Sampling and Analysis Plan for

791/805, 855 and 872 Runnymede Street and 875 O'Conner Street,
East Palo Alto, California

Prepared for the City of East Palo Alto by the Brownfields
Technology Support Center

October 7, 2003

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Ms. Lily Lee,
Environmental and Economic Development Coordinator,
City of East Palo Alto

For EPA use:

Approved by EPA Project Manager:	Date:
_____	_____
Expedited Review? ☐ Yes	☐ No
Received by QA Office:	Date:
_____	_____
Reviewed by:	Date:
_____	_____
Approved:	Date:

Region 9 Quality Assurance Manager	

TABLE OF CONTENTS

Tables

Figures

APPENDICES

1.0 INTRODUCTION

Four residential parcels are located in a former agricultural area where pesticides have been used. The City of East Palo Alto and the property owners are working together to redevelop these parcels. It is anticipated that each parcel will be subdivided for residential development. Earlier investigations at similar properties in the region have shown that organochlorine pesticides were used in the region, and therefore the four parcels will be investigated. This investigation will generate a collaborative data set (combining both field and laboratory analytical methods) to meet project goals.

1.1 Site Name or Sampling Area

The following sites will be the subjects of this investigation:

- 791/805 Runnymede Street
- 855 Runnymede Street
- 872 Runnymede Street
- 875 O'Conner Street

1.2 Site or Sampling Area Location

The areas associated with this preliminary investigation are located in East Palo Alto, California. The parcels surrounding the sites were generally used for agricultural applications from the late 1930s through the early 1940s. Existing residential structures date back to 1916 through the 1960s.

1.3 Responsible Agency

Innovative & Creative Environmental Solutions (ICES) will conduct the investigation under contract with the City of East Palo Alto. The U.S. Army Corps of Engineers (USACE) will provide technical support and oversight of the field activities.

1.4 Project Organization

Environmental and Economic Development Coordinator - Lily Lee is the representative of The City of East Palo Alto for this project. Ms. Lee is the primary decision maker for this investigation and is the primary contact for the project.

San Francisco Regional Water Quality Control Board – Mark Johnson is the Water Board Remedial Project Manager and will provide oversight of the investigation and remedial activities at these sites.

San Mateo County Environmental Health – Charles Ice is the Hazardous Materials Specialist overseeing project sites in East Palo Alto.

Innovative & Creative Environmental Solutions (ICES) Project Manager – Peng Leong is the project manager and will conduct the investigation fieldwork and will prepare the final reports.

USACE Technical Advisors - Both Kira Lynch and Brad Call are designated U.S. Army Corps of Engineers technical advisors for this project. They will be the prime designers of this investigation and will coordinate the decision making process during the field effort and ensure that project quality goals are met.

USACE QA Chemist – John Yaremchuk, QA Chemist, USACE will write the SAP and facilitate the laboratory coordination/review with the EPA Quality Assurance Office.

USEPA Project Officer – Roberta (Bobbie) Kahan is the EPA Project Officer and Brownfields Coordinator.

USEPA QAO – Gail Jones, of the EPA quality assurance staff will provide QA review of this plan.

Project personnel contact information is provided in Table 1.1 below:

Table 1.1 Project Personnel Contact Information

Title/Responsibility	Name	Phone Number
Environmental and Economic Development Coordinator-East Palo Alto	Lily Lee	650-853-3122
San Francisco Regional Water Quality Control Board, RPM	Mark Johnson	510-622-2493
San Mateo County Environmental Health	Charles Ice	650-363-4565
Technical Advisor, USACE	Brad Call Kira Lynch	916-557-6649 206-764-6918
QA Chemist, USACE	John Yaremchuk	916-557-7504
EPA Project Officer	Roberta Kahan	415-744-2191
EPA Quality Assurance Manager	Vance Fong	415-972-3798
EPA Quality Assurance Officer	Gail Jones	415-972-3807
EPA Regional Sample Coordinator	Carl Brickner	415-972-3814
EPA Laboratory Field Services	Greg Nagle	510-412-2334
Project Manager, Innovative & Creative Environmental Solutions	Peng Leong	510-652-3222

1.5 Statement of the Specific Problem

- Determine if "hot spot" or application residual pesticides are present
- Define the extent of pesticide contaminated soil at any hot spot

- Define extent and average pesticide concentrations for low level application residuals if present above action levels.
- Specify and confirm detected pesticides and metals
- Obtain laboratory data for contaminated soil profiling in the event of future remediation

2.0 BACKGROUND

The following discussion presents a conceptual site model (CSM) for the four residential parcels that are the subject of this investigation. Each of these parcels contains a residential building constructed between 1916 and 1956. The properties lie along the eastern side of the San Francisco Peninsula at an approximate elevation of 15 feet above sea level. Previous investigators have reported that groundwater in the area is generally encountered from 5 to 10 feet below the ground surface.

Agricultural activities took place on these properties or adjacent to them. These activities potentially include the preparation and application of pesticides. Information gathered from earlier investigations suggest that organochlorine pesticides were used in the area and that the depth of tilling ranged form 18 to 24 inches. Organochlorine pesticides have low water solubility, have low vapor pressures and are resistant to degradation. As a result of these properties they are persistent in soils. These pesticides also have a high affinity to sorb to soil, and therefore are relatively immobile in the environment.

The preparation of pesticides for application can result in accumulations of relatively high concentrations in a specific location that is often referred to as a "mixing area". Mixing areas may not be encountered at all four of these properties. These mixing areas, if they exist, are expected to be relatively localized (i.e. hot spots) and to contain high concentrations of pesticides. In addition, the application of pesticides on the

parcel, or on adjoining properties, would result in the relatively uniform distribution of low concentrations of these chemicals (from direct application and airborne drift). The pesticides would not be expected to affect the groundwater unless a mixing area is present with a significant mass of these chemicals.

Previous investigators have also suggested that arsenic, mercury, and lead may be linked to the agricultural activities, perhaps related to pesticide formulations, at some of these sites. While available data tends not to support this proposition, it is possible that these metals may be present. As with the organochlorine pesticides, these metals will be relatively immobile under typical environmental conditions and would not contaminate groundwater.

This project entails the assessment of four properties located on Runnymede Street and O'Conner Street in Palo Alto, CA (see Figure 2.1). Phase I site investigations (ICES, 2003a; ICES, 2003b; ICES, 2003c; Lowney Associates, 2002) document either known contamination or the possibility of pesticide use at these sites, and therefore a more detailed preliminary site investigation is proposed. The property at 872 Runnymede Street has an additional consideration in that fill material was apparently imported and placed on the undeveloped portion of the parcel. This has resulted in the accumulation of a 1 to 1-1/2 foot fill layer at the surface overlying the native soil. This fill material was placed after the peak years of agricultural activities in the area. Previous investigations of the 872 Runnymede Street property suggest that this fill layer contains organochlorine pesticides, with much lower concentrations present in the underlying soil. Due to the potential presence of contaminants at these sites, organochlorine pesticides, arsenic and lead will be investigated.

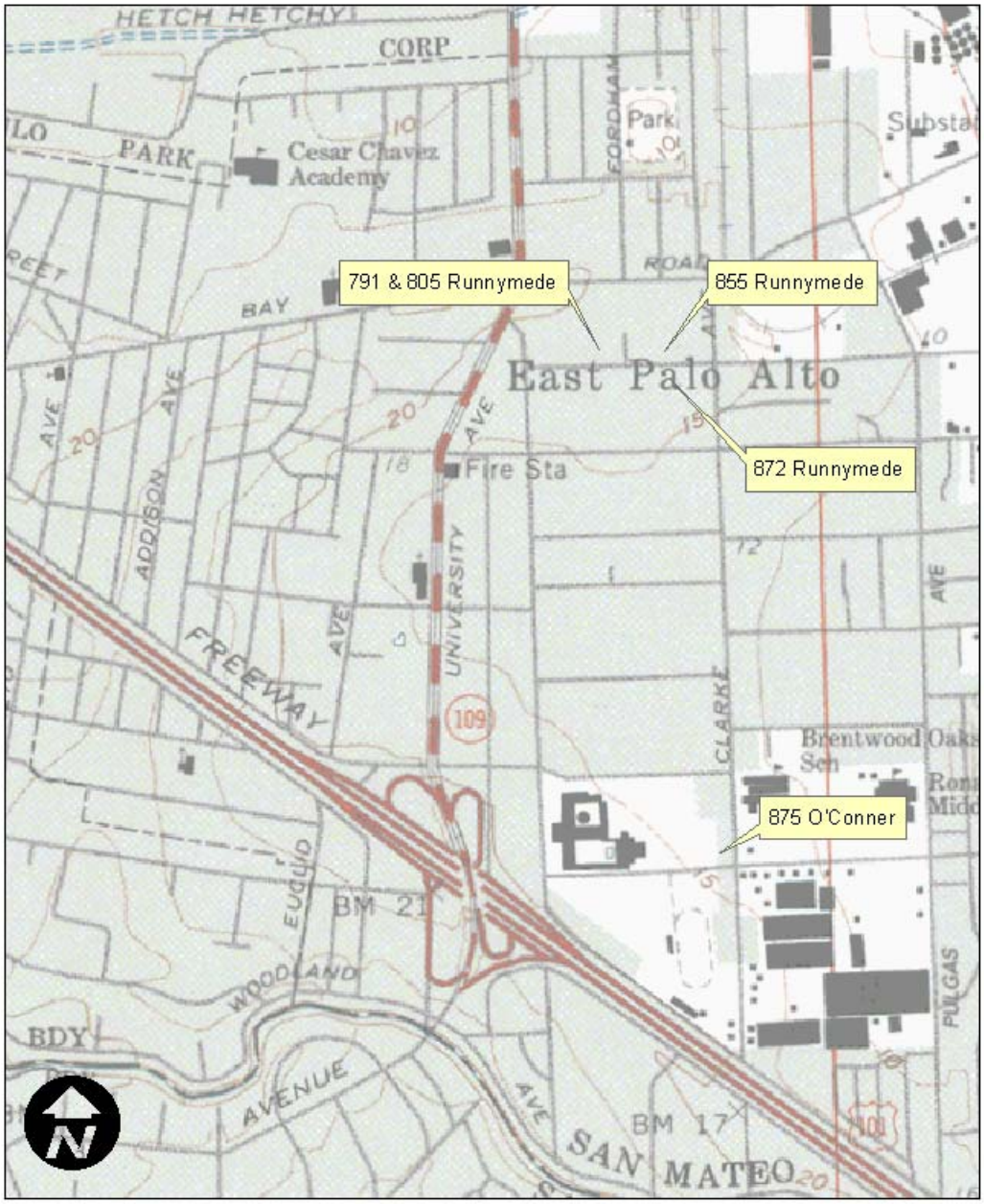
791 and 805 Runnymede Street are contiguous properties with one residential structure on each parcel. A series of greenhouses are

located to the northwest of the site. A review of government records shows no releases at the sites (ICES, 2003a).

855 Runnymede Street consists of a rectangular parcel with a two-story structure located at the central portion of the site. This structure has existed since the 1930s and has been used for residential purpose since that time. A review of government records shows no releases at the site (ICES, 2003c).

872 Runnymede Street is located in a residential area with the on-site residence present since 1916. A greenhouse was present during the 1970s along the back of the property and was removed prior to 1981. The owner of the property has stored construction equipment on site and in 1997 was cited for accumulating construction debris on this property. Fuel and pesticide concentrations are present (Lowney Associates, 2002).

875 O'Conner Street consists of a single structure located on the southern portion of the site. The structure has existed since 1956, prior to that time the property was vacant. A review of government records shows no releases at the site (ICES, 2003b).



Scale: 1" = 1000'

0 1,100 2,200 4,400 Feet

Figure 2.1
Location Map

East Palo Alto, California
Pesticide Investigation
September 2003

2.1 Site or Sampling Area Description

791 and 805 Runnymede Street

The site occupies 1.38 acres in a residential area (see Figure 2.1). The site (Figure 2.2) is bordered on the north by residential structures, on the west by residential structures, on the south by Runnymede Street and residential construction, and on the east by the Faith Baptist Church. The site consists of two parcels, which are located on the north side of Runnymede Street, between Clark and Cooley Avenues. 791 Runnymede Street occupies the western parcel. The eastern parcel is occupied by 805 Runnymede Street.

Two residential structures constructed by 1956 are located at the southern portion of the site. A series of vacant portable structures circa 1990 are located at the eastern portion of the site and were never occupied. A paved basketball court is located at the central portion of the site, directly adjacent to the north of the residential structures.

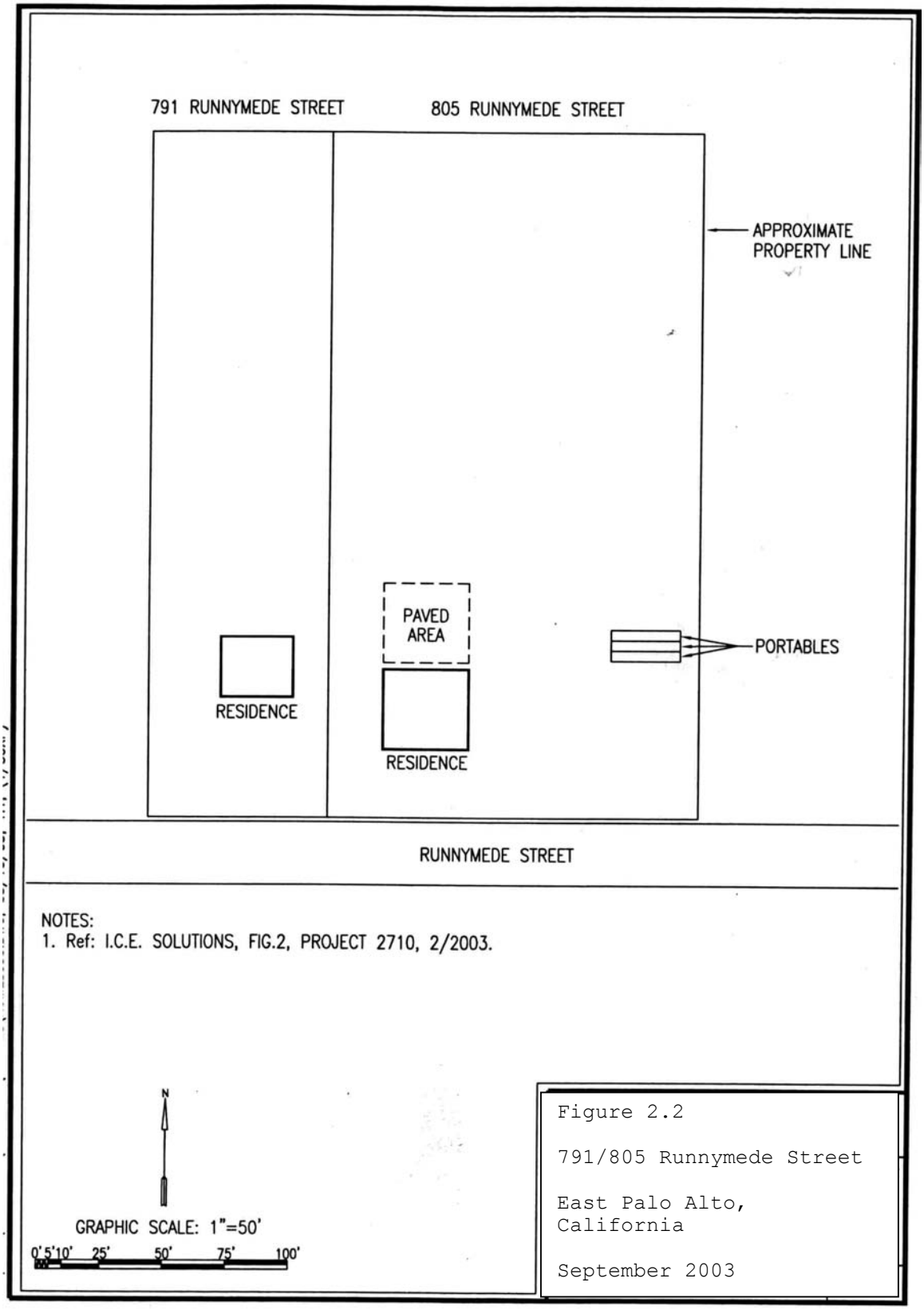


Figure 2.2

791/805 Runnymede Street

East Palo Alto, California

September 2003

855 Runnymede Street

The site occupies 0.52 acres in a residential area (see Figure 2.1). The site (Figure 2.3) is bordered on the north and west by the Faith Missionary Baptist Church, on the east by residential structures and on the south by residential development and a day care center. The site is located on the north side of Runnymede Street, between Clarke and Cooley Avenues.

The existing structure located at the central portion of the site has been present since the 1930s. During the 1930s through 1970, the structure had been used for residential applications. In 1970, Nairobi High School occupied the structure followed by Runnymede Head Start in 2002. The two-story structure is currently occupied by the Faith Center. An unpaved driveway is located along the western perimeter of the structure.

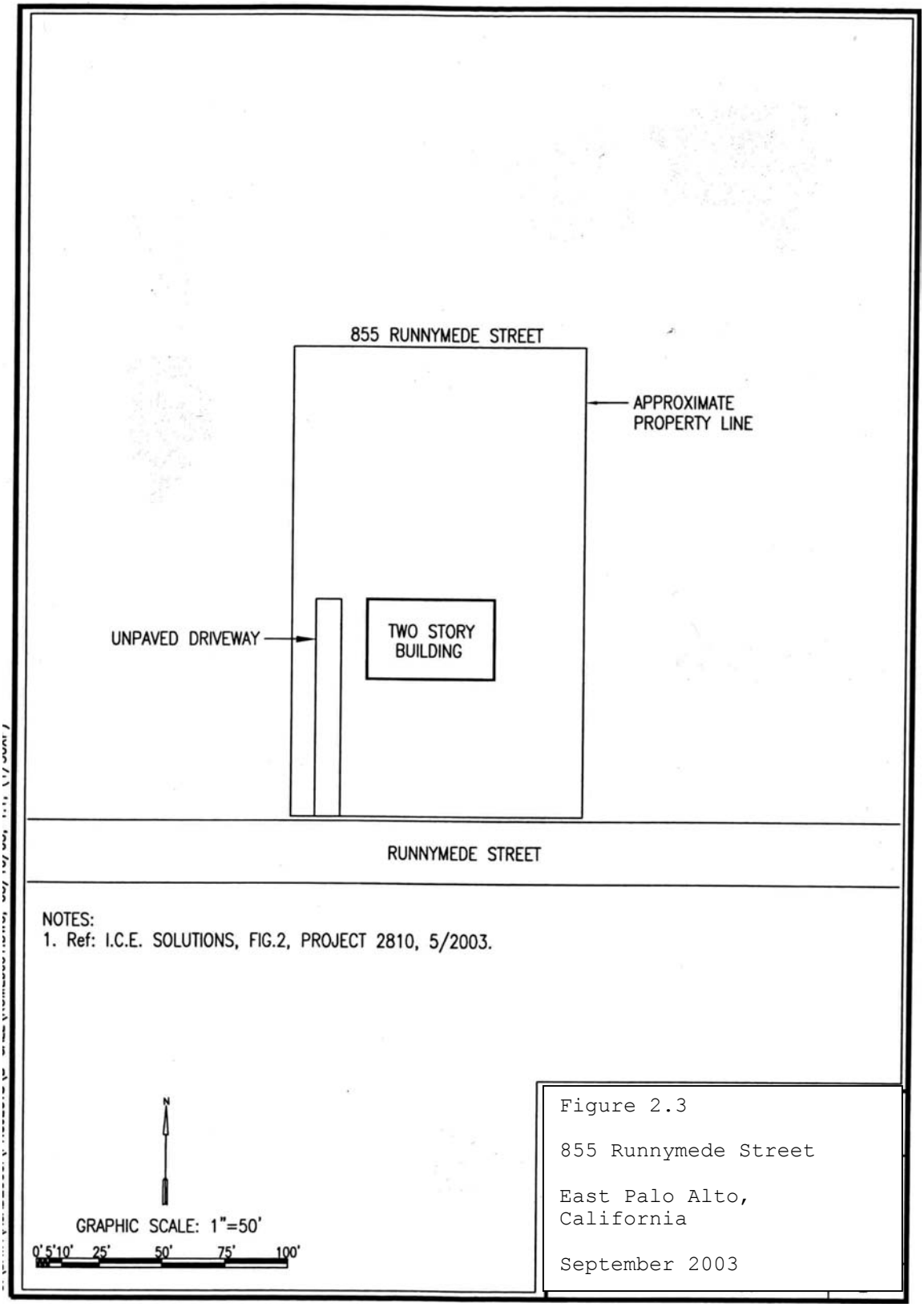


Figure 2.3

855 Runnymede Street

East Palo Alto, California

September 2003

872 Runnymede Street

The site occupies 0.84 acres in a residential area (see Figure 2.1). The site (Figure 2.4) is bordered on the north by residential structures and a children's educational facility (Head Start), on the south by residential structures, on the west by residential structures and on the east by residential structures. The site is located on the south side of Runnymede Street, between Clarke and Cooley Avenues.

The on-site residence has been present since 1916 with the remainder of the site undeveloped. Greenhouses were observed on-site in the 1970s and removed by 1981. A workshop was added to the site circa 1991, after the present owner purchased site. Areas associated with construction debris are detailed on Fig 2.4. Soil sampling was conducted at this site in 2002 and low levels of petroleum and organochlorine pesticides were found (Lowney Associates, 2002). The soil sampling results are provided in Appendix 1.

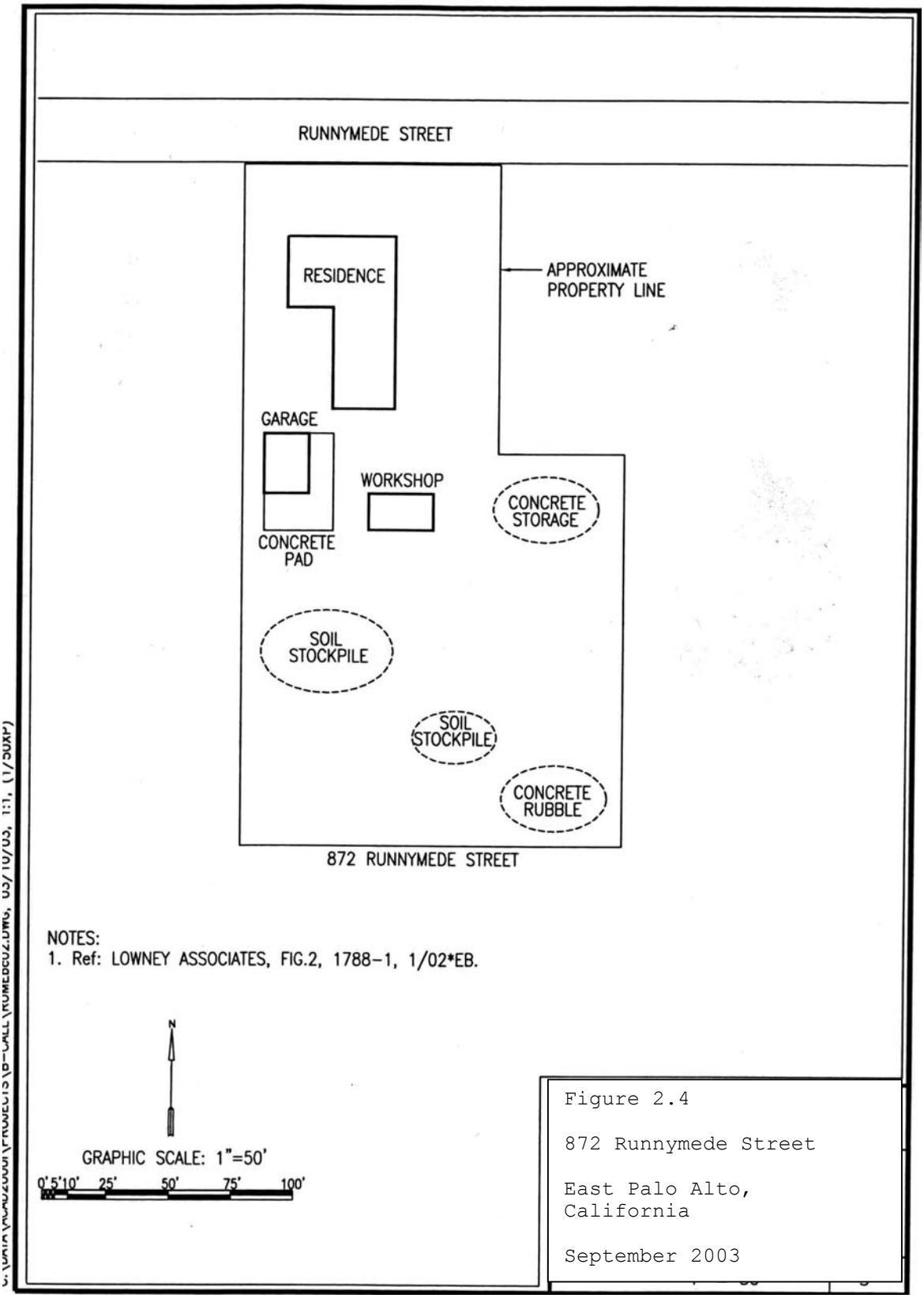


Figure 2.4

872 Runnymede Street

East Palo Alto, California

September 2003

875 O'Conner Street

The site occupies approximately 0.87 acres in a residential area (Figure 2.1). The site (Figure 2.5) is bounded on the south by O'Conner Street. The parcels located directly north and east of the site are occupied by residential structures. A shopping plaza is located west and south of the site. A vacant lot and the San Francisco 49ers Academy is located on the east side of Clarke Avenue, east and northeast of the site. The area southeast of the site is occupied by residential structures, the Grace Temple and the Grace Temple Daycare Center.

The existing structure located at the southeastern portion of the site was used as a residence from the mid 1950s through 1970. The Cummings Temple C.M.E. Church is currently using the structure.

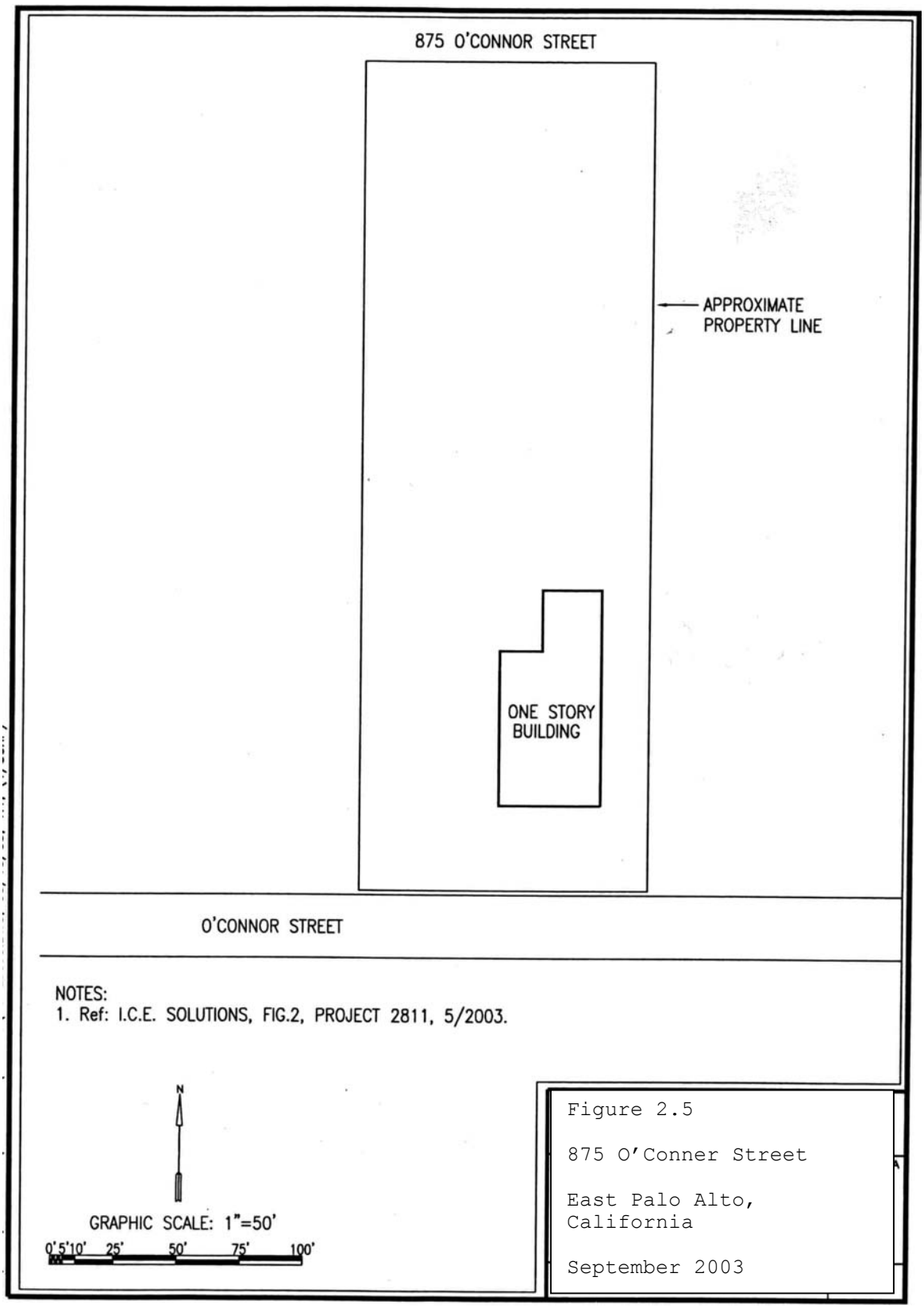


Figure 2.5

875 O'Conner Street

East Palo Alto, California

September 2003

2.2 Operational History

872 Runnymede Street

The East Palo Alto Building Department documents indicate that vehicles were maintained on-site, assorted construction-related materials were stockpiled, and a mixture of soil and construction debris were placed across the site.

2.3 Previous Investigations and Regulatory Involvement

791 and 805 Runnymede Street

A phase I environmental site assessment was conducted on behalf of the City of East Palo Alto by ICES in 2003. No regulatory involvement concerning this property has been documented.

855 Runnymede Street

A phase I environmental site assessment was conducted on behalf of the City of East Palo Alto by ICES in 2003. No regulatory involvement concerning this property has been documented.

