53	10.7	80.3	754	24.4
05/08/2002	22:00	2	65	10.5	80.6	754	19.4
05/08/2002	22:15	3	51	10.2	80.9	754	11.2
05/08/2002	22:30	1.7	58	10.2	80.9	754	14.5
05/08/2002	22:45	1.8	65	9.8	82.3	754	20.6
05/08/2002	23:00	2.9	75	9.6	82.6	754	12.6
05/08/2002	23:15	3.6	46	9.4	82.5	754	13.8
05/08/2002	23:30	2.2	75	9.1	83.6	754	17.5
05/08/2002	23:45	1.4	88	9.1	84.6	754	17.6
05/08/2002	24:00:00	2.9	76	9.0	84.1	754	17.9
05/09/2002	0:15	4.3	54	8.9	83.9	754	9.5
05/09/2002	0:30	4.4	40	8.6	84.6	754	7.1
05/09/2002	0:45	2.7	44	8.5	84.7	754	16.8
05/09/2002	1:00	2.5	62	8.3	85.2	753	10.5
05/09/2002	1:15	1.6	64	8.0	84.5	753	42.1
05/09/2002	1:30	2.7	57	8.3	85.8	753	11.6
05/09/2002	1:45	3.2	67	8.3	85.0	753	14.4
05/09/2002	2:00	4.1	53	8.1	85.3	753	9.1
05/09/2002	2:15	3.8	52	8.2	85.4	753	9
05/09/2002	2:30	6	42	8.3	86.2	753	9.6
05/09/2002	2:45	7.4	27	8.1	86.2	753	3.3
05/09/2002	3:00	7.3	20	8.1	86.3	753	5.7
05/09/2002	3:15	7.9	19	8.3	86.8	753	4
05/09/2002	3:30	6.6	23	8.1	86.3	753	7.3
05/09/2002	3:45	4.4	36	7.8	86.4	753	13.5
05/09/2002	4:00	5.4	28	7.8	86.4	753	7.3
05/09/2002	4:15	3.9	33	7.6	86.7	753	17.1
05/09/2002	4:30	2.4	49	7.5	86.3	753	27.4
05/09/2002	4:45	3.4	35	7.7	86.6	753	31.7
05/09/2002	5:00	6	30	7.9	86.2	753	6.6
05/09/2002	5:15	6.7	29	7.8	86.6	753	6.4
05/09/2002	5:30	4.8	41	8.0	86.4	753	13.2
05/09/2002	5:45	3.7	62	8.4	84.5	753	15.8
05/09/2002	6:00	2.9	73	8.7	83.4	753	21.8
05/09/2002	6:15	2.8	94	8.7	84.1	754	15.1
05/09/2002	6:30	2.7	82	9.9	81.5	754	19.9
05/09/2002	6:45	2.1	109	10.6	80.4	754	23.3
05/09/2002	7:00	1.5	185	11.6	78.2	754	49
05/09/2002	7:15	3.1	212	12.4	76.2	754	23.1
05/09/2002	7:30	4.6	199	12.9	74.6	755	22.3
05/09/2002	7:45	3.5	203	13.5	73.5	755	32.7

Export Filename : C:\MICROMET\CARBAP\EXPORT\02050711.TXT

Export data for station : Metam Sodium App

Printing Date : 2002/05/16

		RWS	RWD	AT	RH	BP	Sigma
		(mph)	(Deg)	(Deg C)	(%)	(mmHg)	(Deg)
05/09/2002	8:00	2.3	216	13.8	72.6	755	45.5
05/09/2002	8:15	5.1	223	14.4	71.8	755	26
05/09/2002	8:30	6	215	15.2	71.1	755	27.9
05/09/2002	8:45	8.8	231	15.7	71.4	755	12.7
05/09/2002	9:00	10.1	234	16.0	69.8	756	18.9
05/09/2002	9:15	8.7	239	16.3	68.6	756	15.8
05/09/2002	9:30	7.4	220	16.7	67.1	755	25
05/09/2002	9:45	8.3	220	17.4	65.4	755	18.8
05/09/2002	10:00	8.5	220	17.6	64.3	756	18.8
05/09/2002	10:15	9.4	229	18.0	64.1	756	17.8
05/09/2002	10:30	10.5	241	18.2	63.4	756	12.8
05/09/2002	10:45	12.1	229	18.5	62.9	756	13.4
05/09/2002	11:00	13.3	241	18.8	61.2	756	14.1
05/09/2002	11:15	13.6	231	18.7	63.5	756	15.6
05/09/2002	11:30	14.3	238	18.6	63.7	756	14.7
05/09/2002	11:45	14.7	237	18.4	64.1	756	16.1
05/09/2002	12:00	16.2	237	18.6	62.7	756	13.2
05/09/2002	12:15	18	223	18.3	63.4	755	14.7
05/09/2002	12:30	15.4	230	18.2	63.7	755	11.4
05/09/2002	12:45	16.2	233	18.0	63.4	755	15.3
05/09/2002	13:00	14.5	229	18.2	62.5	755	16.8
05/09/2002	13:15	14.9	237	18.7	58.2	755	17.3
05/09/2002	13:30	15.2	235	18.6	58.8	755	15.7
05/09/2002	13:45	15.2	237	18.5	57.9	755	16.1
05/09/2002	14:00	15.4	238	18.2	59.2	755	15.9
05/09/2002	14:15	15.7	229	18.4	58.5	755	12.8
05/09/2002	14:30	15.6	230	18.1	58.0	755	14.4
05/09/2002	14:45	14.4	222	17.8	56.3	754	12
05/09/2002	15:00	12.6	227	18.0	56.2	754	15.5
05/09/2002	15:15	11.6	231	18.0	57.6	754	19.7
05/09/2002	15:30	11.6	220	17.9	57.0	754	12.8
05/09/2002	15:45	11	225	17.9	55.5	754	17
05/09/2002	16:00	9.5	209	18.1	54.7	754	15.8
05/09/2002	16:15	9	216	18.4	53.9	754	14.2
05/09/2002	16:30	9.1	209	18.2	55.0	754	17.8
05/09/2002	16:45	9.1	213	17.8	56.6	753	17.3
05/09/2002	17:00	9.6	215	17.4	58.8	753	16.7
05/09/2002	17:15	8.6	230	16.8	60.9	753	10.6
05/09/2002	17:30	9.1	219	16.7	61.4	753	12.7
05/09/2002	17:45	9.3	232	16.4	63.2	753	11.2
05/09/2002	18:00	6.5	222	15.8	63.5	753	12.6
05/09/2002	18:15	6.1	213	15.7	64.7	753	10.3
05/09/2002	18:30	6.2	209	15.3	67.6	753	9.8
05/09/2002	18:45	6	219	14.9	69.5	753	12.4
05/09/2002	19:00	5.3	209	14.5	71.8	752	12.3
05/09/2002	19:15	4.8	208	14.3	73.5	752	6.7
05/09/2002	19:30	3.4	214	14.2	73.3	752	13.3

