



DEPARTMENT OF
NATURAL RESOURCES

MAY 01 2017

COMMISSIONER'S OFFICE
ANCHORAGE

May 1, 2017

Andrew Mack, Commissioner
Alaska Department of Natural Resources
550 West 7th Avenue, Suite 1400
Anchorage, AK 99501

Re: Request for Reconsideration—MLUP No. 6118 (APMA No. A20176118)

Dear Commissioner Mack:

On October 13, 2016, the Pebble Limited Partnership (PLP) submitted an Application for Permits to Mine in Alaska (APMA) to DNR for continued care and maintenance activities at the Pebble Project near Iliamna, AK. APMA No. A20176118 and PLP's 2017 Work Plan (submitted March 24, 2017) detail the extent of activity authorized by Miscellaneous Land Use Permit (MLUP) No. 6118. DNR issued a notice of decision renewing MLUP No. 6118 on April 11, 2017.

During your recent telephone conversation with John Shively prior to the issuance of the permit, you indicated that there were two different approaches to request reconsideration. The first was to request a formal hearing while the second was to request a less formal meeting that allows for a more conversational dialogue. Following the second option, PLP formally requests reconsideration of certain terms and conditions of the April 11, 2017 decision as allowed under 11 AAC 02. We are available to meet with you as your schedule permits.

PLP requests reconsideration of the three items discussed below. These modifications would incorporate necessary flexibility while clarifying the controlling language of MLUP No. 6118 for compliance purposes.

1. Completion Schedule

In Appendix 4, Permanent Borehole Closure and Reclamation, the permit states

By December 31, 2017, permanently close and reclaim 138 boreholes identified within the 2017 Borehole Retention Schedule for abandonment and full reclamation.

PLP submitted APMA #A20176118 on October 13, 2016 for a two-year term (2017-2018). Accordingly, the work plan submitted in March 2017 was prepared assuming two work seasons, rather than the single season under the MLUP. PLP's stated objective is to fully address the identified sites during the 2017 field season. However, experience has shown that factors beyond PLP's control such as weather, safety concerns and other on-the-ground considerations may delay our intended schedule. Likewise, site conditions may require additional consultation with DNR and other agencies before proceeding with full abandonment.

PLP maintains regular and frequent communication with DNR throughout the year, with enhanced outreach during each field season. We routinely apprise staff of any changes or delays that are common to any Alaska field program. Over the past several years, we have voluntarily implemented additional inspection and reporting measures to provide DNR with the information necessary to address public concerns. PLP intends to continue this approach for the 2017 field season.

PLP cannot be in a position where it is out of compliance with the explicit terms of the permit for reasons beyond its control. Accordingly, we request that DNR modify this stipulation to allow for unforeseen circumstances beyond PLP's control, subject to consultation with, and approval from, DNR staff.

The following statement inserted into Appendix 4 would address PLP's concern: "The 2017 Borehole Retention Schedule may be modified by DNR if PLP demonstrates that compliance with the approved schedule cannot be fully implemented due to circumstances reasonably beyond control of PLP." This degree of flexibility – common in many permits – would enable DNR staff to continue to evaluate PLP's compliance and authorize modifications as necessary based on their experience, expertise and in-field observations.

2. Closure Information

In Appendix 4, Permanent Borehole Closure and Reclamation, the permit states, in part,

For each [of the 138] boreholes, report the following to DNR:

8. Geologic log of well, limited to consolidation of formation and static water level if known

9. Abandonment design, including the following:

- a. Depths of sealing or fill material*
- b. Sealing or fill material description*
- c. Mixing ratio of sealant of [sic] fill material*
- d. Volume of material used*

The 2017 MLUP is the first instance where the information listed above has been required of PLP or its predecessors at the site. All 138 inactive boreholes identified for full abandonment were grouted and plugged during previous field seasons, with some work dating back as far as 1992. PLP has information on the plugging and abandonment status for each of these holes, however not to the level of detail requested by DNR. PLP cannot be required to produce information it does not possess and which was not a requirement of the past MLUPs.

Accordingly, we request that DNR revise these stipulations to clarify that PLP is not obligated to provide information that it does not possess. Our concern could be addressed through a slight revision to the language of Appendix 4 as follows: "For each [of the 138] boreholes, report the following to DNR (to the extent that PLP has maintained records of these activities)."

3. Appendix 5—Response to Public Comments

On page 7, the permit states, in part,

Attached to and included as material terms of the Permit are five appendices as referenced herein and described below:

- 5. Appendix 5 – the comprehensive review and response to material submitted to the Department in the course of public process conducted by the Department.*

Appendix 5 contains more than 50 pages of responses to public comments on a range of issues related to the application. In our experience, we have never seen a permit where the agency characterizes its response to comments (RTC) as "terms of the Permit." The RTC is part of the permit record and serves the important purpose of explaining the agency's decision. However, the

RTC was not drafted in the typical language of permit terms, and it would be difficult for us to reduce it to concrete action items relevant to compliance.

PLP also notes that the RTC makes numerous references to a report by Center for Science in Public Participation (CSP2) and funded by United Tribes of Bristol Bay (UTBB)¹. The CSP2 report claims to document evidence of environmental impact, but its authors repeatedly ignore accepted methods and practices that are a hallmark of a rigorous scientific investigation. PLP believes these deficiencies are significant and undermine the conclusions reached by CSP2 and UTBB. For your review, I have enclosed an assessment of the CSP2 report prepared by Argon, Inc. This information was not available during the comment period as the report was completed in April, and, thus, should be considered new information.

Accordingly, PLP requests that DNR clarify that Appendix 5 is not an actual “permit term.” If there are key items in Appendix 5 that should be incorporated into the permit as terms, then we believe those terms should be moved into the body of the permit. If DNR is unwilling to make these changes, we believe it is imperative that the permit have language to the effect that the permit is controlling if there is any discrepancy between the RTC and the permit terms (MLUP #6118 Sections 1 through 17, Special Stipulations A through K, and Appendices 1 through 4).

In closing, we appreciate your consideration of PLP’s request and would appreciate the opportunity to have a short meeting with you and your staff to clarify the permit terms to ensure PLP is in a position to maximize success on the ground this year. The modest changes to the permit we are requesting will not trigger the need for additional public comment under relevant regulations or the terms of the *Nunamta* litigation. The changes we are seeking are a logical outgrowth of PLP’s permit application, and the reconsideration is a continuation of the administrative process (which is complete when there is a final decision subject to appeal in Superior Court).

