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MEMORANDUM

TO: Doug Okumura
Acting Assistant Director
Department of Pesticide Regulation

FROM: William Loscutoff, Chief
Monitoring and Laboratory Division

DATE: March 3, 1999

SUBJECT: QUALITY ASSURANCE EVALUATION OF LOMPOC PESTICIDE
MONITORING



In October 1998, the Department of Pesticide Regulation (DPR) requested that staff of the Air Resources Board (ARB) conduct an evaluation of the laboratories involved in the 1998 Lompoc pesticide monitoring program. Findings and recommendations from the laboratory evaluations are in the enclosed report.

The laboratory evaluations consisted of reviewing quality assurance procedures and documentation, and an on-site inspection of each laboratory. Quality Assurance Section staff of the ARB were assisted by Mr. Lynn Baker from the ARB's Project Support Section, Mr. Mathew Plate from the U.S. Environmental Protection Agency Region IX, and Ms. Kathy Orr from the DPR's Worker Health and Safety Branch.

If you have any questions, please call me at (916) 445-3742.

Enclosure

T10N2QFB/DLF

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STATE OF CALIFORNIA
MONITORING AND LABORATORY DIVISION
QUALITY ASSURANCE SECTION

LOMPOC PESTICIDE MONITORING PROGRAM - PHASE I

SYSTEM EVALUATION REPORT

OF

TRACE ANALYTICAL LABORATORY
UNIVERSITY OF CALIFORNIA, DAVIS,

CENTER FOR ENVIRONMENTAL SCIENCES AND ENGINEERING
UNIVERSITY OF NEVADA, RENO,

AND

INORGANICS LABORATORY SECTION
AIR RESOURCES BOARD

MARCH 3, 1999

LOMPOC PESTICIDE MONITORING PROGRAM - PHASE I

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I. EXECUTIVE SUMMARY

In 1997, the California Department of Pesticide Regulation (DPR) formed an interagency working group to investigate respiratory illnesses in Lompoc. In 1998, the DPR coordinated ambient air monitoring of the area for 12 specific pesticides as well as certain metals which could possibly indicate elevated levels of 3 metal-based pesticides. The data for the 12 specific pesticides were intended to be quantitative; the metals data were expected to be a possible indicator of high usage of the 3-metal-based pesticides. The monitoring was conducted between August 17 and September 14, 1998. The DPR requested that the Quality Assurance Section (QAS) of the Air Resources Board (ARB), in conjunction with staff from the United States Environmental Protection Agency (U.S. EPA) Region IX and the DPR's Worker Health and Safety Branch, conduct a system audit of the participating laboratories by reviewing quality assurance/quality control (QA/QC) procedures. The request was not finalized until after completion of the monitoring and after nearly all of the analyses were completed. This review was not typical of a QAS audit; no performance audits were conducted, no set criteria for QA/QC were established prior to the study, ambient data could not be revealed or compared in this report at the time of the first drafts, and the reviewers were not asked to participate until most of the work was completed. Therefore, this report should be considered to be an evaluation, not an audit.

The purpose of the system evaluation was to evaluate the practices and procedures used by each laboratory which might lessen or increase the validity of the data produced, as well as to recommend any practices and procedures that could be improved in future studies. The system evaluations consisted of: 1) a review of documentation (e.g., analytical protocol, calibrations, etc.); 2) a review of questionnaires completed by each laboratory about their procedures; and 3) on-site inspections (December 15-17, 1998) of the laboratories, including review of calibration documents, logbooks, chain-of-custody forms, and QA/QC records.

Three laboratories were involved in the analysis of the collected samples: the Trace Analytical Laboratory at the University of California, Davis (UCD), the Center for Environmental Sciences and Engineering at the University of Nevada, Reno (UNR), and the Inorganics Laboratory Section of the ARB. The UCD undertook the majority of the analytical work by reporting on the levels of 10 specific pesticides. The UNR was responsible for analysis of samples for methyl bromide and methyl isothiocyanate (MITC), the primary breakdown product of metam sodium. The ARB's task was to analyze particulate air samples for the metals usually screened in the ARB's statewide ambient air toxic monitoring network.