872 Runnymede Street

A phase I environmental site assessment (ESA), soil quality evaluation and health risk assessment were reported by Lowney Associates on April 2002. The ESA was performed for the owner of the property Mr. Trennie Miles to document recognized environmental conditions at the site related to current and historic use.

The soil quality evaluation entailed a soil investigation across the site for organochlorine pesticides (EPA test method 8081) and arsenic, lead and mercury (EPA test methods 6010/7000). Other samples were additionally analyzed for polyaromatic hydrocarbons (PAHs)(EPA test method 8310), polychlorinated biphenyls (PCBs) (EPA test method 8082), a fuel fingerprint scan (EPA test method 8015M), and California Assessment Metals (CAM17)(EPA test method 6010/7000). The analytical results for these tests are included in Appendix 1. The data quality and usability of the data could not be assessed since the raw laboratory data was not available.

875 O' Conner Street
A phase I environmental site assessment was conducted on behalf of City of East Palo Alto by ICES in 2003. No regulatory involvement concerning this property has been documented.

2.4 Geological Information

Not applicable

2.5 Environmental and/or Human Impact

There is currently no evidence that pesticides have been released to the environment or that any adverse health affects have occurred at 791/805 Runnymede, 855 Runnymede and 875 O'Conner Streets. Low levels of pesticides and petroleum hydrocarbons are known to have been released to the environment at 872 Runnymede Street and there have been no reported adverse health effects.

Groundwater is designated as a potential drinking water source in this area. However given the nature of the pesticides and the low likelihood that they could be transported to groundwater, only soil will be sampled as a part of this investigation. This decision will be reevaluated should a mixing area be found that has the potential to affect groundwater.

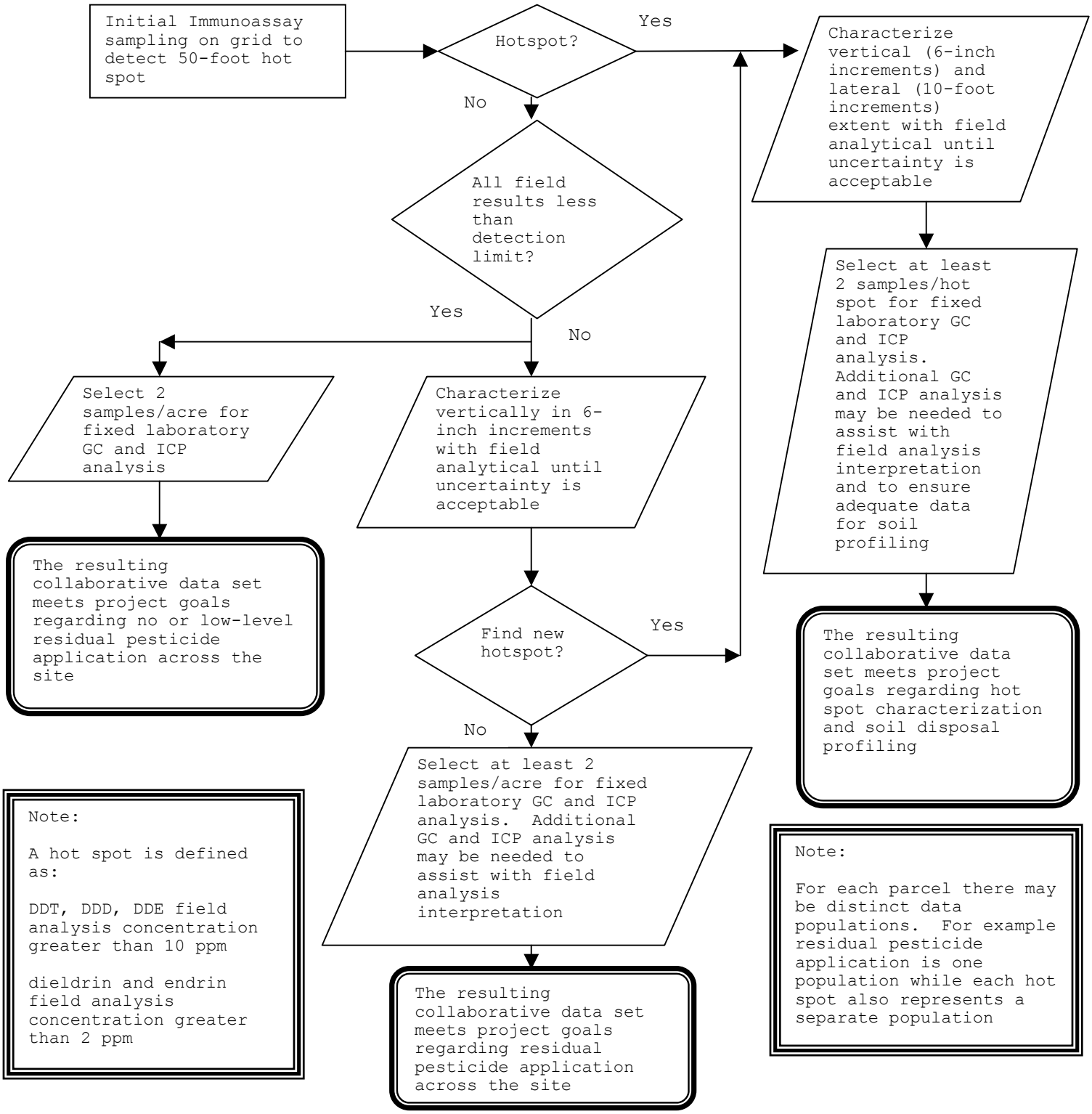
3.0 PROJECT DATA QUALITY OBJECTIVES

3.1 Project Task and Problem Definition

The data generated from this investigation will be used to determine if past agricultural practices have resulted in soil contamination with organochlorine pesticides above risk based screening levels. The primary organochlorine pesticides of concern have been identified as DDT, DDD, DDE, endrin, and dieldrin because they are the most persistent pesticides that

were most widely used during the time period of concern. Arsenic and lead will also be evaluated in a subset of samples found to contain elevated levels of pesticides to confirm that these contaminants are not present above background or action levels. Field analytical methods (using the immunoassay technology) will be used to provide near real-time data and support the dynamic work plan strategy. A dynamic work plan strategy utilizing on site field analysis is being used to manage uncertainty associated with soil contaminant heterogeneity (i.e. sampling uncertainty). GC and ICP methods will be used to confirm the presence of specific analytes, to assist with interpretation of immunoassay data, and manage analytical uncertainty. The combined collaborative data set (field and laboratory analysis) will be used to achieve project goals. The project decision logic is shown diagrammatically in Figure 3.1.

Figure 3.1 Project Decision Logic



3.2 Data Quality Objectives (DQOs)

The goals of this particular investigation are defined below:

1. State the Problem:
    - Determine if "hot spot" or application residual pesticides are present.
    - Define the extent of pesticide contaminated soil at any hot spot.
    - Define extent and average pesticide concentrations for low level application residuals if present above action levels.
    - Specify and confirm detected pesticides/metals
    - Obtain laboratory data for contaminated soil profiling in the event of future remediation

2. Identify the Decision (Refer to Figure 3.1):
    - If field analysis detects DDT, DDD and DDE concentrations greater than 10 ppm, or dieldrin and endrin concentrations greater than 2 ppm, then a soil hot spot will have been located.
    - If a hot spot is found it will be characterized vertically and laterally using field analytical. Saturated zone soils will not be characterized. Professional judgment and knowledge regarding costs associated with soil remediation options (i.e. on site management vs. off-site disposal) will be used to determine when uncertainty is acceptable.
    - For each hot spot, laboratory analysis will be used to obtain specific analyte information and to determine if arsenic and lead are present above background and action levels.
    - If the concentration found in the soil is less than hot spot criteria then this is judged to represent residual pesticide application. Professional judgment and

knowledge regarding costs associated with soil remediation options (i.e. on site management vs. off-site disposal) will be used to determine when residual pesticide contamination has been adequately characterized and uncertainty is acceptable.

- If the field analysis results are positive then fixed laboratory analysis by GC will be used to identify specific pesticides of concern and allow for more complete interpretation of the immunoassay results. In addition the GC analyses will provide concentration information below the field analytical detection limits.
- Use of fixed laboratory data will provide profile data for potential off-site removal.

3. Identify Inputs to the Decision:

- Immunoassay field analysis will be used to quantify organochlorine pesticides.
- Further investigation of an identified hot spot by use of field analysis will define the extent of contamination.
- Use of field analytical methodology allows a higher sampling density to be achieved, which can better define the area of both hot spots and general application residue.
- Use of the EPA methods 8081A and 6010B will provide quantitative and analyte specific results that will be used collaboratively with immunoassay data to make site decisions regarding extent of pesticide contaminated soil and potential remediation options.

4. Define the Boundaries of the Study:

Each of the four parcels will eventually be subdivided for residential use. The average lot size can be estimated to be approximately 7000 square feet. This results in the following:

- 791/805 Runnymede Street – 1.38 acres – 8.6 lots
- 855 Runnymede Street – 0.52 acres – 3.2 lots
- 872 Runnymede Street – 0.84 acres – 5.2 lots
- 875 O'Conner Street – 0.87 acres – 5.4 lots

The initial grid sampling will be conducted using a 50-foot triangular spacing to ensure detection of hot spots. This will result in the following approximate number of samples in the initial grid:

- 791/805 Runnymede Street – 24 samples
- 855 Runnymede Street – 9 samples
- 872 Runnymede Street – 15 samples
- 875 O'Conner Street – 15 samples

Sampling loctions are shown at Appendix 9. The temporal boundaries for this project can fluctuate depending on the data obtained. The field effort is scheduled for October 14 - 17, 2003.

This investigation will assess the potential contamination of the four parcels of land located in East Palo Alto from historic pesticide applications. To determine if there is a human health risk, criteria were gathered from U.S. EPA Region 9 Preliminary Remedial Goals (PRGs) and the Region 2 Regional Water Quality Control Board Environmental Screening Levels (ESLs) (recent name change from Risk-Based Screening Levels (RBSLs)) guidance. The Region 2 values were given precedence and the dieldrin and endrin action levels were modified after discussions with Mr. Roger

Brewer, the ESL coordinator (U.S. Army Corps of Engineers, 2003). This modification resulted from the observation that these two chemicals were not likely to be transported to groundwater, and therefore the Tier 1 ESLs were unnecessarily conservative. The action levels to be applied to the project are listed in Table 3.2.

Table 3.1

ANALYTE	ACTION LEVEL CONCENTRATION, mg/kg	SOURCE
DDT	1.7 mg/kg	PRG & ESL
DDD	2.4 mg/kg	PRG & ESL
DDE	1.7 mg/kg	PRG & ESL
Dieldrin	0.030 mg/kg	mod ESL
Endrin	3.7 mg/kg	mod ESL
Arsenic	8 mg/kg	ESL (bg)
Lead	150 mg/kg	ESL

Note: PRG indicates EPA Region 9 Preliminary Remedial Goal criteria and ESL indicates Water Board Region 2 Environmental Screening Level criteria.

It should also be noted that the data obtained from the field analysis (immunoassay technology) will be biased high, which provides an additional level of conservatism. The action levels for the immunoassay results to define the extent of residual and or hot spot soil contamination above the environmental action levels may be modified in the field based on comparison with analyte specific GC results.

If residual pesticide soil contamination is found (< 10 ppm DDT, DDE, and DDD or < 2 ppm endrin and dieldrin) the project team will be considering a combination of on site contaminated soil management and offsite disposal. If hot spot soil contamination is found (> 10 ppm DDT, DDE, and DDD or > 2 ppm endrin and dieldrin) soil will be removed and taken off-site to a regulated facility. Decisions regarding soil remediation will be made individually for each property and will consider the future land use, which will be residential property development with an

average lot size of 7000 square feet. These remedial action options and associated costs will be considered by the project team when making real-time decisions regarding collecting additional samples for immunoassay and/or fixed laboratory analyses to manage uncertainty.

3.3 Data Quality Indicators (DQIs)

Data quality indicator goals (DQIs) for this investigation were developed following guidelines presented in EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5 Final. The tables listed in Appendix 2 represent the quality control acceptance criteria for selected laboratory analytical methods. These limits reflect the USEPA Region 9 specifications to ensure that laboratory-generated analytical data is of acceptable quality for use in environmental decision-making. Appendices 3 and 4 provide the DQIs for the field analytical methods.

3.4 Data Review and Validation

A collaborative data set will be created using a combination of field and laboratory analysis. Both the field and laboratory data will be reviewed to ensure that quality control measures were properly conducted and that the measures did not identify individual or systematic errors that could compromise the intended use of the data. The field analysis will be evaluated on a daily basis. The review will follow the concepts detailed in *Laboratory Documentation Requirements for Data Validation*, 9-QA-07-97, Draft, USEPA R9. The validation of data requires that appropriate quality assurance and quality control (QA/QC) procedures be followed, and that adequate documentation be included for all data generated both in the laboratory and in the field. Professionals trained in data validation procedures will review this information, "flag" data with qualifiers when QA/QC criteria are not met, and prepare the data validation report. The validation reports are then used as sources of data quality indicators, which are used to conduct a data quality assessment relative to the pre-established DQOs.

3.5 Data Management

Field analytical samples will be given a unique location identifier based on the specific parcel, sample number and sample depth (bottom of depth interval in inches) (791/805 Run_1_6, which represents location 1, a sample interval of 0 to 6 inches collected at 791/805 Runnymede Street, for example). The site map will be annotated as to the sample number to allow for later association of specific results to the exact location on the ground. Specific sample locations will be identified in the field with a tape measure or with a GPS unit. As each sample is collected the sample identifier, sample date/time, and number of sample containers will be logged in both the field logbook and on a master list. This master list will be maintained by the field chemist and will be annotated with the appropriate sample delivery group and related quality control information.

Samples to be submitted for laboratory analysis will be logged on a chain-of-custody form as discussed in Section 9.3. Sampling information will also be described in the logbook, as discussed in Section 9.1. Samples will be kept secure in the custody of the sampler at all times. Samples will be transferred to the analytical laboratories via a certified carrier in a properly custody-sealed container with COC information as discussed in Section 9.3. The laboratories will note any evidence of tampering upon receipt. Completed laboratory data reports will be submitted by the laboratories to the City of East Palo Alto.

3.6 Assessment Oversight

Oversight of the site investigation will be the responsibility of the QA officer designated by the project manager. This individual will be present on-site to ensure that the quality assurance requirements as defined within this sample and analysis plan are adhered to. It is the project manager's responsibility to require the personnel working on the project are familiar with the quality assurance guidelines. If the QA person identifies

field or analytical problems contrary to the guidelines of the SAP, he/she will document such instance and report to the project manager. Appropriate corrective action procedures will be documented and implemented.

4.0 SAMPLING RATIONALE

4.1 Soil Sampling

This investigation will assess the potential contamination of four parcels of land located in East Palo Alto from historic pesticide applications. Contaminants of concern for this study are: DDD, DDE, DDT, endrin, dieldrin, arsenic and lead. The CSM suggests that pesticide contamination will either be confined to the upper soil (tilled) zone (from application or over spray), or will be found from the surface to deeper depths at potential mixing zones (liquid pesticide saturation of soils from mixing activities). These potential scenarios suggest that collection of a 6-inch soil sample using a trowel or hand auger should provide adequate sample support. The rationale for collecting a 6-inch soil sample is that this is the minimum soil thickness that can be reasonably excavated with a backhoe.

Field analytical techniques will enable all pesticide related unresolved site uncertainties to be immediately investigated and resolved. Soil sampling will begin with the 0 to 6 inch soil layer and will continue down in 6-inch increments if pesticides are detected with the field analysis. Soil sampling will stop upon encountering the groundwater (saturated zone). The initial density of surface soil sampling was determined based on the decision to detect a 50-foot hot spot. A triangular sampling grid using a 50-foot sampling radius will be used (USEPA, Methods for Attainment of Clean-up Standards). At those locations where it is necessary to define the lateral extent of a hot spot this will be done by collecting additional samples radially away in all directions from the initial detection in 10-foot increments.

For the 872 Runnymede Street site soil sampling will include both the fill layer and the underlying “native” soil. The fill is assumed to cover approximately 50% of the parcel.

Fixed laboratory samples will be utilized collaboratively with the immunoassay data to make site decisions regarding options for handling contaminated soil. Samples for fixed laboratory analyte specific GC analyses will be strategically selected to specifically help understand and control for bias in the immunoassay results. Soil samples submitted for fixed laboratory analysis will be selected by the field team to accomplish the following:

- confirm a portion of the field analytical non-detects
- confirm a portion of the field analytical detections in the region of the action level
- provide analyte specificity for field analytical detection
- provide soil profile data suitable for acceptance of pesticide or metal contaminated soils at a treatment or disposal facility

Soil samples submitted for laboratory analysis will be homogenized sample splits and will not be selected until the field analysis results are available. Initially sufficient soil will be collected, homogenized, and archived for each sample location so any soil sample could be selected for off site laboratory analyses without having to return to the site.

The general soil-sampling scheme is presented in Table 4.1 below:

Table 4.1

Location	Field Analytical (DDT and cyclodienes via immunoassay)			Laboratory Analytical (GC and ICP)		
	Initial grid samples	Contin-gency samples	Field dups	Minimum	Contin-gency samples	Lab dups
791/805 Runnymede Street	24	12	3	3	6	1
855 Runnymede Street	9	5	1	1	2	1
872 Runnymede Street	30	15	3	2	4	1
875 O'Conner Street	15	8	2	2	4	1
Totals	78	40	9	8	16	4
Grand total	127			28		

Note that at 872 Runnymede the 30 samples consist of the initial grid for the entire site (15) and 15 additional samples needed for the fill material. The grand total amount represents the worst-case scenario for the entire investigation; actual number of samples should be less but cannot be quantified at this time.

4.2 Sediment Sampling

Not applicable

4.3 Water Sampling

Not applicable

4.4 Biological Sampling

Not applicable

4.4.1 Biological Samples for Chemical Analysis

Not applicable

4.4.2 Biological Sample for Species Identification and Habitat Assessment

Not applicable

5.0 REQUEST FOR ANALYSES

5.1. Analyses Narrative

McCampbell Analytical Inc will perform fixed laboratory analysis for this project. The Quality Assurance Program for McCampbell Analytical is included at Appendix 6, while the SOPs for organochlorine pesticide and metal analysis are included at Appendices 7 and 8, respectively.

6.0 FIELD METHODS AND PROCEDURES

6.1 Field Equipment

6.1.1 List of Equipment Needed

- Stainless steel trowels and buckets
- Stainless steel hand auger
- Decontamination fluids, collection drums
- Personal PPE (i.e. gloves, etc.)

Adequate quantities of the above will be accounted for prior to mobilization to the site.

6.1.2 Calibration of Field Equipment

This investigation will not be using any calibrated equipment other than the land surveying equipment, which is pre-calibrated.

6.2 Field Measurement Technologies

Two immunoassay field analytical methods will be employed at the sites, DDT in soil and cyclodienes in soil test kits. Detailed EPA Region 9 SOPs for each method are included in Appendix 3 and 4. Details regarding analysis and decision logic for the field analytic methods are discussed in Sections 3.2 and 4.1.

6.3 Soil

6.3.1 Surface Soil Sampling

The initial soil sampling grid locations will be measured in the field with a tape measure or with a GPS unit. Subsequent sample locations, if needed to characterize hot spots, will also be established with a tape measure or GPS unit. Soil sample locations will be recorded in the field logbook as sampling is conducted. A sketch of the sample location will be entered into the logbook and any physical reference points will be labeled. If possible, distances to the reference points will be given.

Surface soil samples will initially be collected as grab samples (independent, discrete samples) from a depth of 0 to 6 inches below ground surface (bgs). Surface soil samples will be collected using a stainless steel hand trowel. Samples will be placed in a pre-cleaned stainless steel pail and homogenized with a trowel. Material in the pail will be transferred with a trowel from the pail to the appropriate sample containers (three 8-oz glass jars). Samples identified as duplicates will be placed into six, 8-oz glass jars. Sample containers will be filled to the top, taking care to prevent soil from remaining in the lid

threads prior to being closed to prevent potential contaminant migration to or from the sample. The master sample list and field logbook will be annotated regarding the number of sample containers prepared for each location. Each sample glass jar will be labeled with the identification number, the sample depth (bottom of the sample interval in inches) and the container number. Sample containers will be closed as soon as they are filled, placed in a secure area, and chilled to 4□°C if appropriate. Excess soil will be returned to the site.

As mentioned above, primary soil samples will be placed into three glass jars. One jar is for the field analysis, while the remaining two are reserve samples to be archived for potential future laboratory GC and ICP analysis. The duplicate samples follow a similar approach, with two of the six jars destined for field replicate analysis, and the remaining four archived for potential future use for laboratory GC and ICP analysis. The field team will determine which samples are submitted for laboratory analysis based on the results of the field analysis.

6.3.2 Subsurface Soil Sampling

Subsurface sample locations will be handled exactly the same as the surface samples (Section 6.3.1). Subsurface samples will be collected with a trowel, a hand auger, or potentially with a backhoe depending on the depth required. Excess set-aside soil from the above the sampled interval will then be repacked into the hole.

6.4 Sediment Sampling

Not applicable to this investigation.

6.5 Water Sampling

Not applicable to this investigation.

6.5.1 Surface Water Sampling

Not applicable to this investigation.

6.5.2 Groundwater Sampling

Not applicable to this investigation.

6.5.2.1 Water-Level Measurements

Not applicable to this investigation.

6.5.2.2 Purging

Not applicable to this investigation.

6.5.2.3 Well Sampling

Not applicable to this investigation.

6.6 Biological Sampling

Not applicable to this investigation.

6.6.1 Biological Sampling for Chemical Analysis

Not applicable to this investigation.

6.6.1.1 Fish Samples

Not applicable to this investigation.

6.6.1.2 Foliage Samples

Not applicable to this investigation.

6.6.2 Biological Sampling for Species Assessment

Not applicable to this investigation.

6.7 Decontamination Procedures

The decontamination procedures that will be followed are in accordance with approved procedures. Decontamination of sampling equipment must be conducted consistently as to assure the quality of samples collected. All equipment that comes into contact with potentially contaminated soil or water will be decontaminated. Disposable equipment intended for one-time use will not be decontaminated, but will be packaged for appropriate disposal. Decontamination will occur prior to and after each use of a piece of equipment. All sampling devices used, including trowels and

augers, will be steam-cleaned or decontaminated according to EPA Region 9 recommended procedures.

The following, to be carried out in sequence, is an EPA Region IX recommended procedure for the decontamination of sampling equipment:

- Non-phosphate detergent and tap water wash, using a brush if necessary
- Tap-water rinse
- 0.1 N nitric acid rinse
- Deionized/distilled water rinse
- Pesticide-grade solvent (reagent grade hexane) rinse in a decontamination bucket
- Deionized/distilled water rinse (twice)

Equipment will be decontaminated in a pre-designated area on pallets or plastic sheeting, and clean bulky equipment will be stored on plastic sheeting in uncontaminated areas. Cleaned small equipment will be stored in plastic bags. Materials to be stored more than a few hours will also be covered.

7.0 SAMPLE CONTAINERS, PRESERVATION AND STORAGE

7.1 Soil Samples

Soil samples for organochlorine pesticides will be homogenized and transferred from the sample-dedicated homogenization pail into 8-ounce (oz), wide-mouth glass jars using a trowel. The number of sample containers per location is detailed in Section 6.3.1.

METALS. Surface soil samples to be analyzed for metals will be homogenized and transferred from the sample-dedicated homogenization pail into 8-oz, wide-mouth glass jars. The number of sample containers per location is detailed in Section 6.3.1.

Table 7.1

Method and Analysis	Containers and Sample Size	Chemical Preservation	Temperature Preservation
SW8081 (OC pesticides)	2 – 1-liter amber glass bottle with screwcap (water)	none	cool 4^0C
	1 – 8 oz Glass Jar (Certified Pre-Cleaned)	none	
SW6010 or SW6020 (lead, arsenic)	1 – 500-mL polyethylene bottle (water)	HNO3, pH<2	cool 4^0C
	1 – 8 oz Glass Jar (Certified Pre-Cleaned)	none	

7.2 Sediment Samples

Not applicable to this investigation.

7.3 Water Samples

Not applicable to this investigation.

7.4 Biological Samples

Not applicable to this investigation.

7.4.1 Fish Samples

Not applicable to this investigation.

7.4.2 Foliage Samples

Not applicable to this investigation.

7.4.3 Biological Sampling for Species Assessment

Not applicable to this investigation.

8.0 DISPOSAL OF RESIDUAL MATERIALS

In the process of collecting environmental samples at the four sites, the ICES sampling team will generate different types of potentially contaminated IDW that include the following:

- Used personal protective equipment (PPE)
- Disposable sampling equipment
- Decontamination fluids
- Unused soil samples

The EPA's National Contingency Plan (NCP) requires that management of IDW generated during sampling comply with all applicable or relevant and appropriate requirements (ARARs) to the extent practicable. The sampling plan will follow the Office of Emergency and Remedial Response (OERR) Directive 9345.3-02 (May 1991), which provides the guidance for the management of IDW. In addition, other legal and practical considerations that may affect the handling of IDW will be considered.

- Used PPE and disposable equipment will be double bagged and placed in a municipal refuse dumpster. These wastes are not considered hazardous and can be sent to a municipal landfill. Any PPE and disposable equipment that is to be disposed of which can still be reused will be rendered inoperable before disposal in the refuse dumpster.

- Decontamination fluids that will be generated in the sampling event will consist of dilute nitric acid, pesticide-grade solvent, deionized water, residual contaminants, and water with non-phosphate detergent. The volume and concentration of the decontamination fluid will be sufficiently low to allow disposal at the site or sampling area. The water (and water with detergent) will be poured onto the ground or into a storm drain. Pesticide-grade solvents will be allowed to evaporate from the decontamination bucket. The nitric acid will be diluted and/or neutralized with sodium hydroxide and tested with pH paper before pouring onto the ground or into a storm drain.

- Excess or unneeded soil samples will be returned to the site from which they were collected.

9.0 SAMPLE DOCUMENTATION AND SHIPMENT

9.1 Field Notes

9.1.1 Field Logbooks

Field logbooks will document where, when, how and from whom any vital project information was obtained. Logbook entries will be complete and accurate enough to permit reconstruction of field activities. A separate logbook will be maintained for each project. Logbooks are bound with consecutively numbered pages. Each page will be dated and the time of entry noted in military time. All entries will be legible, written in ink and signed by the individual making the entries. Language will be factual, objective and free of personal opinions. At a minimum, the following information will be recorded, if applicable, during the collection of each sample;

- Sample location and description
- Site or sampling area sketch showing sample location and measured distances
- Sampler's name(s)
- Date and time of sample collection
- Designation of sample as composite or grab
- Type of sample (soil)
- Type of sampling equipment used
- Field instrument readings and calibration
- Field observations and details related to analysis or integrity of samples (e.g., weather conditions, noticeable odors, colors, etc.)
- Preliminary sample descriptions (e.g., for soils: clay loam, very wet)
- Sample preservation

- Lot numbers of the sample containers, sample identification numbers and any explanatory codes, and chain-of-custody form numbers
- Shipping arrangements (overnight air bill number)
- Name(s) of recipient laboratory(ies)

In addition to the sampling information, the following specific information will also be recorded in the field logbook for each day of sampling:

- Team members and their responsibilities
- Time of arrival/entry on site and time of site departure
- Other personnel on site
- Summary of any meetings or discussions with tribal, contractor, or federal agency personnel
- Deviations from sampling plans, site safety plans, and QAPP procedures
- Changes in personnel and responsibilities with reasons for the changes
- Levels of safety protection
- Calibration readings for any equipment used and equipment model and serial number

9.1.2 Photographs

Photographs will be taken at the sampling locations and at other areas of interest on site or sampling area. They will serve to verify information entered in the field logbook. For each photograph taken, the following information will be written in the logbook or recorded in a separate field photography log:

- Time, date, location, and weather conditions
- Description of the subject photographed
- Name of person taking the photograph

9.2 Labeling

Each sample container will be labeled with the following information:

- location identification number (parcel, sample number, depth)
- date and time
- container number (i.e. 1 of 3)

9.3 Sample Chain-Of-Custody Forms and Custody Seals

ICES (or those provided by the laboratory) chain-of-custody record forms will be used to document sample collection and shipment to fixed laboratories for analysis. Samples collected for analysis in the field and archive will be tracked in field logbooks and in an excel database that will be maintained by the field analytical lead.

A chain-of-custody record will accompany all sample shipments for off site laboratory analyses. A copy of the form will be completed and sent with the samples for each laboratory and each shipment (i.e., each day). A copy of the chain of custody form will be retained by ICES and remain with the project field sampling logbook. If multiple coolers are sent to a single laboratory on a single day, form(s) will be completed and sent with the samples for each cooler.

The sample database maintained by the field analytical chemist and the chain-of-custody forms will identify the contents of each shipment and maintain the custodial integrity of the samples. Generally, a sample is considered to be in someone's custody if it is either in someone's physical possession, in someone's view, locked up, or kept in a secured area that is restricted to authorized personnel. Until the samples are shipped, the custody of the samples will be the responsibility of ICES. The sampling

team leader or designee will sign the chain-of-custody form in the "relinquished by" box and note date, time, and air bill number.

A self-adhesive custody seal will be placed across the lid of each sample. The shipping containers in which samples are stored (usually a sturdy picnic cooler or ice chest) will be sealed with self-adhesive custody seals any time they are not in someone's possession or view before shipping. All custody seals will be signed and dated.

9.4 Packaging and Shipment

All sample containers will be placed in a strong-outside shipping container (a steel-belted cooler). The following outlines the packaging procedures that will be followed for low concentration samples.

1. When ice is used, pack it in zip-locked, double plastic bags. Seal the drain plug of the cooler with fiberglass tape to prevent melting ice from leaking out of the cooler.

2. The bottom of the cooler should be lined with bubble wrap to prevent breakage during shipment.

3. Check screw caps for tightness and, if not full, mark the sample volume level of liquid samples on the outside of the sample bottles with indelible ink.

4. Secure bottle/container tops with clear tape and custody seal all container tops.

5. Affix sample labels onto the containers with clear tape.

6. Wrap all glass sample containers in bubble wrap to prevent breakage.

7. Seal all sample containers in heavy duty plastic zip-lock bags. Write the sample numbers on the outside of the plastic bags with indelible ink.

8. Place samples in a sturdy cooler(s) lined with a large plastic trash bag. Enclose the appropriate COC(s) in a zip-lock plastic bag affixed to the underside of the cooler lid.