Lastly, we understand under relevant DNR regulations that a request for reconsideration and an informal meeting will not result in a stay, but we want to reiterate that PLP is not, through this request for reconsideration, seeking to effect a stay of the permit. The MLUP should remain in effect as contemplated under Section 17.

We appreciate your attention to the Pebble Project and look forward to meeting with you to discuss these items further. Please contact me at your convenience to schedule a meeting. I can be reached by email (timhavey@pebblepartnership.com), telephone (907.339.2626), or by regular mail at the address below. Any legal questions may be directed to Eric Fjelstad at 907.263.6973 or EFjelstad@perkinscoie.com.

Sincerely,



Tim Havey, Environmental Manager
Pebble Limited Partnership
3201 C Street, Suite 404
Anchorage, AK 99503

¹ Zamzow, K and D Chambers. 2016. Investigation of Reclaimed Drill Sites, Pebble Prospect, 2016. Kendra Zamzow, Ph.D., and David M Chambers, Ph.D., P. Geop., Center for Science in Public Participation, Bozeman, MT, for the United Tribes of Bristol Bay, Dillingham, AK, October, 2016.



Attachments: A Review of CSP2 Investigation of Reclaimed Drill Sites Pebble Prospect, 2016.
Argon, Inc. April 5, 2017.

Cc: John Shively, PLP
Tom Collier, PLP
Eric Fjelstad, Perkins Coie

A Review of CSP2 Investigation of Reclaimed Drill Sites Pebble Prospect, 2016

by
Jane Whitsett
Argon, Inc.
April 5, 2017

Introduction

In August 2016, a Center for Science in Public Participation (CSP2) team visited the Pebble Deposit and inspected 107 sites, collecting samples at several locations. CSP2 presented their work in a report entitled "Investigation of Reclaimed Drill Sites, Pebble Prospect, 2016," Kendra Zamzow, Ph.D. and David M. Chambers, Ph.D, P. Geop., Center for Science in Public Participation, Bozeman, MT, for United Tribes of Bristol Bay, Dillingham, AK, October, 2016.

This review consists of three parts. The summary provides overall findings. Section 1 focuses on the report's methods with respect to QA/QC procedures, site selection and data validation. Section 2 assesses reporting errors, interpretation of results, appropriateness of data comparisons and overall rigor of the report and its conclusions.

Summary

Argon, Inc. conducted this review to assess the integrity of the study design, results and conclusions presented in the CSP2 report. As discussed below, inadequate documentation, improper data comparisons, and discrepancies between reported results and conclusions make it difficult to accept the CSP2 report as presented. Any conclusions or assertions made by the authors must be viewed within that context.

Study Design and Documentation

Rigorous environmental field studies involve careful determination of study objectives that are then planned in sufficient detail to meet the stated objectives. The Environmental Protection Agency (EPA) publishes several guidance documents specific to planning and executing a scientific study followed by validation of results to ensure that any conclusions are rigorous and defensible (See References).

The CSP2 team did not adhere to EPA guidelines in general. There are no references to standard documents such as a field sampling plan or quality assurance plan. Even though there were at least ten laboratory methods performed, the CSP2 report only cited two methods (EPA 200.8 and EPA 3050); there are no other references to EPA protocols in the report. In contrast, the multi-year Environmental Baseline Document (EBD) published by the Pebble Limited Partnership (PLP) comprises data collected and validated according to rigorous standards and practices that are clearly documented in the public record.

Other omissions from the CSP2 report include:

- Field equipment decontamination procedures
- Sample preservation methods
- Use of pre-cleaned sample containers
- Chain of custody
- Unique sample identification
- Sample holding times

The CSP2 report's lack of adherence to EPA requirements is such that the reported results and ensuing conclusions are questionable at best. See Section 1.A QA/QC Procedures for more detail.

The selection of site and sample locations is also driven by EPA procedures to obtain data that are representative of the environment. There is no mention of any rationale in the "Site selection" discussion toward collection of data that characterizes the environment in a statistically meaningful way. See Section 1. B. Site Selection and Sample Locations.

Prior to reporting results, data are normally reviewed for any sample collection or laboratory issues that may cause the data to be rejected altogether or qualified as estimated results. This review is completely missing from the CSP2 report. Hence, it is impossible to ascertain the validity of any of the results and associated conclusions. See Section 1.C. Data Validation for more discussion.

Data Comparison and Interpretation

The CSP2 report attempts to make numerous comparisons between their data collected in 2016 and data collected several years ago by PLP or USGS. Normally, this is a straightforward approach as long as the field and laboratory procedures utilized by each entity are comparable. However, since the CSP2 report includes very little of this required information, it is not possible to accept the comparisons as valid. Even within the CSP2 report the pH in soil data are not comparable due to the very different (and unexplained) procedures used in the field and laboratory. See Section 2.A Appropriateness of Data Comparisons for details.

The CSP2 report's lack of documentation makes it impossible to determine what samples were submitted for which analyses. According to the report, samples were analyzed for total organic carbon, fluoride, chloride and nitrate but there are no results for these analytes anywhere in the report. Only nine metals were reported among several tables whereas there are 25 metals in the list of analyses. Investigators must report all valid results and provide explanation for results that are not valid. The CSP2 report is incomplete in this regard. This in turn further renders interpretation of results, data comparisons and conclusions suspect. See Section 2.B. Interpretation of Results for more details.

When comparing data, CSP2 occasionally identifies some values in bold to indicate an order of magnitude difference between CSP2 results and PLP or USGS results. As noted above, such comparisons are not considered valid. Furthermore, this is an arbitrary and unexplained approach to presenting the significance of the CSP2 results, one which CSP2 inconsistently applies throughout the report. These tables should be reviewed with caution so as not to be influenced by incorrect or misleading information. There are numerous examples of a lack of accuracy under Section 2. C. Overall Scientific Rigor and Conclusions.

Section 1

Scientific studies must be designed and executed in a manner that allows the work to be fully assessed by other scientists. This is a hallmark of any scientific study that seeks to add scientifically sound and defensible results to a body of knowledge. For environmental studies, quality assurance (QA) and quality control (QC) procedures and requirements are an integral part of the field and laboratory methods. The QA/QC process establishes data quality objectives and the quality of individual sample results. Application of QA/QC procedures in the field and laboratory allows investigators and data users to confirm the accuracy and precision of results and in turn evaluate the validity of conclusions.