Lack of funding prevented the DPR from establishing contracts, which define data objectives, analytical requirements, and QA/QC procedures, with the participating laboratories until immediately prior to the initiation of sampling. Laboratory and field sampling personnel should have had contracts well before any monitoring was scheduled to occur to allow adequate time for preparations. Each laboratory had its own level of accountability for the data it produced which resulted in varied QA/QC procedures.

The pesticide monitoring results produced by the UCD are of good quality. Good laboratory practices were used, with minor deficiencies.

The MITC data and the expected results for methyl bromide from the UNR seem questionable because of sample handling practices and insufficient QA/QC safeguards. No samples were shipped to the laboratory until after all samples were collected. The UNR stability studies, as reported in their contract with the DPR, indicated 80-90% recovery of methyl bromide after 3-5 months storage at -20°C. Spikes provided by the DPR will be analyzed after the field samples to confirm this previous study. An earlier DPR study (March 1996) reports an average recovery of 62% of methyl bromide after 4 weeks storage. National Institute of Occupational Health and Safety (NIOSH) Method 2520 reports recoveries of methyl bromide at less than 70% after storage at -10°C for 6 days.

The UNR contract with the DPR also reported 79% recovery of MITC after 2+ months storage at -20°C. Concurrent stability studies indicated greater than 80% MITC recovery after about 2 months storage at -20°C. Stauffer Chemical Company Method RRC-82-35 reported an average recovery of 85% after 14 days storage under refrigeration.

This raises the question of the storage stability of both compounds, especially methyl bromide. Additionally, chain-of-custody forms were not used, only a single point flow verification was performed by the UNR of the sampling flow meters (this is recommended, not required), which were calibrated by the factory, and there was incomplete laboratory documentation of sample handling and laboratory practices. It should be noted, however, that no applications of methyl bromide were made in the area and the samples were collected solely for background purposes.

The QA/QC practices were sufficient for the use of the ARB's metals data as an indicator of the presence of metals in the environment. A qualitative assessment was the intent of analyzing the metals in this study.

II. INTRODUCTION

Since the mid-1990s, citizens living in the Lompoc area have expressed concern about respiratory illnesses in their community. The Office of Environmental Health Hazard Assessment has documented higher than expected rates of lung cancer and other respiratory illnesses in Lompoc. As a result, the DPR formed an interagency working group to investigate the illnesses. Legislation was passed that funded ambient air monitoring of pesticides in Lompoc; however, because of delays in passing the State budget, contracts were not approved until August 11, 1998. The DPR coordinated the monitoring for 12 specific pesticides as well as certain metals which could possibly indicate elevated levels of 3 metal-based pesticides. The data for the 12 specific pesticides were intended to be quantitative; the metals data were expected to be a possible indicator of high usage of the 3 metal-based pesticides. The monitoring was conducted between August 17 and September 14, 1998.

Three laboratories were involved in the analysis of the collected samples: the Trace Analytical Laboratory at the UCD, the Center for Environmental Sciences and Engineering at the UNR, and the Inorganics Laboratory Section of the ARB. The UCD undertook the majority of the analytical work by reporting on the levels of 10 specific pesticides: alachlor, chlorothalonil, chlorpyrifos, diazinon, dimethoate, disulfoton, fenamiphos, fonofos, oxydemeton-methyl, and permethrin. The UNR was responsible for analysis of samples for methyl bromide and methyl isothiocyanate (MITC), the primary breakdown product of metam sodium. At the time of the on-site evaluation, none of the methyl bromide samples had been analyzed. The ARB's task was to analyze particulate air samples for the metals usually screened in the statewide ambient air toxic monitoring network.

The DPR requested that the QAS of the ARB, in conjunction with staff from the U.S. EPA Region IX and DPR's Worker Health and Safety Branch, conduct a system audit of the participating laboratories and to review QA/QC procedures. Questionnaires were mailed by the QAS to the laboratories on October 20, 1998, and on-site inspections were conducted at each laboratory during the week of December 14-18, 1998.