Export Filename : C:\MICROMET\CARBAP\EXPORT\02050711.TXT

Export data for station : Metam Sodium App

Printing Date : 2002/05/16

		RWS (mph)	RWD (Deg)	AT (Deg C)	RH (%)	BP (mmHg)	Sigma (Deg)
05/09/2002	19:45	2.8	190	13.8	74.8	752	14.2
05/09/2002	20:00	3.4	200	13.6	76.4	752	14.9
05/09/2002	20:15	4.9	218	13.6	76.5	752	10.6
05/09/2002	20:30	4.1	207	13.5	77.5	752	10.7
05/09/2002	20:45	2	191	13.2	78.2	752	25.3
05/09/2002	21:00	1.3	118	12.9	79.6	752	31.8
05/09/2002	21:15	1.7	149	12.7	80.0	752	29.2
05/09/2002	21:30	1.1	260	12.6	78.7	752	94.6
05/09/2002	21:45	1.1	92	12.6	79.2	752	22.9
05/09/2002	22:00	0.9	26	12.6	78.2	752	32.5
05/09/2002	22:15	1.5	74	12.5	79.0	752	16.9
05/09/2002	22:30	2.3	21	12.5	78.6	752	22.4
05/09/2002	22:45	1.3	91	12.1	79.8	752	14.3
05/09/2002	23:00	0.8	357	12.0	79.8	752	41.9
05/09/2002	23:15	1.1	272	11.5	83.7	752	27.6
05/09/2002	23:30	0.8	92	11.5	81.0	752	44.4
05/09/2002	23:45	1	47	11.5	81.6	752	56.5
05/09/2002	24:00:00	1.7	39	11.0	81.9	752	15.3
05/10/2002	0:15	2.1	65	10.9	82.2	752	28.3
05/10/2002	0:30	2.4	55	10.7	81.8	751	9.6
05/10/2002	0:45	2.7	71	10.2	83.4	751	18.3
05/10/2002	1:00	3.7	44	10.0	83.9	751	10.7
05/10/2002	1:15	2.2	56	9.6	85.0	751	21.8
05/10/2002	1:30	2.1	59	9.6	85.0	751	11.9
05/10/2002	1:45	3.2	78	9.1	84.8	751	19.7
05/10/2002	2:00	3.8	75	9.0	85.6	751	21.3
05/10/2002	2:15	3.3	63	9.2	85.8	751	18.6
05/10/2002	2:30	3.7	47	8.8	85.1	751	17.8
05/10/2002	2:45	2.8	64	8.7	85.2	751	11.7
05/10/2002	3:00	4.5	45	8.7	86.1	751	9.6
05/10/2002	3:15	4.6	41	8.7	86.4	751	9.8
05/10/2002	3:30	2.7	51	8.6	86.4	751	21.7
05/10/2002	3:45	3.1	47	8.9	86.9	751	13
05/10/2002	4:00	1.1	103	8.1	87.5	751	26.4
05/10/2002	4:15	1.7	58	8.1	88.5	751	25.9
05/10/2002	4:30	3.3	45	8.4	88.1	751	37
05/10/2002	4:45	3.2	21	8.5	88.1	751	18.7
05/10/2002	5:00	1.9	63	8.3	87.7	751	37.5
05/10/2002	5:15	3.6	77	8.1	88.9	751	26.1
05/10/2002	5:30	2.3	58	8.1	86.8	751	27
05/10/2002	5:45	3.8	72	8.3	86.2	752	21.9
05/10/2002	6:00	4.9	42	8.9	86.0	752	11.1
05/10/2002	6:15	5.7	38	9.3	85.6	752	9.9
05/10/2002	6:30	5.2	43	10.2	84.6	752	9.7
05/10/2002	6:45	4	63	11.0	82.5	752	16.3
05/10/2002	7:00	1.8	152	11.7	82.4	752	68.6
05/10/2002	7:15	1.7	176	12.1	82.2	753	38.5

Export Filename : C:\MICROMET\CARBAPI\EXPORT\02050711.TXT

Export data for station : Metam Sodium App

Printing Date : 2002/05/16

		RWS (mph)	RWD (Deg)	AT (Deg C)	RH (%)	BP (mmHg)	Sigma (Deg)
05/10/2002	7:30	3.5	229	12.7	80.3	753	25.2
05/10/2002	7:45	4.1	212	13.1	77.5	753	28.6
05/10/2002	8:00	5.7	204	13.6	75.9	753	17.3
05/10/2002	8:15	5.7	221	14.0	75.1	753	18.9
05/10/2002	8:30	7.8	235	14.4	74.5	754	17.5
05/10/2002	8:45	8.5	229	14.9	71.1	754	17.9
05/10/2002	9:00	8.7	219	15.5	68.3	754	18
05/10/2002	9:15	9.8	211	15.9	66.9	754	17.6
05/10/2002	9:30	11.3	225	16.3	64.8	754	15.4
05/10/2002	9:45	11.7	217	16.5	64.2	754	15.5
05/10/2002	10:00	12.9	222	16.7	63.8	754	15.7
05/10/2002	10:15	13.8	224	16.4	64.8	754	13.6
05/10/2002	10:30	14.1	227	16.7	62.0	754	17.5
05/10/2002	10:45	14.2	243	16.7	60.4	754	17.2
05/10/2002	11:00	13.9	215	16.9	60.6	754	19.2
05/10/2002	11:15	14.3	211	16.9	61.6	754	14.9
05/10/2002	11:30	14.4	222	16.9	60.6	754	13.3
05/10/2002	11:45	15.3	223	17.0	57.6	754	17.8
05/10/2002	12:00	15.4	228	16.9	57.4	754	19.4
05/10/2002	12:15	16	223	17.0	56.1	754	19.7
05/10/2002	12:30	14.8	209	17.2	55.2	753	17.4
05/10/2002	12:45	15	207	17.5	52.2	753	16.4
05/10/2002	13:00	17.2	210	17.3	52.5	753	14.2
05/10/2002	13:15	15.8	206	17.3	57.6	753	14
05/10/2002	13:30	16.8	215	17.0	56.8	753	13.6
05/10/2002	13:45	17	224	17.3	56.9	753	16.3
05/10/2002	14:00	16.8	215	17.2	58.2	753	16.4
05/10/2002	14:15	16.3	227	17.0	57.8	753	16.8
05/10/2002	14:30	15.3	209	17.3	56.8	752	15.8
05/10/2002	14:45	15.5	222	17.0	57.9	752	15
05/10/2002	15:00	15	206	17.2	58.0	752	13.1
05/10/2002	15:15	13.8	198	16.7	59.1	752	16.6
05/10/2002	15:30	13.9	198	16.2	61.6	752	11.6
05/10/2002	15:45	14.2	201	16.1	61.7	752	15.8
05/10/2002	16:00	14.9	207	16.0	62.4	752	14.9
05/10/2002	16:15	14.7	215	15.8	62.3	752	12.1
05/10/2002	16:30	13.1	211	15.8	62.7	752	14.3
05/10/2002	16:45	13.6	208	15.8	63.5	752	13.4
05/10/2002	17:00	13.8	201	15.6	64.2	752	11.7
05/10/2002	17:15	12.7	205	15.3	64.9	752	13.7
05/10/2002	17:30	11.9	206	15.0	65.9	752	12.4
05/10/2002	17:45	10.4	196	14.8	66.7	751	15.6
05/10/2002	18:00	9.1	194	14.6	66.8	751	13.4
05/10/2002	18:15	9.5	187	14.4	67.1	751	12.2
05/10/2002	18:30	8.9	185	14.2	67.7	751	12.5
05/10/2002	18:45	8.6	199	14.0	68.2	751	10.9
05/10/2002	19:00	9.2	192	13.8	68.8	751	11.2