A. QA/QC Procedures

For environmental studies, it is standard practice to prepare a project-specific field sampling plan (FSP) and a quality assurance project plan (QAPP) in accordance with EPA guidance for environmental data collection (EPA, 2002). This ensures that results are defensible and of known quality. The QAPP specifically addresses quantitative and qualitative measures including precision, accuracy, representativeness, comparability, completeness and sensitivity (EPA, November 2002). These are defined in Appendix A of this review.

At a minimum, the following information is specified in the FSP and QAPP:

- Study objectives
- Field sampling procedures
- Sample handling including sample containers, preservation and holding time requirements
- Laboratory analysis including methods, accuracy, precision and sensitivity (detection limits) criteria and reporting requirements
- Data quality objectives and criteria
- Data validation and quality assurance reports

It is standard practice to include the FSP and QAPP as appendices in environmental reports. These documents are not included in the CSP2 report. In fact, there are no stated study objectives in the CSP2 report. Without this basic information, there is no way to design a study with the needed rigor to develop valid conclusions. At a minimum, the report should define study objectives as well as how, when and where the work will be conducted.

Details on how samples are preserved in the field, shipped to laboratories, analysis methods, detection limits, dates of collection and holding times are largely absent in the CSP2 report. There is no mention of using a chain of custody (COC) form to document sample identification, preservation, date and time collected, test methods required and individuals in custody of the samples.

Field QA/QC Practices and Samples

Field QA/QC practices and samples are necessary to address potential cross contamination of samples, representativeness, sample chain of custody and precision of results. For example, sampling equipment is washed with detergent followed by a deionized water rinse between collection of each field sample. The deionized rinse water is collected in a pre-cleaned sample container and is submitted to the laboratory as the equipment blank. The most important reason to collect equipment blanks is to assess the presence of metals in soil and water samples due to cross contamination.

The CSP2 report mentions equipment blanks under “Sample collection for laboratory analysis” (see page 9). However, equipment blank results are not included in the CSP2 report. Metals for environmental soil and water samples are reported as low as parts per billion when analyzed by an inductively coupled plasma – mass spectrometer (ICP-MS) using EPA 200.8. Hence, there is considerable potential for cross contamination during sampling due to the very low detection limits. Since equipment blank results are not reported it is impossible to determine if reported results are biased high or false positives for metals.

Proper sample collection, preservation and handling procedures ensure that each sample represents the environment (representativeness) and is not tampered with while being shipped to the laboratory. For example, water samples for some analysis require preservation by chilling the samples to 4° C in coolers for shipment to the laboratory. There is no evidence in the CSP2 report that samples were collected in laboratory provided pre-cleaned sample containers and shipped at 4° C. Further, COC procedures are required to document sample names, dates collected, names of sample collectors, sample preservation and tests required for each sample. These procedures do not appear to have been completed for the CSP2 study.

The CSP2 report indicates that field trip blanks, equipment blanks and duplicates were collected. However, there is no discussion of how field equipment was decontaminated between samples or how equipment rinsates were collected. Neither trip blank nor equipment blank results are provided in the CSP2 report.

Field duplicates were collected for soil and water samples only. There were no field duplicates collected for sediment or vegetation samples. Unfortunately, field duplicate results are not clearly identified in the report. For these reasons, it is not possible to assess field precision of the reported results.

There is no description in the report of how vegetation samples were collected in the field. Further, there are no results for vegetation samples in the CSP2 report with the exception of Figure 12. Assessing vegetation results cannot be done due to the lack of information.

Throughout the CSP2 report, results are presented in terms of sites using names such as DDH 7382 for a PLP drill hole as defined by PLP. The absence of unique sample names makes it difficult to follow the results and conclusions of the study. Under “field methods” the report should provide a table listing all samples collected with the following information:

- Unique sample ID
- Sample location
- Date collected
- Sample preservation and
- Tests performed by each laboratory

This provides clear, complete sample information and supports the reports discussion and conclusions. As presented, it is very difficult to assess the CSP2’s reports’ completeness in terms of samples collected and tests performed. For example, the report lists three vegetation samples submitted for analysis of multiple parameters (pg. 18 under “Laboratory results and comparison to background”). However, there is no information on vegetation sample collection methods or results. This speaks to the need for unique sample names and complete chain of custody documents to facilitate correct tracking of samples from collection through analysis and reports.

Holding Times

Sample holding time is the maximum time a sample can be stored before analysis to be considered valid. Holding times are specific to the tests being conducted. For example, pH in water is an unstable parameter, therefore EPA requires that field samples be analyzed immediately. In this study pH is measured to indicate acidity and is a key parameter in the

report. The acidity information (pH data) in this report is being used to support claims of “elevated” or “contamination” at the Pebble Prospect.

The first paragraph of the Executive Summary states:

“Sulfidic drill holes that are not properly reclaimed could go acid, and potentially allow groundwater to carry metals to the surface.”

Hence, pH results must be carefully reviewed for accuracy, precision, comparability and representativeness of the environment to assess the validity of pH results for developing conclusions about acidic conditions and “elevated” concentrations of metals. Since pH must be analyzed immediately it is highly unlikely that the ASET laboratory (located in Anchorage) pH results are valid. The time required to transport the samples to the laboratory is several hours at a minimum. The absence of sample collection and analysis dates and times makes it impossible to determine the length of time between sample collection and analysis for all reported results. Laboratory pH results for water samples are especially suspect since the sample reacts quickly with the atmosphere making it more acidic than what naturally occurs at the site. pH in water results should not be deemed usable without full review of sample collection and analysis dates and times. Field pH in water results, assuming they are valid, should be the only pH data used for developing conclusions in this report. With more information on the laboratory procedure for pH in soil analysis this data may be valid (See Section 2, Example 2 for details).

B. Site Selection and Sample Locations

Site selection and sample locations are driven by project objectives. The lack of clearly stated objectives and rationale for selection of sites and samples to meet these objectives is a basic flaw in the design of the CSP2 study to investigate reclaimed drill sites at the Pebble Prospect.

For site characterization, the process of site selection and sample locations are normally designed to provide a statistically meaningful list of sites and sample locations (EPA, December 2002). Unfortunately, the actual selection of sites is such that there is no statistical process carried out to obtain a known degree of confidence in characterization of a particular area. Within some sites there are multiple samples collected with no unique sample identification or location information.

