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DATA VALIDATION MEMORANDUM

TO: Sharon Fair, Branch Chief
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FROM: Stephanie Lewis
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DATE: May 2, 2006

SUBJECT: Summary of the Level II Data Validation for Advanced Technology Laboratory Report ATV5976, Dated April 25, 2006

INTRODUCTION

This memorandum summarizes the finding of a Level II data validation for analytical results of 25 soil samples (including three duplicates) and two water samples (one equipment rinsate blank and one investigative derived waste classification sample) collected from the Justice Elementary School site (Site) in the West Hills community. Los Angeles Unified School District (LAUSD) built and opened the school Site at 23350 Justice Street, Canoga Park, in 1959. A surface drainage is located north of Justice Street along the Site. The drainage originates approximately 2 miles to the west. A concrete lined flood control channel was built along the original earth drainage ditch in 1966. The drainage ditch has always been well below its surrounding grade and no overflow from the drainage ditch is anticipated. The West Hills community requested that the Department of Toxic Substances Control (DTSC) sample the Site for potential presence of perchlorate. As such, DTSC prepared a Sampling and Analysis Plan (SAP) for a limited soil investigation at the Site. A quality assurance and quality control (QA/QC) plan was included in the SAP to provide an appropriate level of assurance regarding the reliability and usability of the data generated during the proposed soil investigation. On April 10, 2006, DTSC implemented the SAP and collected samples as planned. All samples were delivered to Advanced Technology Laboratories (ATL) for perchlorate analysis by EPA Method 314.0, as modified if necessary. EPA Method 314.0 is known as "Determination of Perchlorate in Drinking Water Using Ion Chromatography (IC)." On April 25, 2005, an analytical report (ATV5976) comparable to previous USEPA Level II contract lab documentation was submitted by ATL to DTSC.

DATA VALIDATION

The QA objectives of the investigation are to assure that sampling, analysis and reporting activities provide data that are accurate, precise, representative, and legally defensible. QC represents the specific steps and procedures followed during the course of the project to achieve QA. The QA/QC Plan was implemented as specified in the SAP. The primary QC features included the collection and analysis of QC samples, a field audit, and the data validation.

Data validation is a process of evaluating the performance of data collection against the pre-determined method, procedural, or contractual requirements specified in the SAP. It routinely evaluates how closely the SAP has been followed during data generation in the field and laboratory. It checks for improper practices, abuse and warning signs shown during the investigation. It determines if the available data satisfies the project's data quality objectives (DQOs) and data use requirements by evaluating the data reports for field sampling procedures, laboratory performance and error checks.

DTSC conducted this Level II data validation for ATL's analytical results, including review of project QC program, sampling procedures, analytical procedures, data reports, and DQOs. Each review is presented below.

REVIEW OF PROJECT QC PROGRAM

To ensure that chemical data is of the highest confidence and quality, the review of QC program was divided into two parts: basic QC procedures and QC samples. No findings were identified affecting the quality of the samples collected or the resulting data results.

Basic QC Procedures: Basic QC evaluation criteria include field decontamination, supplies, holding times, equipment calibration and maintenance, and standards.

- **Field Decontamination:** Non-dedicated equipment was decontaminated before and/or after each sample is collected.
- **Supplies:** All supplies were certified clean by the suppliers, inspected by DTSC prior to their use and monitored by ATL through the use of standards and blank samples.
- **Holding Times:** Compliance with holding time requirements was verified.
- **Equipment Calibration and Maintenance:** ATL stated that analytical equipment calibration and maintenance were properly performed as recommended by the manufacturers and described in the ATL's QA/QC Plan. ATL's documentation of compliance and raw data will be made available to DTSC upon request and may be subject to audit by ELAP inspectors through ELAP certification process.

- **Standards:** ATL stated that standards used for calibration or to prepare samples were currently certified by or traceable to National Institute of Standards and Technology (NIST) or other equivalent source. ATL's documentation of compliance will be made available to DTSC upon request and may be subject to audit by ELAP inspectors through ELAP certification process.