9. Fill empty space in the cooler with bubble wrap or Styrofoam peanuts to prevent movement and breakage during shipment. Vermiculite should also be placed in the cooler to absorb spills if they occur.

10. Ice used to cool samples will be double sealed in two zip lock plastic bags and placed on top and around the samples to chill them to the correct temperature.

11. Each ice chest will be securely taped shut with fiberglass strapping tape, and custody seals will be affixed to the front, right and back of each cooler.

10.0 QUALITY CONTROL

10.1 Field Quality Control Samples

The QA/QC samples described in the following subsections will be collected during this investigation.

10.1.1 Assessment of Field Contamination (Blanks)

10.1.1.1 Equipment Blanks

Equipment rinsate blanks will not be collected because the contaminants of interest bind strongly to soil. Careful equipment decontamination combined with visual observation of the sampling equipment will be used to prevent cross contamination.

10.1.1.2 Field Blanks

Field blanks will not be collected during this investigation.

10.1.1.3 Trip Blanks

Not applicable to this investigation.

10.1.1.4 Temperature Blanks

For each cooler that is shipped or transported to an analytical laboratory a 40 mL VOA vial will be included that is marked "temperature blank." This blank will be used by the sample custodian to check the temperature of samples upon receipt.

10.1.2 Assessment of Sample Variability (Field Duplicate or Co-located Samples)

Homogenized duplicate soils samples will be collected at sample locations determined at the site. Duplicate samples will be collected from these locations to assess contaminant variability and as a method quality control measure. Duplicates for both field and laboratory analysis will be collected. Exact locations will be determined in the field based on field analytical results.

Duplicate samples will be preserved, packaged, and sealed in the same manner as other samples of the same matrix. A separate sample number and station number will be assigned to each duplicate, and it will be submitted blind to the field analytical location or fixed laboratory.

10.2 Background Samples

No background samples are planned for this investigation.

10.3 Field Confirmation, and Split Samples

10.3.1 Field Samples

Field analysis will be performed using immunoassay test kits in soil for DDT and cyclodienes. QA parameters include initial demonstration of capability, and multi-point calibration to verify method performance. Quality control parameters are established for both an initial demonstration of capability and for routine quality control.

- Initial demonstration of capability
  - performance evaluation sample (or approved substitute)
  - site specific matrix spike near concentrations of interest
- Routine quality control
  - standards
  - negative controls (as specified by the manufacturer)
  - duplicates

Detailed SOPs regarding both tests particularly in regards to the QC are included in Appendix 3 and 4.

10.3.2 Laboratory Samples

The field analytical effort will be supported by fixed laboratory analysis. Comparability will be assessed relative to the decision to be made using linear regression evaluation and by assessing the methods relative to false positives/false negatives. Overall the quality control evaluation will seek to determine if the correct decisions was made regarding; "has the extent of pesticide contamination been established?"

The collaborative data set quality will be evaluated using linear regression by plotting the pairs of results as a scatter plot. A correlation coefficient will also be computed and the strength of the correlation determined. Field analysis false positives and negatives will also be determined relative to fixed laboratory results. This project seeks to obtain less than 5% false negatives and less than 10% false positives.

10.3.3 Split Samples

There are no split samples proposed for this investigation.

10.4 Laboratory Quality Control Samples

A routinely collected soil sample (a full 8-oz sample jar or two 120-mL sample vials) contains sufficient volume for both routine sample analysis and additional laboratory QC analyses. Therefore, a separate soil sample for laboratory QC purposes will not be collected.

The laboratory should be alerted as to which sample is to be used for QC analysis by a notation on the sample container label and the chain-of-custody record or packing list.

At a minimum, one laboratory QC sample is required per 14 days or one per 20 samples (including blanks and duplicates), whichever is greater. If the sample event lasts longer than 14 days or involves collection of more than 20 samples per matrix, additional QC samples will be designated.

For this project the selection for QA/QC samples will be determined in the field and this decision will be made after evaluation of the field data.

11.0 FIELD VARIANCES

As conditions in the field may vary, it may become necessary to implement minor modifications to sampling as presented in this plan. When appropriate, the QA Office will be notified and a verbal approval will be obtained before implementing the changes. Modifications to the approved plan will be documented in the sampling project report.

12.0 FIELD HEALTH AND SAFETY PROCEDURES

The Health and Safety plan proposed for this investigation is attached as Appendix 5 of this SAP.

13.0 RERERENCES

Innovative & Creative Environmental Solutions (ICES), 2003a. *Phase 1 Environmental Site Assessment, 791 and 805 Runnymede Street, East Palo Alto, California*. February 24.

ICES, 2003b. *Phase 1 Environmental Site Assessment, 875 O'Conner Street, East Palo Alto, California*. May 20.

ICES, 2003c. *Phase 1 Environmental Site Assessment, 855 Runnymede Street, East Palo Alto, California*. May 20.

Lowney Associates, 2002. *Phase 1 Environmental Site Assessment, Soil Quality Evaluation and Health Risk Assessment, 872 Runnymede Street, East Palo Alto, California*. April 26.

Lowney Associates, 2003. Soil Quality Evaluation, 872 Runnymede Street, *East Palo Alto, California.* August 19.

U.S. Army Corps of Engineers, 2003. *Memorandum for File. Subject: Chemicals of Concern, Subject Properties and Soil Exposure Criteria, Pesticide Properties, East Palo Alto Brownfields Project*. June 16.

U.S. Environmental Protection Agency (USEPA), not dated. *Methods for Evaluating the Attainment of Clean-up Standards, Volume 1*. Soils and Solid Media. Statistical Policy Branch. PB89-234959. EPA 230-02-89-042.

Appendix 1

LOWNEY ASSOCIATES
Environmental/ Geotechnical/ Engineering Services

Phase I Environmental Site Assessment, Soil Quality Evaluation, and Health Risk Assessment

872 Runnymede Street

East Palo Alto, California

This report has been prepared for:

T. Miles Enterprises

872 Runnymede Avenue, East Palo Alto, CA 94303

April 16, 2002

Project No. 1788-1/1A

Belinda P. Blackie, P.E., R.E.A.
Principal Environmental Engineer

Ron L. Helm, R.G., C.E.G.
Senior Principal Geologist

5.2 Further Soil Quality Evaluation

To further evaluate on-site soil quality for residual pesticides and other potential contaminants, eight soil samples (TP-1 through TP-8) were collected from fill material from the surface to ½-foot depth from eight locations randomly selected across the site. In addition, eight native soil samples (TP-1 through TP-8 at varying depths) were collected from immediately beneath the fill/native soil interface at depths beginning at 1 to 1½ feet beneath the surface to depths of approximately 3½ to 4 feet beneath the surface (Figure 2). All soil samples were collected from test pits excavated using a backhoe. Fill material placed across the project site by the current property owner appeared to extend to depths of 1 to 1½ feet beneath the ground surface.

The 24 samples were submitted to a state-certified analytical laboratory and analyzed for organochlorine pesticides (EPA Test Method 8081) and pesticide-related metals arsenic, lead, and mercury (EPA Test Method 6010/7000). Two fill samples were additionally analyzed for poly aromatic hydrocarbons (PAHs) (EPA Test Method 8310), polychlorinated biphenyls (PCBs) (EPA Test Method 8082), a fuel fingerprint scan (EPA Test Method 8015M), and California Assessment Metals (CAM 17) (EPA Test Method 6010/7000). These analyses were selected to evaluate whether residual pesticides from previous agricultural use of the site remain present in native on-site soil and to evaluate whether imported fill material placed on-site contains contaminants from site uses where it originated.

5.3 Analytical Results for Soil Quality Evaluation

Analytical results for the initial and further soil quality evaluations are presented together in Tables 5 through 10. Copies of the analytical reports and chain of custody documentation are presented in Appendix D.

Table 5. Analytical Results of Soil Samples
Pesticide-Related Metals Analyses
(concentrations in parts per million)

Sample Number	Depth (feet)	Arsenic	Lead	Mercury
SS-1	0-1/2 foot	**3.6**	17.0	0.051
SS-2	0-1/2 foot	**3.4**	33.0	0.058
SS-3	0-1/2 foot	**3.5**	13.0	0.073
SS-4	0-1/2 foot	**3.0**	15.0	0.33
TP-1	0-1/2 foot	**3.0**	17.0	0.067
TP-1	1½-2 foot	**3.4**	13.0	0.022
TP-1	3½-4 foot	**3.2**	5.4	0.033
TP-2	0-1/2 foot	**5.5**	27.0	0.080
TP-2	1½-2 foot	**4.0**	19.0	0.54
TP-2	3½-4 foot	**3.2**	6.1	0.032
TP-3	0-1/2 foot	**6.6**	22.0	0.062
TP-3	1-1½ foot	**4.0**	24.0	0.34
TP-3	3½-4 foot	**3.4**	6.3	0.023

Table 5. Analytical Results of Soil Samples
Pesticide-Related Metals Analyses (continued)
(concentrations in parts per million)

Sample Number	Depth (feet)	Arsenic	Lead	Mercury
TP-4	0-1/2 foot	**3.7**	43.0	0.041
TP-4	1½-2 foot	**3.6**	23.0	0.41
TP-4	3½-4 foot	**2.9**	5.6	0.041
TP-5	0-1/2 foot	**4.7**	19.0	0.44
TP-5	1-1½ foot	**4.0**	19.0	0.064
TP-5	3½-4 foot	**3.0**	5.6	0.023
TP-6	0-1/2 foot	**3.7**	17.0	0.049
TP-6	1½-2 foot	**3.5**	15.0	0.053
TP-6	3½-4 foot	**3.2**	6.0	0.039
TP-7	0-1/2 foot	**3.9**	16.0	0.064
TP-7	1½-2 foot	**2.9**	11.0	0.043
TP-7	3½-4 foot	**2.8**	5.7	0.020
TP-8	0-1/2 foot	**3.7**	25.0	0.23
TP-8	1½-2 foot	**3.1**	30.0	0.056
TP-8	3½-4 foot	**3.1**	5.8	0.021
Residential PRG*		0.39	400	23
RBSL**		0.39	200	4.7
95% UCL Concentration		4.23	24.6	0.24

UCL Upper confidence limit
Concentrations shown in bold print exceed either PRG or RBSL values for that compound
* Preliminary Remediation Goal-EPA Region 9, November 2000
** Risk-Based Screening Levels – California Regional Water Quality Control Board, 2000

Table 5a. Analytical Results of Soil Samples
Organochlorine Pesticides
(concentrations in parts per million)

Sample Number	Depth (feet)	Dieldrin	Chlordane	Total DDT	Others
SS-1	0-1/2 foot	**0.012**	0.130	0.052	Aldrin = 0.004
SS-2	0-1/2 foot	**0.0080**	0.730	0.046	Heptachlor epoxide = 0.0057
SS-3	0-1/2 foot	<0.002	0.026	0.011	Beta-BHC = 0.0014
SS-4	0-1/2 foot	**0.0023**	0.061	0.0022	ND
TP-1	0-1/2 foot	0.013	<0.02	0.0194	ND
TP-1	1½-2 foot	<0.002	<0.02	<0.006	ND

Table 5a. Analytical Results of Soil Samples Organochlorine Pesticides (continued)

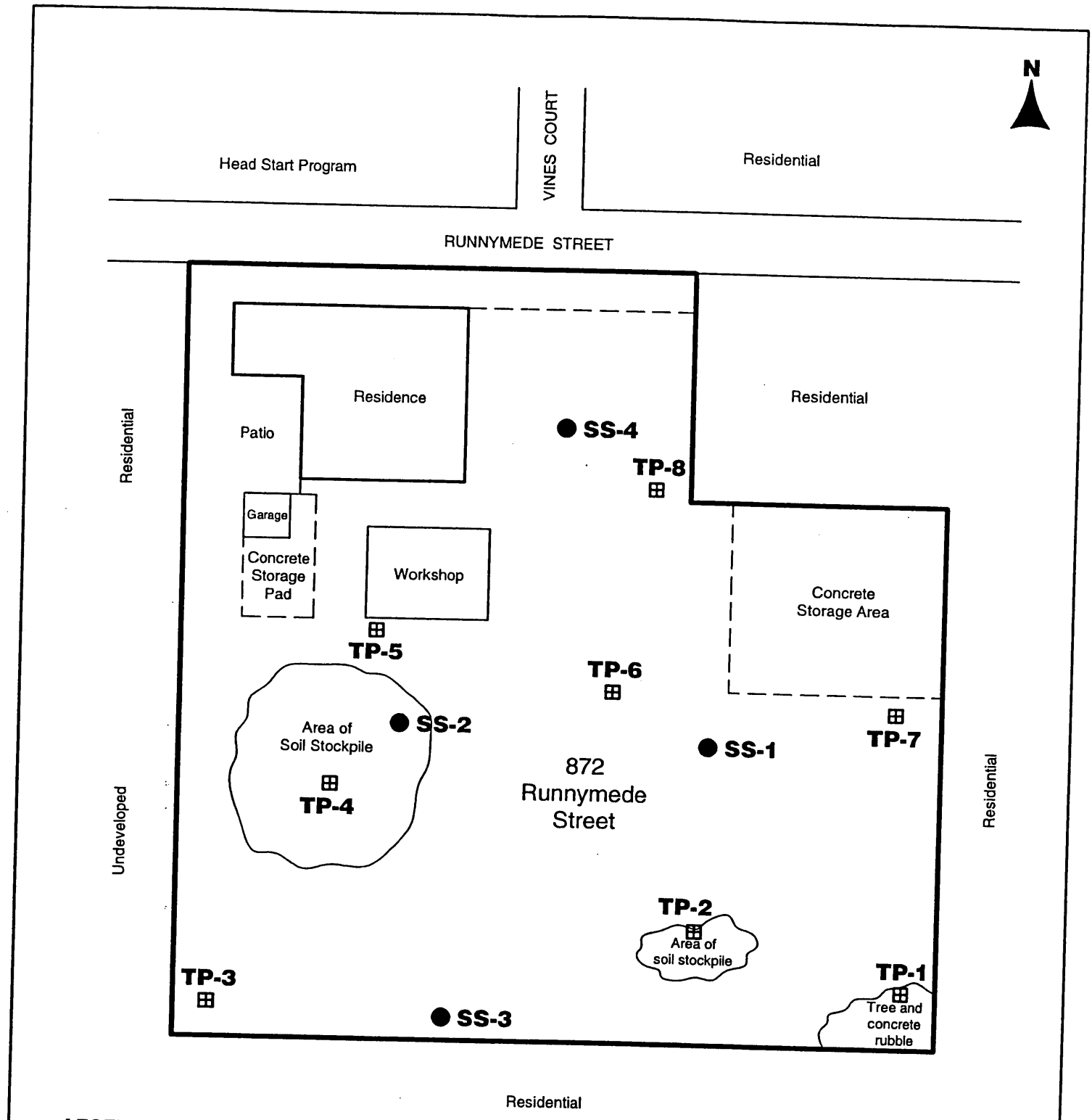
(concentrations in parts per million)

Sample Number	Depth (feet)	Dieldrin	Chlordane	Total DDT	Others
TP-1	3½-4 foot	<0.002	<0.02	<0.006	ND
TP-2	0-1/2 foot	0.018	<0.02	0.0314	ND
TP-2	1½-2 foot	<0.002	<0.02	0.236	ND
TP-2	3½-4 foot	<0.002	3.2	6.1	0.032
TP-3	0-1/2 foot	**0.027**	**0.570**	0.042	ND
TP-3	1-1½ foot	**0.140**	<0.02	0.109	ND
TP-3	3½-4 foot	<0.002	<0.02	<0.006	ND
TP-4	0-1/2 foot	**0.081**	0.550	0.0172	Aldrin = 0.0063
TP-4	1½-2 foot	<0.002	<0.02	0.251	ND
TP-4	3½-4 foot	<0.002	<0.02	<0.006	ND
TP-5	0-1/2 foot	**0.0085**	0.560	0.169	ND
TP-5	1-1½ foot	**0.018**	<0.02	0.228	ND
TP-5	3½-4 foot	<0.002	<0.02	<0.006	ND
TP-6	0-1/2 foot	**0.016**	0.089	0.0243	ND
TP-6	1½-2 foot	**0.028**	<0.02	0.063	ND
TP-6	3½-4 foot	<0.002	<0.02	<0.006	ND
TP-7	0-1/2 foot	**0.024**	0.390	0.063	Aldrin = 0.0025
TP-7	1½-2 foot	<0.002	<0.02	0.0026	ND
TP-7	3½-4 foot	<0.002	<0.02	<0.006	ND
TP-8	0-1/2 foot	**0.003**	0.610	0.0357	Heptachlor epoxide = 0.0065

Table 5a. Analytical Results of Soil Samples
Organochlorine Pesticides (continued)
(concentrations in parts per million)

Sample Number	Depth (feet)	Dieldrin	Chlordane	Total DDT	Others
TP-8	1½-2 foot	**0.0047**	0.038	0.0050	ND
TP-8	3½-4 foot	<0.002	<0.02	<0.006	ND
Residential PRG*		0.03	1.6	1.7	Aldrin = 0.029 Beta-BHC = NE Heptachlor epoxide = 0.053
RBSL**		0.002	0.47	1.7	Aldrin = 0.029 Beta-BHC = NE Heptachlor epoxide = 0.014
95% UCL Concentration		0.033	0.290	0.093	Aldrin = 0.0017 Beta-BHC = NC Heptachlor epoxide = NC

Only organochlorine pesticides detected are shown; all other organochlorine pesticides were detected beneath laboratory detection limits
UCL Upper confidence limit
ND None of the constituents detected over laboratory detection limits
NC Not calculated due to low concentrations detected and/or due to low frequency of detection
Concentrations shown in bold print exceed either PRG or RBSL values for that compound.
* Preliminary Remediation Goal-EPA Region 9, November 2000
** Risk-Based Screening Levels – California Regional Water Quality Control Board, 2000
NE Not Established
< Compound not detected at stated detection limit



LEGEND

● - Approximate location of surface soil sample

⊞ - Approximate location of test pit

Base approximated from Lowney Associates field notes dated 1/7/02.

Not To Scale

1/02*EB

SITE PLAN

872 RUNNYMEDE STREET
East Palo Alto, California

LOWNEY ASSOCIATES
Environmental/Geotechnical/Engineering Services

FIGURE 2
1788-1

Appendix 2a

ORGANOCHLORINE PESTICIDES AND POLYCHLORINATED BIPHENYLS (PCBs)

SW-846 Method 8081 or 8080

Table 1A. Summary of Holding Times and Preservation for Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs)

Analytical Parameter [a]	**Technical and Contract Holding Times**	**Preservation**
Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) in Water Samples	Technical for Extraction: 7 days from collection; Contract for Extraction: 5 days from receipt at laboratory Technical and Contract for Analysis: 40 days from extraction	Cool to 4°C ±2°C
Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) in Soil Samples	Technical for Extraction: 14 days from collection; Contract for Extraction: 10 days from receipt at laboratory Technical and Contract for Analysis: 40 days from extraction	Cool to 4°C ±2°C

[a] Individual target compounds are listed in Table 1B.

Data Calculations and Reporting Units:

Calculate the calibration factors (CF) of single component pesticides according to Section 7.4.2 of SW-846 Method 8000A. Calculate sample results using the analyte CFs from the midpoint standard of the associated initial calibration curve. Perform sample quantitation for multiple components pesticides according to Section 7.6 of SW-846 Method 8080A or 8081.

Report water sample results in concentration units of micrograms per liter (μg/L). Report soil sample results on a dry-weight basis in micrograms per kilogram (μg/kg).

For rounding results, adhere to the following rules:
a) If the number following those to be retained is less than 5, round down;
b) If the number following those to be retained is greater than 5, round up; or

c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

TABLE 1B. Target Compound List, CAS Numbers, and Contract Required Quantitation Limits (CRQL) for SW-846 Method 8081 or Method 8080

COMPOUND	CAS No.	CRQL Water µg/L	CRQL Soil µg/kg
alpha-BHC	319-84-6	0.05	2
beta-BHC	319-85-7	0.05	2
delta-BHC	319-86-8	0.05	2
gamma-BHC (Lindane)	58-89-9	0.05	2
Heptachlor	76-44-8	0.05	2
Aldrin	309-00-2	0.05	2
Heptachlor epoxide	1024-57-3	0.05	2
Endosulfan I	959-98-8	0.05	2
Dieldrin	60-57-1	0.1	3
4,4'-DDE	72-55-9	0.1	3
Endrin	72-20-8	0.1	3
Endosulfan II	33213-65-9	0.1	3
4,4'-DDD	72-54-8	0.1	3
Endosulfan sulfate	1031-07-8	0.1	3
4,4'-DDT	50-29-3	0.1	3
Methoxychlor	72-43-5	0.5	17
Endrin ketone	53494-70-5	0.1	3
Endrin aldehyde	7421-93-4	0.1	3
alpha-Chlordane	5103-71-9	0.05	2
gamma-Chlordane	5103-74-2	0.05	2
Toxaphene	8001-35-2	5	170
Aroclor-1016	12674-11-2	1	33
Aroclor-1221	11104-28-2	2	67
Aroclor-1232	11141-16-5	1	33
Aroclor-1242	53469-21-9	1	33

Aroclor-1248	12672-29-6	1	33
Aroclor-1254	11097-69-1	1	33
Aroclor-1260	11096-82-5	1	3

Table 2. Summary of Calibration Procedures for Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) by SW-846 Method 8081 or 8080

Calibration Element	**Frequency**	**Acceptance Criteria**	**Corrective Action**
Initial Calibration (minimum blank + 3 points for each analyte) (ICAL) [a, b,c]	Initially; whenever required, due to failure of CCV	RSD for CFs ≤20% (≤30% for Surrogate compounds)	1. Terminate analysis 2. Re-calibrate and verify before sample analysis
Continuing Calibration Verification (CCV) at midpoint of ICAL	Beginning of each day, after every 10 samples, and end of run	%D between CF of CCV and avg CFs from ICAL ≤25%	1. Re-calibrate and verify 2. Re-analyze samples back to last good CCV
Endrin and 4,4'-DDT Breakdown	Beginning and end of analytical sequence	≤20% each or ≤30% combined	1. Investigate source of the problem and document 2. If either Endrin, 4,4'-DDT, or their breakdown products were detected, re-analyze the samples

[a] The ICAL low standard must be above but near the CRQL. The low ICAL standard must have a signal to noise ratio ≥5:1. If this requirement cannot be met, the laboratory must submit a MDL study as part of the data package.

[b] ICAL Prepare initial calibration individual standard mixtures A and B (IND A and IND B) containing the single component pesticides specified in Table 9 of SW-846 Method 8081 at three concentration levels. For multiple response pesticides, including toxaphene and Aroclors (except 1016 and 1260), prepare separate initial calibration standards at the following concentration levels: Aroclors (except 1221)at 100 ng/mL; Aroclor-1221 at 200 ng/mL; and toxaphene at 500 ng/mL. Aroclor-1016 and Aroclor-1260 may be combined into a single standard solution. Spike all calibration standards with the surrogate compounds discussed in Table 3 at a concentration of 20 ng/mL.

[c] Report the retention time window for each analyte. For multiple component pesticides, calculate the retention time window for 5 major peaks from the initial calibration standard analysis.

Determine retention time windows for both single and multiple component pesticides using the following guidelines:

Column Type	Retention Time Window in Minutes
Packed Column	≤± 2%
Mega bore or wide bore capillary column	· ±0.05 for tetrachloro-m-xylene through Aldrin · ±0.07 for compounds which elute after Aldrin · ±0.1 for decachlorobiphenyl

Table 3. Summary of Internal Quality Control Procedures for Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) by SW-846 Method 8081 or 8080

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per Batch or SDG[a] (1 per 20 samples minimum)	< CRQL for each compound	1. Investigate source of contamination and document 2. Re-extract and re-analyze all samples processed with a non-compliant method blank
Surrogate [b]	Every standard, sample, method blank and QC sample at 10 times CRQL	60-150% of expected value	1. Re-analyze all samples with non-compliant surrogate recoveries
Matrix Spike and Matrix Spike Duplicate (MS/MSD)[c]	One MS/MSD set per batch or SDG (1 MS/MSD set per 20 samples minimum)	50-135% of expected value; ≤30 RPD between MS and MSD	1. Address in narrative

[a] SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.

[b] Spike each standard, sample, and blank with 1mL of a solution containing 0.2 μg/mL each of tetrachloro-m-xylene and decachlorobiphenyl

[c] Spike MS/MSD samples with 1mL of a solution containing the following compounds and levels:

Target compound	Concentration (μg/mL)	Target Compound	Concentration (μg/mL)
γ-BHC	0.5	Heptachlor	0.5
4,4'-DDT	1.0	Aldrin	0.5
Endrin	1.0	Dieldrin	1.0

Dilute and re-analyze samples with one or more analytes at concentrations exceeding the range of the

calibration curve. Results for such re-analyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.

Second column confirmation is required for all positive results. Perform confirmation analyses on a column of a phase different from that used for quantitation. Confirmation analyses must meet all instrument calibration criteria and blank acceptance criteria specified in Table 2, above.

Appendix 2b

Title 22 Metals by Inductively Coupled Plasma (ICP), Graphite Furnace Atomic Absorption (GFAA), and Cold Vapor Atomic Absorption (CVAA)

SW-846 Method 6010: Inductively Coupled Plasma (ICP);
SW-846 Method 7000: Graphite Furnace Atomic Absorption(GFAA); and
SW-846 Method 7470/7471: Mercury Analysis by CVAA;

Table 1A. Summary of Holding Times and Preservation for Title 22 Metals

Analytical Parameter [a]	**Technical and Contract Holding Times**	**Preservation**
Metals in water (except Mercury)	Technical: 180 days from date of collection; Contract: 35 days from sample receipt at laboratory	pH <2 (with nitric acid)
Metals in soil (except Mercury)	Technical: 180 days from date of collection; Contract: 35 days from sample receipt at laboratory	Cool to 4°C ±2°C
Mercury in water	Technical: 28 days from date of collection; Contract: 26 days from sample receipt at laboratory	pH <2 (with nitric acid)
Mercury in soil	Technical: 28 days from date of collection; Contract: 26 days from sample receipt at laboratory	Cool to 4°C ±2°C

[a] Individual target compounds are listed in Table 1B.

Sample Preparation:

Water samples are to be prepared following the protocol presented in SW-846 Method 3010. Soil samples are to be prepared following the protocol presented in SW-846 Method 3050.

Data Calculations and Reporting Units:

Calculate the sample results according to the protocol of the appropriate analytical method used.

Report water sample results in concentration units of micrograms per liter (μg/L), and soil sample results in concentrations units of milligrams per kilogram (mg/kg) on a dry weight basis. Report percent solids to the nearest percent.

For rounding results, adhere to the following rules:
a) If the number following those to be retained is less than 5, round

down;
  b) If the number following those to be retained is greater than 5, round up; or
  c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.
All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

TABLE 1B. Target Compound List, CAS Numbers, and Contract Required Detection Limits for Title 22 Metals by ICP, GFAA, and CVAA

COMPOUND	CAS No.	CRDL for Water (µg/L)	CRDL for Soil (mg/Kg)
Aluminum	7429-90-5	50	10.0
Antimony	7440-36-0	6	2.5
Arsenic	7440-38-2	2	1.0
Barium	7440-39-3	20	4.0
Beryllium	7440-41-7	1	0.25
Cadmium	7440-43-9	1	0.5
Chromium	7440-47-3	10	2.0
Cobalt	7440-48-4	20	4.0
Copper	7440-50-8	20	4.0
Lead	7439-92-1	5	1.0
Mercury	7439-97-6	0.2	0.1
Molybdenum	7439-97-6	20	4.0
Nickel	7440-02-0	10	4.0
Selenium	7782-49-2	5	1.0
Silver	7440-22-4	20	4.0
Thallium	7440-28-0	1	1.0
Vanadium	7440-62-2	20	4.0
Zinc	7440-66-6	20	5.0

Table 2A. Summary of Calibration Procedures for Title 22 Metals by ICP

Calibration Element	**Frequency**	**Acceptance Criteria**	**Corrective Action**
Initial Calibration (minimum blank + 1 calibration standard) (ICAL)	Initially, Daily; whenever required, due to failure of CCV	Acceptable ICV, CRDL, and ICB standards	1. Terminate analysis 2. Re-calibrate and verify before sample analysis
Initial Calibration Verification (ICV) at midpoint of ICAL (Different source from ICAL standards)	Daily, immediately following ICAL and prior to sample analysis	±10% from expected concentration	1. Terminate analysis and identify and document problem 2. Reprep and re-analyze ICV and all associated samples 3. Re-calibrate and re-analyze reprepped ICV and all associated samples
Calibration Blank Verification (ICB, CCB)	After ICV and every CCV	< CRDL	1. Terminate analysis 2. Determine Source of contamination 3. Reprep ICB and CCB 4. Re-analyze all samples associated with a contaminated blank
Continuing Calibration Verification (CCV)	Before samples, after every 10 samples, and end of run	± 10% from expected concentration	1. Re-calibrate and verify 2. Re-analyze samples back to last acceptable CCV
Contract Required Detection Limit Verification Standard (CRI)	After ICV, but before sample analysis	±35% from expected concentration	1. Re-calibrate and verify 2. Re-analyze samples back to last compliant CCV
ICP Interference Check Sample (ICS)	Run at start and finish of daily run or twice per 8 hours	± 20% from true value concentration	1. Reprep and re-analyze standard 2. Re-calibrate, verify and re-analyze all associated samples

Table 2B. Summary of Calibration Procedures for Title 22 Metals by GFAA

Calibration Element	**Frequency**	**Acceptance Criteria**	**Corrective Action**
Initial Calibration (minimum blank + 3 standards for each analyte) (ICAL) [a]	Initially, each analytical batch; whenever required, due to failure of CCV	r ≥ 0.995	1. Terminate analysis 2. Re-calibrate and verify before sample analysis
Initial Calibration Verification (ICV) at midpoint of ICAL (Different source from ICAL standards)	Daily, immediately following ICAL and prior to sample analysis	±10% from expected concentration	1. Terminate analysis and identify and document problem 2. Reprep and re-analyze ICV and all associated samples 3. Re-calibrate and re-analyze reprepped ICV and all associated samples
Calibration Blank Verification (ICB, CCB)	After ICV and every CCV	< CRDL	1. Terminate analysis 2. Determine Source of contamination 3. Reprep ICB and CCB 4. Re-analyze all samples associated with a contaminated blank
Continuing Calibration Verification (CCV)	Following ICV and before sample analysis; after every 10 samples and end of run	± 10% from expected concentration	1. Re-calibrate and verify 2. Re-analyze samples back to last acceptable CCV
Contract Required Detection Limit Verification Standard (CRA)	After ICV, but before sample analysis	± 35% from expected concentration	1. Reprep and re-analyze standard 2. Re-calibrate and verify

[a] The ICAL low standard must be between the CRDL and 2X CRDL.