Over 300 sites were identified and assigned a priority of 1 - 5. However, context for categorization was only presented for priority sites #1 and #2. Unfortunately, the “Site selection” (page 8) discussion cannot be correlated with field observations, field data and or laboratory data. Further, the discussion of field and laboratory results do not indicate which of the five priorities the sites are in. Consequently, the sites in Priority #1 and #2 (presumably the highest priorities) cannot be discerned in the text or tables.

C. Data Validation

There is no indication of any data validation having been conducted on the CSP2 results. The data validation report is normally a standalone section to the main report. Since this was not provided it is not possible to evaluate the quality of the data. The discussion below addresses this in more detail followed by examples of basic flaws that render the investigation of reclaimed drill sites inadequate and inconclusive.

A primary goal of data validation is to ensure that environmental sampling programs and decisions are supported by data of the type and quality needed and expected for their intended use (EPA 2000). Data validation is a systematic process for evaluating a data set's performance and compliance when compared to a set of standards to ascertain its completeness, correctness, and consistency using the methods and criteria defined in the project documentation. To do so properly requires a set of activities conducted during the planning, implementation, and reporting phases of an environmental data collection project.

As a result of this review, data are qualified to inform data users of any results that may be deemed an estimated result, biased result or a rejected result. This review is completed before preparation of a report to ensure data quality is acceptable and defensible.

A key piece of information to begin data validation is a complete record of all samples collected with location, sample ID, sample collection dates, analysis dates and analysis conducted on each sample. This information does not exist in the CSP2 report. Table A, below, lists examples where there are multiple field samples for a given site. This table illustrates the difficulty in tracking actual samples for any given site in the report.

The Sample ID and Sample Date columns are included to emphasize the need for unique sample IDs and dates in an environmental study. It is evident that several sites have multiple samples collected from them. The fact that there are no unique sample IDs associated with the sites makes it very difficult to have confidence that the results across the seven data tables in the CSP2 report correctly reflect individual, unique samples. Likewise, the absence of sampling and analysis dates makes it impossible to assess adherence to EPA holding times.

The meaning of #1 and #2 for Location DDH 3129 (CSP2 Figure 12) is not explained in the report. Possibly these are unique samples but this is not clear from the report. The same applies to the terms "area 1" and "area 2" for DDH 6355. Field duplicate results are not clearly identified anywhere in the report.

There is mention of three vegetation samples collected (See page 18). However, there is no mention under Methods in the CSP2 report as to field sampling method or laboratory analyses for vegetation samples. How were these samples collected in the field, preserved and analyzed by what laboratory? This is a significant omission for these samples.

Interestingly, total organic carbon (TOC) is listed as a test for water samples under "Sample collection for laboratory analysis" yet there are no TOC results in the report. Also, there are 25 metals and metalloids analyzed for all samples. Yet each table only lists results for as few as three metals. While it is common to report limited results in various tables for the purposes of discussion it is not acceptable to conduct analyses that are not presented anywhere in the report. These tables can be very lengthy and are often included as an appendix to the report. There is no such tabulation of all results in the CSP2 report. These are notable omissions that make data validation impossible. Consequently, the data and conclusions are suspect and in some cases (vegetation) unusable in the CSP2 report.

Table A Summary of Sample Information in CSP2 Report

Site Name	Sample ID	Date Collected	Date Analyzed	Matrix	Analysis
DDH 3129	NA	NA	NA	Soil (drill cuttings)	
DDH 3129 #1	NA	NA	NA	Soil	?
DDH 3129 #2	NA	NA	NA	Soil	?
DDH 6355 area 1	NA	NA	NA	Soil (drill waste discharge)	?
DDH 6355 area 2	NA	NA	NA	Soil (drill waste discharge)	?
DDH 6348/GH08-111 area	NA	NA	NA	Soil	?
	NA	NA	NA	Vegetation	?
DDH 7382	NA	NA	NA	Water	?
	NA	NA	NA	Soil	?
	NA	NA	NA	Sediment	?
	NA	NA	NA	Grout	?
	NA	NA	NA	Gravel	?
DDH 4202	NA	NA	NA	Sediment	?
	NA	NA	NA	Water	?
DDH 5330	NA	NA	NA	Sediment	?
	NA	NA	NA	Water	?
DDH 7365	NA	NA	NA	Water (standpipe)	?
	NA	NA	NA	Water (pond)	?
DDH 11540	NA	NA	NA	Sump Material	?
	NA	NA	NA	Water	?

Key:

NA – not available

Section 2

Section 2 is an assessment of appropriateness of data comparisons, interpretation of results, and overall rigor of the report, errors and its conclusions.

A. Appropriateness of Data Comparisons

The CSP2 report makes several comparisons of the 2016 study results to PLP and USGS results. Several factors must be considered when making data comparisons. The samples must be collected in the same manner and analyzed using comparable laboratory methods. For spatial comparison of results, there must be a rationale presented for why the comparison is valid. There is not enough information on analytical methods employed for this investigation to determine acceptability of CSP2 data for comparison to PLP or USGS data. Below are examples from the CSP2 report where data comparisons are made without the necessary supporting information to validate comparison with results from previous studies.

Example 1: Soil Trace Elements Data Comparison

In Table 8-Soil Chemistry, the CSP2 authors include analytical results for certain parameters taken from the Pebble EBD (Chapter 10, Trace Elements) to serve as ‘background’ for comparison to new data. CSP2 soil data in Table 8 were compared to “four PLP samples from the eastern edge of the ore body, two samples on the western zone and two USGS sites

between 50' and 800' from our samples sites" (See page 21.). Hence, the range presented in Table 8 for PLP is actually just six soil samples. Further, if one is to select samples for comparison there must be a clearly stated rationale for why the samples were selected and that comparison to them is valid. At a minimum, the Pebble EBD sample names for selected samples should be listed in the report.

Some samples in Table 8 are not actually "soil" samples. For example, drill cuttings are samples above vegetation which are not samples of soil. Drill cuttings are from depths far below the 1 - 6" depths for the CSP2 "soil" samples in Table 8. Consequently, the results are not comparable to PLP baseline soil samples which were collected from depths up to 6". Further, soils in the deposit area often contain peat which are naturally more acidic than soils without peat. The CSP2 report does not support the comparison of their "soil" sample results to six selected PLP soil sample results with any scientific rigor.