The request for the QAS participation was not finalized until after completion of the monitoring and after nearly all of the analyses were completed. The review that follows, therefore, is not typical of a QAS audit; no performance audits were conducted, no consistent QA/QC criteria existed to guide the study, ambient data could not be revealed or compared in this report at the time of the first drafts, and the reviewers were not engaged formally until most of the work was completed. This report more closely follows practices typical of an evaluation rather than a rigorous audit.

Discussions of the work to be conducted began with the laboratories in early spring of 1998. The contracts, which funded the work and detailed specific requirements for QA/QC procedures, were not approved until one week before sampling began. The UCD undertook all of the preliminary QA/QC work (minimum detection limits, storage stability, trapping efficiencies, etc.) without funding. The UNR's contract appeared to have been based on earlier contracts and the QA/QC procedures outlined were not generally suited for this monitoring program. The ARB had no contract; the laboratory was verbally requested to handle the samples per its normal ambient air toxic sampling program protocols. Each laboratory had its own level of accountability for the data it produced. This resulted in the use of varied QA/QC procedures.

III. EVALUATION OBJECTIVES

The purpose of the system evaluation was to assess the practices and procedures used by each laboratory which might lessen or increase the validity of the data produced, and to help determine uncertainty or variability with the data. The evaluation team considered the manner in which the data would be used and how well the laboratory fulfilled the expectations of the objective in arriving at its conclusions. The system evaluation consisted of:

1. Review of documentation (e.g., analytical protocol, calibrations, etc.);
2. Review of questionnaires completed by each laboratory about their procedures; and
3. On-site inspections of laboratories, including review of calibration documents, logbooks, chain-of-custody forms, and QA/QC records.

Also of major interest were the practices and procedures that could be modified to improve the usefulness of the data in future studies. To this end, the evaluation team reviewed the field sampling QA/QC procedures, as known, and made comments/recommendations. The evaluation team was not asked to evaluate the QA/QC procedures used during field sampling; however, certain practices were made evident during the laboratory evaluation. When these practices affected data quality, the evaluation team made appropriate comments. The purpose of this system evaluation was not to evaluate each of these laboratories by a pre-set standard of QA/QC criteria (e.g., "good laboratory practices" criteria or the ARB's "Quality Assurance Plan for Pesticide Monitoring") since each laboratory has its own QA/QC objectives based on its individual data needs.

IV. FINDINGS

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The resin (XAD-4) samples were collected by a private contractor and delivered to the UCD for analysis by one of two methods. Flame photometric detection (FPD) and mass spectrometry detection (MSD) were used in conjunction with gas chromatography (GC). Some samples were oxidized prior to analysis, others extracted from the sample medium and then analyzed directly.

The data produced by the UCD were found to be reliable and followed good QA/QC procedures. The QA/QC procedures were in place to support the laboratory's normal workload. Good QA/QC procedures were used by field sampling and handling personnel. Rotometers used to regulate sample flow rates were calibrated prior to use in the field and after the monitoring was completed. Samples were stored on dry ice, and the laboratory noted that dry ice was always present with the samples upon receipt at the laboratory. Data loggers to record temperatures in the ice chests were used, but failed to operate properly. Samples were shipped to the laboratory by Federal Express on a weekly basis. Log sheets were used to identify each sample, list start/stop times, and note unusual circumstances. Chain-of-custody forms were assigned to all samples collected; however, the water-soluble ink used smeared when the forms became wet. This did not significantly affect the legibility of the forms. Field blanks were provided with each batch of samples sent to the laboratory.