Table 2C. Summary of Calibration Procedures for Mercury by CVAA

Calibration Element	**Frequency**	**Acceptance Criteria**	**Corrective Action**
Initial Calibration (minimum blank + 5 standards) (ICAL) [a]	Initially, each analytical batch; whenever required, due to failure of CCV	r ≥ 0.995	1. Terminate analysis 2. Re-calibrate and verify before sample analysis
Initial Calibration Verification (ICV) at midpoint of ICAL (Different source from ICAL standards)	Daily, immediately following ICAL and prior to sample analysis	±20% from expected concentration	1. Terminate analysis and identify and document problem 2. Reprep and re-analyze ICV and all associated samples 3. Re-calibrate and re-analyze reprepped ICV and all associated samples
Calibration Blank Verification (ICB, CCB)	After ICV and every CCV	< CRDL	1. Terminate analysis 2. Determine Source of contamination 3. Reprep ICB and CCB 4. Re-analyze all samples associated with a contaminated blank
Continuing Calibration Verification (CCV)	Before Samples, after every 10 samples, and end of run	±20% from expected concentration	1. Re-calibrate and verify 2. Re-analyze samples back to last acceptable CCV
Contract Required Detection Limit Verification Standard (CRA)	After ICV, but before sample analysis	± 35% from expected concentration	1. Reprep and re-analyze standard 2. Re-calibrate and verify

[a] The ICAL low standard must be at the CRDL.

Table 3. Summary of Internal Quality Control Procedures for Title 22 Metals by ICP, CVAA, and GFAA

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per batch or SDG [a, b]	< CRDL	1. If lowest sample concentration is more than 10X the blank conc., no action 2. If samples are non-detected, no action 3. If detected sample concentrations are less than 10X blank conc., all affected samples must be prepared again with another method blank and re-analyzed
Duplicate Sample (DUP)	One per batch or SDG [a, b]	**Waters:** RPD <± 20% for samples >5X CRDL; ± CRDL for samples <5X CRDL **Soils:** RPD <± 35% for samples >5X CRDL; ± 2xCRDL for samples <5X CRDL	1. Flag associated data with an "*"
Matrix Spike Sample (MS)	One per batch or SDG [a, b]	± 25% from expected value [c]	1. Flag associated data with an "N"
Laboratory Control Sample (LCS)	One per batch or SDG [a, b]	**Waters:** ± 20% from expected concentration **Soils:** within control limits of certified solid LCS or ± 20% from expected spike concentration	1. Terminate analysis and identify and document the problem 2. Re-analyze all associated samples
Serial Dilution Sample (5 X Dilution) (ICP only)	One per batch or SDG [a, b]	± 10% difference from original results for analytes greater than 50 X IDL	1. Flag associated data with a "B"

Table 3. (cont) Summary of Internal Quality Control Procedures for Title 22 Metals by ICP, CVAA, and GFAA

QC Element	Frequency	Acceptance Criteria	Corrective Action
Duplicate Injections (GFAA only)	All samples	Duplicate results within ± 20% RPD (or CV)	1. Rerun sample once 2. Flag associated data with an "E" if acceptance criteria are not met after second run
Analytical Spike Sample (2 X CRDL) (GFAA only)	All samples	Spike recovery ± 15%	1. If spike recovery is <40%, dilute sample by a factor of 5 to 10 and run again. If recovery is still <40%, report data and flag with an "E" to indicate interference problems 2. If sample concentration is **<**50% of recovered spike value and spike recovery is >40% and <85% or >115%, report result down to IDL and flag result with a "W" 3. If sample concentration is **>**50% of recovered spike value and spike recovery is >40% and <85% or >115%, quantitate by MSA
Method of Standard Addition (GFAA only)	As determined by analytical spike recovery results	3 samples spiked at 50%, 100%, and 150% of sample concentration and r ≥ 0.995	1. Rerun samples only once 2. Flag associated data with a "+" if acceptance criteria are not met after second run

[a] SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.

[b] Minimum requirement is the analysis of 1 QC sample per 20 samples.

[c] An exception to this rule is granted in situations where the sample concentration exceeds the spike concentration by a factor of 4. In such an event, the data shall be reported unflagged.

Dilute and reanalyze samples with concentrations exceeding the range of the calibration curve. Results for such re-analyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.

Appendix 3

U.S. EPA REGION IX LABORATORY
RICHMOND, CALIFORNIA

STANDARD OPERATING PROCEDURE #930

Immunoassay (ELISA) Method for DDT Detection

Prepared by: _____ _____
Greg Nagle, Field and Biology Team Date

Reviewed by: _____ _____
K. W. Hendrix, QA Officer Date

Approved by: _____ _____
Brenda Bettencourt, Lab Director Date

Periodic Review:

Signature	Title	Date
_____	_____	_____
_____	_____	_____
_____	_____	_____

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

1.1 This SOP describes a procedure used for screening soils to determine 1,1,1-trichloro-2,2-bis(4-chlorophenyl) ethane (DDT) and its breakdown products (DDD, DDE and DDA) concentrations using a competitive enzyme-linked immunosorbent assay (ELISA). The immunoassay technique provides a single quantitative result, reported as DDT, for all compounds detected. The procedure described is based on EPA SW-846 Standard Method 4042.

1.2 The products used in this procedure are commercially available from Strategic Diagnostics, Inc. (SDI).

1.3 The method detection limit (MDL) submitted by the manufacturer of the testing product is 0.2 ppm for DDT in soil matrix samples. The actual detection limit may be depend on the more specific sample matrix (i.e. sediment, sludge, etc.) and analyst's performance. This method is appropriate for detection of DDT concentrations in the range of 0.2 to 10 ppm.

1.4 Immunoassay techniques use antibody molecules that bind to the target analyte (in this case, DDT) as well as other chemicals. Hence, an immunoassay has a tendency to overestimate the concentration of the target analyte when other analytes that may bind with the antibody are present. Thus, the specificity of this procedure for DDT is partly a function of the cross-reactivity of those other compounds (see Section 4, Table 1).

1.5 For large numbers of samples, this immunoassay technique may be preferable to traditional Gas Chromatography (GC) methods because of its, quickness (approximately 2 hour total analysis time for 30 samples), ease of sample preparation, and relatively low cost (at least 20% less than GC). We estimate that approximately 60 samples can be analyzed in one day.

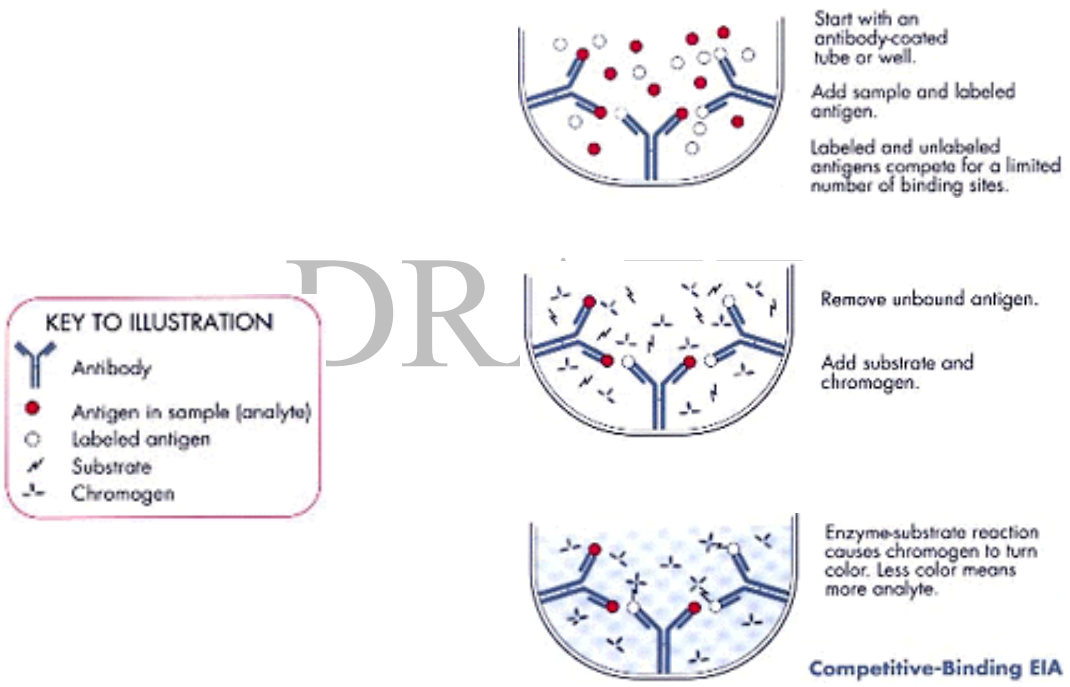
1.6 Immunoassays are to performed in the EPA Region 9 Laboratory Bioassay Laboratory, Room 308, or in the field.

2.0 SUMMARY OF METHOD

An accurately measured volume of sample is mixed with a volume of enzyme-DDT conjugate reagent in a test coated with anti-DDT antibodies. The conjugate "competes" with the DDT present in the sample for binding to the anti-DDT antibody immobilized on the walls of each tube. The mixture is incubated at room temperature. Unbound conjugate and sample analyte that may be present in tubes are removed by rinsing with washing solution. A signal-generating substrate/chromogen reagent is added and the plate is incubated again at room temperature. A stop solution is added to the wells of the plate to terminate the signal-generating activity of the enzyme- conjugate reagent. The absorbance is measured at a wavelength of 450 nm. The test is interpreted by measuring the signal produced by a sample and determining the concentration from a dose-response curve constructed from standards tested at the same time. The color (signal) developed during the test is inversely

proportional to the concentration of DDT in the sample. Thus, the darker the solution, the lower the concentration of DDT in the sample.

Figure 1. Competitive ELISA.
[Source: Strategic Diagnostics, Inc.
http://www.sdxi.com/techov.html]



3.0 DEFINITIONS

Accuracy: A measure of the bias in a system when compared to a reference procedure.

Antibody: A serum-based protein or ascites which specifically recognizes and binds to an antigen or set of antigens.

Antigen: A substance that elicits an immune response and reacts specifically with an antibody. For most immunoassays used for environmental monitoring, the antigen consists of a hapten (target analyte or analog) covalently linked to a protein carrier.

Coefficient of Variation (CV): A term for analytical precision defined as the standard deviation divided by the mean and multiplied by 100, also known as percent relative standard deviation.

Comparability: An expression of the confidence with which one data set can be compared to another. For example, at least 10% of the immunoassay results should be compared with results generated by GC/MS.

Completeness: A measure of the amount of valid data obtained from a measurement system compared with the amount of valid data expected.

Cross-Reactivity: A measure of a compound's affinity for binding with antibodies for another analyte. Many compounds may have similar traits as the desired analyte and may be detected (in varying degrees) by the anti-analyte antibodies, giving false positive results.

Data Quality: The totality of features and characteristics of data that bear on the ability of the data to satisfy a given purpose. The characteristics of major importance are accuracy, precision, completeness, representativeness, and comparability.

Data Validation: Systematic process for reviewing a body of data against a set of criteria, including data editing, screening, checking, auditing, verification, certification, and review.

Detectability: Sensitivity of a method to accurately measure a substance in a specified concentration range. Also known as Limits of Detection, or LOD.

Immunoassay: An analytical tool which employs a wide range of methods to quantify antigens or antibodies.

Interferences: Substances or conditions found in an environmental matrix that interfere with accurate measurements of the target analyte.

Optical Density (OD): Unit of measurement in spectrophotometry, also called absorbance.

Precision: Standard deviation is a measure of precision. It is more statistically correct to refer to standard deviation as a measure of scatter or dispersion. An evaluation of replicate measurements in a single run, in

day-to-day runs, and runs by different laboratories, all under prescribed similar conditions.

Quality Assurance (QA): The total integrated program for assuring the reliability of monitoring and measurement data. A system for integrating the quality planning, quality assessment, and quality improvement efforts to meet user requirements.

Quality Control (QC): The routine application of procedures to determine if a method is performing within established criteria.

Representativeness: An expression of the degree to which data accurately and precisely represent a characteristic of a population.

Specificity: The ability of an antibody to recognize and attach to corresponding antigens and no other unrelated antigen.

Standard Operating Procedure (SOP): A document which details an operation, analysis, or action whose mechanisms are established and an acceptable method for performing certain procedures.

4.0 INTERFERENCES

4.1 Cross-Reactivity

4.1.1 The Enviroguard DDT is Soil Test Kit will not differentiate between DDT and other structurally similar compounds, but will detect their presence to differing degrees. Compounds that are chemically similar to DDT may cause a positive test result (false positive) for DDT. This phenomenon is known as cross-reactivity. The ELISA kit used in this procedure has been evaluated for cross-reactivity by the manufacturer. Table 1 shows the compounds and the approximate concentration at which known cross-reactants will yield a positive result at the low calibration standard and in this case the method detection limit. It also shows the concentration required to inhibit one-half of the color developed by the Negative Control (IC50)

4.1.2 The presence of cross-reacting compounds will result in an increase in the calculated concentration of the sample being analyzed and therefore influence the incidence of false positive results. Confirm a certain percentage (usually 10%) of positive results using another analytical technique such as GC/Electron Capture (EPA Method 8081A). False negative results are generally not a concern with immunoassay techniques.

Table 1. Cross-Reactivities of DDT Compounds.

Compound	MDL (ppm)	IC50 (ppm)
p,p'-DDT	0.2	1.25
p,p-DDD	0.05	0.3
p,p'-DDE	0.6	3.6
o,p'-DDT	14.9	93
o,p-DDD	1.76	11
o,p'-DDE	14.9	93
DDA	0.01	0.04
Chloropropylate	0.01	0.08
Chlorobenzilate	0.06	0.35
Dicofol	0.3	2
Thiobencarb	5.0	52
Tebuconazole	7.0	95
Neburon	17	284
Chloroxuron	24	216
Monolinuron	25	714
Diclofop	70	>1000
Tetradifon	1.2	14

The following analytes are not detected at or above 100 ppm:		
2,4-D	4-Chlorophenoxyacetic acid	Chlordane
Penatchlorophenol	Chlorobromuron	Chlortoluron
Dicamba	Diflubenzuron	Diuron
Lindane	Linuron	MCPA acid
MCPB	Mecoprop	Gasoline
Diesel	2,4,6-Trinitrotoluene	Toxaphene

4.2 Matrix Effects

4.2.1 Non-specific interferences such as sample pH, temperature, osmolarity, solvents, surfactants, and the

presence of metal ions can effect immunoassay performance.

4.2.2 ELISA tests are prone to matrix effects due to the presence of interference agents (e.g. suspended solids) that interfere with antibody binding events. Since ELISAs default to false positives, the effect of these interference agents leads to a false positive basis in analytical results. Matrix effects need to be considered if the assay described below is used on "dirty" matrices such as sludges.

5.0 EQUIPMENT/APPARATUS

< Various calibrated pipettes
< Multi-channel pipettor and pipette tips
< Waste beaker
< Parafilm® or acetate tape
< Timer
< Orbital shaker
< Marking pen
< Tape
< KimWipes®
< Protective Gloves
< Sorbent paper (Paper towels)

6.0 REAGENTS AND STANDARDS

NOTE: Due to the sensitive nature of their materials, ELISA kits must be handled with special care.

- Store all kit components at 4EC to 8EC when not in use.
- Unused plate strips should be stored in a resealable plastic bag with desiccant.
- Allow all reagents and samples to reach ambient temperature (18E to 27EC) before beginning analysis.
- Do not store kit components at room temperature for more than 8 hours.
- Check the expiration date on each kit before using. Do not use expired materials.
- Do not mix reagents kits with different lot numbers.

6.1 Reagents/Supplies

Included in Test Kit:
< 20 Antibody Coated Tubes
< 1 vial Assay Dilutent
< 1 vial Negative Control (Methanol)
< 1 vial 0.2 ppm DDT Calibrator (in Methanol)

< 1 vial 1.0 ppm DDT Calibrator (in Methanol)
< 1 vial 10.0 ppm DDT Calibrator (in Methanol)
< 1 vial Enzyme Conjugate
< 1 vial Substrate
< 1 vial Stop Solution
< 1 -20 place Test Tube Rack
< 22 Yellow (2-25ml) Gibson Microman® positive displacement pipette tips

Included in Extraction Kits: (required, available through vendor)
< 12 Extraction Jars with screw caps (each bottle contains 3 stainless steel mixing beads)
< 12 filter modules (tops and bottoms)
< 12 Ampul crackers
< 12 Wooden spatulas
< 12 Weigh Canoes
< 12 Bulb Pipettes
< 12 Disposable Transfer Pipettes
< 12 Ampules containing 10 mL each of 100% Methanol

Included in Accessory Kit (required, available through vendor)
< GibsonM-25 Microman® positive Displacement Pipettor
< Eppendorf™ Repeater® Pipettor
< Electronic timer
< Polystyrene test tubes, 12X75 mm (for blanking spectrophotometer)
< Portable balance capable of weighing 10 grams
< Wash Bottle
< 5.0 mL Combitips® for Repeater pipettor
- for 0.1 mL to 0.5 mL dispensing volumes (3)
< 12.5 mL Combitips® for Repeater pipettor
- for 0.25 mL to 1.25 mL dispensing volumes (6)
< 50.0 mL Combitips® for Repeater pipettor
- for 0.1 mL to 0.5 mL dispensing volumes (1)
< Thirty position foam racks (2)
< Artel differential photometer - allows you to measure results in the form of optical density (OD) values. These values can be used for objective record keeping and quality assurance.

6.2 Standards
Standards are provided in the kit. At least 3 calibrators should be used to ensure a good fit for the standard curve.

6.2.1 Allow the DDT standard solutions to come to room temperature. Swirl vials to mix solution before pipetting.

7.0 PROCEDURES

7.1 Sample Collection and Preservation

7.1.1 Samples should be collected in pre-cleaned glass containers. Soils obtained from areas adjacent to standing water, surface soils collected immediately after rain or snow, or any soils with relatively high amounts of water ($30% by weight) should be dried before testing.

7.1.2 The immunoassay testing products employ 10 gram sample volumes. Distribution of DDT in soils may be highly variable. This variability can be minimized through use of a composite sampling technique. Sample collection procedures should focus on the volume necessary to ensure that the sample represents the source.

7.2 Sample Preparation/Extraction

7.2.1 Take care to remove excess twigs, organic matter and rocks or pebbles from the soil sample to be tested. Using the wooden spatulas, weighing canoes and portable balance weigh approximately 10 grams (±0.1 grams).

7.2.2 Add the 10 gram sample to the screw top extraction jar containing the stainless-steel mixing beads and 10 mL of methanol. Shake vigorously for 2 minutes. If the sample soaks up all the methanol leaving no excess liquid to filter, add an additional 10 mL of methanol and shake for an additional 2 minutes. **Note any dilution factors for use in the final calculations.** Allow the sample to settle for one minute or until a liquid solvent layer is observed above the sample.

7.2.3 Filter the extract by inserting the bulb pipette into the top (liquid) layer in the extraction jar (being careful not to draw up some of the sample. Transfer at least ½ bulb capacity into the bottom portion of the filtration unit. **Do not use more than one full bulb.**

7.2.4 Press the top portion of the filtration unit (which is the piece with the cap and filter) into the bottom portion (containing the sample) until it snaps together or until the majority of the liquid has passed through the filter. Place on a flat surface.

7.3 Sample Analysis

7.3.1 Allow all the test kits components to come to ambient temperature (at least 1 hour) before use.

7.3.2 Remove the Antibody coated test tubes from the foil pouch and label as follows (no more than twenty tubes/assay).

Tube Label	Tube Contents
NC	Negative Control
C1	0.2 ppm Calibrator
C2	1.0 ppm Calibrator
C3	10.0 ppm Calibrator
S1	Sample 1
S2	Sample 2, etc...

7.3.3 Place the test tubes in the test tube rack pressing down firmly on each tube so that they are secured.

7.3.4 Position the Repeater pipettor at setting 2 and use the **12.5 mL** syringe to **500 FL** of Assay Diluent to all test tubes.

7.3.5 Attach a clean yellow pipette up to the positive displacement pipette and adjust the dial to **"250"** to pipette **25 FL**.

7.3.6 Use the positive displacement pipettor to add the Negative Control (methanol), the DDT Calibrators, and the Sample extracts to the appropriate test tubes. **Use a clean pipette tip for each addition. Replace the cap(s) on the calibrator vials immediately after use to minimize evaporation.**

7.3.7 Attach the **5.0 mL** Combitip labeled "Conjugate" to the repeater pipettor and adjust the dial to 1 to a **100 FL** of the DDT-Enzyme Conjugate to each tube.

7.3.8 Gently shake the test tube rack to mix the 10 to 15 seconds. Leave the tubes undisturbed for **15 minutes.**

7.3.9 Vigorously shake out the test tube contents into a sink or suitable container. Fill the test tubes to overflowing with cool tap, or distilled water, then decant and vigorously shake out any remaining water. Repeat this step three more times, being certain to shake out as much water as possible on each wash. After the final wash, remove as much water as possible by tapping the inverted tubes on sorbent paper.

7.3.10 Position the Repeater pipettor at setting 2 and use a clean **125 mL** Combitip to add **500 FL** of Substrate to all test tubes. Briefly shake the test tube rack to mix, then incubate for **10 minutes** . *If a blue color does not develop within 10 minutes after you add the substrate solution, the test is invalid and the entire test must be repeated.*

7.3.11 Position the Repeater pipettor at setting 2 and use a 12.5 mL syringe to add 500 FL of Stop Solution (1.0 N Hydrochloric Acid) to all test tubes. This will turn the color from blue to yellow.

8.0 Interpretation/Calculation

While results can be visually interpreted 10 minutes after adding the Substrate to each tube, a more precise determination of the concentration shall be performed using a photometer after adding the stop solution. All sample concentrations will be determined by photometer within 30 minutes of the addition of the stop solution as follows.

8.1 Dry the outside of all assay tubes prior to the photometric analysis.

8.2 Place a blank test tube (from the EnviroGard Field Accessory Kit) containing 1.5 mL of deionized water in the left (reference well) of the differential photometer.

8.3 Place the Negative Control test tube into the right (sample) well. Record the optical density (OD) of the Negative Control.

8.4 Remove the Negative Control test tube and replace it with 0.2 Calibrator test tube to reactivate the photometer. Record the result. Repeat this step to determine the OD for each of the remaining calibrators and for each sample.

8.5 If the sample OD is equal to the OD of a calibrator, the sample contains DDT at a concentration approximately equal to the calibrator.

8.6 If the sample OD is greater to the OD of a calibrator, the sample contains DDT at a concentration less than the calibrator.

8.7 If the sample OD is lower to the OD of a calibrator, the sample contains DDT at a concentration greater than the calibrator.

Tube	OD	Interpretation
NC	1.07	
C1 (0.2 ppm)	0.80	
C1 (0.2 ppm)	0.57	
C1 (0.2 ppm)	0.32	
S1	0.64	>0.2 ppm < 1ppm
S2	0.16	>10 ppm

8.8 If an assay result indicates that a soil sample contains greater than 10 ppm total DDT, and more specific information is needed, the soil extract may be diluted up to 1:100 in methanol and assayed again. You must multiply the re-assay by 100 to determine the approximate extract concentration.

9.0 QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

9.1 Initial Demonstration of Capability
The following tests should be run for new matrices (e.g., sediment, sludge, etc.).

9.1.1 Performance Evaluation sample–analyze a performance evaluation sample or lab quality assurance officer approved substitute and submit the results as raw data to the lab QA officer.

9.1.2 A site specific matrix spike (MS) sample should be analyzed to demonstrate the efficiency of the method prior to mobilization to the field. The matrix spike sample should contain DDT at concentrations expected to be found in the bulk of the samples, or at the regulatory limit of interest (if sampling is done for regulatory purposes). The sample chosen for spiking should be representative of the field samples being analyzed. Spike recovery should fall within 65-135% .

9.2 Routine Quality Control

Routine quality control procedures associated with this procedure include the analyses of standards, negative controls (as specified by the manufacturer) and duplicate analyses for each analytical batch (i.e., >20 samples). All of the analyses described below must be conducted simultaneously, e.g., as part of the same batch of samples.

9.2.1 The matrix duplicate (DUP) sample must conducted simultaneously as the primary sample.

9.3 Sample Dilutions

If the sample concentration is outside of the calibrated range demonstrated by the initial calibration and as specified by the manufacturer, then the sample must be diluted to within the calibration range and re-tested. Given the nature of the competitive immunoassay, the sample cannot be diluted *after* color development. Thus, a diluted aliquot of the original sample extract re-analyzed.

9.4 Validation of Results

Ten percent of all positive samples should be verified by an outside party using EPA Method SW 8081A.

9.5 Other Quality Control Considerations

9.5.1 Do not use testing products past their expiration date.

9.5.2 Do not mix the equipment, supplies, and reagents from the testing products for different analytes, or from the testing products from different manufacturers.

9.5.3 Use the testing products within the storage temperature and operating temperature limits specified by the manufacturer.

10.0 DATA VALIDATION

For any project, the data by this analysis is checked to determine whether it meets the objectives stated for the seven basic elements of data quality: representativeness, completeness, comparability, accuracy, precision, specificity, and detectability.

Statistical analysis is an integral part of assessing the data quality in terms of the data quality objectives (DQO). Choice of the correct statistical approach is extremely important, because it is the primary means of providing estimates of the reliability of the data. One important analysis is determination of the coefficient of variation (CV), which can be determined from the components of variance. This gives a statistical estimate of the variation of means of duplicates for multiple assay runs and helps to determine the precision of the method employed.

One problem in evaluating the precision of immunoassay experiments is that the experimental error variance may not be constant. If the relation between the error variance and the true value of standard reference samples is distinct, then that relation can be used to evaluate the precision of the immunoassay.

11.0 HEALTH AND SAFETY

No extraordinary safety measures are required. However, safety procedures consistent with good laboratory practices should be employed. Some reagents may contain dilute acid solutions. Avoid contact with eyes, skin, and mucous membranes. Personal protective equipment including lab coat, safety glasses or goggles, and gloves should be worn at all times.

12.0 REFERENCES

SDI Product Profile, EnviroGard DDT in Soil Test Kit.

USEPA. 1996. SW-846 Method 4042: Soil Screening for DDT by Immunoassay.

Appendix 4

U.S. EPA REGION IX LABORATORY
RICHMOND, CALIFORNIA

STANDARD OPERATING PROCEDURE #935

Immunoassay (ELISA) Method for Cyclodiene Detection

Prepared by: _____ _____
Greg Nagle, Field and Biology Team Date

Reviewed by: _____ _____
K. W. Hendrix, QA Officer Date

Approved by: _____ _____
Brenda Bettencourt, Lab Director Date

Periodic Review:

Signature	Title	Date
_____	_____	_____
_____	_____	_____
_____	_____	_____

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

1.1 This SOP describes a procedure used for screening soils to determine cyclodiene concentrations using a competitive enzyme-linked immunosorbent assay (ELISA). The cyclodiene insecticides encompass a large group of polychlorinated cyclic hydrocarbons with a endomethylene bridged structures. Among them are two pairs of stereoisomers: aldrin and isodrin, deldrin and endrin. Other cyclodiene compounds of interest include heptachlor, chlordane, endosulfan and isobenzan. The immunoassay technique provides a single quantitative result, reported as Dieldrin, for all compounds detected. The procedure described is based on EPA SW-846 Standard Method 4042.

1.2 The products used in this procedure are commercially available from Strategic Diagnostics, Inc. (SDI).

1.3 The Cyclodienes RaPID Assay® does not differentiate between cyclodienes and other related compounds. The method detection limit (MDL) submitted by the manufacturer of the testing product is 0.045 ppb as dieldrin. The Cyclodience RaPID Assay® has a range of detection in soil of 100 ppb to 2000 ppb (as dieldrin). The actual performance may be depend on the more specific sample matrix (i.e. sediment, sludge, etc.) and analyst's performance.

1.4 Immunoassay techniques use antibody molecules that bind to the target analyte (in this case, cyclodienes) as well as other chemicals. Hence, an immunoassay has a tendency to overestimate the concentration of the target analyte when other analytes that may bind with the antibody are present. Thus, the specificity of this procedure for cyclodienes is partly a function of the cross-reactivity of those other compounds (see Section 4, Table 1).

1.5 For large numbers of samples, this immunoassay technique may be preferable to traditional Gas Chromatography (GC) methods because of its, quickness (approximately 2 hour total analysis time for 30 samples), ease of sample preparation, and relatively low cost (at least 20% less than GC). We estimate that approximately 60 samples can be analyzed in one day.

1.6 Immunoassays are to performed in the EPA Region 9 Laboratory Bioassay Laboratory, Room 308, or in the field.

2.0 SUMMARY OF METHOD

An accurately measured volume of sample is mixed with a volume of enzyme-cyclodienes conjugate reagent in a test coated with anti-cyclodienes antibodies. The conjugate "competes" with the cyclodienes present in the sample for binding to the anti-cyclodienes antibody immobilized on the walls of each tube. The mixture is incubated at room temperature. Unbound conjugate and sample analyte that may be present in tubes are removed by rinsing with washing solution. A signal-generating substrate/chromogen reagent is added and the plate is incubated again at room

temperature. A stop solution is added to the wells of the plate to terminate the signal-generating activity of the enzyme- conjugate reagent. The absorbance is measured at a wavelength of 450 nm. The test is interpreted by measuring the signal produced by a sample and determining the concentration from a dose-response curve constructed from standards tested at the same time. The color (signal) developed during the test is inversely proportional to the concentration of cyclodienes in the sample. Thus, the darker the solution, the lower the concentration of cyclodienes in the sample.

3.0 DEFINITIONS

Accuracy: A measure of the bias in a system when compared to a reference procedure.

Antibody: A serum-based protein or ascites which specifically recognizes and binds to an antigen or set of antigens.

Antigen: A substance that elicits an immune response and reacts specifically with an antibody. For most immunoassays used for environmental monitoring, the antigen consists of a hapten (target analyte or analog) covalently linked to a protein carrier.