QC Samples: Appropriate QC samples include field QC samples, background samples, field testing confirmation samples and laboratory QC samples.

- **Field QC Samples:** Field QC samples included one (1) equipment rinsate blank (for assessment of field contamination) and three (3) field duplicates (for assessment of sampling variability due to sampling technique, instrument performance or the heterogeneity of the matrix being sampled), at a rate not less than one per matrix per day when equipment was decontaminated in the field.
- **Background Samples:** No background samples were planned because perchlorate was not considered a naturally occurring compound.
- **Field Test Confirmation Samples:** No confirmation samples were planned because no field test was conducted.
- **Laboratory QC Samples:** Laboratory QC sample types included method blanks, laboratory duplicates, laboratory control samples (LCS), matrix spikes (MS), and matrix spike duplicates (MSD). ATL analyzed three (3) soil samples and one (1) water sample for each laboratory QC sample type to monitor the precision and accuracy of its analytical procedures, at a rate not less than one laboratory QC sample per type per batch of up to 20 samples (including blanks and duplicates).

REVIEW OF SAMPLING PROCEDURES

John Naginis, a DTSC senior engineering geologist and a Professional Geologist registered in California, supervised the sampling activities on April 10, 2006. Field activities were planned, conducted and completed in a manner consistent with the SAP and were monitored through a field audit and photo documentation by Dr. Ann Chang, a DTSC project manager. No specific findings were identified affecting the quality of the samples collected or the resulting data results.

- **Field Documentation:** Field logs and other documentations were reviewed regarding sampling procedures (e.g., sample containers, collection, preservation, packaging, transportation, receipt, handling and storage, chain of custody, holding time, and decontamination procedures) conducted on April 10, 2006. DTSC project manager delivered all collected samples to ATL on the same day.
- **Boring Logs:** Due to the nature of the sampling activities and sampling depths, no boring logs were prepared as originally planned.

- **Sample Conditions:** Upon receipt, ATL inspected the condition of the sample containers and reported the information accordingly on the chain-of-custody forms (which were attached to the analytical report). If conditions or problems were identified which would require immediate resolution, ATL would immediately notify DTSC. Such conditions may include wrong sample container, container breakage, water leaks, missing or improper chain-of-custody, exceeded holding times, improper preservation, missing or illegible sample labeling, or temperature excursions. DTSC did not receive any such notification from ATL.
- **Observations of Significance:** The sample identification (ID) for Sample JES-S07-0.5 collected from Sample Location JES-S07 was inadvertently labeled as JES-S07-3 in the field. Subsequently, correction of this error has been made on Table 3-4 of the field logs, Chain of Custody Record and ATL's analytical report.

REVIEW OF ANALYTICAL PROCEDURES

DTSC only evaluated criteria of analytical method, instrument calibration and reporting limits (RLs). All analyses were performed as specified in ATL's standard operating procedures (SOPs), EPA Method 314.0 requirements and DTSC Method 955-M. Review of surrogates, retention time window and tentative identified compounds (TICs) is not necessary because they are only applicable for organic analyses. No findings were identified affecting the quality of the samples collected or the resulting data results.

- **Analytical Method:** Analytical method for perchlorate analysis is EPA Method 314.0. However, EPA Method 314.0 is designed for water samples. ATL was instructed to follow DTSC Method 955-M for soil sample extraction. All collected soil and water samples were analyzed for perchlorate, using EPA Method 314.0, as modified if necessary.
- **Laboratory Certification:** All samples were analyzed by ATL, an ELAP certified laboratory. No subcontracted laboratory was used. ATL's QA/QC manual and SOPs are maintained in project files. ATL was instructed to report any estimated values with a "J" qualifier, i.e., between the method detection (MDL) and RL, and no "J" flagged value was reported.
- **Calibrations:** Instrument calibrations shall be checked as specified in the applicable method and the laboratory's QA/QC Plan prior to analysis. Analyte concentrations can be determined with either calibration curves or response factors, as defined in the method. ATL has maintained records of standard preparation and instrument calibration (procedures, frequency and results). As discussed in the Review of Project QC Program (Equipment Calibration and Maintenance), ATL's documentation and raw data will be made available to DTSC upon request and may be subject to audit by ELAP inspectors through ELAP certification process. Records unambiguously trace the preparation of standards and their use in calibration and quantitation of sample results.