Example 2: Soil and Sediment pH Data Comparison

Given the significance of acidity in soil and water in CSP2's investigation, pH should be rigorously measured and analyzed in the field or laboratory. However, it is not possible to use the CSP2 laboratory water pH or field soil pH data for the reasons cited below and in Section 1 A. Holding Times.

There are various procedures for measuring soil pH. Some of the details that vary from one laboratory to the next are: soil:solution ratio, use of a salt solution rather than water, method of mixing, time of standing before reading, etc. In addition, there are many factors that affect soil pH as measured in the field or laboratory. Generally the pH of most soils increases with dilution in water, and becomes constant at a soil:water ratio of about 1:5. Therefore, when reporting soil pH, it is essential to include a detailed description of the procedure followed.

Under "Field methods" for soil pH, 2-3 samples were homogenized and mixed in a glass beaker with a small amount of distilled water; the mixture was allowed to sit for several minutes until the pH stabilized (See page 9). Under "Sample collection for laboratory analysis" pH was determined from 1:5 water extracts (See page 9). Hence, the laboratory procedure gives a far more dilute sample than the field method. (Note: The use of "distilled" water is highly unlikely as environmental laboratories use "deionized" water not distilled water.) This is all the information that is provided for analysis of pH in soil and sediment.

Based on the variation in field vs. laboratory pH in Table B below and the differences in the procedures it is inappropriate to compare the two sets of results. The bold results and text are the most dramatic examples of poor reproducibility between the field and laboratory data. These data should not be reported without a clear explanation for the very large differences in the results. The lack of rigor and attention to data quality renders all of the results suspect.

Table B. Summary of Field and Laboratory pH in Soil and Sediment from CSP2

Site Name	Soil Field pH	Soil Lab pH	Comments
DDH 7392	2.60	3.0	
DDH 3129	2.78	3.2	
DDH 6355 #2	2.90	6.6	Field acid concentration >10,000 laboratory
GH05-60	3.00	3.5	
6348/GH08-111 area	3.67	4.3	
DDH 5324	4.10	6.2	Field acid concentration >100 laboratory
DDH 12557	4.87	5.1	
DDH 9462	5.14	None	
DDH 6355 #3	5.30	None	
GH05-60	5.70	None	
DDH 5326	6.40	None	
DDH 4171	7.09	5.7	
DDH 6355 #1	7.30	2.7	Field acid concentration <10,000 laboratory
	Sediment Field pH	Sediment Lab pH	
Unknown Artesian	4.94	None	
DDH 11540	4.96	None	
DDH 4202	6.28	5.0	
DDH 5330	7.16	6.2	
DDH 7382 (Gravel)	6.50	9.5	Field acid concentration >1,000 laboratory
DDH 7382 (Artesian sediment)		6.3	

Example 3: Misleading Data Comparisons for Petroleum Hydrocarbons in Soil

The soil data summarized in the Pebble EBD (Chapter 10) is based on data collected over a 400-square mile area from more than 100 individual locations. The CSP2 authors fail to explain why aggregated data is appropriate for use as “baseline” for individual data points. In addition, there is naturally occurring organic matter in soils that have the same chemical signature as petroleum hydrocarbons. Of greater concern is the authors’ willingness to shift how that baseline data is presented in furtherance of their overall argument.

Table 8 in the CSP2 report cites EBD data as a *range* of values that are in fact just six soil samples, which is inconsistent with Table 6, 7 and 9. For example, in Table 9 (DRO/RRO), the authors shift to citing the EBD data as the *mean* of values. Why? Consider how the soil sample collected by CSP2 looks in comparison to the mean (using the authors’ arbitrary ‘order of magnitude’ standard, in bold):

From CSP2, Table 9:

Sample ID	Matrix	Units	DRO	RRO
PLP mean	Soil	mg/kg	209	2,028
DDH 7359 (CSP2)	Soil	mg/kg	3470	12,800

and then versus the range:

CSP2, Table 9 with ranges from PLP EBD:

Sample ID	Matrix	Units	DRO	RRO
PLP range	Soil	mg/kg	11.7 - 1300	32.7 – 12,300
DDH 7359 (CSP2)	Soil	mg/kg	3470	12,800

With the ranges included, neither value looks implausible because they fail the ‘order of magnitude’ test. But by using the mean, the authors create a false impression of greater impact despite there being none.

B. Interpretation of Results

A report’s credibility rests on the ability of others to evaluate the underlying data and confirm or reject any of its conclusions. All data should be presented, at least in summary. The author must also accurately cite or quote information from other studies, particularly when those other reports are meant to serve as a “baseline” for comparison. Furthermore, when comparing results from different studies, the author is obligated to note how data may differ and whether it is suitable for a direct comparison.

The CSP2 report, however, does not provide the necessary contextual information for the data comparisons. In addition, data from Pebble’s EBD are quoted inconsistently and used to make the false claim of “contamination” resulting from exploration activities.

Scientific rigor and QA/QC practices are intended to remove as much bias as possible from results and conclusions. As these are missing from the CSP2 report, its credibility is greatly reduced and its conclusions appear to be biased. By the way data are presented to the reader, the authors have created false ‘standards’ of harm that they themselves apply inconsistently throughout the report. To any objective reader, it is clear that the CSP2 report is written to support conclusions the authors reached prior to this study.

Example 4: Arbitrary and Inconsistent Standards of Impact

The CSP2 authors adopt several arbitrary standards by which they attempt to dramatize differences in sample results and thus create a false impression of harm. It is worth noting that these standards are not used for regulatory compliance and are only marginally appropriate for definitive use in a scientific study. Even when correctly applied, their value is anecdotal at best.

A somewhat plausible standard used by the authors is the ‘order of magnitude’ test. The most common order of magnitude test is base 10, although the authors do not explicitly define what it means. They often apply it incorrectly, thus enabling them to highlight numbers and falsely imply something meaningful. The use of this test raises further questions about whether the

authors deliberately cited incorrect background data (See Examples 2 and 3) to further dramatize harm that doesn't exist.

It is all the more curious to use 'elevated' as a standard of comparison when the authors have collected only one sample rather than the thousands of samples collected under the EBD. Only a thorough, longitudinal analysis like the EBD can establish rigorous baseline trends that can be used for comparison.

Consider Table 10 in the CSP2 report. In the discussion, the authors state that "bold indicates concentrations an order of magnitude above nearby sites or prior measurements" yet the table shows bold values that clearly fail any reasonable order of magnitude test. The bold copper and molybdenum results are not an order of magnitude above 'background', while the specific conductance value is bold *even though it is compared to nothing*.