Coefficient of Variation (CV): A term for analytical precision defined as the standard deviation divided by the mean and multiplied by 100, also known as percent relative standard deviation.

Comparability: An expression of the confidence with which one data set can be compared to another. For example, at least 10% of the immunoassay results should be compared with results generated by GC/MS.

Completeness: A measure of the amount of valid data obtained from a measurement system compared with the amount of valid data expected.

Cross-Reactivity: A measure of a compound's affinity for binding with antibodies for another analyte. Many compounds may have similar traits as the desired analyte and may be detected (in varying degrees) by the anti-analyte antibodies, giving false positive results.

Data Quality: The totality of features and characteristics of data that bear on the ability of the data to satisfy a given purpose. The characteristics of major importance are accuracy, precision, completeness, representativeness, and comparability.

Data Validation: Systematic process for reviewing a body of data against a set of criteria, including data editing, screening, checking, auditing, verification, certification, and review.

Detectability: Sensitivity of a method to accurately measure a substance in a specified concentration range. Also known as Limits of Detection, or LOD.

Immunoassay: An analytical tool which employs a wide range of methods to quantify antigens or antibodies.

Interferences: Substances or conditions found in an environmental matrix that interfere with accurate measurements of the target analyte.

Optical Density (OD): Unit of measurement in spectrophotometry, also called absorbance.

Precision: Standard deviation is a measure of precision. It is more statistically correct to refer to standard deviation as a measure of scatter or dispersion. An evaluation of replicate measurements in a single run, in day-to-day runs, and runs by different laboratories, all under prescribed similar conditions.

Quality Assurance (QA): The total integrated program for assuring the reliability of monitoring and measurement data. A system for integrating the quality planning, quality assessment, and quality improvement efforts to meet user requirements.

Quality Control (QC): The routine application of procedures to determine if a method is performing within established criteria.

Representativeness: An expression of the degree to which data accurately and precisely represent a characteristic of a population.

Specificity: The ability of an antibody to recognize and attach to corresponding antigens and no other unrelated antigen.

Standard Operating Procedure (SOP): A document which details an operation, analysis, or action whose mechanisms are established and an acceptable method for performing certain procedures.

4.0 INTERFERENCES

4.1 Cross-Reactivity

4.1.1 The RaPID Assay Cyclodiene is Soil Application does differentiate between Cyclodienes and other structurally similar compounds, but will detect their presence to differing degrees. Compounds that are chemically similar to Cyclodienes may cause a positive test result (false positive) for Cyclodienes. This phenomenon is known as cross-reactivity. The ELISA kit used in this procedure has been evaluated for cross-reactivity by the manufacturer. Table 1 shows the compounds and the approximate concentration at which known cross-reactants will yield a positive result at the low calibration standard and in this case the method detection limit. It also shows the concentration required to inhibit one-half of the color developed by the Negative Control (IC50)

4.1.2 The presence of cross-reacting compounds will result in an increase in the calculated concentration of the sample being analyzed and therefore influence the incidence of false positive results. Confirm a certain percentage (usually 10%) of positive results using another analytical technique such as GC/Electron Capture (EPA Method 8081A). False negative results are generally not a concern with immunoassay techniques.

Table 1. Method Detection Limits (MDLs), Limit of Quantitation (LOQ) and Cross-Reactivities of Cyclodiene Compounds.

Compound	MDL (ppb)	LOQ (ppb)	IC50 (ppb)
Dieldrin	45	100	894
Aldrin	220	88	788
Isodrin	32	107	954
Heptachlor-endo-epoxide	47	199	1780
Heptachlor	50	178	1590
Endrin	51	138	1230
Chlordane	81	301	2690
" - Endosulfan	87	499	4460
Isobenzan	104	512	4580
Toxaphene	195	1298	11600
Lindane	1520	9460	84600
Chlorthalonil	159000	NR	NR
p,p-DDD	204000	NR	NR
Phosmet	394000	NR	NR
Propenfos	500000	NR	NR
p,p-DDT	656200	NR	NR
p,p-DDE	NR	NR	NR
Perthane	NR	NR	NR

Note: The IC50 is the concentration in soil required to inhibit one-half of the color produced by the negative control. It is also used to calculate cross-reactivity values to similar compounds.

4.2 Matrix Effects

4.2.1 Non-specific interferences such as sample pH, temperature, osmolarity, solvents, surfactants, and the presence of metal ions can effect immunoassay performance.

4.2.2 ELISA tests are prone to matrix effects due to the presence of interference agents (e.g. suspended solids) that interfere with antibody binding events. Since ELISAs default to false positives, the effect

of these interference agents leads to a false positive basis in analytical results. Matrix effects need to be considered if the assay described below is used on "dirty" matrices such as sludges.

5.0 EQUIPMENT/APPARATUS

< Various calibrated pipettes
< Multi-channel pipettor and pipette tips
< Waste beaker
< Parafilm® or acetate tape
< Timer
< Orbital shaker
< Marking pen
< Tape
< KimWipes®
< Protective Gloves
< Sorbent paper (Paper towels)

6.0 REAGENTS AND STANDARDS

NOTE: Due to the sensitive nature of their materials, ELISA kits must be handled with special care.

- Store all kit components at 4EC to 8EC when not in use.
- Unused plate strips should be stored in a resealable plastic bag with desiccant.
- Allow all reagents and samples to reach ambient temperature (18E to 27EC) before beginning analysis.
- Do not store kit components at room temperature for more than 8 hours.
- Check the expiration date on each kit before using. Do not use expired materials.
- Do not mix reagents from kits with different lot numbers.

6.1 Reagents/Supplies

Included in Test Kit:
< 1 vial Antibody Coupled Magnetic Particles
< 1 Box of 100 test tubes
< 1 vial Assay Dilutent
< 1 vial Negative Control (Methanol)
< 1 vial 100 ppb Cyclodienes Calibrator (in Methanol)
< 1 vial 750 ppb Cyclodienes Calibrator (in Methanol)
< 1 vial 2000 ppb Cyclodienes Calibrator (in Methanol)
< 1 vial Enzyme Conjugate

< 1 vial Substrate
< 1 vial Stop Solution
< 1 -60 Position Test Tube Rack
< 1 -Magnetic Base
< 22 Yellow (2-25ml) Gibson Microman® positive displacement pipette tips

Included in Extraction Kits: (required, available through vendor)
< 12 Extraction Jars with screw caps (each bottle contains 3 stainless steel mixing beads)
< 12 filter modules (tops and bottoms)
< 12 Ampul crackers
< 12 Wooden spatulas
< 12 Weigh Canoes
< 12 Bulb Pipettes
< 12 Disposable Transfer Pipettes
< 12 Ampules containing 10 mL each of 100% Methanol

Included in Accessory Kit (required, available through vendor)
< Gibson M-25 Microman® positive Displacement Pipettor
< Eppendorf™ Repeater® Pipettor
< Electronic timer
< Polystyrene test tubes, 12X75 mm (for blanking spectrophotometer)
< Portable balance capable of weighing 10 grams
< Wash Bottle
< 5.0 mL Combitips® for Repeater pipettor
- for 0.1 mL to 0.5 mL dispensing volumes (3)
< 12.5 mL Combitips® for Repeater pipettor
- for 0.25 mL to 1.25 mL dispensing volumes (6)
< 50.0 mL Combitips® for Repeater pipettor
- for 0.1 mL to 0.5 mL dispensing volumes (1)

Included in the Rapid Assay® Accessory Kit
< RPA-1 Analyzer (Spectrophotmeter)
< Domestic Power Cord/Mains Transformer
< Program Cartridge
< Printer paper
< RPA-1 Analyzer Operator's Manual

6.2 Standards
Standards are provided in the test kit. Three calibration standards at 100, 750 and 2000 ppb should be used to ensure a good fit for the standard curve.

6.2.1 Allow the Cyclodiene standard solutions to come to room temperature. Swirl vials to mix solution before pipetting.

7.0 PROCEDURES

7.1 Sample Collection and Preservation

7.1.1 Samples should be collected in pre-cleaned glass containers. Soils obtained from areas adjacent to standing water, surface soils collected immediately after rain or snow, or any soils with relatively high amounts of water ($30% by weight) should be dried before testing.

7.1.2 The immunoassay testing products employ 10 gram sample volumes. Distribution of Cyclodienes in soils may be highly variable. This variability can be minimized through use of a composite sampling technique. Sample collection procedures should focus on the volume necessary to ensure that the sample represents the source.

7.2 Sample Preparation/Extraction

7.2.1 Take care to remove excess twigs, organic matter and rocks or pebbles from the soil sample to be tested. Using the wooden spatulas, weighing canoes and portable balance weigh approximately 10 grams (±0.1 grams).

7.2.2 Add the 10 gram sample to the screw top extraction jar containing the stainless-steel mixing beads and 10 mL of methanol. Shake vigorously for 2 minutes. If the sample soaks up all the methanol leaving no excess liquid to filter, add an additional 10 mL of methanol and shake for an additional 2 minutes. **Note any dilution factors for use in the final calculations.** Allow the sample to settle for one minute or until a liquid solvent layer is observed above the sample.

7.2.3 Filter the extract by inserting the bulb pipette into the top (liquid) layer in the extraction jar (being careful not to draw up some of the sample. Transfer at least ½ bulb capacity into the bottom portion of the filtration unit. **Do not use more than one full bulb.**

7.2.4 Press the top portion of the filtration unit (which is the piece with the cap and filter) into the bottom portion (containing the sample) until it snaps together or until the majority of the liquid has passed through the filter. Place on a flat surface.

7.3 Sample Analysis

7.3.1 Allow all the test kits components to come to ambient temperature (at least 1 hour) before use.

7.3.2 Remove the upper rack from the magnetic base. Label all test tubes for standards, controls and prepared samples. For example...

Tube Label	Tube Contents
BLK	Blank
C1	100 ppb Standard
C2	750 ppb Standard
C3	2000 ppb Standard
S1	Sample 1
S2	Sample 2, etc...

7.3.3 Deliver 250 FL of standards, controls or prepared samples to the bottom of each test tube by inserting the pipet tip all the way into the tube without touching the sides or the bottom of the tube.

7.3.4 Deliver 250 FL of enzyme conjugate down the inside wall of each tube by aiming the pipet tip ¼" to ½" below the tube rim without touching the rim or tube wall with the pipet tip: deliver the liquid gently.

7.3.5 Deliver 500 FL of thoroughly mixed cyclodiene antibody coupled magnetic particles down the inside of each tube by aiming the pipet tip ¼" to ½" below the tube rim without touching the rim or tube wall with the pipet tip: deliver the liquid gently. Vortex for 1-2 seconds (at low speed to minimize foaming

7.3.6 Incubate for 30 minutes at room temperature (15E-30EC)..

7.3.7 Combine the upper rack with the magnetic base; **press all tubes into base** ; allow 2 minutes for the particles to separate.

7.3.8 Gently shake the test tube rack to mix the 10 to 15 seconds. Leave the tubes undisturbed for **15 minutes.**

7.3.9 Do not separate upper rack from the lower base. Using a smooth motion invert the combined rack assembly over a sink and pour out the tube contents; keep inverted and **gently** blot the test tubes rims on several layers of paper toweling.

7.3.10 Add **1mL** of washing solution down the inside wall of each tube. Vortex each tube. Wait 2 minutes. Using a smooth motion invert the combined rack assembly over a sink and pour out the tube contents; keep inverted and gently blot the test tube rims on several layers of paper toweling.

7.3.11 Lift the upper rack (with its tubes) off the magnetic base; add 500 FL of the color reagent down the inside wall of each tube by aiming the pipet tip ¼" to ½" below the tube rim without touching the rim or tube wall with the pipet tip: deliver the liquid gently. Vortex for 1 to 2 seconds at low speed to minimize foaming.

7.3.12 Incubate for 20 minutes at room temperature (15E-30EC). During this period, add 1 mL of washing solution into a clean test tube for use as an instrument blank.

7.3.13 Add 500 FL of the stopping solution down the inside wall of each tube by aiming the pipet tip ¼" to ½" below the tube rim without touching the rim or tube wall with the pipet tip: deliver the liquid gently. Read results at 450 nm within 15 minutes after adding the Stopping Solution.

8.0 Interpretation/Calculation

Results are interpreted/calculated using a spectrophotometer provided by the vendor. The RPA-1 analyzer is a benchtop based single wavelength, dual beam, microprocessor controlled spectrophotometer. Start-up instructions are provided as Attachment I. To obtain soil results from the Cyclodiene Rapid Assay® test kit on the RPA-1 use the following parameter settings:

Data Reduction	:	Linear Regression
Xformation	:	Ln/LogitB
Read Mode	:	Absorbance
Wavelenght	:	450 nm
Units	:	PPB
# Rgt Blank	:	0

Calibrators

# of Cals	:	4
# of Reps	:	2

Concentrations

#1: 0.00 ppb
#2: 100 ppb
#3: 750 ppb
#4: 2000 ppb

Range	:	100-2000
Correlation	:	0.990
Rep. %CV	:	10%

8.1 Dry the outside of all assay tubes prior to the photometric analysis.

8.2 If an assay result indicates that a soil sample contains greater than 2000 ppb total Cyclodienes, and more specific information is needed, the soil extract may be diluted up to 1:100 in methanol and assayed again. You must multiply the re-assay by 100 to determine the approximate extract concentration.

9.0 QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

9.1 Initial Demonstration of Capability

The following tests should be run for new matrices (e.g., sediment, sludge, etc.).

9.1.1 Performance Evaluation sample–analyze a performance evaluation sample or lab quality assurance officer approved substitute and submit the results as raw data to the lab QA officer.

9.1.2 A site specific matrix spike (MS) sample should be analyzed to demonstrate the efficiency of the method prior to mobilization to the field. The matrix spike sample should contain cyclodienes at concentrations expected to be found in the bulk of the samples, or at the regulatory limit of interest (if sampling is done for regulatory purposes). The sample chosen for spiking should be representative of the field samples being analyzed. Spike recovery should fall within 65-135% .

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9.2.1 The matrix duplicate (DUP) sample must conducted simultaneously as the primary sample.

9.3 Sample Dilutions

If the sample concentration is outside of the calibrated range demonstrated by the initial calibration and as specified by the manufacturer, then the sample must be diluted to within the calibration range and re-tested. Given the nature of the competitive immunoassay, the sample cannot be diluted *after* color development. Thus, a diluted aliquot of the original sample extract re-analyzed.

9.4 Validation of Results

Ten percent of all positive samples should be verified by an outside party using EPA Method SW 8081A.

9.5 Other Quality Control Considerations

9.5.1 Do not use testing products past their expiration date.

9.5.2 Do not mix the equipment, supplies, and reagents from the testing products for different analytes, or from the testing products from different manufacturers.

9.5.3 Use the testing products within the storage temperature and operating temperature limits specified by the manufacturer.

10.0 DATA VALIDATION

For any project, the data by this analysis is checked to determine whether it meets the objectives stated for the seven basic elements of data quality: representativeness, completeness, comparability, accuracy, precision, specificity, and detectability.

Statistical analysis is an integral part of assessing the data quality in terms of the data quality objectives (DQO). Choice of the correct statistical approach is extremely important, because it is the primary means of providing estimates of the reliability of the data. One important analysis is determination of the coefficient of variation (CV), which can be determined from the components of variance. This gives a statistical estimate of the variation of means of duplicates for multiple assay runs and helps to determine the precision of the method employed.

One problem in evaluating the precision of immunoassay experiments is that the experimental error variance may not be constant. If the relation between the error variance and the true value of standard reference samples is distinct, then that relation can be used to evaluate the precision of the immunoassay.

11.0 HEALTH AND SAFETY

No extraordinary safety measures are required. However, safety procedures consistent with good laboratory practices should be employed. Some reagents may contain dilute acid solutions. Avoid contact with eyes, skin, and mucous membranes. Personal protective equipment including lab coat, safety glasses or goggles, and gloves should be worn at all times.

12.0 REFERENCES

SDI Product Profile, EnviroGard Cyclodienes in Soil Test Kit.

USEPA. 1996. SW-846 Method 4000: Immunoassay.

Attachment I

Rapid Assay Start-up Manual

INTRODUCTION

This manual is intended to act as a guide for first time users of the RaPID Assay® kits and the associated equipment and as a reference for experienced users. It contains information on how to set-up the required equipment and run SDI's RaPID Assays. For more detailed explanation, refer to the operating manual for individual pieces of equipment and to the package insert for each assay kit.

Before running the first assay, read thoroughly those sections referring to each piece of equipment to be used (Sections 1 - 8). Next proceed to Sections 9 and 10 to run the assay. Section 11 is provided to assist the operator in resolving problems which might be encounterd.

If any of the material contained in this manual is unclear or if problems are encountered, please feel free to call SDI's Technical Support at (800) 544-8881.

SECTION 1 - RPA-I™ ANALYZER

The RPA-I Analyzer is a laboratory benchtop-based, single wavelength, dual beam, microprocessor-controlled analyzer. It can read the absorbances of calibrators and samples, perform mathematical computations, and report raw absorbances and sample concentrations with statistics. For a complete and detailed description of the RPA-I, please refer to the *RPA-I RaPID Analyzer Operations Manual* (Part No. A00046).

ENVIRONMENT

- 5° C to 33° C
- 10% to 85% humidity
- Flat, level surface away from strong sources of electromagnetic interference.
- No direct sunlight or drafts.
- Removed from sources of direct heat and moisture.
- Ventilation space at least 6 inches on sides and back.

UNPACKING AND INSTALLATION

1. Inspect the carton for visible signs of damage and note the condition of the SHOCK-WATCH indicator on the side of the carton. If damage has occurred, or a part is missing, immediately contact SDI.

2. Open the carton and remove the brown rectangular box from the grey packing material. (Save all boxes.) This box contains the power transformer, roll of paper, and Program Cartridge. Refer to Figure 1 for identification of shipping carton contents.

3. Lift off the gray packing material to reveal the photometer. Remove it from the carton.

4. Insert the Program Cartridge (with the white label facing up) into the Program Cartridge Holder found on the rear panel of the instrument. Push in until the white label is no longer visible (Refer to Figure 2).

5. With the power **OFF** to the instrument, (bottom of the white toggle power switch should be depressed) insert the round end of the Power Transformer (notched end facing up) into the AC Power Connector found on the rear panel of the instrument. Plug the square end of the power cable into a grounded AC outlet.

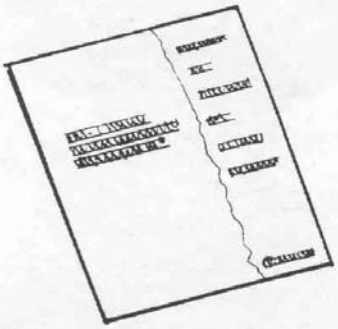
6. The instrument is then activated by depressing the top of the white toggle power switch. The instrument will perform a "Self Test." During this short test, the various electronic components of the RPA-I are automatically analyzed. This includes checks of EPROM and RAM memory. If there are any abnormalities in these areas, the RPA-I will alert the operator with an "ERROR" message. If all the parameters are satisfactory, the "Select Command" prompt will appear and the operator may continue.

SHIPPING CARTON CONTENTS

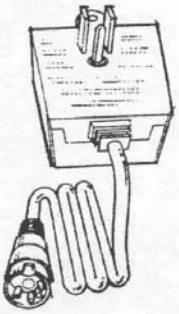
The shipping carton should contain the following items:

RPA-I Analyzer with a 450/600nm filter block.

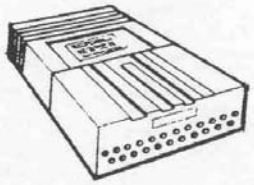
RPA-I Analyzer Operator's Manual



Domestic Power Cord/Mains Transformer



Program Cartridge



Printer Paper

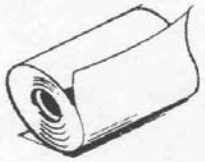


Figure 1. Shipping Carton Contents

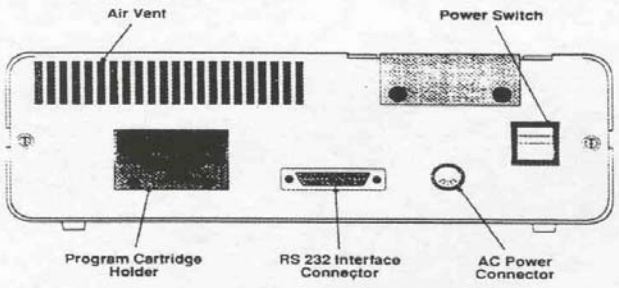


Figure 2. Rear Panel

SHORT OPERATING PROCEDURE FOR THE RPA-I

ALLOW THE RPA-I TO WARM UP FOR 30 MINUTES PRIOR TO USE. Avoid analyzing samples with air bubbles, foam, scratches, or foreign matter. The RPA-I performs a self test first. If all parameters are satisfactory, the "Select Command" prompt will appear. If there are abnormalities, an "Error" message will appear.

The RPA-I reports all results on a thermal paper printout. The unit is turned off by switching the power switch in the rear of the unit to the off position.

INSTRUMENT DISPLAY	OPERATOR RESPONSE
SELECT COMMAND	Press RUN
RUN PROTOCOL: Aldicarb, Atrazine, Alachlor, etc.	Scroll using the YES [§] or NO [›] until the desired protocol appears. Press ENTER.
SPL. REPLICATES: (1-5)	Press 1 (Press 2 if analyzing samples in duplicate, etc.). Press ENTER.
BLANK TUBE INSERT TUBE EVALUATING TUBE REMOVE TUBE (Beep)	Insert tube with 1 mL of washing solution Remove tube
CAL. #1 REP. #1 INSERT TUBE EVALUATING TUBE REMOVE TUBE (Beep)	Insert first standard replicate (0 ppb calibrator/tube #1). Remove Tube
Follow the prompts on the instrument display.	Note: Tube order is important here. The RPA-I expects to see the standards/calibrators in ascending order in duplicate, starting with 0 ppb.

After all the standards (calibrators) have been evaluated, the instrument will display:

PRINTING DATA LISTING XFORM DATA PRINTING CURVE	Data will print. Curve will print only if programmed to print (See Section 3 Special Functions - Instrument Functions: Print Curve).
CTRL. #1 REP. #1 INSERT TUBE EVALUATING TUBE REMOVE TUBE (Beep)	Insert Control Tube. Remove Tube.
EDIT CALIBRATORS YES/NO	Press NO if it is not necessary to edit the calibrators, press YES to edit (See Section 3 Run).
SPL. #1 REP. #1 INSERT TUBE EVALUATING TUBE REMOVE TUBE (Beep)	Insert first Sample Tube. Remove Tube.

Follow the prompts on the instrument display. After all the samples have been evaluated, press STOP.

EXPLANATION OF DATA

Bolded areas are explained in the right hand column.

```
04-12-91  12:36:38

******** SDI ********

PROTOCOL :  ATRAZINE

TECH ID :_____________
LOT #   :_____________
EXP DATE:_____________

Data Reduct:Lin.Regression
Xformation:          Ln/LgtBÀ
Read Mode  :      Absorbance
Wavelength :          450 nm
Units      :             PPB

EQUATION OF LINE :
------------------
Slope      =     -Ø.842      À
Intercept  =     -Ø.1Ø6      À
Corr (r)   =     Ø.9929      À

Transformed Data :
------------------
   Conc        Abs
--------------------
  -2.3Ø      1.926          À
   Ø.ØØ     -Ø.334          À
   1.61     -1.327          À


Calibrator Data:
----------------

  Conc     Abs    CV   Predic
           Diff         Diff
-------------------------------
  Ø.ØØ   1.Ø32
         1.Ø24
  Mean   1.Ø28   Ø.5

  Ø.1Ø   Ø.889          Ø.1ØÀ
        -Ø.ØØ2          -2.4À
         Ø.9Ø6           Ø.Ø8
        -Ø.Ø18         -22.7
  Mean   Ø.897   1.4     Ø.Ø9À
        -Ø.Ø11         -11.8

  1.ØØ   Ø.442           1.23
         Ø.234          18.9
         Ø.416           1.39
         Ø.393          Ø8.2
  Mean   Ø.429   4.2     1.31À
         Ø.311          23.7

  [illegible]
         Ø.2Ø3           4.67
        -Ø.328           -7.Ø
  Mean   Ø.216   8.4      4.26
        -Ø.736          -17.3

-------------------------------
```

Data Reduction

Method of transformation for data. Example, Ln refers to the natural log of the concentration and LgtB refers to the logit function of the absorbance divided by the absorbance at zero concentration.

Equation of Line

These values are the coefficients which describe a "best fit" or linear regression straight line where Logit (B/B_O) = slope x Log_e (conc. in ppb) + intercept. The Corr(r) is the correlation coefficient which indicates "goodness of fit" of the data to the best fit line. The square of this value represents the proportion of variance (on the y axis) that is explained by the linear regression.

Transformed Data

This section shows the average "transformed data" for each standard point. For example,
Log_e (0.1 ppb) = -2.30
Logit ($\frac{0.897}{1.028}$ or $\frac{B}{B_O}$) = 1.926

Calibrator Data

0.889 = observed absorbance
0.10 = observed concentration
-0.002 = known conc.(0.10) - observed conc.(0.10)*
-2.4 = concentration diff (-0.002) ' observed conc. (0.10) x 100*
1.4 = standard deviation of observed absorbances ' mean (0.897) x 100
4.2 = coefficient of variation (%CV) is calculated using absorbances

*For accuracy, the data reduction software of the RPA-I utilizes seven significant digit numbers although only three are displayed or printed.

```
Control Data :
--------------

Ctrl#    Abs      Conc
-------------------------
  1    Ø.274     2.93

ID:_____________________


Samples Data :
--------------

Spl#     Abs      Conc       CV
-------------------------------
  1    Ø.482     1.Ø2
       Ø.46Ø     1.13
Mean   Ø.471     1.Ø8      7.3Ã

ID:_____________________

  2    Ø.36Ø     1.84
       Ø.368     1.76
Mean   Ø.364     1.8Ø      3.Ø

ID:_____________________

  3    Ø.925     Ø.Ø7
       Ø.93Ø     Ø.Ø6
Mean   Ø.928     Ø.Ø6      4.7

ID:_____________________

  4    Ø.991     Ø.Ø2nd      Ã
       Ø.998     Ø.Ø1nd
Mean   Ø.995     Ø.Ø2nd17.7*Ã

ID:_____________________

  5    Ø.927     Ø.Ø6
       Ø.921     Ø.Ø7
Mean   Ø.924     Ø.Ø7      5.4

ID:_____________________

  6    Ø.23Ø     3.86
       Ø.233     3.78
Mean   Ø.232     3.82      1.4

ID:_____________________

  7    1.Ø38       nd        Ã
       1.Ø36       nd
Mean   1.Ø37       nd

ID:_____________________
END OF RUN
Ø4-12-91  12:39:24
```

Control Data

Displays absorbance and concentration of control sample. This concentration should be compared to the reported range located on the control vial label to assure the quality of the run.

Sample Data

7.3 = this %CV is calculated using the sample concentrations

"nd" indicates concentration below the "Normal Range Low" value entered during the protocol setup. This value is the least detectable dose (LDD) for RaPID Assay protocols.
"*" indicates the %CV exceeds the parameter setting limit.

An "nd" without a concentration indicates the absorbance measured is greater than the absorbance of the 0 ppb standard therefore a concentration cannot be calculated.

Appendix 5

HEALTH AND SAFETY PLAN

791/805, 855 and 872 RUNNYMEDE STREET AND 875 O'CONNER STREET EAST PALO ALTO, CALIFORNIA

SEPTEMBER 16, 2003

Prepared for:

Ms. Lily Lee
US Environmental Protection Agency
2415 University Avenue
East Palo Alto, California 94303

CONTENTS

CONTENTS (continued)

PAGE

FIGURE 1 : SITE LOCATION AND HOSPITAL ROUTE

OSHA NOTICE

Health and Safety Plan
791/805, 855 and 872 Runnymede Street and 875 O'Conner Street
East Palo Alto, California

1.0 INTRODUCTION

This Health and Safety Plan ("HSP") addresses the hazards associated with the planned field activities at 875 O'Conner Street located in East Palo Alto, California ("the Site"; Figure 1). It presents baseline health and safety requirements for establishing and maintaining a safe working environment during the course of work. The planned field activities at the Site include soil sample collection under the supervision of Innovative and Creative Environmental Solutions (ICES) personnel.

If work plan specifications change during or after the preparation of this HSP, or if site conditions differ as the result of more information, the ICES Health and Safety Director shall be informed immediately and appropriate changes shall be made to this HSP.

At a minimum, all contractor/subcontractor personnel working on site must:

- Have read and understood the specifications of this HSP
- Have completed all training requirements in 29 Code of Federal Regulations (CFR) 1910.120
- Provide their own health and safety equipment as indicated in this HSP, and comply with the minimum requirements established by this HSP. If the contractor/subcontractor has prepared his/her own HSP, it must minimally meet requirements contained herein and all applicable Federal, State, and local health and safety requirements.

This HSP shall be read and approved by the ICES Health and Safety Director, the ICES Project Manager, and a ICES Quality Assurance Reviewer.

A copy of this HSP shall be kept on site, easily accessible to all employees and government inspectors, and another in ICES

files.
This HSP was prepared using the following documents:

- 29 CFR 1910 -- Occupational Safety and Health Standards, 1990

- 29 CFR 1926 -- Safety and Health Regulations for Construction

- 29 CFR 1910.1000 -- OSHA Air Contaminants - Permissible Exposure Limits, 1990

- Title 8, California Code of Regulations, Occupation Health and Safety Standards.

- American Conference of Governmental Industrial Hygienists (ACGIH). Threshold Limit Values and Biological Exposure Indices for 1990 - 1991. Cincinnati, Ohio, ACGIH.

- California Department of Health Services (DHS), Toxic Substances Control Division (TSCD), Technical and Support Unit, Region 3, Los Angeles, California, August 1988. Site Safety Plan Guidance Document.

- National Institute for Occupational Safety and Health (NIOSH); Occupational Safety and Health Administration (OSHA); U.S. Coast Guard (USCG); U.S. Environmental Protection Agency (EPA), October 1985. Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities. Washington D.C.: U.S. Government Printing Office.

- NIOSH/OSHA, 1981. Occupational Health Guidelines for Chemical Hazards.

- Sax, N. Irving, 1984, Dangerous Properties of Materials, 6th edition, Van Nostrand Reinhold Company, Inc., New York, New York.