Export Filename : C:\MICROMET\CARBAP\EXPORT\02050711.TXT

Export data for station : Metam Sodium App

Printing Date : 2002/05/16

		RWS (mph)	RWD (Deg)	AT (Deg C)	RH (%)	BP (mmHg)	Sigma (Deg)
05/10/2002	19:15	6.6	196	13.6	69.4	751	12.2
05/10/2002	19:30	4.3	182	13.3	70.3	751	16.3
05/10/2002	19:45	3.8	188	13.1	71.0	751	17.1
05/10/2002	20:00	5.8	217	13.1	71.1	751	10.4
05/10/2002	20:15	5.6	225	12.9	71.7	751	11.6
05/10/2002	20:30	4.3	228	12.5	73.2	751	15
05/10/2002	20:45	3.2	237	12.1	75.6	751	18.1
05/10/2002	21:00	0.9	68	11.8	74.5	751	62.4
05/10/2002	21:15	1.7	39	11.4	75.2	751	15.8
05/10/2002	21:30	3.3	40	11.2	76.5	751	18.8
05/10/2002	21:45	6.5	39	10.6	78.8	751	7.1
05/10/2002	22:00	7.2	40	10.4	79.2	751	4.3
05/10/2002	22:15	4.8	22	10.3	79.5	751	14.8
05/10/2002	22:30	4.6	35	10.2	80.0	751	16.1
05/10/2002	22:45	2.6	77	9.7	81.4	751	37.3
05/10/2002	23:00	5.3	35	9.9	81.0	751	8.8
05/10/2002	23:15	4.7	30	9.7	81.8	751	8.3
05/10/2002	23:30	5.2	23	9.7	81.6	751	9.9
05/10/2002	23:45	3.8	54	9.8	81.7	751	17.1
05/10/2002	24:00:00	3.8	65	9.9	81.4	752	14.7
05/11/2002	0:15	2	107	9.1	82.6	752	43.7
05/11/2002	0:30	2.6	88	9.1	82.8	752	12.4
05/11/2002	0:45	2.3	33	9.0	82.2	752	38.4
05/11/2002	1:00	1.3	52	8.9	82.4	752	43.7
05/11/2002	1:15	2	42	9.1	82.0	752	32.8
05/11/2002	1:30	3.8	56	9.1	81.1	752	24.4
05/11/2002	1:45	5.1	41	8.5	82.8	752	27.8
05/11/2002	2:00	2.7	60	8.4	83.9	751	22.4
05/11/2002	2:15	4	44	8.2	83.9	751	8.8
05/11/2002	2:30	3.4	45	8.0	84.5	751	16.8
05/11/2002	2:45	2.9	51	7.7	84.7	751	27.2
05/11/2002	3:00	1.4	43	7.3	85.6	751	33.6
05/11/2002	3:15	2.8	74	7.3	86.2	751	20.5
05/11/2002	3:30	4.3	24	7.6	86.6	751	5.9
05/11/2002	3:45	4.4	29	7.6	85.9	751	8.1
05/11/2002	4:00	4.4	33	7.5	85.9	751	7.8
05/11/2002	4:15	5.4	38	7.5	85.8	751	8.2
05/11/2002	4:30	6.8	26	7.4	86.0	751	3.3
05/11/2002	4:45	4.9	44	7.1	85.7	751	16.2
05/11/2002	5:00	3.7	58	7.2	85.7	751	11.8
05/11/2002	5:15	3.9	57	7.2	85.5	751	11.5
05/11/2002	5:30	3.6	93	6.7	86.5	751	14.4
05/11/2002	5:45	4.2	52	7.1	85.9	751	41.4
05/11/2002	6:00	2.3	253	7.5	84.0	752	85.2
05/11/2002	6:15	2.6	67	8.5	82.6	752	27.7
05/11/2002	6:30	1.1	306	9.7	81.5	752	49.2
05/11/2002	6:45	2.8	94	10.2	80.8	752	52.9

Export Filename : C:\MICROMET\CARBAPI\EXPORT\02050711.TXT
 Export data for station : Metam Sodium App
 Printing Date : 2002/05/16

		RWS	RWD	AT	RH	BP	Sigma
		(mph)	(Deg)	(Deg C)	(%)	(mmHg)	(Deg)
05/11/2002	7:00	3.1	188	11.2	80.1	753	24
05/11/2002	7:15	4.5	237	11.5	80.1	753	21.2
05/11/2002	7:30	4.3	226	12.1	77.8	753	24.8
05/11/2002	7:45	5.5	230	12.8	75.2	753	13.7
05/11/2002	8:00	7.9	224	13.6	72.8	754	16.3
05/11/2002	8:15	8.9	212	14.2	70.5	754	14.9
05/11/2002	8:30	8.1	212	14.8	68.4	754	18.1
05/11/2002	8:45	7.9	228	15.3	65.7	754	16.1
05/11/2002	9:00	7.6	223	15.8	62.6	754	18.8
05/11/2002	9:15	8.3	217	16.3	61.0	754	13.1
05/11/2002	9:30	10.1	222	16.9	59.6	754	15.9
05/11/2002	9:45	11	228	17.5	58.1	754	16
05/11/2002	10:00	11.6	229	18.0	54.9	754	19.9
05/11/2002	10:15	12.5	238	18.2	51.1	754	16.1
05/11/2002	10:30	12	238	18.5	48.7	754	17.2
05/11/2002	10:45	12.3	240	18.5	48.8	755	14.2
05/11/2002	11:00	11.9	233	19.3	44.4	754	14.3
05/11/2002	11:15	12.4	226	19.4	41.8	754	14.5
05/11/2002	11:30	14.1	236	19.7	38.6	754	12.8
05/11/2002	11:45	15	241	19.9	39.2	754	10.9
05/11/2002	12:00	14.9	235	20.2	37.9	754	12
05/11/2002	12:15	15.6	230	20.4	39.5	754	14.9
05/11/2002	12:30	15.9	222	20.5	37.7	754	12.6
05/11/2002	12:45	16.8	231	20.3	36.6	754	10.5
05/11/2002	13:00	16.4	236	20.3	35.8	754	12.8
05/11/2002	13:15	18.5	236	20.5	35.0	754	10.8
05/11/2002	13:30	20.1	246	20.7	34.0	754	7.8
05/11/2002	13:45	21	242	20.6	34.4	754	8.3
05/11/2002	14:00	20.4	235	20.8	34.1	754	11.7
05/11/2002	14:15	19.2	224	20.5	33.9	754	10.4
05/11/2002	14:30	18.6	238	20.5	34.5	754	13.7
05/11/2002	14:45	18.6	233	20.4	35.2	754	10.8
05/11/2002	15:00	19	236	20.2	36.2	754	9
05/11/2002	15:15	17.2	229	20.4	35.9	754	9.9
05/11/2002	15:30	16.6	228	21.0	31.4	754	11.4
05/11/2002	15:45	15.6	224	21.2	34.7	754	11.7
05/11/2002	16:00	16.1	231	21.0	36.9	754	10.5
05/11/2002	16:15	16.4	235	20.6	38.3	754	9.2
05/11/2002	16:30	15.6	224	20.6	38.9	754	11.3
05/11/2002	16:45	15.1	225	20.0	44.9	754	10.1
05/11/2002	17:00	14.5	228	19.7	47.1	754	9.4
05/11/2002	17:15	13.7	222	19.4	50.0	754	8.8
05/11/2002	17:30	13.1	227	18.7	52.8	754	10.1
05/11/2002	17:45	10.3	217	18.4	54.6	754	16.4
05/11/2002	18:00	9.1	234	17.9	56.1	754	9.6
05/11/2002	18:15	8	229	17.4	58.0	754	9.8
05/11/2002	18:30	7.1	227	17.0	59.5	754	13.6

Export Filename : C:\MICROMET\CARBAPI\EXPORT\02050711.TXT

Export data for station : Metam Sodium App

Printing Date : 2002/05/16

		RWS (mph)	RWD (Deg)	AT (Deg C)	RH (%)	BP (mmHg)	Sigma (Deg)
05/11/2002	18:45	7.4	222	16.6	62.0	754	11.5
05/11/2002	19:00	8.3	231	15.8	65.9	755	10.7
05/11/2002	19:15	4.9	199	15.0	68.8	755	13.9
05/11/2002	19:30	4.2	226	14.8	69.6	755	13.7
05/11/2002	19:45	2.9	274	14.0	73.5	755	14.5
05/11/2002	20:00	1.6	105	13.8	74.1	755	91.6
05/11/2002	20:15	1.3	45	14.0	73.1	755	38.4
05/11/2002	20:30	1.6	72	13.4	72.5	755	28.9
05/11/2002	20:45	4	45	13.2	73.4	755	9
05/11/2002	21:00	5.2	25	13.1	72.4	755	8.7
05/11/2002	21:15	5.6	24	12.8	74.5	755	10.4
05/11/2002	21:30	8	37	12.8	74.8	755	6.2
05/11/2002	21:45	7.8	33	12.5	75.0	755	5.7
05/11/2002	22:00	6.4	40	12.4	75.2	755	7.8
05/11/2002	22:15	8.7	38	12.6	72.7	755	5.8
05/11/2002	22:30	10.4	38	12.5	71.7	755	4.4
05/11/2002	22:45	9.1	41	12.5	69.9	755	6.8
05/11/2002	23:00	9.7	41	12.3	69.4	755	5
05/11/2002	23:15	8.9	37	12.2	69.1	755	6.6
05/11/2002	23:30	8.9	37	12.2	68.4	755	6.2
05/11/2002	23:45	8.5	36	11.9	68.8	755	4.2
05/11/2002	24:00:00	8	33	11.9	68.3	755	5

APPENDIX VI

APPLICATION FIELD LOG SHEETS

CARTRIDGE FIELD LOG SHEET MITC

Project: MITC Application Air Monitoring in ~~San Luis Obispo~~ ^{VENTURA} County