From CSP2, Table 10:

		Field & Lab pH	Field & lab SC μ S/cm	Al mg/L	As μ g/L	Cu μ g/L	Fe mg/L	Mn μ g/L	Mo μ g/L	Zn μ g/L
Sediment and sump material										
2016	Sump material	6.3	1,150	10,000	4	475	20,000	233	38	33
2008	PB159 sed.	--	--	86,000	19	218	56,000	616	9	102
2008	PB161 sed.	--	--	82,000	11	109	45,000	978	5	91

Example 5: Unclear Comparisons to PLP and USGS Results

According to CSP2 bold values in Table 7 are an order of magnitude above background. This criteria does not seem to be true for Cu. As shown in the table below three sites have bolded Cu results indicating they are ten times greater than background. However, it is not clear what data is deemed "background" since concentration ranges are given for both PLP, mine area and PLP, nearby sites.

Site Name	Cu, mg/kg
PLP, nearby sites	9-20
PLP, mine area	1-200
DDH 5330 sediment, 2016	621
DDH 7380/7386, artesian, sed., 2016	599
DDH 7382, artesian sed., 2016	186

C. Overall Scientific Rigor and Conclusions

The CSP2 report makes many incorrect allegations of "contamination" due to exploration activities by directly comparing the results of their own sampling effort with studies published separately by USGS in 2011 and PLP in 2012. A direct comparison can only be valid, however, when *all aspects* of the programs are actually comparable. CSP2 results cannot be simply taken as being directly comparable to previous studies. A closer inspection of the CSP2 program reveals significant flaws when compared to the stringent standards PLP adopted for the Environmental Baseline program. Nearly all of the CSP2 data would be rejected under PLP's rigorous protocols.

Example 6: Incomplete Documentation

The report notes in paragraph 3 under “field methods”:

“Field meters were calibrated each morning and checked each evening in pH buffers (pH 4.01, 7.00, 10.01) and conductivity solution (447 μS/cm). On August 4, the YSI 556 did not correctly measure pH buffers during the evening check; laboratory measurements were utilized for site data collected on that day.”

However, nowhere in the report text or tables are there any dates given for sample collection and field measurements such as pH or specific conductivity. Hence, one cannot discern which samples are the August 4 samples or where they were collected. Clearly, there is no way to verify that the investigators reported the correct pH values for samples collected for pH analysis on August 4th.

Example 7: Soil Sample Collection Methodology

The CSP2 report describes soil sample collection procedures as:

“Soil and sediment were collected from 2-3 locations within a few feet of each other and homogenized in a Ziploc bag; the sample was double-bagged for storage and transport.” (CSP2, p. 9)

The CSP2 report provides no other information about sample collection, sampling depth, preservation or chain of custody, all of which are essential for producing valid results. Analytical tests are very sensitive and can reflect contamination or spoiling from things like improperly cleaned equipment, technicians with dirty hands, improper sample containers, or failure to meet preservation methods and holding times. Table C compares how some of the basic sampling protocols are implemented in each program.

Table C. Comparison of Soil Sampling Programs

	CSP2	PLP Field Sampling Plan for Trace Elements (EBD 2012, Appendix F)
Program Guidelines	?	<i>Soil Sampling, US EPA SESDPROC-300; Field Sampling Guidance, ADEC</i>
Sample Containers	Ziploc bags	Laboratory-provided 4 or 8 oz. pre-cleaned glass jars with laboratory seals
Collection Instruments	?	Disposable polyethylene trowels or stainless steel trowels
Cross-contamination prevention	?	Technician uses new pair of nitrile gloves for each sample; samples are sealed and segregated at time of collection
Equipment Decontamination	?	Between samples, all reusable collection equipment is decontaminated with deionized water and detergent.
Sample Preservation	?	4° C using gel packs
Sample Hold Times	?	28 days (metals),immediately (pH in water), 7 days (DRO/RRO in water)
Sample Date and Time	?	Technician records exact time sample is collected to verify hold times
Analysis Methods	Incomplete	Analytical methods recorded on the chain of custody form & lab reports
Chain of Custody Procedures	?	COC documents all necessary information to ensure sample meets QA/QC standards, including technician name, exact location of sample, preservation methods, date and time of transfer to shipper, lab, etc.

In addition, all PLP data are subject to a rigorous QA/QC review before any result can be considered valid and certified for the final data set (see EBD 2012, Appendix G). The CSP2 report does not indicate that any QA/QC protocols were followed to scrutinize methods or results. As shown in Example 2, seemingly insignificant omissions build upon each other and culminate with CSP2 including highly suspect results in its final report without any effort to explain their validity.

Example 8: Incomplete and Obscure Information

The CSP2 authors claim that data collected near drill site DDH 11540 in 2011 and 2016 show “potential mobilization of Cu and Mo from the sump to the wetland” (CSP2, Table 10 and p. 26). As evidence, the authors cite results from their sampling effort as well as data from a 2011 CSP2 report (“Water Quality at Pebble Prospect Drill Rig #6”) that appear to show copper values in a wetland spring increasing between 2011 and 2016 (0.25 ug/L to 1.8 ug/L). The 2011 report also showed a nearby “wetland pool” with copper ranging from 71-137 ug/L while sump water registered 435 ug/L.

Although the authors do not clearly state it, the copper values shown above represent the *total recoverable* concentration rather than the *dissolved* fraction (Cu water quality criteria, and effects on aquatic life, are based on the dissolved fraction). In fact, the dissolved copper value from the sump water was 2.7 ug/L, which is completely in line with the 1.8 ug/L value observed in 2016. By omitting the dissolved data, the authors clearly intended to mislead the reader into assuming that high copper in the sump is somehow “mobilizing” and influencing water quality in the spring.