- U.S. EPA, Office of Emergency and Remedial Response, Hazardous Response Support Division, November 1984. Standard Operating Safety Guides.

2.0 SITE CHARACTERISTICS

Site Name: Cummings Temple C.M.E. Church
875 O'Conner Street
East Palo Alto, California

The Site is located on the north side of O'Conner Street, west of Clarke Avenue. The relatively flat Site consists of a rectangular parcel measuring approximately 120 feet wide by 330 feet long. A one-story structure occupied by the Cummings Temple C.M.E. Church is located at the southeastern portion of the Site. A stockpile of soil is located at the western portion of the Site. The remaining areas of the Site are either unpaved or landscaped. At the time of the site visit, several automobiles were parked at the eastern portion of the Site.

Site Name: 791/805 Runnymede Street
East Palo Alto, California

The Site is located on the north side of Runnymede Street, between Clarke and Cooley Avenues. The relatively flat Site consists of two rectangular parcels measuring approximately 220 feet wide by 270 feet long. There is a residential structure on each parcel. The residence on the eastern parcel is used as the Faith Baptist Church.

Site Name: 855 Runnymede Street
East Palo Alto, California

The Site is located on the north side of Runnymede Street, between Clarke and Cooley Avenues. The relatively flat Site consists of a rectangular parcel measuring approximately 116 feet wide by 185 feet long. The two-story structure on the parcel is used by the Faith Center.

Site Name: 872 Runnymede Street
East Palo Alto, California

The Site is located on the south side of Runnymede Street, between Clarke and Cooley Avenues. The relatively flat Site consists of an irregularly shaped parcel measuring approximately 150 feet wide by 272 feet long. The parcel includes a one-story residence and a workshop. Construction debris and fill soil have been stockpiled on Site.

2.1 Background

The Site was vacant and undeveloped prior to 1956. In 1956, the

existing structure located at the southeastern portion of the Site appeared to have been constructed. Two structures occupied the southwestern portion of the Site in 1965. The two structures were removed by 1982.

The existing structure located at the southeastern portion of the Site was used for residential applications from the mid 1950s through 1970. The structure is currently being used by the Cummings Temple C.M.E. Church.

A Phase I Environmental Site Assessment was performed by ICES in May 2003. Based on the findings of the ESA, ICES recommended that a preliminary investigation be initially conducted within the Site to assess the potential presence of contaminants associated with activities formerly conducted within the neighboring area of the Site that could have potentially impacted the Site.

Specifically, ICES recommended that soil samples be collected from the surficial soil within the Site to assess the potential presence of organochlorine pesticides, arsenic, lead, and mercury.

3.0 WORK DESCRIPTION

Tasks to be performed at the Site include soil sampling. All work to be performed at the Site will be conducted during daylight hours.

4.0 KEY PERSONNEL AND RESPONSIBILITIES

4.1 Site Safety Personnel

Name	Responsibilities
Peng Leong	Project Manager
Derek Wong	Site Safety Officer
Peng Leong	Health and Safety Director

4.2 ICES Personnel and Responsibilities

The responsibilities of the ICES personnel listed in Section 4.1 are outlined below.

4.2.1 ICES Project Manager

The ICES Project Manager, Peng Leong, has the ultimate responsibility for the health and safety of ICES personnel on site. As part of his duties, Mr. Leong shall be responsible for:

- Keeping the ICES Health and Safety Director informed of project developments
- Ensuring that on-site ICES personnel receive the proper training, and are informed of potential hazards anticipated at the Site and procedures and precautions to be implemented on the job
- Ensuring that contractors and subcontractors are informed of the expected hazards and appropriate protective measures at the Site. (Subcontractors should also be given a copy of ICES's HSP for review.)
- Ensuring that resources are available to provide a safe and healthy work environment for ICES personnel.

4.2.2 ICES Health and Safety Director

The ICES Health and Safety Director, Peng Leong, shall be responsible for:

- Monitoring the health and safety impacts of this project for on-site ICES personnel
- Assessing the potential health and safety hazards at the Site
- Recommending appropriate safeguards and procedures
- Modifying the HSP, when necessary
- Approving changes in safeguards used or operating procedures employed at the Site.

The ICES Health and Safety Director shall have the authority to:

- Require that additional safety precautions or procedures be implemented
- Order an evacuation of the Site, or portion of the Site, or shut down any operation, if she believes a health or safety hazard exists
- Deny unauthorized personnel access to the Site
- Require that any worker obtain immediate medical attention
- Approve or disallow any proposed modifications to safety precautions or working procedures.

4.2.3 ICES Site Safety Officer

The ICES Site Safety Officer (SSO), Derek Wong, has fulfilled the 40-hour health and safety training requirements pursuant to 29 CFR 1910.120.

The SSO, or a trained designated alternate, will be present at the Site during work activities. The SSO shall be notified of and approve activities in which persons may be reasonably expected to be exposed to contaminated soils and/or ground water.

The SSO shall be responsible for:

- Ensuring that on-site ICES personnel comply with the

requirements of the HSP
- Limiting access to the Site
- Reporting unusual or potentially hazardous conditions to the ICES Health and Safety Director and the ICES Project Manager
- Reporting injuries, exposures, or illnesses to the ICES Health and Safety Director and the ICES Project Manager
- Communicating proposed changes in work scope or procedures to the ICES Health and Safety Director for approval
- Recommending to the ICES Health and Safety Director and the ICES Project Manager additional safety procedures or precautions that might be implemented.

The SSO shall have the authority to:

- Order an evacuation of the Site, or portion(s) of the Site, or shut down any operation if he believes a health or safety hazard exists
- Deny site access to unauthorized personnel
- Require that any worker, including the contractor's or subcontractor's personnel, obtain immediate medical attention.

5.0 HAZARD ANALYSIS

Potential chemical and general safety hazards during the field activities at the Site include the following:

- Chemical hazards
    - Respiratory (exposure to fugitive dust)
    - Dermal (contact with pesticide-affected soil)
- Physical hazards
    - Noise
    - Electric shock
    - Heavy equipment
    - Heat stress

Work procedures to protect workers from chemical and physical hazards are discussed in Section 6.0.

5.1 Chemical Hazards

The primary chemical hazards is exposure to chemical compounds from the soil. Of particular concern is the potential for workers to be exposed to contaminants during the drilling and sampling activities. The primary contaminants include organochlorine pesticides (specifically p,p'-dichlorodiphenyl trichloroethane [DDT], chlordane, dieldrin, and endrin), arsenic, lead, and mercury. Inhalation exposures are the primary exposure pathway of concern.

Description of the chemicals of concern including physical and odor recognition characteristics, effects of short-term exposure for use in the field, and the Time-weighted Average (TWA) over an eight hour period for the permissible exposure limit (PEL) (OSHA Standard 29 CFR 1910.1000) are presented below.

5.1.1 Chemical Description of DDT

DDT is a colorless crystal or white to slightly off-white powder with a slightly aromatic odor. When present on a soil matrix, DDT is indistinguishable.

Short-term exposure to high concentrations of DDT can cause a prickly sensation of the tongue, lips, and face, a general feeling of ill health, headache, fatigue, vomiting, dizziness, tremors, convulsions, partial paralysis of the hands, and coma. DDT also can irritate the eyes and skin.

The TWA of the PEL for DDT is 1.0 mg/m^3.

5.1.2 Chemical Description of Chlordane

Chlordane is a amber-colored viscous liquid with a pungent, chlorine-like odor.

Short-term exposure to chlordane can cause blurred vision, delirium, coughing, nausea, vomiting, irritability, convulsions, and abdominal pains.

The PEL for chlordane is 0.5 mg/m^3.

5.1.3 Chemical Description of Dieldrin

Dieldrin is a light brown crystal with a mild chemical odor.

Short-term exposure to dieldrin can cause hyperirritability, headaches, dizziness, nausea, vomiting, blood in the urine, tremors, convulsions, and coma.

The PEL for dieldrin is 0.25 mg/m^3.

5.1.4 Chemical Description of Endrin

Endrin is a colorless to tan solid with a mild chemical odor.

Exposure to endrin may cause sudden convulsions that may occur from 30 minutes to 10 hours after exposure. Headaches, dizziness, drowsiness, weakness, and loss of appetite may occur two to four weeks after exposure.

The PEL for endrin is 0.1 mg/m^3.

5.1.5 Chemical Description of Arsenic

Metallic arsenic is most commonly a grey, brittle, crystalline solid. It can also be in a black or yellow amorphous form. Arsenic is also commonly found in its volatile white trioxide form. Arsenic is also used in several insecticides, herbicides, silvicides, defoliants, desiccants, and rodenticides and appears in a variety of forms.

Arsenic is classified by the U.S. Environmental Protection Agency as a known human carcinogen.

Short-term exposure to arsenic can cause marked irritation of the stomach and intestines with nausea, vomiting, and diarrhea. In severe cases the vomiting and stools are bloody and the exposed individual goes into collapse and shock with weak, rapid pulse, cold sweats, coma, and death. Inorganic arsenicals are more toxic than organic arsenicals, and the trivalent form is more toxic than the pentavalent form. Acute arsenic poisoning usually results from ingestion exposures.

The PEL for arsenic is 0.01 mg/m^3 and for organic arsenic the PEL is 0.5 mg/m^3.

5.1.6 Chemical Description of Lead

Lead (inorganic) is a bluish-white, silver, or grey odorless solid.

Short-term exposure to lead can cause decreased appetite, insomnia, headache, muscle and joint pain, colic, and constipation.

The PEL for lead is 0.05 mg/m^3.

5.1.7 Chemical Description of Mercury

Mercury is a silvery, mobile, odorless liquid.

Short-term exposure to inhaled mercury vapors may cause headache, cough, chest pains, chest tightness, and difficulty in breathing. In addition, it may cause soreness of the mouth, loss of teeth, nausea, and diarrhea. Liquid mercury may irritate the skin.

The TWA of the PEL for mercury is 0.1 mg/m^3.

5.2 Physical Hazards

The potential physical hazards at the Site during the planned activities stem from heavy machinery use and the hazardous nature of drilling work. The potential for heat stress caused by the use of personal protective equipment (PPE) and high mid-day temperatures, has been minimized by specifying the use of lighter PPE in this HSP. The anticipated physical hazards at the Site are listed in Section 5.0. Work procedures to protect workers from chemical and physical hazards are discussed in Section 6.0.

Noise

Noise results primarily from regrading activities and operation of field equipment and other machinery.

Electric Shock

Electrical equipment for field activities and surface and subsurface utility lines pose the potential for electric shock.

Heavy Equipment

Regrading and compacting equipment pose a physical injury hazard to on-site personnel.

Heat Stress

Heat stress could pose a hazard to on-site personnel due to the need for PPE and the potentially high mid-day temperatures.

6.0 WORK REQUIREMENTS

6.1 Respiratory Protection

Field operations will be initiated in Level D. The primary route of potential exposure for chemicals is inhalation of fugitive

dust.

Inhalation hazards due to volatilization will be monitored visually. If on-site dust levels impair visibility during soil sampling activities, work shall be temporarily stopped to wet the area responsible for generating dust. If dust problems continue, a temporary stop work order will be observed and the ICES Health and Safety Officer shall be notified.

6.2 Dermal Protection

Unless adequate precautions are taken, chemicals may contact the skin or clothing. Potential physical contact with chemicals of concern are possible under the following circumstances:

- drilling and sampling activities

6.2.1 Personal Protective Equipment

ICES and contractor/subcontractor personnel will wear the following protective clothing on site:

- Hard hats
- Steel-toed/steel-shank boots
- Inner and outer disposable PVC gloves for soil sampling (to be changed immediately after sampling is completed)
- Safety glasses
- Uncoated Tyvek coveralls (if the potential for splashing exists)

6.3 Action Levels

6.3.1 Action Levels for a Temporary Stop Work

The SSO shall impose a temporary stop work and contact the ICES Health and Safety Director immediately if the following conditions are observed, or if there is a question about site conditions:

- Uncontrolled dust generation
- Indications of heat stress

- Changes in the general health profile of on-site personnel, including headaches, dizziness, breathing difficulties, irritation to the eyes, nose, throat, and hands

6.4 Protection Against Physical Hazards

6.4.1 Noise

Noise results primarily from drilling equipment and other machinery. Workers will wear ear plugs when operating heavy machinery to avoid noise that may exceed the 85 decibel Threshold Limit Value (TLV) established by the American Conference of Governmental Industrial Hygienists. However, based on previous field experience, expected noise level should not exceed 85 decibels.

6.4.2 Electric Shock

All electrical equipment to be used during field activities will be suitably grounded and insulated.

6.4.3 Heavy Equipment

Hazards related to drilling will necessitate securing the work area. All relevant requirements pursuant to 29 CFR 1926.602 and Subpart W, Rollover Protective Structures; Overhead Protection, shall be observed during the course of drilling/sampling activities.

All field personnel not directly involved in the drilling/sampling work will be kept at safe distances from areas where heavy equipment are in use. Unauthorized visitors will not be permitted near areas where heavy equipment are in use regardless of whether the area has been designated as an exclusion zone.

6.4.4 General Safety

All ICES and contractor/subcontractor personnel will wear approved head protection while working around heavy equipment in the site area. Fire hydrants, electrical and underground lines and pipes will be identified before drilling operations begin. Two 10-pound fire extinguishers will be kept on site near the exclusion zone.

6.5 Entry Procedures

At a minimum, all visitors entering the exclusion zone must wear

the protective clothing and equipment worn by ICES and contractor/subcontractor personnel. Permission to enter the work area must be obtained from at least one of the personnel named in Section 4.0. Each visitor's name and purpose of visit will be recorded in the field notes.

7.0 WORK ZONE AND DECONTAMINATION PROCEDURES

A site must be controlled to reduce the possibility of exposure to any contaminants present and to limit their transport from the site by personnel or equipment.

7.1 Control

A control system is required to ensure that personnel and equipment working on hazardous waste sites are subjected to appropriate health and safety surveillance and site access control.

The possibility of exposure or translocation of contaminants can be reduced or eliminated in a number of ways, including:

- Setting security or physical barriers at control points to regulate access to and/or exclude unnecessary personnel from the general area
- Minimizing the number of personnel and equipment on site consistent with effective operations
- Establishing work zones within the site
- Conducting operations in a manner which will reduce the exposure of personnel and equipment
- Minimizing the airborne dispersion of contaminants (utilizing dust control procedures)
- Implementing appropriate decontamination procedures for both equipment and personnel.

7.2 Field Operations Work Areas

Work areas (zones) will be established based on anticipated contamination. Within these zones, prescribed operations will occur utilizing appropriate Personal Protective Equipment (PPE). Movement between areas will be controlled at checkpoints. The planned zones are:

- Exclusion (contaminated)
- Contamination Reduction

- Support (noncontaminated).

7.2.1 Exclusion Zone

The Exclusion Zone is the innermost area of the three concentric rings and is considered contaminated, dirty, or "hot." Within this area, the prescribed protection must be worn by any personnel upon entering. An entry checkpoint will be established at the periphery of the exclusion zone to control the flow of personnel and equipment between contiguous zones, and to guarantee that the procedures established to enter and exit the zones are followed.

The Exclusion Zone boundary will be established initially on the presence of the contaminant(s) within the area. Subsequent to initial operations, the boundary may be readjusted based on observations and/or measurement. The boundary will be physically secured and posted.

7.2.2 Contamination Reduction Zone

Between the Exclusion and the Support Zone is the Contamination Reduction Zone. The purpose of this zone is to provide an area to prevent or reduce the transfer of contaminants which may have been picked up by personnel or equipment returning from the Exclusion Zone. All decontamination activities occur in this area. The boundary between the Support Zone and the Contamination Reduction Zone is the contamination control line. This boundary separates the potentially contaminated area from the clean area. Entry into the Contamination Reduction Zone from the clean area will be through an access control point. Personnel entering at this station will be wearing the prescribed PPE for working in the Contamination Reduction Zone. Exiting the Contamination Reduction Zone to the Clean Area requires the removal of any suspected or known contaminated PPE, and compliance with the established decontamination procedures.

7.2.3 Support Zone

The Support Zone is the outermost of the three rings and is considered decontaminated, or Clean Area. It contains the Command Post (CP) for field operations and other elements necessary to support site activities. Normal street or Level D work clothes are the appropriate apparel to be worn in this area.

7.3 Zone Dimensions

Considerable judgement is needed to ensure safe working distances for each zone, balanced against practical work considerations. Physical and topographical barriers may constrain ideal locations. Field/laboratory measurements combined with meteorological conditions and air dispersion calculations will assist in establishing the control zone distances. When not working in areas that require the use of chemical-resistant clothing, work zone procedures may still need to limit the movement of personnel and retain adequate site control.

7.4 Decontamination Procedures

As part of the system to prevent or reduce the physical transfer of contaminants by people and/or equipment from the site, procedures will be instituted for decontaminating anything leaving the Exclusion Zone and Contamination Reduction Zone. These procedures include the decontamination of personnel, protective equipment, monitoring equipment, clean-up equipment, etc. Unless otherwise demonstrated, everything leaving the Exclusion Zone should be considered contaminated. In general, decontamination at the site consists of rinsing equipment with detergent/water solution. Reusable decontaminated PPE will be stored for air drying.

Decontamination is addressed in two ways: the physical arrangement and control of contamination zones, and the effective use of decontamination procedures.

The decontamination process uses cleaning solutions, followed by rinse solutions. Used solution, brushes, sponges, and containers must be properly disposed of.

Decontamination Solution

Description	Usage
3 cups Alconox 1 cup sodium carbonate 5-8 gallons water	Light contamination
Commercial Detergent - Full strength or diluted	Organic contaminants

As with all alkaline cleaners, continuous or repeated contact with the skin should be avoided. If an employee's skin becomes contaminated, he/she will move to the decontamination area and

remove contaminated clothing, and wash with a mild soap/detergent and water to remove any contaminant from the skin. He/she will then see a physician for possible medical treatment.

A rinse solution will be used to remove the contamination solution and neutralize any excess decontamination solution.

All personnel will follow these decontamination procedures:

1. When returning from the Exclusion Zone, remove heavy soil, as necessary, from boots, gloves, and clothing by using a towel or hose before entering the Contamination Reduction Zone.

2. At the decontamination area, step into decontamination tub(s) and brush boots and gloves clean.

3. Remove disposable suit and discard in proper container.

4. Step into rinse tub(s), then remove boots.

5. Remove outer gloves and dispose of properly.

6. Remove hard hat.

7. Remove inner gloves and dispose of properly.

Decontamination procedures may be modified, if necessary, with the approval of the Site Safety Officer.

7.4.1 Personal Decontamination During Medical Emergencies

In the event of personal injury, first-aid personnel must decide if the victim's injuries are potentially the type that would be aggravated by movement. If there is any doubt, or if the victim is unconscious and cannot respond, no attempt should be made to move the victim to the decontamination area. Only off-site paramedics may move such victims. If the paramedics approve, the victim's PPE will be cut off in the Decontamination Reduction Zone. If the decision is made not to remove the victim's protective clothing, he/she will be wrapped in a tarp or similar object to protect the ambulance and crew during transportation. If the victim is contaminated with materials that threaten to cause additional injury or immediate health hazards, the PPE will be carefully removed and the victim washed appropriately.

8.0 EMERGENCY PROCEDURES

8.1 General Injury

- Step 1: Use first-aid kit on site, if appropriate.
- Step 2: Use off-site help and/or assistance if appropriate.
- Step 3: Notify SSO, Project Manager and Health and Safety Director.

8.2 Specific Treatments

- Eye Exposure: flush eye with eye wash, call ambulance.
- Skin Exposure: wash immediately with soap and water; call ambulance, if necessary.
- Fire (localized): use fire extinguisher and activate alarm system, if necessary.
- Fire (uncontrolled): call Fire Department.
- Chemical Spill: call Fire Department and National Response Center for Toxic Chemical and Oil Spills.
- Explosion: call Fire Department if potential for additional explosions or fire danger exists.
- Inhalation: move affected person(s) to fresh air and cover source of vapors, if appropriate.
- Swallowing: call ambulance.

8.3 Emergency Phone Numbers

Medical/General Service Numbers

Police Department	911
Fire Department	911
Ambulance	911

Hospital

Stanford University Medical Center (650) 723-4000
300 Pasteur Drive
Palo Alto, California 94305

From the Site, proceed south on Clarke Avenue. Turn right on East Bayshore Road and proceed west. Continue west on East Bayshore Road and turn left on University Avenue. Proceed south on University Avenue and continue south on Palm Drive. Turn right on Arboretum Road. Proceed west on Arboretum Road and turn left on Sand Hill Road. Proceed south on Sand Hill Road and turn left on Pasteur Drive. Stanford University Medical Center is located at 300 Pasteur Drive (Figure 1).

Hazardous Materials Response/Reporting

National Emergency Response Center	(800) 424-8802
California State Office of Emergency Services	(800) 852-7550
Regional Water Quality Control Board	(510) 622-2300

8.4 Accident Reporting Procedures

In the event of an emergency, contact the following:

ICES :		(510) 652-3222
Peng Leong (Health and Safety Director)	Cell	(707) 803-3222
Peng Leong (Project Manager)	Cell	(707) 803-3222
Derek Wong (Site Safety Officer)	Cell	(510) 282-3525

If an exposure or injury occurs, work shall be temporarily halted until the SSO, in consultation with the Health and Safety Director, decides it is safe to continue work.

9.0 DOCUMENTATION

The SSO will record field observations of health and safety procedures by workers conducting the planned activities outlined in Section 3.0, including deviations from the recommended health and safety procedures.

10.0 MEDICAL MONITORING

Appropriate medical monitoring will be required of ICES personnel to:

- Meet requirements of 29 CFR 1910.120 (f).
- Meet requirements for respirator use.
- Meet other legal requirements.

A signed physician's statement qualifying the individual for the work to be performed will be required as part of the medical monitoring program.

11.0 TRAINING PROGRAM

1. The ICES SSO shall have fulfilled all appropriate training requirements indicated by 29 CFR 1910.120 (e), including the 40-hour training requirement and required refresher courses.

2. A tailgate session to discuss this HSP will be held before field activities begin. All ICES personnel and contractor/subcontractor employees shall receive, at a minimum, the following information:

- The names of personnel and alternates responsible for site safety and health
- Safety, health, and other hazards at the Site
- Instruction in the use of personal protective equipment
- Action levels
- Employee work practices to minimize risks from on-site hazards
- Instruction in the safe use of engineering controls and equipment on site
- Site control measures
- Emergency plans
- Proposition 65 warnings.

12.0 PROPOSITION 65

Under California's Safe Drinking Water and Toxic Enforcement Act of 1986 (Proposition 65), individuals who may be exposed in the work place to chemicals that may cause cancer or birth defects must be warned of such hazards pursuant to California Health and Safety Code (HSC) Section 25249.6. At this Site, the chemicals that may cause cancer or reproductive abnormalities, and their respective warnings, are listed below.

12.1 Carcinogens and Reproductive Toxicants

Chemicals known to the State of California as reproductive toxicants, as listed in Title 22, CCR Section 12000(b), which may be present at the Site include some of the organochlorine pesticides, arsenic, lead, and mercury.

12.2 Warnings

Pursuant to HSC Section 25249.6 and CCR Sections 12601(c)(3)(A) and 12601(c)(3)(B), the following warnings must be made:

"This area contains chemicals known to the State of California to cause cancer."

13.0 SIGNATURES

13.1 ICES Personnel

This HSP for the planned field activities to be conducted at 875 O'Conner Street located in East Palo Alto, California, is approved by the following ICES personnel:

_____ _____

Peng Leong Date
Health and Safety Director

_____ _____

Peng Leong Date
Project Manager

_____ _____

Derek Wong Date
Site Safety Officer

13.2 Contractor and Subcontractor Personnel

Contractor and Subcontractor Agreement:

1. Contractor certifies that the following personnel noted below to be employed on the planned field activities at 875 O'Conner Street located in East Palo Alto, California, have met the requirements of the OSHA Hazardous Waste Operations and Emergency Response Standard 29 CFR 1910.120 and other applicable OSHA Standards.

2. Contractor certifies that in addition to meeting the OSHA requirements, it has received a copy of this HSP, and will ensure that its employees are informed and will comply with both OSHA requirements and the guidelines in this HSP.

3. Contractor further certifies that it has read, understands and will comply with all provisions of this HSP, and it will take full responsibility for the health and safety of its employees.

Contractor	Signature	Date
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

Appendix 6

McCAMPBELL ANALYTICAL INC.

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Quality Assurance Program

for

McCampbell Analytical Incorporated

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Table of Contents

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Organization, Responsibility and Goals

McCampbell Analytical currently has a staff of approximately thirty. The lab director Edward Hamilton, lab/QA manager Angela Rydelius, and shift supervisors share final responsibility for all decisions.

The lab/QA manager is also responsible for data review and has authority to approve or disapprove specific analyses and final reports. They are responsible for advising on all aspects of QA/QC including: assuring proper QA/QC procedures are employed during data generation; periodically reviewing QA/QC procedures; and, if problems are detected, making recommendations to ensure that appropriate corrective actions are taken.

The Supervisory Chemists are considered competent and proficient in a wide variety of analyses and instrument troubleshooting/repair and serve as technical advisors to the less experienced technicians and chemists. They are responsible for training new employees and ensuring that analytical methods and instruments are working properly. Other chemists and technicians may also be experienced and proficient enough to conduct training and instrument troubleshooting and maintenance but are generally knowledgeable in fewer analyses.

New employees are trained by veteran employees. Training includes hands-on instrumental operation, becoming familiar with the appropriate SOPs and completing a satisfactory initial demonstration of proficiency when appropriate. In addition, the trainer will review the trainee's data until proficiency has been established. Proficiency is defined as being able to independently and satisfactorily perform an analysis error free for a period of one month.

The pursuit of quality is one of the primary goals of this laboratory and this document outlines some of the specific steps taken to achieve this goal. While no QA program can achieve absolute perfection, the identification of problems and subsequent corrective actions will move the laboratory steadily towards this goal.

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Chain of Custody for Samples

Samples shall be delivered to the lab by the contractor or a third party, and will not be collected in the field by McCampbell Analytical personnel. The chain of custody will record the following:

a) the time and date of sampling and the sampler's signature,
b) the time and date when the samples were relinquished to the lab,
c) the signatures of persons who relinquished and received the samples,
d) a description of each sample matrix,
e) a unique identifier for each sample,
f) the type of analysis requested,
g) a description of the condition of the samples, for example, whether or not they were kept cool, the presence of head space, preservatives, or the smell of fuel products, and
h) client name.

Additional useful information includes Client Contact name, Project ID, TAT, billing, phone, fax and email information.

Clients shall be informed immediately of any sample identity discrepancies between the COC and actual sample container, or if hold time, preservatives, or containers are invalid, or if sampling times are not present on the COC, or if testing instructions are ambiguous.

Samples may be couriered to the lab by a third party service. In such instances, the courier must formally receive and relinquish the samples by signing the COC. Some courier companies such as FedEx, UPS and CA Overnite will not participate in the chain of receiving and relinquishing and MAI recommends that the client seal the cooler with a tamper proof seal prior to shipment. In such instances, MAI personnel will note on the COC the name of the courier service, and if found true by observation, that the samples were received sealed and intact.

Our laboratory shall note the following on the sample's chain of custody: ice, head space, appropriate containers, preservative, sample handling procedures prior to sample storage such as dechlorination and filtration, and approximate sediment content of each water sample. The sediment content of a water sample is based upon observation of one or more of its clear and transparent containers. Dissolved metals rather than total metals shall be assumed for the analysis of water samples containing significant sediment unless otherwise specified on the COC. Whenever samples are removed from the lab, it is also noted on the chain of custody.

Each sample will be assigned a unique number, which will be used to identify it within the laboratory. Once the sample is given a lab ID, the sample is either refrigerated when required by method or given to our extraction department,

depending upon the flow of work within the lab. When the extraction department is finished with the sample it is returned to a refrigerator or other appropriate storage area. Our staff is trained to not allow any sample requiring refrigeration to remain un-refrigerated longer than is necessary, with a maximum time of 2 hours. Samples are handled in accordance with regulations set forth by the State of California and the federal government, the EPA, the LUFT manual, SW-846 and other publications.

Samples will be stored for a minimum of one month, and will be stored separately from the standards. Water and soil blanks will be placed in refrigerators that contain samples in order to assess the possibility of vapor phase cross contamination. Samples will be discarded in accordance with local, state and federal regulations.

Maintenance and Calibration of Simple Machines

Refrigerator and Freezer temperatures will be recorded daily and drying ovens as used and the thermostats of these appliances will be adjusted as necessary.
The accuracy of the gravimetric balance will be checked monthly against class S weights and a record kept of these measurements. The manufacturer will be consulted for corrective action if discrepancies greater than 2% are observed.
All pipettes will be tested for accuracy monthly. Pipettes with an error greater than 2% will be refurbished or replaced.

Analytical Procedures

The analytical procedures and methodologies that are used here are described in EPA SW-846, 600/4-79-020, 600/4-84-017, 600/4-82-057, CFR40 (parts 260-299), Standard Methods for the Examination of Water & Wastewater, the California LUFT manual, and California State Title 22 as well as other published paper and internet documents. When ambiguity exists in these sources, common sense and good scientific practices are followed.

a) GC, GC-MS, HPLC, IC and IR Analyses

TPH (g/ss) (8015), volatile aromatics (8020/ 602), volatile halocarbons (8021/ 8010/ 601/ 502) and VOCs (8240/ 8260/ 624/ 524) solids and liquids are direct loaded or extracted with methanol, polyethylene glycol or other suitable solvents. Semi-volatiles including TPH (d/k/mo) (8015), Oil & Grease (SM5520), TRPH (418.1), EDB-DBCP-TCPA (504.1/ 8011), endothall (548), phenyl ureas (532), Diquat-Paraquat (549.2), PNAs (550/ 550.1/ 8310), HAAs (552.1/ 552.2), aldehydes / carbonyls (554/ 8315), anions (300.0/ 300.1), hexachrome (218.6), perchlorate (314.0), chlorinated pesticides and PCBs (8082/ 8081/ 608/ 505/ 508), SVOCs (8270/ 625/ 525/ 526/ 528), NP pesticides (8141/ 507), nitroaromatics & nitramines (8330), chlorinated herbicides (8151/ 515) solids and liquids are solid-liquid, liquid-liquid, or liquid-SPE extracted with methylene chloride, hexane, diethylether, acetone, MTBE, deionized water, or trichlorotrifluoroethane according to EPA methods 3510, 3520, 3550, 418.1 or the relevant analytical method and

derivitized when proscribed by the method. All volatiles are extracted using purge & trap (EPA method 5030) or whole container (EPA method 5035) methodology. Aqueous samples testing for acrolein-acrylonitrile-acrylamide (8316), glyphosate (547), carbamates (531.1/ 8318) or hexachrome (218.6) are directly loaded for HPLC analysis; glyphosate and carbamates, as proscribed by method, are derivitized on line prior to detection. These procedures are documented in our company SOPs, which are derived from published methods.