Project #: P-02-001 On Flow: 2.50 ±0.02lpm Off Flow: 2.50 lpm ±10%

2.28 = 2.50 lpm

Log #	Sample Name	Sampler ID Number	Date On	Time On	Counter On	Flow On	Comments	Weather	Initials
			Off	Off	Off	Off		K,P,C,F&R	On
001	MIWBC-FS-1	MA3	05/07/02	0943	527.4	2.28		C	JRS
			5/8/02	0937	551.3	2.08		K	ASm
002	MIWB-1	MN3	05/07/02	0943	188.8	2.28		C	JRS
			5/8/02	0943	212.8	2.04		K	ASm
003	MISBC-FS-1	MO3	5/7/02	1000	213.9	2.28		C	JRS
			5/8/02	1018	238.2	2.20		K	ASm
004	MISB-1	MR3	5/7/02	1000	345.4	2.28		C	JRS
			5/8/02	1018	369.7	2.20		K	ASm
005	MIEBC-FS-1	MT2	5/7/02	1013	205.5	2.28		C	JRS
			5/8/02	1034	229.8	2.16		K	ASm
006	MIEB-1	MP1	5/7/02	1013	112.5	2.28		C	JRS
			5/8/02	1034	136.8	2.15		K	ASm
007	MINBC-FS-1		5/7/02	1025	4.7	2.28		C	JRS
			5/8/02	1054	29.2	2.21		K	ASm
008	MINB-1	MW4	5/7/02	1025	199.9	2.28	BATTERY DOA	C	JRS
			5/8/02	1054	222.8	ND		K	ASm
009	MIW-2	MA-3	5/8/02	0703	551.3	2.28	APPLICATION START TIME: 0720 (PST)	K	ASm
			5/9/02	1810	562.4	2.26		K	ASm
010	MIS-2	MR-3	5/8/02	0708	369.7	2.28	APPLICATION STOP TIME: 1000 (PST)	K	ASm
			5/9/02	1830	381.2	2.26		K	ASm
011	MIEC-2	MP-3	5/8/02	0712	136.8	2.28		K	ASm
			5/9/02	1908	148.7	2.05		K	ASm
012	MIE-2	MT-4	5/8/02	0712	229.8	2.28		K	ASm
			5/9/02	1900	241.6	2.08		K	ASm
013	MIN-2	MU-3	5/8/02	0718	29.2	2.28		K	ASm
			5/9/02	1935	41.4	2.21		K	ASm
014	MINW-2	MN-3	5/9/02	0700	212.8	2.28		K	ASm
			5/9/02	1800	223.9	2.29		K	ASm
015	MINE-2	MW-4	5/9/02	0715	222.8	2.28		K	ASm
			5/9/02	1923	234.9	2.11		K	ASm
016	MISE-2	MQ-4	5/9/02	0710	51.0	2.28		K	ASm
			5/9/02	1850	62.7	2.25		K	ASm
017	MISW-2	MO-2	5/9/02	0705	238.2	2.28		K	ASm
			5/9/02	1820	249.5	2.00		K	ASm

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CARTRIDGE FIELD LOG SHEET

Project: MITC Application Air Monitoring in ^{Ventura} ~~San Luis Obispo~~ County
 Project #: P-02-001 On Flow: 2.50 ±0.02lpm Off Flow: 2.50 lpm ±10%

Log #	Sample Name	Sampler ID Number	Date On	Time On	Counter On	Flow On	Comments	Weather	Initials
			Off	Off	Off	Off		K,P,C,F&R	On Off
018	MINW-3	MN-3	5-09-02	1807	223.8	2.28		K	ash
			5-10-02	0548	235.5	2.04		K	ash
019	MIW-3	MA-3	5-09-02	1818	223.8 574.1	2.28	562.4	K	ash
			5-10-02	0557	574.1	2.17	K	ash	
020	MISW-3	MO-2	5-09-02	1825	249.5	2.28		K	ash
			5-10-02	0610	261.2	2.28		K	ash
021	MIS-3	MR-3	5-09-02	1848	381.2	2.28	BATTERY DIED AS FLOW WAS MEASURED	K	ash
			5-10-02	0615	392.1	2.13		K	ash
022	MISE-3	MQ-4	5-09-02	1857	62.7	2.28		K	ash
			5-10-02	0623	74.1	2.29		K	ash
023	MIE-3	MT-4	5-09-02	1909	241.6	2.28		K	ash
			5-10-02	0630	252.8	2.32		K	ash
024	MIEC-3	MP-3	5-09-02	1917	148.7	2.28		K	ash
			5-10-02	0630	160.0	2.24		K	ash
025	MINE-3	MW-4	5-09-02	1930	234.9	2.28		K	ash
			5-10-02	0645	246.2	2.19		K	ash
026	MIN-3	MV-13	5-09-02	1943	41.4	2.28		K	ash
			5-10-02	0653	52.6	2.26		K	ash
027	MINW-4	MU-3	5-10-02	0553	235.9	2.28		K	ash
			5-10-02	1715	246.8	2.33		K	ash
028	MIW-4	MA-3	5-10-02	0600	574.1	2.28		K	ash
			5-10-02	1725	585.5	2.25		K	ash
029	MISW-4	MO-2	5-10-02	0613	261.2	2.28		K	ash
			5-10-02	1735	272.2	2.20		K	ash
030	MIS-4	MR-3	5-10-02	0620	392.1	2.28		K	ash
			5-10-02	1745	403.5	2.19		K	ash
031	MISE-4	MQ-4	5-10-02	0625	74.1	2.28		K	ash
			5-10-02	1757	85.7	2.13		K	ash
032	MIE-4	MT-4	5-10-02	0635	252.8	2.28		K	ash
			5-10-02	1808	264.3	2.11		K	ash
033	MIEC-4	MP-3	5-10-02	0640	160.0	2.28		K	ash
			5-10-02	1815	168.4	0.70		K	ash
034	MINE-4	MW-4	5-10-02	0648	246.2	2.28		K	ash
			5-10-02	1830	257.9	2.20		K	ash
035	MIN-4	MV-3	5-10-02	0655	52.6	2.28		K	ash
			5-10-02	1840	64.4	2.09		K	ash

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CARTRIDGE FIELD LOG SHEET

Project: MITC Application Air Monitoring in ^{Ventura} San Luis Obispo County
 Project #: P-02-001 On Flow: 2.50 ±0.02lpm Off Flow: 2.50 lpm ±10%

Log #	Sample Name	Sampler ID Number	Date On Off	Time On Off	Counter On Off	Flow On Off	Comments	Weather		Initials	
								K,P,C,F&R On	Off	On	Off
036	MINW-5	MN-3	5/10/02	1720	246.8	2.28		K		OSU	
			5/11/02	0544	259.2	2.20		K		OSU	
037	MIW-5	MA-3	5/10/02	1730	585.5	2.28		K		OSU	
			5/11/02	0552	597.8	2.27		K		OSU	
038	MISW-5	MO-2	5/10/02	1740	272.2	2.28		K		OSU	
			5/11/02	0603	284.9	2.07		K		OSU	
039	MIS-5	MR-3	5/10/02	1752	403.5	2.28		K		OSU	
			5/11/02	0610	415.8	2.10		K		OSU	
040	MISE-5	MQ-4	5/10/02	1802	85.7	2.20		K		OSU	
			5/11/02	0622	98.0	2.00		K		OSU	
041	MIE-5	MT-4	5/10/02	1815	264.3	2.28		K		OSU	
			5/11/02	0632	276.6	2.19		K		OSU	
042	MIEC-5	MP-3	5/10/02	1822	168.4	2.28		K		OSU	
			5/11/02	0640	180.7	2.19		K		OSU	
043	MINE-5	MW-4	5/10/02	1835	257.9	2.28		K		OSU	
			5/11/02	0650	270.2	2.08		K		OSU	
044	MIN-5	MV-13	5/10/02	1848	64.4	2.28		K		OSU	
			5/11/02	0659	76.5	2.09		K		OSU	
045	MINW-6	MN-3	5-11-02	0547	259.2	2.28		K		OSU	
			5-11-02	1855	271.7	Battery dead, unable to measure final flows		K		BI	
046	MIW-6	MA-3	5-11-02	0558	597.8	2.28		K		OSU	
			5-11-02	1702	609.0	2.22		K		BI	
047	MISW-6	MO-2	5-11-02	0607	284.9	2.28		K		OSU	
			5-11-02	1727	296.4	2.38		K		BI	
048	MIS-6	MR-3	5-11-02	0618	415.8	2.28		K		OSU	
			5-11-02	1737	427.3	2.16		K		BI	
049	MISE-6	MQ-4	5-11-02	0626	98.0	2.28		K		OSU	
			5-11-02	1750	109.5	2.29		K		BI	
050	MIE-6	MT-4	5-11-02	0638	276.6	2.28		K		OSU	
			5-11-02	1806	288.2	2.14		K		BI	
051	MIEC-6	MP-3	5-11-02	0645	180.7	2.28		K		OSU	
			5-11-02	1816	192.3	1.83		K		BI	
052	MINE-6	MW-4	5-11-02	0655	270.2	2.28		K		OSU	
			5-11-02	1834	281.9	2.20		K		BI	
053	MIN-6	MV-13	5-11-02	0705	76.5	2.28		K		OSU	
			5-11-02	1841	88.2	2.24		K		BI	