The following is a more transparent and scientifically defensible way to present the data, highlighting the more meaningful results:

From CSP2 Table 10, with additional data:

		Cu (total) µg/L	Cu (dissolved) µg/L	TSS mg/L
2016	Wetland spring	1.8	?	?
2011	Wetland spring	0.25	0.8	0.7-12
2011	Wetland pool	71-137	4.0-5.8	807-1510
2011	Sump water	435	2.7	2370

Example 9: Interpretation of Petroleum Hydrocarbons Analysis in Soil, Sediment and Water

The CSP2 report omits a considerable amount of data for the evaluation of petroleum hydrocarbons in soil, sediment and water. The text under “Petroleum” on page 24 of the CSP2 report cites the collection of soil or sediment from six sites, water from two sites and a sample of sump material for a total of nine samples analyzed for DRO and RRO. Soil duplicates were collected at one site and water duplicates were collected at one site. However, Table 9 only reports DRO/RRO for one soil (DDH 7359), and one sediment, water and grout sample from DDH 7382 for a total of four samples. Although the investigators mention samples were analyzed with and without silica gel cleanup they do not indicate which of these results is reported in

Table 9. To make any assertions regarding site contamination without reporting all results and supporting chromatograms is contrary to sound scientific procedures.

For a complete interpretation of results the report should include at a minimum DRO/RRO results for all samples before and after silica gel cleanup. This allows for evaluation of possible biogenic interference. The authors should also include chromatograms of samples alongside DRO/RRO calibration standards and petroleum biomarker chromatograms. This allows for comparison of the petroleum hydrocarbon standards fingerprint against the field samples, and provides a complete and much more rigorous assessment of petroleum hydrocarbon occurrence in soil, sediment and water than what is reported in the CSP2 report.

D. Miscellaneous

The CSP2 report makes numerous statements with little to no data to support the conclusions, including at least 100 examples of unsupported speculation. Selected examples (emphasis is Argon's) include:

- "some indication that a sump *may* be leaking metals" (p.4) yet their own sample results definitely show no "elevated" results (10 times > PB 159 and PB 161 sediments) for the sump results in Table 10 (p. 25).
- "acidic water *could* make its way to the surface" (p.5) despite later acknowledging "artesian water tended to be neutral pH" (p.10).
- "*may* be a risk of release of waters elevated in copper and other metals" (p.17). Statement is made in reference to a drill site that is properly fitted with a plug to stop artesian flow. Authors falsely imply water is elevated in copper, etc. without providing any data supporting the claim (they did not sample this site).
- "There is also a ring of dead vegetation several feet above the floor of the gully. *There is no obvious explanation for this, but it is possible* that several drill sites disposed of waste in the gully, affecting vegetation." (p. 23). There is no basis for this statement.
- "the soil at these sites was acidic and the flushing may not be historical, *it may be continuing* on a periodic basis." (p. 27). This "periodic flushing" has never been observed or documented at any Pebble exploration site.
- "copper concentrations indicate water is being impacted in at least localized areas, but the extent to which copper is moving into fish-bearing waters *is not known*." (p. 29). There is no evidence of any copper migration. See Example 7 above.

Following are examples of discrepancies or errors between results and conclusions:

- **CSP2 Report:** For DDH 4202 comparison to PB177 "Water quality was very similar, with the exception that sulfate and iron were elevated at DDH 4202 (Sulfate at 35 mg/L vs. 22 mg/L and iron at 2.7 mg/L vs. 0.6 mg/L)" (page 19).

Argon Response: Sulfate and iron in the water sample from DDH 4202 are far below the CSP2 criteria of an order of magnitude (10 times greater concentration). The conclusion that sulfate and iron are elevated in water from DDH 4202 is not supported by CSP2 results and criteria for "elevated".

- **CSP2 Report:** “Copper is in concentrations that would be toxic to aquatic life if the standpipe leaked” (page 19 last paragraph, last sentence).

Argon Response: According to EPA’s aquatic life ambient freshwater quality criteria for copper, copper criteria are customized to the particular water under consideration using the biotic – ligand model (BLM). This model includes inputs from temperature, pH, dissolved organic carbon, calcium, magnesium, sodium, potassium, sulfate, chloride and alkalinity (EPA, 2007). There is no indication of applying the BLM to this data and hence no basis for the stated conclusion that there are copper concentrations toxic to aquatic life.

- **CSP2 Report:** For DDH 9475, “...artesian water very high in sodium and sulfate, and elevated in some metals, was flowing downhill about 130 feet into vegetation” (page 20).

Argon Response: There are no results for “some metals” anywhere in the report. Only sodium is “elevated” in Table 6. Table 11 lists sodium as 195 mg/L and Table 6 lists sodium at 196 mg/L.

- **CSP2 Report:** For soil sample results in Table 8 “Site analytes fell within previous concentrations with the exception of one or more of the following elevated in 2016:sulfate” (page 21).

Argon Response: CSP2’s criteria for “elevated” is 10 times “background” concentration. For sulfate in soils the PLP range is 13 – 67 mg/L. *All* soil sulfate results are less than 670 mg/L (10 x 67 mg/L). Hence, the statement is not true for sulfate in soil.

- **CSP2 Report:** Table 8 soil samples DDH 3129, DDH 7392 and DDH 4171 have what appears to be a range of results.

Argon Response: There needs to be an explanation of the actual individual samples that these ranges represent. It is possible that these are listing field duplicate results not a range of results. If this is the case the duplicates for sulfate (DDH 3129) and copper (DDH3129, DDH 4171) show values that render the results questionable owing to the large difference (> 2 times difference) in the field duplicate results for these two analytes.

- **CSP2 Report:** Comparison to 2011 Investigation, “Concentrations of cadmium (not shown) ...were similar to 2011, but...aluminum...were higher than the 2011 spring sample and higher than ponds 715’ and 915’ upgradient” (page 25).

Argon Response: The ponds referred to here are the USGS samples PB159 and PB 161, both of which have no aluminum results listed in Table 10. Hence, comparison of 2011 spring sample is not supported in the report for aluminum. Further the sump material is listed as having elevated copper and molybdenum (page 26, first paragraph). In fact, the sump material copper (475 mg/kg) and molybdenum (38 mg/kg) are both well below ten times the concentrations for these metals in PB 159 and PB 161 sediments (Table 10).

- **CSP2 Report:** “Water surrounding the base of DDH 4202 casing was elevated in aluminum, iron, manganese and copper...” (page 29, last paragraph).

Argon Response: DDH 4202 water sample is reported in Table 12 with no results given for aluminum, iron or manganese for PLP pond water or DDH 4202. The copper concentration for DDH 4202 is 7 ug/L and is just above PLP pond water range of 1-3 ug/L. Hence copper is not “elevated” using the criteria of a ten-times higher than background concentration. The suggestion that “...DDH 4202 is leaking artesian water...” in the CSP2 report is not supported from the limited data (pH, copper, sulfate, molybdenum) listed in Table 12.