Overall, the UCD performed very well. Prior to the monitoring program, storage stability, breakthrough, detection limits, and collection efficiency studies were conducted. All of the preliminary QA/QC studies were completed prior to sampling; however, some of the information

on stability studies and trapping efficiencies had not been reported before analysis began. While this did not compromise the results, these data should have been available for review by the study director prior to the start of the study. All samples were analyzed within the time period confirmed by the stability studies. All standards were prepared from neat (pure), certified materials. One discrepancy was that the certification for the standard, fonofos, had expired on August 26, 1998, in the middle of the sampling period. It is unlikely that the standard had degraded to a significant degree. The other standards were within the certification period.

All samples were checked against field data sheets and chain-of-custody forms when received at the laboratory. Samples were extracted and prepared for analysis within one day of receipt at the laboratory. Replicate analyses were run on all samples. Concurrent field and trip spikes averaged over 90% recovery and collocated samples averaged a difference of less than 10%.

Calibration curves were run with each batch of samples analyzed, and the highest and lowest level standard bracketed any pesticide detected. The calibration acceptance criterion used by the laboratory is an "r" squared of 0.90. The U.S. EPA recommends (EPA SW846, Method 8000b "Determinative Chromatographic Separations," Section 7.5.2) an "r" of 0.99 ("r" squared of 0.98) for quantitative results. All of the observed calibrations for GC/MS met this criterion, but some of the GC/FPD calibrations did not. This lower "r" squared value may have resulted in increased variability in some of the GC/FPD data. All detected pesticides were confirmed with mass spectrometry. By the sample labeling, the UCD knew which samples were collocated, blanks, and spikes; however, they did not know the spike level.

Field spike samples were run with each batch of field samples. Samples were stored in a locked freezer that had a temperature recorder. All documentation was present and available. Chain-of-custody forms were used, and logbooks for instruments and bench work were present with pertinent data recorded. Calibration and sample data were recorded and archived. The in-house QA manager observed the personnel prepare and analyze the samples on at least one occasion.

In addition to the pesticides requested for analysis, the UCD also reported results for cycloate. A calibration standard which was past the certification date was used for quantitation. Because some of the standard may have degraded, cycloate results may be lower than the true air concentrations. Confirmation of cycloate was established through mass spectrometry.

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Samples were collected by a private contractor and delivered to the laboratory. The samples analyzed for MITC were extracted from charcoal sampling tubes with an organic solvent and measured with a nitrogen/phosphorous detector (NPD) in conjunction with GC. Methyl bromide samples were also collected in charcoal tubes. At the time of the on-site evaluation, plans were for the samples to be extracted with benzyl alcohol and measured with an electron capture detector (ECD) in conjunction with GC. Those samples are expected to be analyzed in February 1999.

The UNR laboratory generally supports one-time research projects and does not necessarily require the same QA/QC procedures as would be typical of a monitoring program. Field QA/QC procedures were not as complete or comprehensive as those used by UCD. The flow meters used were purchased just prior to the study and calibrations were provided by the manufacturer. A single point calibration was performed in-house by the laboratory. The flow meters used in the study, along with the calibration data, were apparently lost in the field and not returned to the UNR. All of the UNR's samples were stored in the field in an ice chest with dry ice until the sampling was completed. The samples were returned to Sacramento with the sampling equipment and stored at the DPR's West Sacramento facility. They were shipped to the UNR in two batches; the last shipment was not received by the UNR until October 16, 1998. Field log sheets were sent with the samples, but no chain-of-custody forms accompanied the samples.

Clear goals and expectations for this study were not established between the DPR and the UNR prior to the start of this monitoring program. The contract between the DPR and the UNR, which should have detailed the QA/QC procedures, was based on an earlier study done by the UNR for MITC and did not contain QA/QC procedures appropriate for this study. The samples were not checked against the log sheet when they arrived at the laboratory due to concerns by the analyst for degradation of the samples if they warmed up to room temperature. They were checked when removed from the freezer for analysis and at that time three samples were not accounted for. All samples appeared to be frozen, and dry ice was present when they were received at the laboratory. Samples were stored in an unlocked freezer. Only one field blank accompanied the MITC samples. No spikes were provided for MITC. Samples were not extracted until just prior to analysis, which was often more than a month after receipt. None of the methyl bromide samples had been analyzed by the time of the on-site evaluation, December 17, 1998. Methyl bromide spikes were prepared for the UNR by the DPR. No bound laboratory notebooks with numbered pages for recording sample data were maintained. Notations were made on the computer-generated data sheets. Laboratory blanks and spikes were not requested by the DPR nor prepared. Preliminary QA/QC work (detection limits, stability studies, trapping efficiencies, etc.) was not conducted. The preliminary QA/QC values were based on earlier studies, but no confirmation work was done prior to analysis. Replicate injections of samples were not done to establish method precision. The UNR confirmed MITC results with a separate method and plans to do the same with the methyl bromide samples after completion of analysis.