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CARTRIDGE FIELD LOG SHEET

Project: MITC Application Air Monitoring in ^{Ventura} San Luis Obispo County
 Project #: P-02-001 On Flow: 2.50 ±0.02lpm Off Flow: 2.50 lpm ±10%

Log #	Sample Name	Sampler ID Number	Date		Time		Counter		Flow		Comments	Weather		Initials	
			On	Off	On	Off	On	Off	On	Off		On	Off		
054	MINW-7	MN-3	5-11-02		1900		271.7		2.28			K		BI	
			5-12-02		526		282.2		2.07			K		BI	
055	MIW-7	MA-3	5-11-02		1707		609.0		2.28			K		BI	
			5-12-02		535		609.4		N/A		Dead battery upon arrival	K		BI	
056	MISW-7	MO-2	5-11-02		1730		296.4		2.28			K		BI	
			5-12-02		551		308.6		2.00			K		BI	
057	MIS-7	MR-3	5-11-02		1742		427.3		2.28			K		BI	
			5-12-02		603		436.8		N/A		Dead battery upon arrival	K		BI	
058	MISE-7	MQ-4	5-11-02		1756		109.5		2.28			K		BI	
			5-12-02		611		118.7		N/A		Dead battery upon arrival	K		BI	
059	MIE-7	MT-4	5-11-02		1812		298.2		2.28			K		BI	
			5-12-02		626		298.2		N/A		Dead battery upon arrival	K		BI	
060	MIEC-7	MP-3	5-11-02		1823		192.3		2.28			K		BI	
			5-12-02		627		204.4		1.96			K		BI	
061	MINE-7	MW-4	5-11-02		1839		281.9		2.28			K		BI	
			5-12-02		650		292.6		2.28			K		BI	
062	MIN-7	MV-13	5-11-02		1945		88.2		2.28			K		BI	
			5-12-02		656		100.4		2.10			K		BI	
063	Trip Spike #1		5/11/02		1725										BF
64	" #2		5/11/02		1725										BF
65	" #3		5/11/02		1725										BF
66	" #4		5/11/02		1725										BF

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CARTRIDGE FIELD LOG SHEET

MIC

Project: Metam Sodium Application Air Monitoring in ^{Ventura} San Luis Obispo County

Project #: P-02-001 On Flow: 75 ±2ccm Off Flow: 75 ccm ±10%

73 = 75cc

Log #	Sample Name	Sampler ID Number	Date On	Time On	Counter On	Flow On	Comments	Weather	Initials
			Off	Off	Off	Off		K,P,C,F&R On Off	On Off
001	MSW ^{BC} -1FB ^{IB}	MA1	05/07/02	0943	527.4	74		C	JRR
			5/8/02	0943	551.3	68		K	ASH
002	MSWB-1FB	MN1	05/07/02	0943	188.8	73		C	JRR
			5/8/02	0943	212.8	70		K	ASH
003	MSSBC-FS-1FB	MO2	5/7/02	1000	213.9	73		C	JRR
			5/8/02	1018	238.2	4.8		K	ASH
004	MSSB-1FB	MR2	5/7/02	1000	345.4	73		C	JRR
			5/8/02	1018	369.7	2.6		K	ASH
005	MSEBC-FS-1FB	MT4	5/7/02	1013	205.5	73		C	JRR
			5/8/02	1034	229.8	63		K	ASH
006	MSEB-1FB	MP3	5/7/02	1013	112.5	73		C	JRR
			5/8/02	1034	136.8	57.8		K	ASH
007	MSNBC-FS-1FB	MU2	5/7/02	1025	4.7	73		C	JRR
			5/8/02	1054	29.2	2.8		K	ASH
008	MSNB-1FB	MW2	5/7/02	1025	199.9	73	BATTERY DOA	C	JRR
			5/8/02	1054	222.8	ND		K	ASH
009	MSW-2FB	MA-1	5/8/02	0703	551.3	73	APPLICATION START TIME:	K	ASH
			5/9/02	0810	562.4	68.0	0720 (PST)	K	ASH
010	MSS-2FB	MR-2	5/8/02	0708	369.7	73	APPLICATION STOP TIME	K	ASH
			5/9/02	1830	381.2	70.5	1000 (PST)	K	ASH
011	MSEC-2FB	MP-1	5/8/02	0712	136.8	73		K	ASH
			5/9/02	1900	148.7	68		K	ASH
012	MSE-2FB	MT-2	5/8/02	0712	229.8	73		K	ASH
			5/9/02	1900	241.6	74.0			
013	MSN-2FB	MU-2	5/8/02	0718	29.2	73		K	ASH
			5/9/02	1935	41.4	65.7		K	ASH
014	MSNW-2FB	MN-1	5/9/02	0700	212.8	73		K	ASH
			5/9/02	1800	223.8	69		K	ASH
015	MSNE-2FB	MW-1	5/9/02	0715	222.8	73		K	ASH
			5/9/02	1923	234.9	76.0		K	ASH
016	MSSE-2FB	MQ-1	5/9/02	0710	51.0	73		K	ASH
			5/9/02	1850	62.7	68.3			
017	MSSW-2FB	MO-3	5/9/02	0705	238.2	73		K	ASH
			5/9/02	1820	249.5	63.7		K	ASH

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CARTRIDGE FIELD LOG SHEET

Project: Metam Sodium Application Air Monitoring in ^{Ventura} San Luis Obispo County

Project #: P-02-001 On Flow: 75 ±2ccm Off Flow: 75 ccm ±10%

Log #	Sample Name	Sampler ID	Date On	Time On	Counter On	Flow On	Comments	Weather		Initials	
								K,P,C,F&R	On	Off	On
018	MSNW-3FB	MN-1	5/9/02	1807	223.8	73.3	235.5	K		ASH	
			5/10/02	1818	227.8	74.4		K		ASH	
019	MSW-3FB	MA-1	5/9/02	1818	562.4	73		K		ASH	
			5/10/02	0557	574.1	74.9		K		ASH	
020	MSSW-3FB	MO-3	5/9/02	1825	249.5	73		K		ASH	
			5/10/02	0610	261.2	74.2		K		ASH	
021	MSS-3FB	MR-2	5/9/02	1848	381.2	73	BATTERY DIED AS FLOW WAS MEASURED	K		ASH	
			5/10/02	0615	392.1	69.7		K		ASH	
022	MSSE-3FB	MQ-1	5/9/02	1857	62.7	73		K		ASH	
			5/10/02	0623	74.1	67.0		K		ASH	
023	MSE-3FB	MT-2	5/9/02	1917	241.6	73		K		ASH	
			5/10/02	0630	252.8	69.1		K		ASH	
024	MSEC-3FB	MP-1	5/9/02	1917	148.7	73		K		ASH	
			5/10/02	0630	160.0	55.3		K		ASH	
025	MSNE-3FB	MW-1	5/9/02	1930	234.9	73		K		ASH	
			5/10/02	0645	246.2	70.9		K		ASH	
026	MSN-3FB	MU-2	5/9/02	1943	41.4	73		K		ASH	
			5/10/02	0653	52.6	70.5		K		ASH	
027	MSNW-4FB	MN-1	5/10/02	0553	235.5	73		K		ASH	
			5/10/02	1715	246.8	67.0		K		ASH	
028	MSW-4FB	MA-1	5/10/02	0600	574.1	73		K		ASH	
			5/10/02	1725	585.5	68.3		K		ASH	
029	MSSW-4FB	MO-3	5/10/02	0613	261.2	73		K		ASH	
			5/10/02	1735	272.6	69.0		K		ASH	
030	MSS-4FB	MR-2	5/10/02	0620	392.1	73		K		ASH	
			5/10/02	1745	403.5	67.7		K		ASH	
031	MSSE-4FB	MQ-1	5/10/02	0625	74.1	73		K		ASH	
			5/10/02	1757	85.7	67.7		K		ASH	
032	MSE-4FB	MT-2	5/10/02	0635	252.8	73		K		ASH	
			5/10/02	1808	264.3	70.5		K		ASH	
033	MSEC-4FB	MP-1	5/10/02	0640	160.0	73		K		ASH	
			5/10/02	1815	168.4	22.0		K		ASH	
034	MSNE-4FB	MW-1	5/10/02	0648	246.2	73		K		ASH	
			5/10/02	1830	257.9	72.0		K		ASH	
035	MSN-4FB	MU-2	5/10/02	0655	52.6	73		K		ASH	
			5/10/02	1840	64.4	71.7		K		ASH	