Two separate standards, each made from a stock standard having a different lot number or manufacturer, are utilized. One stock standard is used to calibrate the instrument and to prepare daily matrix spikes and LCS QC. The second stock standard will be run daily as the CCV to confirm that the instrument is still within calibration. This system ensures high quality data by spotting inaccurately prepared (by the manufacturer or analyst) or "aged" standards. A standards logbook will be kept detailing the preparation of working standards and uniquely identifying them.

The variability of gasoline and diesel preclude the use of multi-source standards. However the constancy over time of the FID detector's response is assessed by comparison of the historical calibration to the daily standard and to the matrix spikes.

The GC's are calibrated using a minimum of five concentrations of the same standard. The highest concentration defines the upper working range of the calibration while the lowest concentration equals the working instrumental detection limit. A linear calibration is typically used for all compounds and is considered acceptable if the %RSD of the CF or RF of each target analyte is $\leq 20\%$ as required by the CA DHS and federal EPA. Alternatively, a non-linear calibration may be used. A non-linear calibration is considered acceptable if the coefficient of determination (COD) for each target analyte is ≥ 0.99.

A blank shall be run initially and a daily mid-level standard (continuing calibration verification standards) initially and approximately every 10 samples or 12 hours and evaluated against method criteria. Corrective action includes re-analysis and/ or the instrument re-calibration.

Surrogate standards, when known, are added prior to extraction; this encompasses most of these analyses. Matrix spike and surrogate recoveries must fall within the ranges outlined in the method or corrective action will be taken.

The techniques for quantitating and resolving complex chlorinated mixtures are detailed in EPA method 8081. Dual column confirmation will be done on all positive pesticide samples and will be done on positive volatile analytes (non-GC-MS methods) by request. Dual detector confirmation (example PID-FID or PID-ELCD) is present for most volatile analytes.

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EPA methods 8240/ 8260/ 624? 524 shall be run as follows. A historical five-point calibration shall be conducted. Three surrogates and three internal standards are added to each injection. The system performance check compounds, SPCCs (chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, chlorobenzene) must have RRFs ≤ 0.1, 0.1, 0.25, 0.3, 0.3, respectively, and the calibration check compounds, CCCs (1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene, vinyl chloride) must have %RSDs < 30% in order that the calibration be valid. On a daily basis, the MS is tuned and the mass ratios shown in method 8240 for BFB must be met initially and after every 12 hours (8 hours for 524) of analysis. A mid-range daily standard will be run after 12 hours of analysis; the above-mentioned SPCC criteria must be met and the CCCs must be within 20% of their daily calibration values for the run to continue. Additional continuance criteria are that any internal standard's retention time must not have changed by more than 30 seconds or its area by a factor of two from that last daily calibration unless by design (tuning or column shortening). Criteria for qualitative and tentative identification and quantitation of a compound are detailed in EPA method 824. Each analyst will demonstrate their capability through a precision and accuracy study of four QC samples as outlined in the method. Matrix spike and surrogate recoveries must fall within the ranges outlined in the method or corrective action will be taken.

EPA methods 8270/ 625/ 525/ 526/ 528 shall be run as follows. A historical five-point calibration shall be conducted. Each injection will contain the six recommended internal and six recommended surrogate standards. The MS will be tuned to fulfill the method criteria for DFTPP before a run can be initiated. The system performance check compounds, SPCCs (N-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitro-phenol, 4-nitrophenol) must have RRFs ≥ 0.05, and the calibration check compounds, CCCs (see method 8270) must have % RSDs < 30% in order that the calibration be valid. On a daily basis, the MS is tuned and the mass ratios shown in method 8270 for DFTPP must be met initially and after 12 hours (8 hours for 525/ 526/ 528) of analysis. A mid-range daily standard will be run after 12 hours of analysis; the above-mentioned SPCC criteria must be met and the CCCs must be within 20% of their daily calibration values for the run to continue. Additional continuance criteria are that any internal standard's retention time must not have changed by more than 30 seconds or its area by a factor of two from that last daily calibration unless by design (tuning or column shortening). Criteria for qualitative and tentative identification and quantitation of a compound are detailed in EPA method 8270. Each analyst will demonstrate their capability through a precision and accuracy study of four QC samples as outlined in the method or corrective action will be taken.

For GC and IR analyses in general, a daily LCS and LCSD (and matrix spike and spike duplicate when sufficient sample containers are provided) will be analyzed every 20 samples for each matrix being analyzed on a given instrument. The quantitated value of LCS, LCSD, spike and spike duplicate must be within 60-140% recovery. One method blank must also be run initially for that day's sequence. A volatiles water/air blank is reagent grade water defined as tap water that has been brought to a rolling boil for 30 minutes, cooled and continuously purged with N_2. Method blanks must contain less than the reporting limit of each method analyte. In the event that any of the CCVs or QC samples fail their criteria they should be immediately reanalyzed. If they continue to fail, an investigation must be

conducted to determine the root cause and the sequence scrutinized for validity by an independent QA officer. Some data may be usable depending on the type and severity of the problem. Corrective action should be taken to resolve the problem and the instrument recalibrated if necessary. If it is determined that there is unusable data, the affected samples will need to be reanalyzed in a new sequence.

The failure of standards, surrogates or QC to fall within accepted ranges is not the only criteria for rerunning samples. The suspicion of contamination arising from the previously injected sample, the previous sample in the same purge & trap vessel / port position, or contamination that exists instrument-wide in flow pathways or valves will necessitate that the effected sample(s) be rerun.

The statistical analysis of replicate samples will be used to determine the minimum detection limit for each individual and group analyte and for external standard methods to determine relative retention time windows, as outlined in chapter one and method 8000 of SW-846. An initial demonstration of proficiency will be conducted for each instrument to assess the precision and accuracy of the instrument and operator. On a daily basis, precision and accuracy are found by comparison of the spike and spike duplicate or a chosen sample and its duplicate.

Records shall be kept of all this data for each instrument and updated as new information is generated. This data will be analyzed for trends that may indicate the onset of problems.

b) Metals

Soil, sludge and water samples for metals analysis are digested using EPA methods (200.7, 200.8, 200.9, 3005, 3010, 3020, 3040, 3050, method 245.2 / 7470 / 245.7 / 1631 for mercury) and analyzed according to EPA methods in 600/4-79-020, SW-846 and elsewhere and documented in our company SOPs. All atomic absorption methods (FAA, GFAA, HGAA, CVAA) will be run in the following manner. Each run will be preceded by a minimum three-point calibration and a blank, followed by an independent check standard (± 15% of the calibration curve), followed by samples. A mid-point calibration standard will be run after each set of 10 samples and at the end of each run. A matrix-spike, spike-duplicate, reagent-blank and one serial dilution will be analyzed with each batch (or 20 samples). Background correction will be used unless it is known to degrade the quality of results. All GFAA standards and samples will be matrix matched to whenever possible.

ICP will be run as follows. An initial 5-point calibration will be performed for each metal to define its range of linearity. On a daily basis, a single mid-point standard will be run to "re-slope" the calibration curve, followed by a blank and an instrument performance check standard. The instrument performance check standard must be within 5% of its true value before the run can proceed. A matrix-spike, spike-duplicate and reagent-blank will be analyzed with each batch (or 20 samples). A mid-point calibration standard and calibration blank will be run after each set of 10 samples and at the end

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of each run. The standard must be ± 10% of the true value and the calibration blank must be below the RL for all elements for the run to proceed. Appropriate background corrections will be made for each element.

The statistical analysis of replicate samples will be used to determine the minimum detection limit for each individual and group analyte. Records shall be kept of all QC data for each element and updated as new information is generated. This data will be analyzed for trends that may indicate the onset of problems.

Miscellaneous Tests

pH, cyanide, 5520 Oil & Grease, specific conductivity, RCI, colorimetric hexachrome, ignitability and other miscellaneous tests that are conducted here are performed in accordance with their methods outlined in EPA SW-846, 600/4-84-017, 600/4-82-057, CRF40 (parts 260-299), Standards Methods for the Examination of Water & Wastewater, the California LUFT manual, and the California State Title 22 and detailed in our SOPs. In general, for all QC, a matrix-spike, spike-duplicate and blank are analyzed every 20 samples. The quantitated value of both spike and spike duplicate must be within 60-140% recovery. One method blank must also be run for each matrix being analyzed on that day's sequence and must be less than the reporting limit. Where the analytical technique or sample is not amenable to spiking then one out of every ten samples will be analyzed in duplicate or by serial dilution.

Data Reduction and Reporting

Data will be acquired from all instruments using the manufacturers software, Agilent ChemStation or a LIMS system and analyzed by user-set methods. Formula for external and internal standard calculations are used that is identical to those found in method 8000 of SW-846. The chromatograms are scrutinized and the quantitations are reviewed before being reported in a run log or sent on to the LIMS. High values are double-checked for calculation mistakes and low values for the possibility of contamination. Raw data is converted to standard reporting units by the usual method of numerator and denominator unit cancellation. The report is further reviewed before the data are sent to the client.

All records, including but not limited to instrumental raw data, run logs, analytical reports, instrument maintenance logs, QA documents will be retained in the lab for a period of ten years. Records older than ten years will be destroyed.

External Quality Control Checks

QC samples are solicited from clients and are welcomed from governmental agencies in an ongoing effort to maintain and improve analytical quality. External performance evaluation samples are tested as an external QC check at a minimum of once per year, and when available, for water and soil matrices.

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Corrective Actions

Errors, deficiencies and data that do not pass acceptance criteria will be investigated. Some of these instances may require corrective actions. These corrective actions will be documented in appropriate locations including instrument-specific maintenance logs, run logs and the company error log. Clients will occasionally contest the results of a specific sample and the subsequent re-analysis of this sample provides further feedback on the quality of analytical work. If re-analysis shows that the lab's original results are in error, then the analysis is free of charge and corrective action will be taken. An overall lab error log is kept as record of our laboratory's performance and is available for client inspection.

Quality Assurance Reports

The QA program will be reviewed and reported on at least annually by the lab/QA manager. This report will include an assessment of the overall effectiveness of the program and identify any deficiencies. The report will also include suggestions on how to deal with deficiencies as well as on improvements if necessary.

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QA Program Review

Date reviewed	Changes made	New revision #
December 2, 1999	Created	---
June 6, 2001	Added organizational chart; description of positions, roles; training, general updates	1
July 27, 2001	SS, CCV, extraction parameters generalized for various methods, and updated to include new methods. Minor changes to QC section	2
October 28,2002	SS, CCV, extraction parameters generalized for various methods, and updated to include new methods. Log In section updated by inclusion of information in "SOP for Sample Handling & Receiving".	3
Feb 28, 2003	Minor grammatical changes. RRT windows not needed for IS methods. DK removed from lab hierarchy. GFAA acceptance criteria tightened and GFAA matrix matching specified.	4

Appendix 7

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Organochlorine Pesticides by Gas Chromatography (Method 8081B, Rev. 2, 2000)

1 Introduction:

1.1 This method is used to determine the concentrations of various organochlorine pesticides in extracts from solid and liquid matrices, using fused-silica, open-tubular, capillary columns with electron capture detectors (ECD).

1.2 The PCBs as Aroclors is not included in the list of target analytes under this method. The PCBs was analyzed by using method 8082, which includes specific clean up and quantitation procedures for PCBs analysis.

1.3 This method is not recommended for determining Kepone.

2 Sample Collection, Preservation and Storage:

2.1 Samples should be collected in contaminant free glass containers. [Aqueous samples can be collected in 1L amber glass liter with TLE (Teflon lined enclosure) and 2 g to 30 g for solid samples in glass jars with TLE or metal liners (MAI)].

2.2 Sample extracted within 7 days and extracts are stored at cool 4°C in the dark and should be analyzed within 40 days of extraction.

2.3 For aqueous samples with no residual chlorine present, cool to 4°C. For aqueous samples with residual chlorine present, add 3 mL 10% sodium thiosulfate solution per gallon (or 0.008%) and cool to 4°C. Addition of sodium thiosulfate solution to sample container may be performed in the laboratory prior to field use.

3 Sample Extraction:

3.1 Water samples are extracted using a separately funnel extraction (method 3510C).

3.2 Soil samples are extracted using an ultrasonic extraction (method 3550C).

4 Extract Cleanup:

4.1 Method 3610 (Alumina): use to remove phthalate esters.

4.2 Method 3620 (Florisil): use to separate organochlorine pesticides from aliphatic compounds, aromatics, and nitrogen-containing compounds.

4.3 Method 3630 (Silica gel): use to separate single component organochlorine pesticdes from some interferants.

4.4 Method 3640 (GPC-pesticide option): use if a sample is of biological origin, or contains high molecular weight materials.

4.5 Method 3660 (Sulfur cleanup): use to eliminate sulfur from sample extracts, which may cause chromatographic interference with analytes with analytes of interest.

4.6 Method 3665 (Sulfuric acid/permanganate cleanup): use for the rigorous cleanup of sample extracts prior to analysis for polychlorinated biphenyls (PCBs). This method cannot be used to cleanup extracts for other target analytes. It will destroy most organic chemicals including the pesticides such as Aldrin, Dieldrin, Endrin, Endosulfan (I and II), and Endosulfan sulfate.

5 Definitions:

5.1 Note: Italicized names are formal EPA SW-846 nomenclature. Non-italicized names are used for uniformity to facilitate LIMS processing and reporting of QC data.

5.2 **ANALYSIS BATCH** – A group of up to 20 field samples of the same matrix.

5.3 **CALIBRATION CHECK** – Made from a stock solution which is different from the stock used to prepare the calibration standards to verify the ratio of instrument response to analyte amount.

5.4 **CALIBRATION STANDARD (CAL)** – Prepared from the primary dilution standard solution or stock standard solutions.

5.5 **CONTINUING CALIBRATION VERIFICATION STANDARD (CCV)** – Individual CAL solution analyzed.

5.6 **EQUIPMENT BLANK** –Appropriate reagent grade media is opened in the field and poured appropriately over or through the sample collection device, collected in a sample container and returned to the lab as a sample. Its purpose is to check the cleanliness of the sampling device.

5.7 **FIELD BLANK** – May be a trip blank or equipment blank.

5.8 **INITIAL CALIBRATION STANDARDS** – A series of CAL solutions used for initial calibration.

5.9 **INITIAL CALIBRATION VERIFICATION STANDARD (ICV) –** An individual CAL solution analyzed prior to any sample analysis, which verifies initial/historical calibration curves.

5.10 **INITIAL DEMONSTRATION OF PROFICIENCY (IDOC)** – A method is prepared and determined which generates data of acceptable accuracy and precision for a reference sample containing the target analytes in a clean matrix.

5.11 **LABORARORY CONTROL SAMPLE AND LABORATORY CONTROL SAMPLE DUPLICATE (LCS/LCSD)** *or LABORATORY FORTIFIED BLANK (LFB)* – Two aliquots of clean matrix (reagent water, Ottawa sand, wipe, CT, etc.) are spiked with midrange levels of analyte and run like a sample. They are analyzed as samples, and % recoveries (accuracy) and %RPD (precision) calculated and their purpose is to determine whether the methodology is in control.

5.12 **MATRIX SPIKE AND MATRIX SPIKE DUPLICATE (MS/MSD) or** *LABORATORY FORTIFIED MATRIX (LFM)* – Two aliquots of an environmental sample to which midrange levels of method analytes are added. They are analyzed as samples, and % recoveries (accuracy) and %RPD (precision) calculated with the purpose of determining whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes must be determined in a separate aliquot and subtracted.

5.13 **METHOD BLANK (MB)** *or LABORATORY REAGENT BLANK (LRB)* – For water samples, reagent water is treated exactly as a sample including exposure to glassware, etc. For soils no matrix is used. For wipes, charcoal tubes, etc, an unused sample medium is extracted exactly as a sample. Its purpose is to determine if contamination is present in any of the sample preparation or analytical steps.

5.14 **METHOD QUANTIFICATION LIMIT (MQL)** – The minimum concentration of analyte that can be reported, which can be no lower than the lowest calibration standard.

5.15 **PQL** – The Practical Quantitation limit is the lowest level that can be reliably determined within specified limits of precision and accuracy during routine laboratory operating conditions.

5.16 **QUALITY CONTROL SAMPLE (QCS) –** A solution of method analytes of known concentration that is used to spike an aliquot of LRB or sample matrix. The QCS must be from an external source and different than that of

the calibrations standards.

5.17 **REPLICATE SAMPLE –** Prepare by dividing a sample into two or more separate aliquots.

5.18 **STANDARD CURVE –** A curve which plots concentrations of known analyte standard versus the instrument response to the analyte.

5.19 **SURROGATE –** Compounds that are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are added to all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

5.20 **TRIP BLANK -** Aliquot of reagent water or solvent in sealed containers that is brought to the field and transported together with the field samples back to the lab.

6 Initial/Historical Calibration:

6.1 Prepare a minimum of five calibration curve and establish the calibration range of the method (the lowest standard is the basic of the method reporting limit).

6.2 Any of the following curve types may be used:

A. ***Linear calibration using the average calibration or response factor –*** When the RSD of the mean calibration factor or mean response factor for a single analyte is ≤ 20% over the entire calibration range (RSD = (SD / CF_{avg}) or =(SD / RF_{avg})), then the calibration curve can be assumed to be linear and to pass through the origin, and the average calibration or response factor may be used to determine sample concentrations. Even if the RSD for one or more analyte exceeds 20% the linear calibration using the average CF or RF may still be acceptable for these compounds if the following conditions are met:

a) The mean of the RSD values for ALL analytes in the calibration is ≤ 20%, AND,

b) The user / client must be provided a specific list of the compounds for which the RSD exceeded 20% and the results of their mean RSD calculations.

c) The alternatives to using an average RF (Internal Standard calculation) or CF (External Standard calculation) curve having >20% RSD are to adjust the calibration range until the RSD is ≤ 20% OR to use a different calibration type.

B. ***Linear calibration using a least squares regression –*** If the RSD of the average calibration or response factor is > 20% over the calibration range, then the linearity through the origin cannot be assumed. The analyst may use a linear regression fit that is not forced through the origin, but have the intercept calculated from the data points. The data points should be unweighted and not include the origin (0,0) as a calibration point. Only when combining replicate multi-point calibrations may data points be weighted, as detailed in Method 8000B, section 7.5.2. The use of a linear regression may not be used as a rationale for reporting results outside of the calibration range. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purpose, r ≥ 0.99 IS REQUIRED.

C. ***Non-linear calibration –*** To be suitable for non-linear calibration, the data point curve must be continuous, continuously differentiable and monotonic over the calibration range. The fit equation may not have more than 4 parameters, for example a third order polynomial. Do not force the line through the origin and do not include the

origin (0,0) as a calibration point. Six calibration standards are required for a quadratic fit [this is the ChemStation curve fit] and seven for a cubic fit. The coefficient of the determination (COD) or 'goodness of fit' will equal 1.0 for a "perfect" fit model. In order to be an acceptable non-linear calibration, COD $\geq$ 0.99 IS REQUIRED [COD value is displayed in ChemStation on page 1 of the compound's calibration record].

D. Whatever curve fit option is used, it must yield a unique concentration for every response (area).

6.3 Transfer an aliquot of each standard into an auto sampler vial and cap.

6.4 Load on auto sampler rack and analyze in order of least to most concentrated.

6.5 Calibration curves should be generated for each compound.

7 Calibration verification:

7.1 A CCV must be analyzed after every 12 hours, defined as ending when the last sample injected within the 12 hour window has completely eluted (and at the end of the analysis set if external standard calculation is used). If the response for these calibration verification standards is not ± 15%, the test must be repeated using a freshly made standard. If the freshly made standard also deviates by more than 15% sample analysis must be stopped, the cause determined and the instrument recalibrated. Alternatively, the CCV is acceptable if the average of ALL target analyte values is ±15% of their true values, BUT the user / client must be informed which analytes deviate more than ±15% of their true values. All samples following the last acceptable check standard must be reanalyzed. An to exception to this rule occurs when the samples following the last passing CCV are non-detect for target analytes and compounds failing the ±15% acceptance criteria are >15% of their true value.

7.2 The breakdown of DDT and Endrin should be measured before samples are analyzed and at the beginning of each 12-hour shift. If degradation of either DDT or Endrin exceeds 15%, take corrective action before proceeding with calibration.

8 Quality control:

8.1 IDOC: Each analyst for a given method must analyze at least 4 replicates of a mid-level standard **extracted** from a water matrix using the same procedure as for a water sample. Percent recoveries and RSDs for each analyte may be compared to values published in the method and should meet those criteria if they are from multi-laboratory studies, although this is not a strict requirement, and the development of in house guidelines is strongly recommended. In the absence of recommended acceptance criteria the suggested rule of thumb is 70-130% recovery. [MAI RPD ±20%].

8.2 IDOC: An MDL study must be performed using a minimum of 7 extracted replicates for each matrix.

8.3 Some methods require performance check standards to be analyzed prior to standards or samples.

8.4 One MB must be analyzed with each analysis batch. [MAI prefers to analyze it before processing any samples]. The MB must be carried through the entire extraction process, including any clean up procedures. When samples that are extracted together are analyzed on separate instruments, the method blank associated that batch is analyzed on one instrument and a solvent blank on the other instrument(s). The results must be below the MRLs before continuing the analysis. If not, corrective action must be taken. Blank subtraction is not allowed for GC and HPLC methods.

8.5 Some methods have an Internal Standard requirement and acceptance criteria regarding its constancy during a sequence. [At least one **Internal** standard should be added to each sample, blank, standard or spiked sample prior to its analysis. The use of internal standard may be delayed for methods that do not name acceptable compounds until MAI finds a suitable compound].

8.6 Surrogate recoveries should be monitored and evaluated against in house control limits. Dilution may cause the SS to be inaccurately measured; and if available less dilute injections may be used to evaluate SS recovery in these cases. Samples having unacceptable recoveries should be re-extracted / re-analyzed. If the re-extracted sample also has unacceptable recovery then the sample's data is reported as "estimated concentrations". If hold time expires prior to re-analysis report both sets of data and note the hold time problem. [At least one **surrogate** standard should be added to each sample, blank, standard or spiked sample prior to its processing / extraction. The use of surrogate standard is optional or may be delayed for methods that do not name acceptable surrogate compounds until MAI finds a suitable compound].

8.7 An LCS/LCSD pair containing approximately 10% of the reported target analytes must be prepared and analyzed with each analysis batch to demonstrate the lab's ability to perform the analysis in a clean matrix. The lab should monitor % Recoveries and % RPDs through control charts and compare data to that published in SW846 methods if collected from multi- lab studies. Many methods do not contain recommended acceptance criteria. The range 70-130% recovery is recommended as a general rule, and should be used as a guide to evaluate its in house limits once they are established. If the QC fail to meet these criteria the source of the problem should be identified and resolved before continuing analyses.

8.8 An MS/MSD pair containing approximately 10% of the reported target analytes should be prepared and analyzed for every analysis batch. Calculate the percent recovery and RPD for the MS/MSD pair. The lab should monitor % Recoveries and % RPDs through control charts and compare data to that published in SW846 methods if collected from multi- lab studies. Many methods do not contain recommended acceptance criteria. The range 70-130% recovery is recommended as a general rule, and should be used as a guide to evaluate its in house limits once they are established. The percent recovery should be within 70-130% or matrix induced bias may be a factor. Method 8000B recommends a 5:1 spike-to-background amount to allow for accurate evaluation. [MAI: If the spiked concentration is less than the background concentration, the recovery should not be calculated]. A QCS should be run quarterly. If the determined values are not within "acceptable accuracy" (MAI = ± 20% of the theoretical values) the source of the problem should be identified and corrected before continuing analyses.

8.9 Control chart parameters are calculated and monitored after ~20 samples for LCS & LCSD and MS & MSD and ~20-30 samples for SS, as follows. For each analyte for each matrix calculate the average percent recovery (p) and standard deviation (s). Plot this data along with its upper and lower control limits at the 99% confidence level (p ± 3s) and upper and lower warning limits at the 95% confidence level (p ± 2s). Evaluate subsequent LCS-LCSD, MS-MSD and SS values based on these limits and take appropriate corrective action if analyte recoveries fall outside of their control limits. A matrix effect may be the cause of failed MS-MSD recoveries if that batch's LCD-LCSD recoveries are good. These limits should be updated at least semi-annually. Do not omit data that does not conform to preconceived notions of acceptability, as limits based on this 'censored' data may be unrealistically

narrow. For methods and matrices with very limited data (such as unusual matrices) interim limits may be established using available data or by analogy to similar methods or matrices.

8.10 Retention time (RT) windows must be established for the identification of target analytes in all GC or HPLC methods that do not employ internal standards. MAI uses the internal standard method for all GC and HPLC analyses and retention times of all analytes are referenced to those of their internal standards. [Methods that are used at MAI that temporarily do not use IS, for example newly developed methods, will adopt the default RT window of 0.10 minutes for volatile analyses and 0.06 minutes for semi-volatile analyses, both based on our staff's collective experience. Our peak search windows are wider than this value to ensure that all proximal peaks are considered for identification but the final identification will be based on these windows].

9 Analysis/Calculation:

9.1 Samples are analyzed in a set referred to as a sequence. The sequence always begins with the CCV. Once the CCV has passed the criteria outlined in sec. 7, the instrument is considered to be in calibration and is ready to analyze samples and/or sample extracts.

9.2 All applicable QC samples including method blanks are included in the remainder of the sequence. In order for the sequence to be considered valid, the QC samples must pass all criteria outlined in Quality Control section above.

9.3 In the event that any of the CCVs or QC samples fail their criteria they should be immediately reanalyze.

9.4 Whenever silica gel (method 3630) or florisil (method 3620) cleanups are used, the analyst must demonstrate that the fractionation scheme is reproducible.

9.5 Confirmation of peak identification is required for non-MS detectors when the composition of the sample is not well characterized. [For MAI when site history is not known]. Confirmation techniques include use of a second dissimilar column, MS detection, or "other recognized confirmation techniques". [MAI accepts overspiking of the unknown with amount of known compound when there is a likely coelution mismatch]. For example, a second wavelength on a UV-VIS DAD detector may be used if the lab demonstrates using an established confirmatory method that this technique is capable in typical sample extracts. Method 8000B, section 7.10.4, indicates that two dissimilar detectors may be used for confirmation. **[MAI accepts second detector confirmation in tandem detector systems if there is ±20% agreement between results from the two detectors]**. When second column confirmation is used and "the results differ significantly, for example RPD > 40%", examine the chromatographic pattern for anomalies such as peak coelution or coincidental peak identification based on pattern mismatch, and if not present report the higher of the two values accompanied by an explanatory note to the user / client.

9.6 All calculations are performed by Chemstation software.

9 Interferences:

9.1 Interferences by phthalate esters is a major problem in pesticide determinations. It can be removed prior to analysis using method 3640 or method 3630. Avoiding contact with any plastic material and checking all solvents and reagents for phthalate contamination can also minimize it.

9.2 The presence of sulfur result in broad peaks that interferes with the detection of early-eluting organochlorine pesticides. Sulfur contamination should be expected with sediment samples and can be removed by method 3660.

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Appendix 8

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SOP for Instrumental Analysis of Trace Metals by ICP (EPA 6010C)

1. Scope and Applications:

1.1 This method is used to determine trace elements in aqueous, sludge and solid samples. [MAI uses EPA method 200.7 to determine metals in aqueous, water and waste water]. No digestion is required prior to analysis for dissolved elements in water samples. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial wastes, soils, sludges, sediments, and other solid wastes for which total (acid-leachable) elements are required.

2. Sample Collection, Preservation and Storage:

2.1 Aqueous samples should be collected in polyethylene or fluorocarbon (TFE or PFA) plastic and soils in core tubes or glass jars.

2.2 Sample preservation and holding time are as follows:

A. *Metals excluding hexavalent chromium and mercury*:

a. Aqueous samples: (200 Series define aqueous samples as they have < 1% sediments)

1. Total: HNO_3 to pH<2, 6 months (200 Series call this Total Recoverable and requires a pH check on each sample. Also, if acid is added in the lab, 200 Series require that the sample be acidified 16-18 hours prior to analysis).
2. Dissolved: Filter on site, HNO_3 to pH<2, 6 months.
3. Suspended: Filter on site, 6 months

b. Solid samples:

1. Total: 6 months

B. *Hexavalent Chromium*:

a. Aqueous samples: 24 hours. Store at $4^o \pm 2^oC$ until analyzed (EPA 218.6 calls for sample to be preserved with IC mobile phase concentrate).

b. Solid samples: one month to extraction, 4 days after extraction. Store at $4^o \pm 2^oC$ until analyzed.

C. *Mercury*:

a. Aqueous samples:

1. Total: HNO_3 to pH<2, 28 days.
2. Dissolved: Filter, HNO_3 to pH<2, 28 days.

b. Solid samples:

1. Total: 28 days. Store at $4^o \pm 2^oC$ until analyzed.

3. Sample Digestions:

Note: If mercury is to be analyzed, the digestion procedure must use mixed nitric and hydrochloric acids through all steps of the digestion. Mercury will be lost if the sample is digested when hydrochloric acid is not present. If it has not already been added to the sample as a preservative, Au should be added to give a final concentration of 2

mg/L (use 2.0 mL of 5.3.11 per 100 mL of sample) to preserve the mercury and to prevent it from plating out in the sample introduction system.