The UNR stability studies, as reported in their contract with the DPR, indicated 80-90% recovery of methyl bromide after 3-5 months storage at -20°C . Spikes provided by the DPR will be analyzed after the field samples to confirm this previous study. An earlier DPR study (March 1996) reports an average recovery of 62% of methyl bromide after 4 weeks storage. National Institute of Occupational Health and Safety (NIOSH) Method 2520 reports recoveries of methyl bromide at less than 70% after storage at -10°C for 6 days.

The contract of the UNR with the DPR also reported 79% recovery of MITC after 2+ months storage at -20°C . Concurrent stability studies indicated greater than 80% MITC recovery after about 2 months storage at -20°C . Stauffer Chemical Company Method RRC-82-35 reported an

average recovery of 85% after 14 days storage under refrigeration.

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Teflon filter samples collected by a private contractor were delivered to the ARB and analyzed by non-destructive Energy-Dispersive X-ray Fluorescence (XRF) Spectroscopy for metals. Approximately 30 elements can be determined by this method. Of primary interest for this study were manganese, aluminum, and silicon.

All samples (filters) were received with a chain-of-custody form. A written Standard Operating Procedure (SOP) for Energy-Dispersive X-ray Fluorescent Spectroscopy was followed (MLD034). The ARB's SOP referenced the Desert Research Institute (DRI) XRF SOP #2-205.2 which was the basis for ARB's SOP. Detailed quality control data for the analysis period (Third Quarter 1998) are found in the ARB's Inorganics Laboratory Section Quality Control Report, Section VI, page 20, December 21, 1998. No independent method validation was found in either the ARB's SOP or DRI's SOP. Control samples were run with the field samples and within specified limits. Replicate analyses of the samples in this study compared favorably, considering the use of these data. For the 3 replicate runs, values ranged from +65% to -10% for the 3 elements (Al, Si, and Mn) of concern, excluding one pair where manganese was detected in one run, but not the duplicate run.

Some of the collocated results in disagreement may have been due to a leak in one of the collocated samplers. The ARB sampling protocol specified leak checks prior to sampling, but not at the conclusion of the sampling program.

Lack of space in the laboratory was noted, especially for sample storage. Dedicated laboratory books were not used for instrument conditions and maintenance. Internal review of QA/QC procedures consisted of a review of the results. Spiked samples are not employed in this method per the ARB's Quality Control report. The ARB sampling protocol specified that a blank filter was to accompany the shipment of samples from the field to the laboratory. No blank filter was provided to the laboratory.

V. RECOMMENDATIONS AND COMMENTS

If Phase 2 of the monitoring program is undertaken, it is recommended that an audit be performed prior to any monitoring. The audit team should be given ample time to review contracts with data objectives and QA/QC procedures outlined, so that recommendations can be implemented prior to the study.

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A contract with the UCD that included data objectives and QA/QC requirements should have

been available and reviewed by the UCD ahead of any proposed sampling. The UCD appeared to have a very good QA/QC program in place; however, the QAS recommends that a formal system of internal QA/QC audit and corrective action be implemented by the QA manager. Calibration linearity should have a high correlation ($r^2 = 0.98$). All calibration standards should have been used within their certification time period. A reliable method of verifying the minimum and maximum temperatures in the sample ice chests should have been used. Field personnel should have been instructed to use permanent ink which would not have smeared if it became wet.