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CARTRIDGE FIELD LOG SHEET

Project: Metam Sodium Application Air Monitoring in ^{Ventura} San Luis Obispo County

Project #: P-02-001 On Flow: 75 ±2ccm Off Flow: 75 ccm ±10%

Log #	Sample Name	Sampler ID Number	Date On	Time On	Counter On	Flow On	Comments	Weather	Initials
			Off	Off	Off	Off		K,P,C,F&R On / Off	On / Off
036	MSNW-5FB	MN-1	5/10/02	1720	246.8	73		K	ash
			5/11/02	0544	259.2	78.8		K	ash
037	MSW-5FB	MA-1	5/10/02	1730	585.5	73		K	ash
			5/11/02	0552	597.8	75.0		K	ash
038	MSSW-5FB	MO-3	5/10/02	1740	278.6	73		K	ash
			5/11/02	0603	284.9	73.0		K	ash
039	MSS-5FB	MR-2	5/10/02	1752	403.5	73		K	ash
			5/11/02	0610	415.8	76.8		K	ash
040	MSSE-5FB	MQ-1	5/10/02	1802	85.7	73		K	ash
			5/11/02	0622	98.0	58.0		K	ash
041	MSE-5FB	MT-2	5/10/02	1815	276.6	73	264.3	K	ash
			5/11/02	0632	276.6	67.7		K	ash
042	MSEC-5FB	MP-1	5/10/02	1822	168.4	73		K	ash
			5/11/02	0640	180.7	62.5		K	ash
043	MSNE-5FB	MW-1	5/10/02	1835	257.9	73		K	ash
			5/11/02	0650	270.2	70.0		K	ash
044	MSN-5FB	MU-2	5/10/02	1848	64.4	73		K	ash
			5/11/02	0659	76.5	70.7		K	ash
045	MSNW-6FB	MN-1	5/11/02	0547	259.2	73		K	ash
			5/11/02	1855	271.7	battery dead, unable to measure final flow		K	BI
046	MSW-6FB	MA-1	5/11/02	0558	597.8	73		K	ash
			5/11/02	1702	609.0	68		K	BI
047	MSSW-6FB	MO-3	5/11/02	0607	284.9	73		K	ash
			5/11/02	1727	296.4	66		K	BI
048	MSS-6FB	MR-2	5/11/02	0618	415.8	73		K	ash
			5/11/02	1737	427.3	66		K	BI
049	MSSE-6FB	MQ-1	5/11/02	0626	98.0	73		K	ash
			5/11/02	1750	169.5	66		K	BI
050	MSE-6FB	MT-2	5/11/02	0638	276.6	73		K	ash
			5/11/02	1806	288.2	68		K	BI
051	MSEC-6FB	MP-1	5/11/02	0645	180.7	73		K	ash
			5/11/02	1816	192.3	59		K	BI
052	MSNE-6FB	MW-1	5/11/02	0655	270.2	73		K	ash
		MW-1	5/11/02	1834	281.9	69		K	BI
053	MSN-6FB	MU-2	5/11/02	0705	76.5	73		K	ash
		MU-2	5/11/02	1841	98.2	70		K	BI

CARTRIDGE FIELD LOG SHEET

Project: Metam Sodium Application Air Monitoring in ^{Ventura} San Luis Obispo County

Project #: P-02-001 On Flow: 75 ±2ccm Off Flow: 75 ccm ±10%

Log #	Sample Name	Sampler ID Number	Date On	Time On	Counter On	Flow On	Comments	Weather		Initials
			Off	Off	Off	Off		K,P,C,F&R	On	Off
054	MSNW-7FB	MN-1	5-11-02	1900 1900	271.7 271.7	73		K		BI
			5-12-02	526	282.2	80		K		BI
055	MSW-7FB	MA-1	5-11-02	1707	609.0	73	Dead battery upon arrival	K		BI
			5-12-02	535	609.4	N/A		K		BI
056	MSSW-7FB	MO-3	5-11-02	1730	296.4	73		K		BI
			5-12-02	551	308.6	75		K		BI
057	MSS-7FB	MR-2	5-11-02	1742	427.3	73	Dead battery upon arrival	K		BI
			5-12-02	603	436.8	N/A		K		BI
058	MSSE-7FB	MQ-1	5-11-02	1756	109.5	73	Dead battery upon arrival	K		BI
			5-12-02	611	118.7	N/A		K		BI
059	MSE-7FB	MT-2	5-11-02	1812	288.2	73	Dead battery upon arrival	K		BI
			5-12-02	626	298.2	N/A		K		BI
060	MSEC-7FB	MP-1	5-11-02	1823	192.3	73		K		BI
			5-12-02	627	204.4	62		K		BI
061	MSNE-7FB	MW-1	5-11-02	1839	281.9	73		K		BI
			5-12-02	650	292.6	71		K		BI
062	MSN-7FB	MV-2	5-11-02	1845	88.2	73		K		BI
			5-12-02	656	100.4	71		K		BI
063	Trip Spike #1		5/11/02	1722						BF
064	" #2		5/11/02	1722						BF
065	" #3		5/11/02	1722						BF
066	" #4		5/11/02	1722						BF

APPENDIX VII

JEROME H₂S MONITOR RESULTS

Log #	Sample Name	Date	Pst Time	ppb	Comments + Weather	Initials
001	H ₂ W-1	7/5/02	0950	3	C	BI
002	H ₂ S-1	7/5/02	0956	4	C	BI
003	H ₂ E-1	7/5/02	1009	3	C	BI
004	H ₂ NB-1	7/5/02	1021	2	C	BI
005	H ₂ W-2	7/5/02 8/5/02	0942	3	K	BI
006	H ₂ S-2	5-8-02 5-8-02	1015	3	K	BI
007	H ₂ E-2	5-8-02	1036	3	K	BI
008	H ₂ N-2	5-8-02	1055	2	K	BI
009	H ₂ NW-3	5-9-02	544	2	K	BI
010	H ₂ W-3	5-9-02	549	3	K	BI
011	H ₂ SW-3	5-9-02	653	1	K	BI
012	H ₂ S-3	5-9-02	708	6	K	BI
013	H ₂ SE-3	5-9-02	710	5	K	BI
014	H ₂ E-3	5-9-02	712	4	K	BI
015	H ₂ NE-3	5-9-02	715	4	K	BI
016	H ₂ N-3	5-9-02	718	3	K	BI
017	H ₂ NW-4	5-9-02	1758	3	K	BI
018	H ₂ W-4	5-9-02	1810	1	K	BI
019	H ₂ SW-4	5-9-02	1820	2	K	BI
020	H ₂ S-4	5-9-02	1838	3	K	BI
021	H ₂ SE-4	5-9-02	1847	3	K	BI
022	H ₂ E-4	5-9-02	1910	3	K	BI
023	H ₂ NE-4	5-9-02	1920	2	K	BI
024	H ₂ N-4	5-9-02	1934	2	K	BI

Real-time 5-9-02

* * During application (9:30^{am} - 10:00) Jerome readings were 2-3 ppb taken around perimeter of application area.