- The Discussion section (page 27) of the CSP2 report presents results of selected soil and sediment samples associated with drill cuttings and discharge. There are several aspects of this text and Figure 12 that are unclear and confusing. These are listed below:

CSP2 Report: “Soils at both types of sites were elevated in copper, molybdenum, sulfate and specific conductance”.

Argon Response:

- The sump sample DDH 11540 does not have sulfate reported anywhere else in the report but may have a concentration of 750 mg/L based on Figure 12.
- The only “exceedances” of sulfate appear in Table 8. Soil Chemistry where the bolded values are less than an order of magnitude below the PLP range which are not really exceedances.
- Specific conductance (SC) was analyzed in the field and the laboratory; which of these is reported in Figure 12? Specific conductance is not reported in Table 8 Soil Chemistry or Table 7 Sediment Chemistry. One can only obtain a general understanding of SC from Figure 12.

Conclusions

A scientific investigation’s credibility rests on several key factors: the rigor with which data are collected and analyzed; the thorough presentation of all valid data; and an objective interpretation of that data regardless of any biases the authors may have. While the United Tribes of Bristol Bay-funded report may be titled “Investigation of Reclaimed Drill Sites, Pebble Prospect, 2016” it fails to meet some of the most basic standards of scientific inquiry and thus undermines the definitive “conclusions” it claims to document.

In order to conclude there are impacts due to “contamination” data must go through a rigorous evaluation process. It is not enough to note “exceedances” of a range or mean as grounds for determining impacts. This is more appropriately done using a risk assessment approach to systematically determine if impacts indeed exist as well as their magnitude, duration and extent. The authors did not apply standard, scientifically acceptable risk/impact assessment methodologies, and this invalidates their speculative conclusions. For example: the standard for determining the existence of “problems” is loose and unsubstantiated in this report. More stringent criteria are typically utilized to better characterize whether “impacts” do exist, and if so, then their magnitude, duration, extent, and significance is systematically analyzed – none of which was done in this report.

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APPENDIX A

DEFINITION OF PRECISION, ACCURACY, REPRESENTATIVENESS, COMPARABILITY AND COMPLETENESS

Precision

Precision measures the reproducibility of repetitive measurements. It is a measure of agreement among independent measurements produced by applying the same process under similar conditions. Precision is evaluated by calculating the relative percent difference (RPD) between duplicate samples. The results incorporate variability from the analytical process, sample collection and handling, and matrix factors.

Precision is calculated as the RPD as follows:

$$\text{RPD} = (D1 - D2) / \{(D1 + D2) / 2\} \times 100$$

Where: RPD = Relative percent difference

D1 = First duplicate value

D2 = Second duplicate value

Accuracy

Accuracy measures correctness or the closeness between the true value and the quantity detected. As random error (variability due to imprecision) and systematic error (bias) are included, accuracy reflects the total error associated with a measurement. A measurement is accurate when the reported value is within a set percentage from the true value or known concentration of a spike or standard. Accuracy is expressed as percent recovery and will evaluate the impact of matrix interferences. Accuracy will be measured by calculating the percent recovery or percent drift of known levels of spiked compounds into an appropriate sample matrix as follows:

Percent recovery is calculated for laboratory control samples (LCS) and surrogate spikes as follows:

$$\%R = (Q_d / Q_a) \times 100$$

Where: %R = Percent recovery

Q_d = Quantity detected by analysis

Q_a = True or accepted reference quantity or value

Percent recovery for matrix spike (MS) and matrix spike duplicate (MSD) are calculated as follows:

$$\%R = ((Q_i - Q_c) / Q_t) \times 100$$

Where: %R = Percent recovery

Q_i = Quantity detected initially on spiked sample

Q_c = Quantity detected initially on unspiked sample

Q_t = True value of spike added

Accuracy goals will be presented in the project QAPP for each analytical group. If the accuracy goals for LCS are exceeded, associated project sample results reported above the detection limit will be qualified as high estimates. If a LCS fails recovery criteria with a low bias, associated project sample results will be qualified as low estimates.

If a MS sample has high recoveries, associated concentrations reported above the detection limit will be qualified in the parent sample (and field duplicate sample, if applicable) as having possible matrix affects. If MS samples fail recovery criteria with a low bias, associated sample results will be qualified in the parent sample as having possible matrix affects. Note that accuracy control limits do not apply for when parent sample concentrations are equal to or greater than the spiked amount in MS samples.

Representativeness

Representativeness expresses the degree to which data accurately and precisely represent a characteristic population or parameter variation at a sampling point. Representativeness is a qualitative parameter that depends on the proper design of the sampling program and proper laboratory protocol. Implementing standardized uniform field procedures for data collection and analysis will achieve adequate representativeness of data.

Comparability

Comparability is a qualitative measure of the confidence with which one data set can be compared to another. The number of matrices that will be sampled, the sampling and analytical variations implemented over the entire duration of sampling and analytical activities, and the range of field conditions encountered will be considered in ultimately determining comparability. Comparability will be achieved by using similar collection and analysis techniques, and reporting in conventional units.

Completeness

Completeness is measured as the amount of valid data obtained from a measurement system compared to the amount of valid data that was expected under correct normal conditions. The completeness goal of a project is stated in the project (site)-specific QAPP. It is calculated for each method and matrix for an assigned group of samples using the following formula:

$$C = \quad R/T \times 100$$

Where: C = Percent completeness (field and laboratory)

R = Total usable analytical data
points

T = Total data points possible

Sensitivity

Analytical sensitivity is the capability of a method or instrument to discriminate between measurement responses representing different concentrations of a variable of interest. It is important that all analytical results be reported with adequate sensitivity for project purposes. Laboratory analytical results are typically reported with a corresponding detection limit (DL), limit of detection (LOD), and limit of quantitation (LOQ). The DL is defined as the smallest analyte concentration that can be demonstrated to be different from analytical noise (background) at a 99% level of confidence. At the DL, the false positive (Type I error) rate is 1%. The LOD is the smallest analyte concentration that must be present in a sample in order to be detected at a 99% confidence level, meaning there must be a minimum probability of 99% of reporting a "detection" and a 1% chance of reporting a non-detect (a false negative, Type II error). The LOQ is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias; the LOQ is equal to or greater than the concentration of the lowest calibration standard. Based on the descriptions above, the following is true: $DL < LOD \leq LOQ$.