The stock solution used to prepare spike (fortified) samples should have been from a different source than the standard used to prepare calibration standards. If that was not possible, it would have been preferred that the spike samples be prepared from an independent stock solution. This second stock solution should have been compared to the calibration standards. Blank and spike samples should have been labeled to make them indistinguishable from field samples. Overall, the UCD used good QA/QC practices and all of the data produced are reliable and of good quality. The possible variability of some of the GC/FPD data, due to a low correlation for calibration standards, should be considered in interpreting the data.

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As mentioned above, a contract, including data objectives and all QA/QC requirements, should have been completed and reviewed ahead of the proposed sampling. Flow meters should have been verified with multi-point calibrations independent of the manufacturer's certification, but this is not a requirement. Field samples should have been sent in a timely manner to the laboratory accompanied by a chain-of-custody form. Some method of verifying the minimum and maximum temperatures in the sample ice chests should have been used. Samples should have been checked against chain-of-custody sheets upon arrival at the laboratory so any discrepancies could have been immediately investigated. Samples should have been analyzed as soon as possible after receipt at the laboratory, or extracted from the sampling medium until analysis could have been performed. Laboratory blanks and spikes should have been prepared and analyzed at the same time as the field samples. A bound, numbered laboratory book should have been used to record sample information and unusual occurrences. Detection limits, stability studies, trapping efficiencies, etc. should have been conducted prior to sampling, or if based on earlier studies, at least confirmed prior to sampling. Replicate injections of some samples should have been done to establish precision of the method. It is recommended that storage facilities be secured in order to maintain the integrity of the samples.

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A written statement of work or agreement, including data objectives and all QA/QC requirements, should have been completed and reviewed ahead of any proposed sampling. Although not affecting the XRF data quality, it is recommended that the ARB update its SOP for XRF to include additional information on detection limits, stability studies, trapping efficiencies, linearity, recovery levels, reproducibility, method validation, and calibration frequency, or clearly reference the documents where this information is available. A separate, bound, numbered laboratory book

should have been used for instrument conditions and maintenance. Adequate laboratory space for storage of samples is needed to eliminate the potential for mishandling filters. A blank sample filter should have been provided to the laboratory and analyzed at the same time as the field samples. The other purpose of these data, to evaluate silicon levels, may have significant variability, as this method has a low degree of accuracy due to silicon's atomic number and the silica particle size variations. Finally, field samplers should have been leak checked at the beginning and end of each sampling run.

VI. CONCLUSION

Lack of early funding prevented the DPR from establishing contracts with the participating laboratories to define data objectives, analytical requirements, and QA/QC procedures, until immediately prior to the initiation of sampling. Laboratory and field sampling personnel should have had contracts established among participants before monitoring occurred. Each laboratory had its own level of data accountability and this resulted in varied QA/QC procedures. No performance audits were conducted on the field sampling equipment or on the analytical methodology.

The UCD provided field, trip and laboratory spikes as part of its internal QA/QC program. The evaluation team believes the pesticide monitoring results produced by the UCD are of good quality. Good laboratory practices were used, with only minor deficiencies.

The MITC data and especially the expected results for methyl bromide from the UNR seem questionable due to sample handling practices and insufficient QA/QC safeguards. No samples were shipped to the laboratory until after all samples were collected, and the samples may have been stored longer than the time covered by the stability studies. The different stability study results raises the question of the storage stability of both compounds, especially methyl bromide. Additionally, chain-of-custody forms were not used, only a single point flow verification of the sampling flow meters was performed by the UNR, and there was incomplete laboratory documentation. It should be noted, however, that no applications of methyl bromide were made in the area and the samples were collected solely for background purposes.

The ARB's metals data are suitable as an indicator of ambient air concentrations of manganese, aluminum, and silicon. QA/QC practices, though incomplete in some areas, are not likely to impair the laboratory's ability to assess the presence of the elements of interest in the sampling environment.