- **RLs:** The RLs for perchlorate analyses must be defensible, not less than the results of the ATL's MDL study, and not greater than the DTSC-approved screening levels. DTSC had previously determined the RLs to be 40 micrograms per kilogram (ug/kg) for soil samples and 2.0 micrograms per liter (ug/L) for water samples, respectively. ATL used the approved RLs and ran its lowest calibration standards at or near its RLs.

REVIEW OF DATA REPORTS

Data review was performed to ensure that the data produced were credible, cost-effective, and of known and defensive quality. The data was reviewed in accordance with the SAP, the ATL SOPs, the principles present in *USEPA National Functional Guidelines for Laboratory Data Review - Organics* (EPA, 1999) and *USEPA National Functional Guidelines for Laboratory Data Review - Inorganics* (EPA, 2002), and the professional judgment of the validation team.

All collected samples were analyzed for perchlorate, using EPA Method 314.0. The analytical results were summarized in **Table 1**. A designation of "ND" means not detected at the RLs of 40 ug/kg for soil samples or 2.0 ug/L for water samples. The areas of data review are summarized in **Table 2** and discussed below.

- **Completeness of Laboratory Report:** The analytical report was considered complete because it contained the following information: laboratory/client/sample IDs, ELAP certification number, project name, sample matrix, sample collection/preservation/preparation/extraction/analysis dates, analytical methods, analytes, reporting units/limits, dilution factors, report page numbering system, designated title and signatures.
- **Chain of Custody:** A set of Chain of Custody forms (three pages) was included in the analytical report. The Chain of Custody was properly completed. ATL marked sample conditions on the forms upon receipt. However, wrong sample ID for Sample JES-S07-0.5 was inadvertently labeled and marked as JES-S07-03. As discussed previously in the Review of Sampling Procedures (Observations of Significance), corrections have been made subsequently.
- **Sample Containers and Conditions:** As discussed previously in the Review of Sampling Procedures (Sample Conditions), ATL marked the sample container conditions as normal on the Chain of Custody forms.
- **Holding Times:** All samples were collected and analyzed on April 10, 2006. All analyses were performed within the method-specified holding time (28 days).
- **Preservation:** No specific chemical preservation requirements were required for perchlorate analyses. However, all samples were refrigerated during transport and storage as specified in the SAP.