A. Soil Samples [MAI uses method 3050B]**:**

1. Weigh to the nearest 0.01 g and transfer a 1 g sample (wet weight unless otherwise specify) into a plastic digestion bottle.
2. Add 5 mL of DI water and 5 mL of HNO_3 (nitric acid), mix and cover with a non-ribbed watch glass.
3. Heat the sample at 90-95^oC 10-15minutes without boiling (85^oC for open bottle = 95^oC for covered bottle).
4. Cool the sample then add 5ml concentrated HNO_3, replace the cover, and heat for 30 minutes without boiling. Repeat this step until no brown fumes are given off by the sample. Cover bottle with ribbed watch glass and continue heating at 90-95^oC without boiling for 2 hours or until 5 mL of solution remains, whichever comes first. The bottom of the digestion bottle must be covered by solution at all times or the digestion must be redone.
5. Cool the sample and add 3 mL of 30% H_2O_2 (hydrogen peroxide) and 2ml water; cover with non-ribbed watch glass. If effervescence is strong, add H_2O_2 in 1 mL increments and heat until effervescence is minimal or until the general sample appearance is unchanged.
6. Cover with ribbed watch glass and heat the acid-peroxide digestate without boiling until the volume has been reduced to 5 mL or for 2 hours.
7. Add 10ml concentrated HCl to the sample. Cover with a non-ribbed watch glass and heat at 90-95^oC for 15minutes.
8. Bring to volume of 50 mL with DI water.
9. For GFAA and ICP-MS analysis, dilute sample 1:20 with 1% HNO_3 to make final acidity is 2% of HNO_3 and 1% of HCl.

B. Water Samples [MAI uses method 200 Series]**:**

Note: For all digestions, if antimony (Sb) is positive or silver (Ag) is greater than 100ppb, redigest using lower sample volume**.**

1. <u>Aqueous Sample Preparation – Dissolved Analytes</u>:

a. Filter and acid preserved 50 mL of sample into a 60 mL plastic bottle.

b. Add 1 mL (0.75 mL if sample was taken from preserved plastic container) concentrated HNO_3 and 0.5 mL concentrated HCl to sample bottle so that sample is 2% HNO_3 and 1% in HCl. [MAI requires the HCl to matrix match all water samples].

c. Cap bottle and mix. pH < 2 must be verified by measurement. The sample is now ready for analysis.

2. <u>Aqueous Sample Preparation – Total Recoverable Analytes</u>:

Note: Aqueous sample containing undissolved solids or particulate material [3] 1% (w/v) should be extracted as a solid type sample.

a. If the sample is not already preserved then add 2.5 ml of HNO_3, conc. per 500 mL of sample. If the sample is preserved in the lab then it must sit for 16 hours in the preservative prior to aliquoting and pH < 2 must be verified by measurement prior to aliquoting.

b. Transfer 50 mL of a well shaken sample to a plastic digestion bottle and add 0.75 mL nitric acid and 0.5 mL of hydrochloric acid to the sample bottle so that the sample contains 2% HNO_3 and 1% HCl.
c. Place the sample on a hot plate in a fume hood for solution evaporation. Adjust temperature to approximately but no higher than 85°C (85°C for open bottle = 95°C for covered bottle).
d. Cover the sample bottle with a ribbed watch glass to promote refluxing and to prevent sample contamination from the fume hood environment.
e. Reduce the volume to 10 mL by heating at 85°C without boiling (takes about two hours and volume diminishes rapidly around 10 mL). No part of the bottom of the digestion beaker may be exposed or go to dryness or the digestion must be redone.
f. Cover the lip of the sample bottle with a non-ribbed watch glass to discourage evaporation and gently heat the sample for 30 minutes. (Note: avoid vigorous boiling to prevent loss of the HCl-H_2O azeotrope).
g. Allow sample to cool and make to 50mL volume with DI. Sample is ready for analysis.

3. <u>Solid Sample Preparation – Total Recoverable Analytes</u>: (For aqueous samples with > 1% (w/v) sediment)

a. Transfer an appropriate amount of a well shaken sample (if the sample contains a high water content transfer 50-100ml) to a tared beaker and dry at 60°C. Transfer 1.0 ± 0.01 g of a representative dried sample to a plastic digestion bottle. The dried sample may be disaggregated with a mortar and pestle and sieved through a 5 mesh polypropylene sieve to achieve homogeneity.
b. Add 10 mL of DI water, 2 mL of HNO_3 and 2 mL of HCl. Cover the sample bottle with a non-ribbed watch glass.
c. Place the sample bottle on a hot plate in fume hood and adjusted to a temperature of approximately 85°C (85°C for open bottle = 95°C for covered bottle).
d. Heat the sample and gently reflux for 30 minutes. Avoid vigorous boiling to prevent loss of the HCl-H_2O azeotrope.
e. Allow sample to cool and add DI water to the 50 mL mark.
f. For GFAA and ICP-MS analysis, dilute sample 1:4 with DI water containing 2% HNO_3 so that final acidity is 2% of HNO_3 and 1% of HCl.

4. Definitions:

4.1 Note: Italicized names are used for uniformity to facilitate LIMS processing and reporting of QC data. Non-italicized names are method specific.
4.2 ***Analysis Batch*** – A group of up to 20 field samples.
4.3 ***Calibration Check*** – Made from a stock solution which is different from the stock used to prepare the calibration standards to verify the ratio of instrument response to analyte amount.
4.4 ***Calibration Standard (CAL)*** – Prepared from the primary dilution standard solution or stock standard solutions.
4.5 ***Continuing Calibration Verification Standard (CCV)*** – Individual CAL solution analyzed.

4.6 ***Equipment Blank*** – Appropriate reagent grade media is opened in the field and poured appropriately over or through the sample collection device, collected in a sample container and returned to the lab as a sample. Its purpose is to check the cleanliness of the sampling device.

4.7 ***External Reference Standard (ERS)*** – A midrange standard prepared with stock standards from a different source than those of the calibration standards.

4.8 ***Field Blank*** – May be a trip blank or equipment blank.

4.9 ***Initial Calibration Standards*** – A series of Cal solutions used for initial calibration.

4.10 ***Initial Calibration Verification Standard (ICV)*** – An individual mid-range standard analyzed prior to any sample analysis, which verifies initial/historical calibration curves.

4.11 ***Initial Demonstration Of Proficiency (IDOC)*** – A method is prepared and determined which generates data of acceptable accuracy and precision for a reference sample containing the target analytes in a clean matrix.

4.12 ***Instrument Check Standard (ICS)*** *or Interference Check Solution* – A solution is prepared to contain known concentrations of interfering elements that will demonstrate the magnitude of interferences and provide an adequate test of any corrections.

4.13 ***Instrument Detection Limits (IDLs)*** – The average calculation of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day reports in µg/L.

4.14 ***Instrument Performance Check Solution*** **(IPC)** – A midrange calibration standard.

4.15 ***Laboratory Control Sample and Laboratory Control Sample Duplicate (LCS/LCSD)*** *or Laboratory Fortified Blank (LFB)* – Two aliquots of reagent water spiked with midrange levels of analyte and run like a sample. Their purpose is to assess accuracy, precision, and to determine whether the methodology is in control.

4.16 ***Method Blank (MB)*** *or Laboratory Reagent Blank (LRB)* – Reagent water treated exactly as a sample including exposure to glassware, etc. Its purpose is to determine if contamination is present in any of the sample preparation or analytical steps.

4.17 ***Method Detection Limit (MDL)*** – The minimum concentration of analyte that can be reported, which can be no lower than the lowest calibration standard.

4.18 ***Matrix Spike and Matrix Spike Duplicate (MS/MSD)*** *or Laboratory Fortified Matrix (LFM)* – Two aliquots of an environmental sample to which midrange levels of method analytes are added. They are analyzed as a sample, with the purpose of determining whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes must be determined in a separate aliquot and subtracted.

4.19 ***Method Quantification Limit (MQL)*** – The minimum concentration of analyte that can be reported, which can be no lower than the lowest calibration standard.

4.20 ***Practical Quantitation Limit (PQL)*** – The lowest level that can be reliably determined within specified limits of precision and accuracy during routine laboratory operating conditions.

4.21 ***Quality Control Sample (QCS)*** – A solution of method analytes of known concentration that is used to spike an aliquot of RLB or sample matrix. The QCS must be from an external source and different than that of the calibration standards.

4.22 ***Replicate Sample*** – Prepare by dividing a sample into two or more separate aliquots.

4.23 ***Standard Curve*** – A curve which plots concentrations of known analyte standard versus the instrument response to the analyte.

4.24 ***Surrogate*** – Compounds that are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are added to all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

4.25 ***Trip Blank*** – Aliquot of reagent water or solvent in sealed containers that brought to the field and transported together with the field samples back to the lab.

5. Calibration and Standardization:

5.1 Instrument Setup: Turn argon on by opening valve on dewar. Turn recirculating chiller on. Turn RF generator on at front of instrument. Light the torch using "slow light" (shift + F12 on keyboard). If torch fails to light after several attempts, open torch compartment and look for obvious problems such as dirty or wet torch. If torch is wet, remove and rinse with methanol or acetone and dry using nitrogen or oven. If the torch appears dry but significantly dirty, follow removal, cleaning and replacement procedure in operator's manual. After replacing torch, attempt to light again. Once torch is lit, allow instrument to warm up for at least thirty minutes. While the instrument is warming up, perform a wavelength calibration. Fill rinse reservoir with a solution of DI water and 0.5% HNO_3. During extended use, the rinse should be replenished to avoid running dry. Check waste container. If there is a possibility of overflow during the day, dispose of waste and replace empty waste container.

5.2 Operating procedure: The order in which samples, standards, etc. are analyzed is very important and needs to be kept in mind when preparing sample rack for a sequence. The order for the sample rack is IPC, MIIC, calibration blank (every ten samples and at end of sequence), extraction blank, samples/digests, ERS (every ten samples and at end of sequence), QC. The volume of sample/digest needed is dependent on what method is being used. Generally, CAM17 requires 20mL and all other methods require 10mL. IS (internal standard – 1000ppm Y) must be added to culture tube before adding sample/digest. Add 0.1mL IS for 10mL sample/digests and 0.2mL for 20mL samples/digests. Always pipet IS into the bottom of culture tube. Pipet appropriate volume of sample/digests into tube with enough force to cause thorough mixing with IS. Use sample rack when preparing sample/digests and record necessary information in run log. This information must include Sample ID, tube #, matrix, extraction date if necessary, extraction factor and dilution factor, if any. Once sample rack contains all necessary samples, place on the autosampler. Uncover the appropriate calibration standards vials in calibration rack and top off if necessary. If you are unsure of calibration standard positioning or concentration. Load the appropriate method and refer to the standards menu. The calibration vials should periodically be cleaned with a strong acid solution. Load and start the appropriate sequence. Cover the calibration standards with parafilm ASAP after they have been analyzed. MDLs, RLs and upper ranges of linearity are defined by studies located in the ICP1 binder. The instrumental upper range of linearity for the CAM17 metals is 100ppm for all except Ba and Be, whose upper range is 40ppm. Any sample that exceeds the upper range must be reanalyzed at a dilution. Serial dilutions must be in agreement for the data to be valid. For uncharacterized elements use serial dilutions to validate results that are higher than the calibration standard or dilute to a concentration lower than the calibration standard.

5.3 Mixed Calibration Standard Solutions: [MAI uses 2% HNO_3 plus 1% HCl for all standards and samples]. The calibration standard solutions must contain an appropriate internal standard for each analyte. Prior to preparing the mixed standards, each stock solution must be analyzed separately to determine spectral interferences or the presence of impurities. Transfer the mixed standard solutions to freshly acid-cleaned FEP fluorocarbon bottles for storage. Some typical calibration standard combinations are listed as follows:

Solution	Elements
I	Be, Cd, Mn, Pb, Se and Zn
II	Ba, Co, Cu, Fe and V
III	As and Mo
IV	Al, Ca, Cr, K, Na, Ni, Li and Sr
V	Ag[a], Mg, Sb and Tl
VI	P

5.3 Blanks: Two types of blanks are required for the analysis. The calibration blank is used to flush the system between standards and samples. The calibration blank is used to monitor for possible contamination resulting from the sample preparation procedure and is also matrix matched. The calibration blank will also be used for all initial (ICB) and continuing calibration blank (CCB) determinations. The method blank must be carried throughout the entire sample preparation and analytical process.

5.4 The calibration must be verified initially with a second source ICV and then with a CCV after every ten samples and at the end of the run. Each analyte of interest must be within 10% of its true value or reanalysis or recalibation is required before any further samples may be analyzed. The calibration blank (CCB) must be analyzed at the same frequency [MAI: and it should follow the CCV to estimate carryover]. The CCB must be < 3 times the current IDL.

6. **Quality Control:**

6.1 Determine the instrument detection limit (IDLs) at least every three months and keep with the instrument log book.

6.2 The intensities of all internal standards must be monitored for every analysis. If the intensity of any internal standard in a sample falls below 30% of the intensity in the initial calibration standard, and instrument drift is not the cause, the sample must diluted until the 30% criteria is met. Nebulization efficiency varies with solution viscosity and IS is used to normalize this effect. Total Dissolved Solids < 0.2% is recommended for this reason as well as to prevent solids deposition at the cones.

6.3 One dilution test (serial dilution) must be included for each twenty samples or less of each matrix in a batch. If the analyte concentration is within the linear dynamic range of the instrument and sufficiently high (minimally, a factor of at least 100 times greater than the concentration in the reagent blank), an analysis of fivefold (1+4) dilution must agree within ± 10% of the original determination. If not, an interference effect must be suspected.

6.4 One MB must be analyzed with each analysis batch [MAI prefers to analyze it before processing any sample]. The results must be below the MDL before continuing the analysis. If not, corrective action must be taken.

6.5 An LCS/LCSD pair must be prepared and analyzed with each analysis batch [MAI: if the recovery of any analyte falls outside the control limits of 75-125% or the RSD < 20%, the source of the problem should be identified and resolved before continuing analyses].

6.6 An MS/MSD pair must be prepared and analyzed with each analysis batch. The spike may be added post-digestion. Calculate the percent recovery and RPD for the MS/MSD pair. The percent recovery should be 75-125% and less than 20 relative percent difference (RPD) for precision. Recovery outside of 75-125%, requires that the sample be diluted and the diluted values must be 10% of the original values to permit matrix effects to be assumed. Otherwise the MS/MSD must be redone.

7. Interferences:

7.1 All known interferents (or their derivatives, such as alternate isotope of an interfering element) must be measured in addition to the target analyte. The enclosed table (not ready yet) shows a list of known isobaric interferents.

7.2 Where an interference source is present, the analyte impacted must be flagged for QA completeness to indicate: a) the percent of interference correction applied to the data, or b) an uncorrected interference by virtue of the elemental equation used for quantitation. Observed rather than theoretical (ex: $^{35}Cl/^{37}Cl = 3.13$) should be used to make interferent correction and chelation or collision cells can greatly diminish polyatomic interferents.

8. Data Analysis and Calculations:

7.1 All calculations are performed by instrument software. If dilutions were performed, the appropriate factors must be applied to sample values.

7.2 The quantitative values are reported in micrograms per liter (µg/L) for aqueous samples and milligrams per kilogram (mg/kg) for solid samples.

7.3 The concentrations determined are to be reported on the basis of the actual weight of the sample. If a dry weight analysis is desired, then the percent solids of the sample must be also be provided.

7.4 If percent solids is desired, a separate determination of percent solids must be performed on a homogeneous aliquot of the sample.

Appendix 9

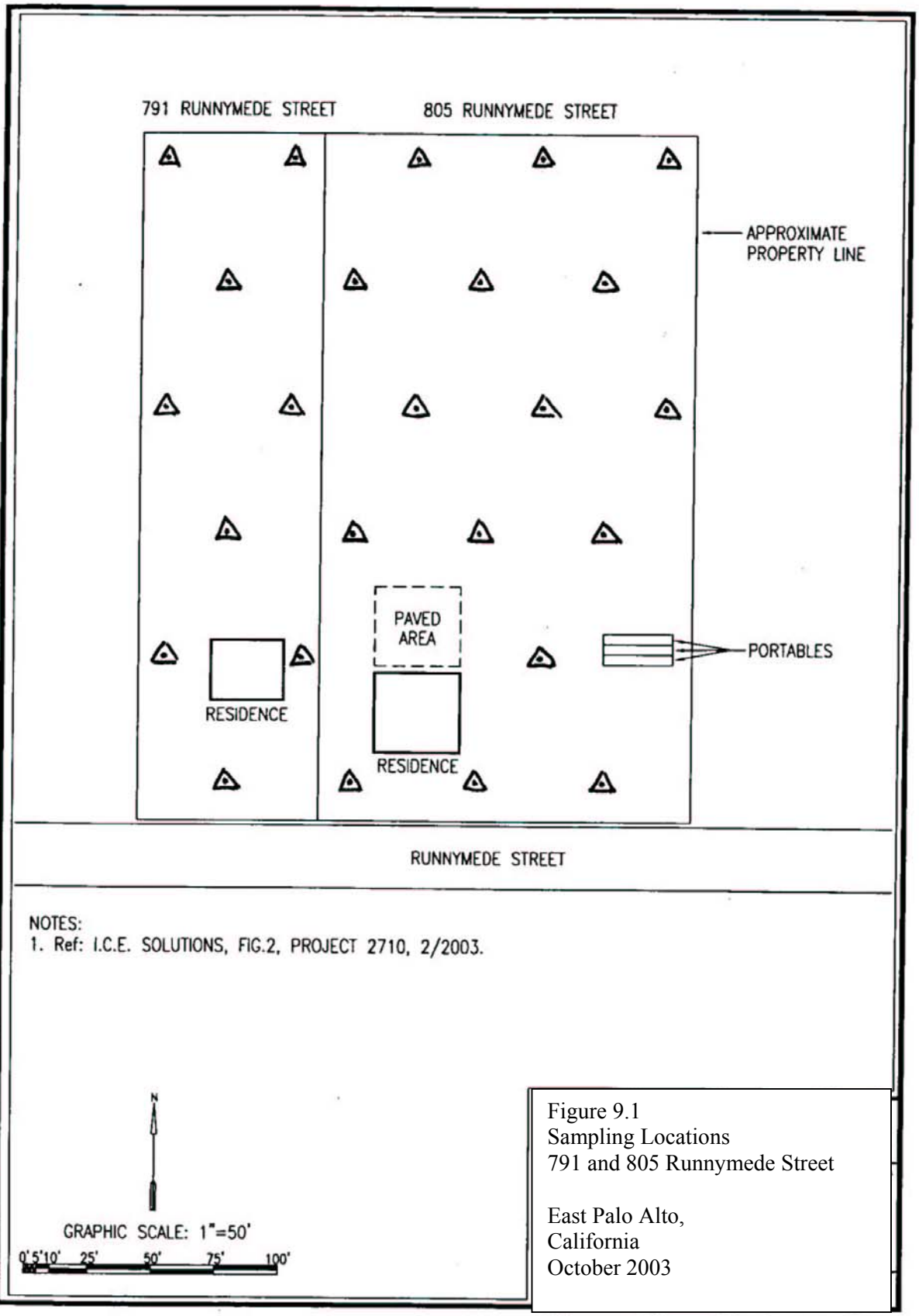

Figure 9.1
Sampling Locations
791 and 805 Runnymede Street

East Palo Alto,
California
October 2003

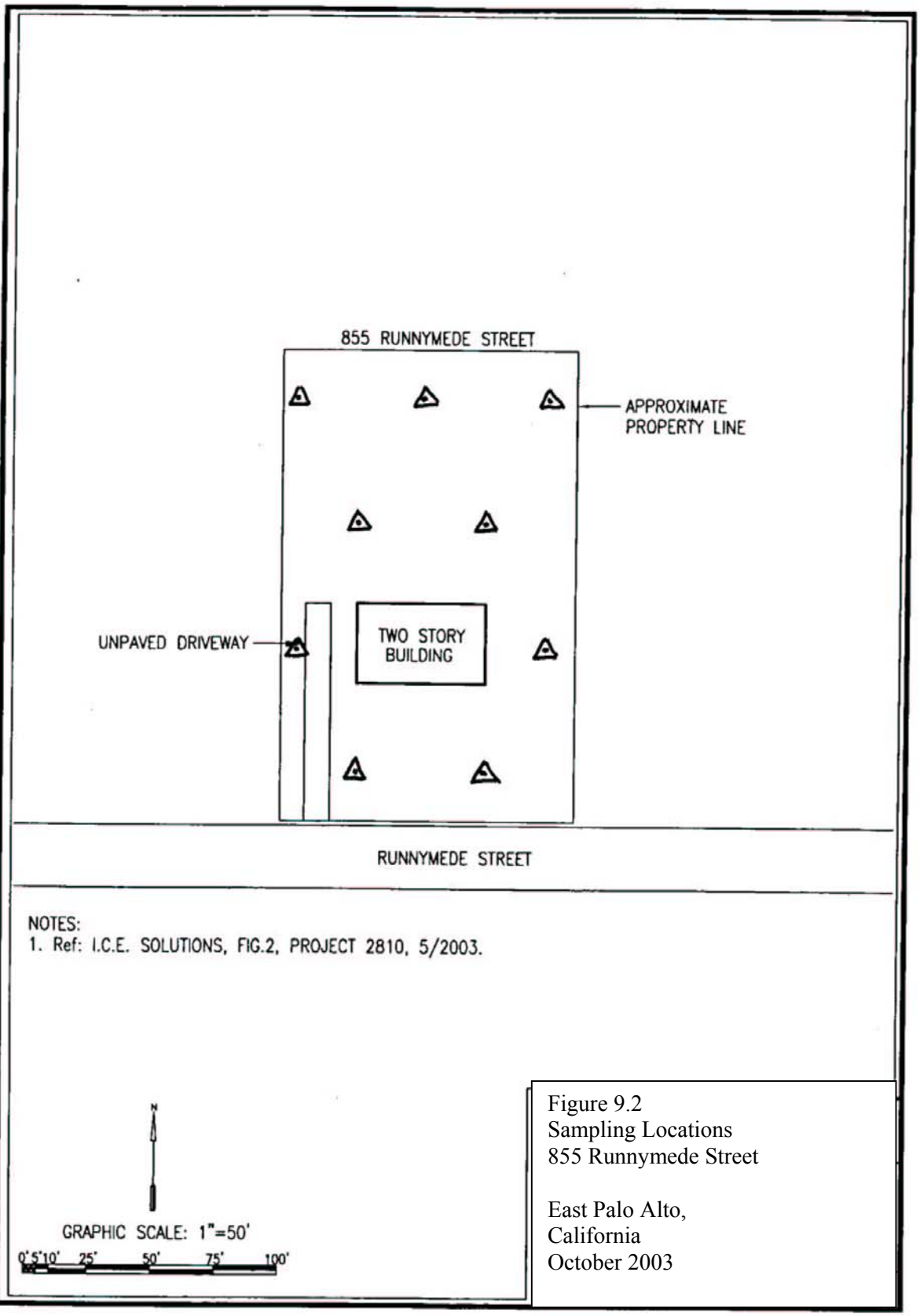

Figure 9.2
Sampling Locations
855 Runnymede Street

East Palo Alto,
California
October 2003

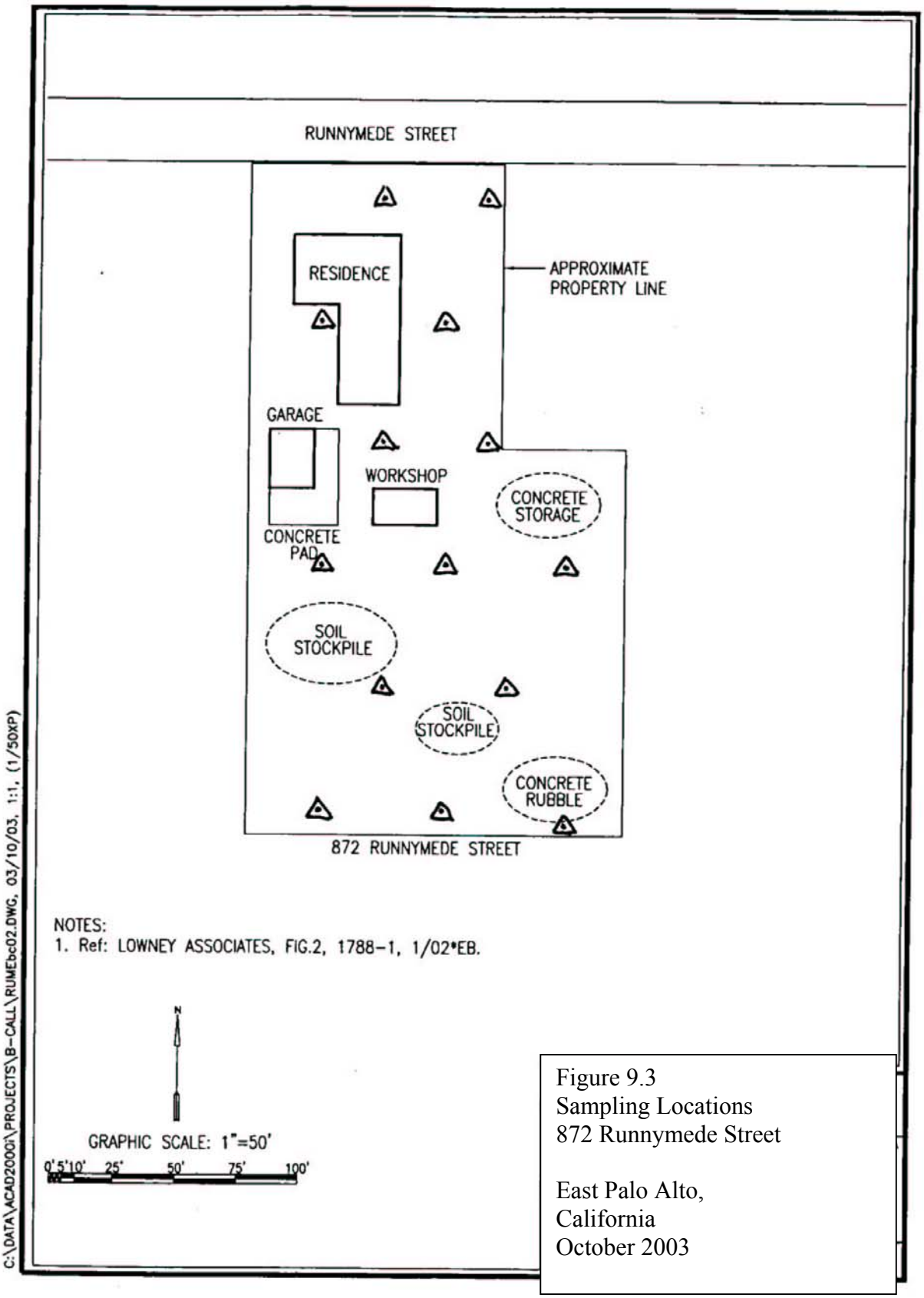

Figure 9.3
Sampling Locations
872 Runnymede Street

East Palo Alto,
California
October 2003

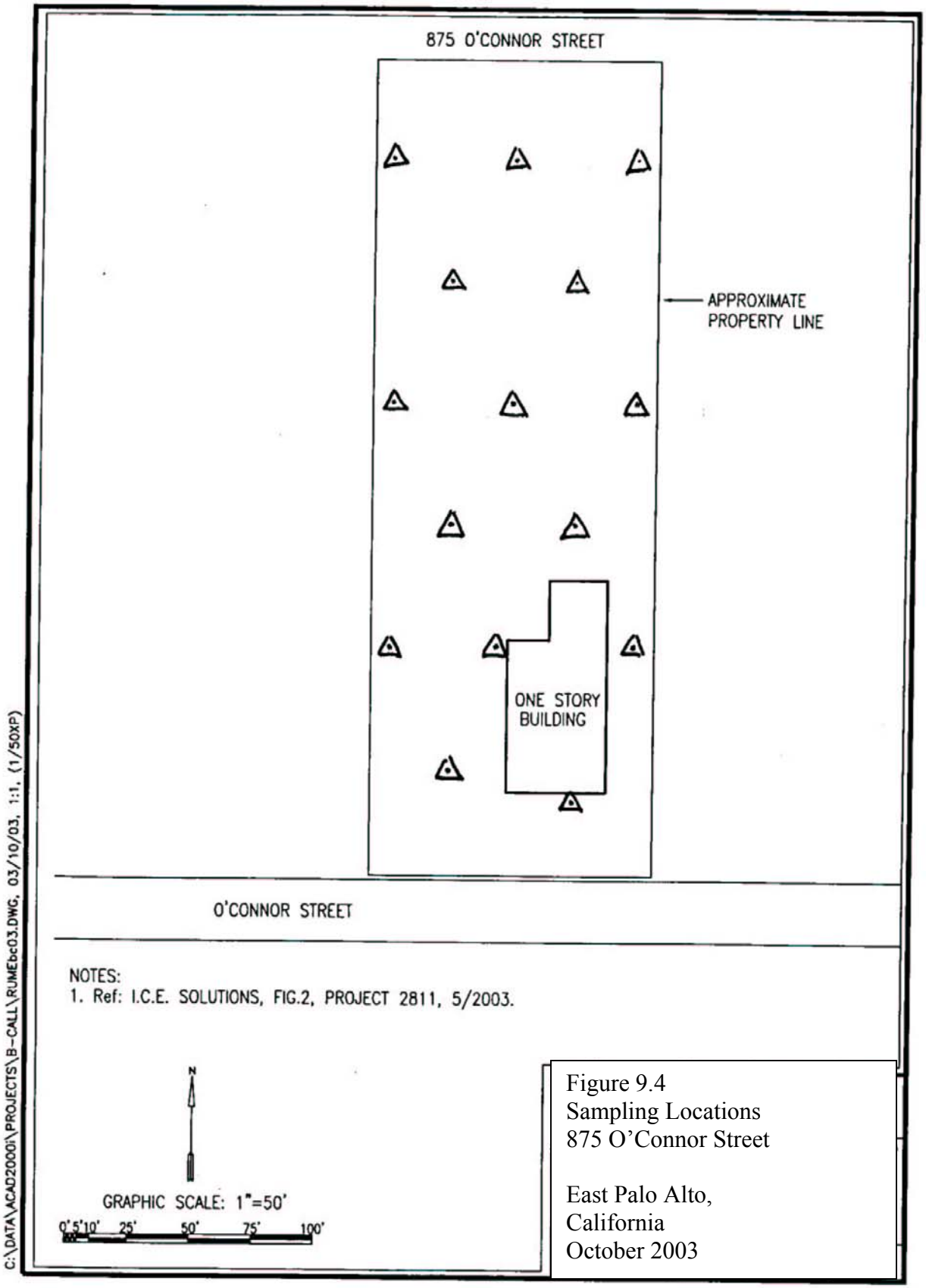

Figure 9.4
Sampling Locations
875 O'Connor Street

East Palo Alto,
California
October 2003