Log #	Sample Name	Date	Pst Time	PPb	Comments + Weather	initials
25	H ₂ NW-5	5-10-02	543 543	2	K	BI
26	H ₂ W-5	5-10-02	554 554	1	K	BI
27	H ₂ SW-5	5-10-02	603	1	K	BI
28	H ₂ S-5	5-10-02	611	3	K	BI
29	H ₂ SE-5	5-10-02	622	3	K	BI
30	H ₂ E-5	5-10-02	629	2	K	BI
31	H ₂ NE-5	5-10-02	639	2	K	BI
32	H ₂ N-5	5-10-02	648	1	K	BI
33	H ₂ NW-6	5-10-02	1716	3	K	BI
34	H ₂ W-6	5-10-02	1728	1	K	BI
35	H ₂ SW-6	5-10-02	1738	2	K	BI
36	H ₂ S-6	5-10-02	1750	3	K	BI
37	H ₂ SE-6	5-10-02	1758	3	K	BI
38	H ₂ E-6	5-10-02	1822	3	K	BI
39	H ₂ NE-6	5-10-02	1832	3	K	BI
40	H ₂ N-6	5-10-02	1844	3	K	BI
41	H ₂ NW-7	5-11-02	544	3	K	BI
42	H ₂ W-7	5-11-02	555	1	K	BI
43	H ₂ SW-7	5-11-02	603	0	K	BI
44	H ₂ S-7	5-11-02	614	3	K	BI
45	H ₂ SE-7	5-11-02	621	3	K	BI
46	H ₂ E-7	5-11-02	644	2	K	BI
47	H ₂ NE-7	5-11-02	651	2	K	BI
48	H ₂ N-7	5-11-02	703	3	K	BI

Log #	Sample Name	Date	Pst Time	PPb Comments * Weather	initials
49	H ₂ NW	5-11-02	1906	2	BI
50	H ₂ W	5-11-02	1720	4	BI
51	H ₂ SW	5-11-02	1735	3	BI
52	H ₂ S	5-11-02	1749	3	BI
53	H ₂ SE	5-11-02	1757	3	BI
54	H ₂ E	5-11-02	1831	3	BI
55	H ₂ NE	5-11-02	1839	3	BI
56	H ₂ N	5-11-02	1851	2	BI
57	H ₂ NW	5-12-02	531	3	BI
58	H ₂ W	5-12-02	545	3	BI
59	H ₂ SW	5-12-02	600	3	BI
60	H ₂ S	5-12-02	609	3	BI
61	H ₂ SE	5-12-02	615	3	BI
62	H ₂ E	5-12-02	637	4	BI
63	H ₂ NE	5-12-02	654	3	BI
64	H ₂ N	5-12-02	704	3	BI

APPENDIX VIII

JEROME H₂S CALIBRATION REPORT

ARB Calibration Report - Jerome Hydrogen Sulfide

Calibration Summary:

ID Information:

Station Name:	Metam Sodium Application	Manufacturer:	Jerome	AS-FOUND:	X
Site #:	Sacramento Warehouse	Model #:	631-X	AS-LEFT:	
Station Address:	New Cuyama	Property #:	1165	Calibration Date:	04/18/02
Agency:	ARB	Trnsfr Std Flow:		Report Date:	04/18/02
		Corrected Flow:	-0.05	Previous Cal. Date:	04/15/02

Calibration Info.:
Calibration Results:

Component:	H2S
Instrument Range (ppb):	0 to 50,000
AS-FOUND Percent Deviation From True (%):	12.8%
AS-FOUND Change From Previous Cal. (%):	N.A.
Slope:	0.857
Intercept:	3.809
Correlation:	0.99992
AS-FOUND Meets $\pm 15\%$ Requirement:	YES

Meteorology:

Temperature ($^{\circ}$C):	25.0
Elevation (Feet.):	25
Pressure (mmHg):	759.8

Compressed Gas Cylinder:

Cylinder #:	JJ732
Assay (ppm):	20.7
Certified Date:	10/19/01
Date Cert. Expires:	10/18/03
Pressure Out (psi):	15

Calibration Standards:

Standard:	I.D. #:	Cert. Date:	Certification Slope:	Intercept
Dasibi 5009 Calibrator 0-10 slpm MFC:	20004557	03/12/02	0.9823	0.0048
Dasibi 5009 Calibrator 0-100 sccm MFC:	20004557	03/12/02	1.0095	0.5042
CSI Clean Air Pack for Dilution Air:	20004749	N.A.	Outlet Pressure:	20
Tylan 0 - 3 slpm MFM	20003937	04/04/02	1.0954	-0.0464

Calibration Data:

Calibration Data:

SO2 Gas Flow		Transfer Standard		Total Flow sccm	SO2 Out ppb	Jerome Reading ppb
Display	sccm	Display	Dilution Flow sccm			
0.0	0.5	9.18	9022.3	9022.8	0	0
90.7	92.1	9.18	9022.3	9114.4	209	240
60.7	61.8	9.18	9022.3	9084.1	141	160
30.7	31.5	9.18	9022.3	9053.8	72	78
10.7	11.3	9.18	9022.3	9033.6	26	27
0.0	0.5	9.18	9022.3	9022.8	0	0

Dial Setpoints:

Zero:	N.A.
Span:	N.A.

Hydrogen Sulfide Regression Data

Percent Difference:

14.8%
13.7%
8.3%
4.2%

Regression Results:

x Coefficient (Slope):	0.8565
y Constant (Intercept):	3.8090
Number of Observations:	4
Correlation:	0.999920

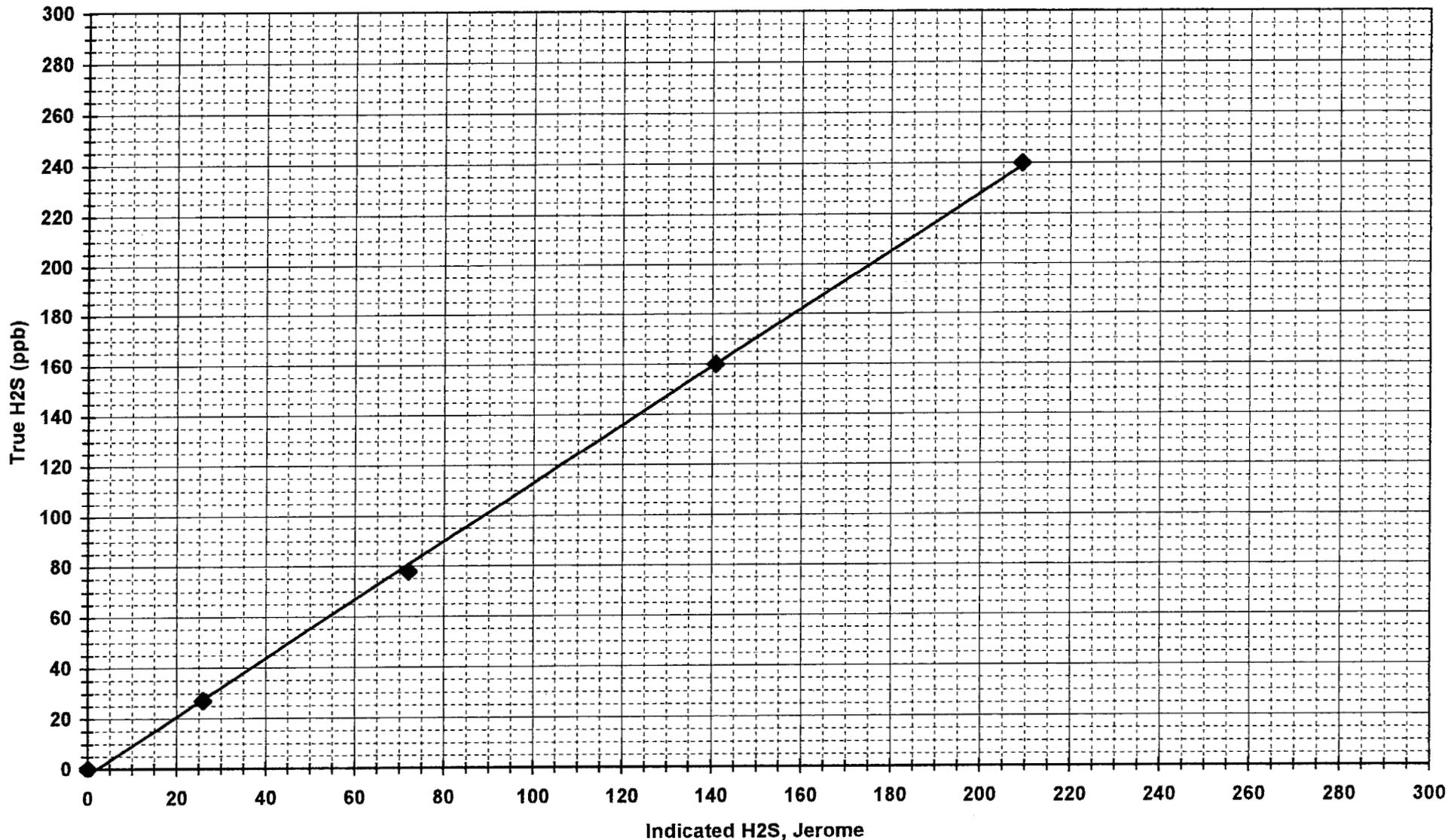
Corrected SO2:

(Display * x) + y
209.38
140.86
70.62
26.94

Comments:	Some irregular readings occurred during the 72 ppb step. Registered 11 ppb 2 of 5 times.		
Calibrated by:	Steve Rider		Checked by:

CALIBRATION GRAPH (AS-FOUND)

Metam Sodium Application Hydrogen Sulfide Analyzer
Station Number: Camarillo Sensor Number: 1165 Cal. Date: 04/19/02



— Linear (True H2S = (Jerome * 0.857) + 3.809)

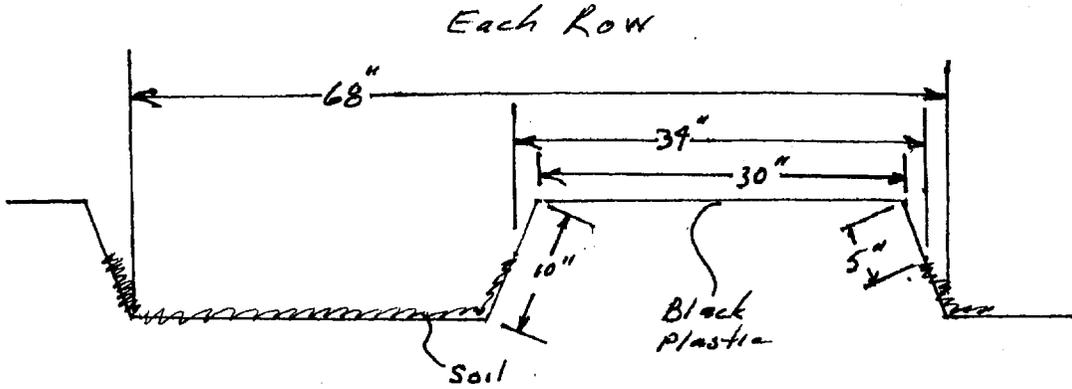
APPENDIX IX

NOTES ON FIELD MEASUREMENTS

Metam Sodium Application
Ventura County
Project #: P-02-001

Row #	Length (inches)						
1	7452	22	8564	43	9691	64	11273
2	7548	23	8606	44	9739	65	11356
3	7628	24	8652	45	9779	66	11420
4	7716	25	8684	46	9830	67	11418
5	7780	26	8758	47	9902	68	11408
6	7836	27	8803	48	9969	69	11404
7	7886	28	8892	49	10055	70	11400
8	7926	29	8933	50	10125	71	11382
9	7986	30	9006	51	10125	72	11364
10	8052	31	9063	52	10281	73	11356
11	8082	32	9120	53	10308	74	11349
12	8124	33	9187	54	10308	75	11342
13	8168	34	9242	55	10521	76	11334
14	8217	35	9290	56	10545	77	11326
15	8263	36	9346	57	10578	78	11317
16	8314	37	9405	58	10744	79	11309
17	8358	38	9463	59	10830	80	11300
18	8400	39	9511	60	10916	81	11292
19	8440	40	9570	61	10998	82	11031
20	8482	41	9616	62	11078	83	10959
21	8520	42	9670	63	11084	84	10916

67,935 ft



Each Row

Total plastic width = $30'' + 2(5'') = 40''$

Total plastic area = width x (row length less 30' row length for road in middle of field)

$$= 40'' (67,935' - \frac{30'}{\text{row}} \times 84 \text{ rows})$$

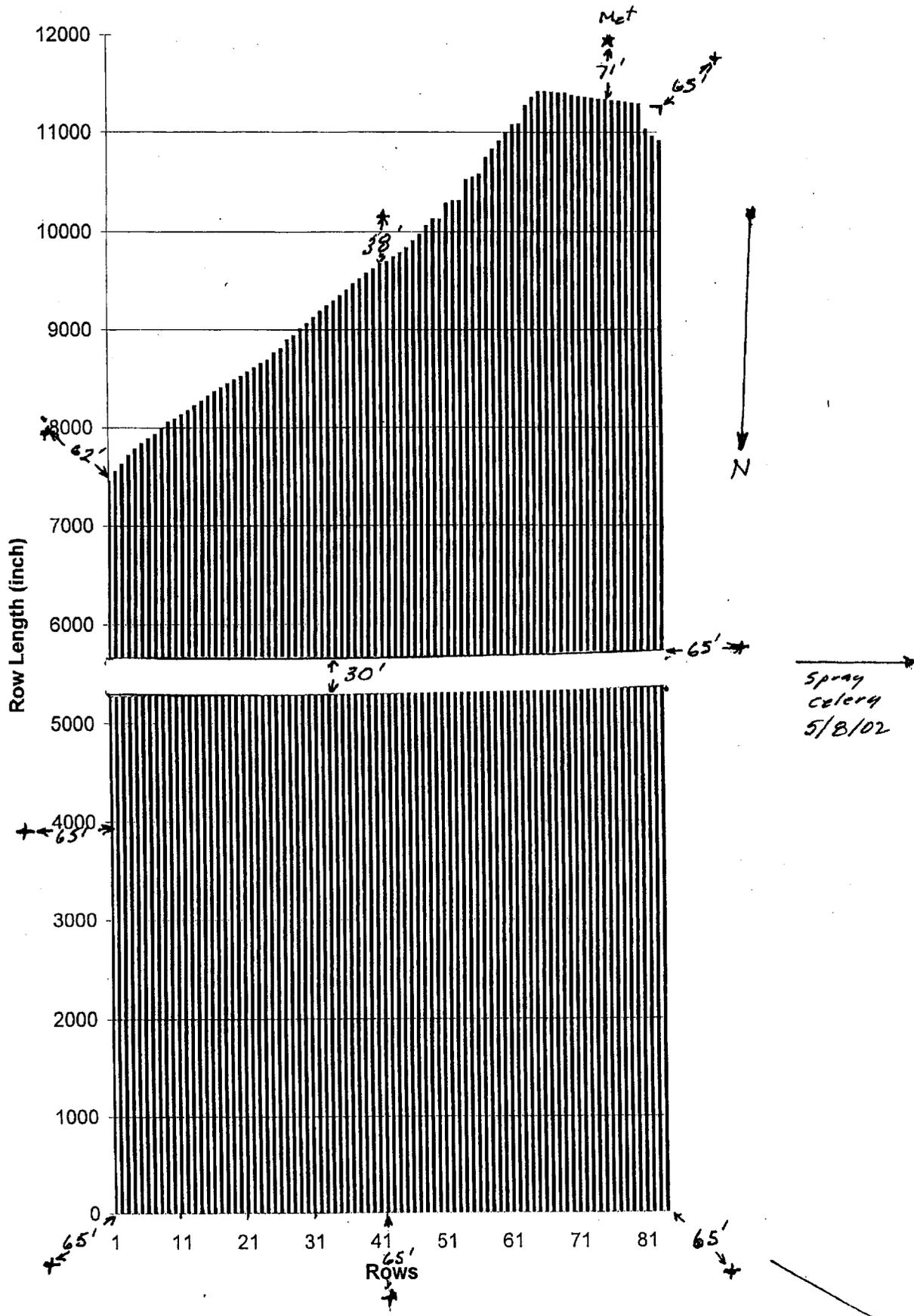
$$= 218,050 \text{ ft}^2 \approx 5.01 \text{ acres}$$

Field area = total length x width

$$= 67,935 \text{ ft} \times 68''$$

$$= 387,965 \text{ ft}^2 \approx 8.87 \text{ acres}$$

Metam Sodium Application
 Ventura County
 Project #: P-02-001



123 Plant Beans