- **Field QC Samples (Equipment Rinsate Blanks):** Sample EQUIPMENT RINSATE was submitted to ATL as an equipment rinsate blank. The ND result met the analytical goal of no detectable analyte and the equipment rinsate blank results were acceptable.
- **Field QC Samples (Field Duplicates):** Samples JES-S03-0.5D, JES-S07-2D and JES-S08-2D were submitted to ATL blind, but designated as field duplicates for Samples JES-S03-0.5, JES-S07-2 and JES-S08-2, respectively. All primary and duplicate samples were reported ND. Since ND samples yield no usable numbers to perform statistical evaluation, they are not generally used to evaluate the precision of analyses. In instances when field duplicates yield no usable numbers, the MS/MSD results are used to evaluate the precision of the analysis. Therefore, the field duplicate results were acceptable.
- **Surrogate Recoveries:** No surrogate recoveries were applicable to perchlorate or non-organic samples.
- **Laboratory QC Samples (Method Blanks):** No target analytes were detected in the associated method blanks. The method blank results were acceptable.
- **Laboratory QC Samples (LCS):** Three (3) LCS soil samples and one (1) LCS water sample were prepared and analyzed by ATL at a frequency higher than the one specified in the SAP. The percent (%) recoveries of all spiked analytes were within the laboratory's acceptance criteria (85 – 115%). The LCS results were acceptable.
- **Laboratory QC Samples (MS/MSD):** Samples JES-S01-0.5, JES-S06-0.5, JES-S10-0.5, and EQUIPMENT RINSATE were utilized for the MS/MSD analyses. The % recovery of 129% for Sample JES-S10-0.5 MSD was outside the control limit of 80 – 120%; and the data was "S" flagged. The RPDs (21.5% and 21.9%) for MS/MSD samples of Sample JES-S06-0.5 and JES-S10-0.5, respectively, exceeded the RPD control limit of 15%; and the data were "R" flagged. ATL also reported that the RPDs were calculated from raw data, rather than the reported values (which were rounded values).
- **Laboratory QC Samples (Duplicates):** ATL prepared laboratory duplicates from Samples JES-S01-0.5, JES-S06-0.5, JES-S10-0.5, and EQUIPMENT RINSATE, respectively. All primary and duplicate samples were reported ND. The calculated RPD between field sample and laboratory duplicate analyses was reported zero for each pair in the analytical report. The laboratory duplicate results were acceptable. However, ND samples are not generally used to evaluate the precision of analyses since they yield no usable numbers to perform statistical evaluation. In such instances, the MS/MSD results are used to evaluate the precision of the analysis.

- **Compound Identification and Quantitation:** The analytical report contained data for the target analyte, perchlorate, using EPA Method 314.0 in ug/kg for soil samples and in ug/L for water samples. Qualitatively, the analyte was documented to be correctly identified and reported. Since all samples were reported ND, there is no need for any quantitation. However, raw data were not reviewed as part of Level II data validation. Result recalculation or transcription error checking from the raw data was conducted separately by ATL. Analytical results were checked, verified and confirmed to be correctly calculated by ATL.
- **Dilution Factors:** No dilutions were performed.
- **Data Qualifiers:** Data validation flags, as defined in the *National Functional Guidelines*, indicate if results are considered anomalous, quantitative, estimated, or rejected. All qualifiers should be discussed prior to utilizing the chemical data for the screening risk evaluation. Only rejected data are unusable for decision-making purposes; however, other qualified data may require further verification. ATL was instructed to report any "J" flagged values if there was any. No "J" flagged values were noted in the analytical report. However, ATL used "R" and "S" qualifiers to flag those QC data with out of control range conditions in its Analytical QC Summary Report.
- **Confirmation of Positive Samples:** Pursuant to the SAP, the presence of perchlorate in all positive soil samples by an ion chromatography (e.g., EPA Method 314.0) should be confirmed and analyzed by an ion chromatography/mass spectrophotometer (IC/MS) method or equivalent, with a RL of 0.2 µg/kg. Since all samples were reported ND, no confirmation analysis by the IC/MS method was necessary.
- **Observations of Significance:** No occurrences which might adversely affect sample integrity or data quality were noted in the analytical report.
- **Case Narrative:** The analytical report included a case narrative describing all variances, deviation or deficiencies encountered during laboratory analyses, possible reasons (with verifications), potential impacts, and corrective actions taken, if any.

REVIEW OF DATA QUALITY OBJECTIVES (DQOs)

The project DQOs were evaluated to determine whether the quantitative and qualitative needs of the sampling and analysis program had been met. DQOs were specified in terms of specific data quality indicators (DQIs), i.e., precision, accuracy, representativeness, completeness, comparability, and RLs. The data generated from the perchlorate investigation may not be considered invalid if the DQOs or criteria are not fully achieved, but variances will trigger the appropriate QA/QC measures needed to evaluate and correct these activities, if necessary.

Quality DQIs: Qualitative DQIs are comparability and representativeness.

- **Comparability:** Comparability expresses the confidence with which one data set can be compared to another. ATL used the specified EPA Method 314.0, consistent with the current standards of practice as approved by USEPA and DTSC. The method specified in the SAP allows the data to be evaluated for trends or changes (in space or time) at the Site. All data were calculated and reported in units (ug/kg and ug/L) consistent with standard procedures so that the results of the analyses can be compared with those of other laboratories, if necessary. The DQO for comparability has been met.
- **Representativeness:** Representativeness is the degree to which data accurately and precisely represent the actual Site conditions (in terms of a population, parameter variations at a sampling point, process condition, or environmental condition. To address representativeness, the SAP specified sufficient and proper number and locations of samples; incorporating appropriate sampling methodologies; specifying and performing proper sample collection and preservation techniques; performing required decontamination procedures; selecting appropriate methods to prepare and analyze soil samples and equipment rinsate blanks; and establishing proper field and laboratory QA/QC procedures for the parameters of interest. Samples were collected and analyzed in accordance with the SAP. The DQO for representativeness has been met.

Quantitative DQIs: Quantitative DQIs are precision, accuracy, completeness, and RLs. Precision and accuracy objectives, based on statistically generated limits established annually by ATL, were viewed as goals, not as criteria. If matrix bias is suspected, the associated data will be qualified and the direction of the bias indicated in the data validation report. The ND results for field duplicates indicated appropriate sample collection and handling procedures.

- **Precision:** Precision measures the reproducibility of repetitive measurements by assessing the RPD between field sample and field duplicate analyses, MS/MSD analyses, and field sample and laboratory duplicate analyses. If the RPD exceeds 15% as set by ATL, data may be qualified. The following two sets of MS/MSD samples failed this RPD standard: Sample JES-S10-0.5 (21.9%) and JES-S06-0.5 (21.5%). Since the LCS and method blank results were acceptable, the associated sample results were ND, and the RPDs (10.7% and 0.842%) of MS/MSD for Samples JES-S01-0.5 and EQUIPMENT RINSATE were below the RPD limit of 15%, the effect of the out-of-control high %RPD (due to potential matrix interference) may be negligible. As a result, the effect does not impact the data and the associated sample results will not be qualified as estimates.
- **Accuracy:** Accuracy is a statistical measurement (the degree of agreement of a measurement with a known or true value) of correctness and includes components of random error (variability due to imprecision) and systematic error.

Laboratory accuracy is expressed as the % recovery by assessing LCS (85 - 115%), MS/MSD (80 - 120%), laboratory duplicate (85 - 115%) and initial and continuing calibrations of instruments. All recoveries of LCS, MS, MSD, and laboratory duplicate were reported within the corresponding control limits with the exception of Sample JES-S10-0.5 MSD which had a % recovery of 129%. Since the LCS results were acceptable and the associated sample results were ND, the effect of the out-of-control high recovery (due to potential matrix interference) may be negligible. The associated sample results will not be qualified as estimates, but will be used with caution for being biased high.

Field accuracy is assessed through the analysis of equipment rinsate blanks to monitor errors associated with the sampling process including equipment decontamination procedures, field contamination, sample preservation, and sample handling. The DQO for equipment rinsate blanks is that all reported values are less than the corresponding RLs for perchlorate. The analytical result of the equipment rinsate blank was ND.

Therefore, the accuracy DQO has been met.

- **Completeness:** Completeness is the amount of valid data obtained compared to the amount expected under ideal conditions. The DQO for completeness is to obtain valid results for at least 90% of the planned data results. Completeness may be affected by such factors as sample bottle breakage and acceptance/non-acceptance of analytical results. The analytical data for the perchlorate samples are 100% complete and the DQO for completeness has been met.
- **RLs:** ATL reported the following MDLs for EPA Method 314.0: 12 ug/kg for soil samples and 1.2 ug/L for water samples, respectively. ATL used the following RLs: 40 ug/kg for soil samples and 2.0 ug/L for water samples, respectively. The SAP requires that ATL report detected concentrations that are above the MDL but below the RL. A provisional screening level of 43,000 ug/kg has been calculated for perchlorate in residential soil. Therefore, these RLs for perchlorate analyses meet the objectives of having sufficient quality data to perform a screening risk evaluation. ATL reported no "J" flagged values and evaluated closely since detections near the MDL can have high variability.

CONCLUSIONS

Based on this Level II validation, all data collected through implementation of the SAP satisfy data quality requirements specified for the perchlorate investigation. The analyses followed the approved method and included acceptable QC procedures. Some matrix effects were noted, which are typical of real environmental samples. The relevant QA/QC results were satisfactory and acceptable. No outstanding issues were identified during the course of the data validation review. Overall, the presented data (including the qualified results) are reliable and useable for project decision making. It appears that no presence of perchlorate exists.

RECOMMENDATION

It is recommended that the data be used to characterize the nature and extent of any contamination, support screening risk evaluation, evaluate the response action need, or assist in determination of additional actions.

TABLE 1

Justice Elementary School (JES) Perchlorate Sampling
Perchlorate Concentrations in $\mu\text{g}/\text{kg}$
Samples Collected 4/10/2006 and Analyzed by EPA Method 314.0

| Depth | Sample Location | | | | | | | | | | | | |
|-------|-----------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|--------|---------|---------|
| | SO1 | SO2 | SO3 | SO4 | SO5 | SO6 | SO7 | SO8 | SO9 | S10 | S03-DP | S07-2DP | S08-2DP |
| 0.5 | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | - | - |
| 1* | - | - | - | - | - | - | ND | ND | - | - | - | - | - |
| 2 | ND | ND | ND | ND | ND | ND | ND | ND | ND | ND | - | ND | ND |

Key:

* The one-foot samples were collected to evaluate the root zone conditions in the garden areas.

ND = Not detected at the RLs of 40 $\mu\text{g}/\text{kg}$ for soil samples or 2.0 $\mu\text{g}/\text{L}$ for water samples

- = No samples collected

DP = Duplicate sample

Depths were measured in feet below existing grade

TABLE 2

| QUALITY INDICATOR | ACCEPTABILITY | |
|--|--|---|
| | SOIL | WATER |
| | modified EPA Method 314.0 Target Analyte: perchlorate | EPA Method 314.0 Target Analyte: perchlorate |
| Completeness of Laboratory Reports (e.g., laboratory, client, and sample identifications; ELAP certification number, project name, sample matrix, sample collection, preservation, preparation, extraction, analysis dates; analytical methods; analytes; reporting units and limits; dilution factors; report page numbering system; designated title and signatures) | Y | Y |
| DTSC Method 955-M for Sample Extraction | Y | NA |
| Reporting Limit (RL) | Y (40 ug/kg) | Y (2 ug/L) |
| Chain of Custody | Y | Y |
| Sample Containers and Conditions | Y | Y |
| Holding Time (< 28 days) | Y (1 day) | Y (1 day) |
| Sample Preservation | Y | Y |
| Equipment Rinsate Blanks | Y | Y |
| Field Duplicates | See discussion | See discussion |
| Field QC Samples – Others | NA | NA |
| Surrogate Recoveries | NA | NA |
| Method Blanks | Y | Y |
| LCS % Recovery | Y | Y |
| MS/MSD % Recovery | See discussion | See discussion |
| MS/MSD % RPD | See discussion | See discussion |
| Laboratory Duplicates | See discussion | See discussion |
| Laboratory QC Samples – Others | NA | NA |
| Compound Identification | Y | Y |
| Compound Quantitation | Y | Y |
| Dilution Factors | Y | Y |
| Data Qualifiers | Y | Y |
| Confirmation of Positive Samples | NA | NA |
| Observations of Significance | NA | NA |
| Case Narrative | Y | Y |
| Instrument Tuning | NA | NA |
| Initial Calibration | Lab | Lab |
| Calibration Verification | Lab | Lab |
| Interference Check Standard | NA | NA |
| Others | NA | NA |

NOTES:

Y = acceptable or in compliance

NA = not applicable

See Discussion = see discussions in the section of Review of Data Reports

Lab = responsible by the Laboratory