

## Chapter 4 CHEMICAL QUALITY ASSURANCE REPORTS

4-1. Purpose. The purpose of the CQAR is to provide the data user with a timely review of chemical data quality. This is achieved through the inspection and analysis of QA samples, and through an examination of the corresponding project sample data. The exact format of the document is not as important as its content. The CQAR author should feel free to arrange the document in whatever format he/she is comfortable with as long as the essential information is conveyed in a succinct and timely fashion. The following format is suggested as a guide only. Whatever format is chosen should encompass at a minimum the same content that is specified below.

4-2. Cover Memorandum. The purpose of this attachment to the CQAR is to route the CQAR to its primary audience (the PM or TM). The standard memorandum format usually is adequate, which would identify the office symbol of the originating organization; the date of the transmittal; the facility name and project feature; major findings; and a point-of-contact (POC) and telephone number. The cover memorandum should be signed by the QA director whenever possible. Where local requirements for routing signed documents through the chain of command would delay delivery of the CQAR to the client, it is recommended that an unsigned advanced copy be sent to the client while the formal signed copy proceeds through channels. The cover memorandum should always refer the reader to the text for details (*i.e.*, to find out explicitly which sample results were affected), and should always advise the reader to have the district project chemist evaluate the data useability using the project DQOs.

4-3. Cover Page. The cover page should identify the title of the document, the report status (*i.e.*, draft, interim, final), its origin (*i.e.*, the name of the firm producing it), the project facility name (*i.e.*, Fort Green), project feature involved (*i.e.*, Lagoon Area), the date of preparation, and the name and signature of the responsible party.

4-4. Report Contents. The CQAR should contain the items listed below, although not necessarily in the format nor in the order presented. The format should present the information in an organized fashion which the reader can easily comprehend. The information below assumes that QA samples were collected and analyzed as part of the project QA effort.

a. Project Information. This section should contain any pertinent reference information to aid the reader in assessing the relevance of this report. This could include such things as funding documents (*i.e.*, MIPR Nos.), related report numbers and dates for the primary and QA laboratory data, sample receipt dates, and previous CQARs. The name, office symbol, and telephone number of a POC is also helpful.

b. Executive Summary.

(1) A summary description of the QA/QC effort expended on this data should be presented. Suggest citing the number, matrices, and types of samples tested (*i.e.*, 10 soils, 1 TB, 2 EBs, 1 QC soil, 1 QA soil), as well as the tests performed. Example statements might be, "Five soil samples were collected and analyzed in triplicate (project, QC, and QA); one for lead, one for mercury, and five for PCBs. A complete assessment of the data quality could not be made because there were no QC or QA samples collected for explosives and pesticides." The identities of the laboratories performing the various project tests should be cited. Any tables and/or attachments provided in the report which are not specifically referred to in later text should be referenced here (*i.e.*, all tables and attachments should be referenced somewhere in the text). Suggest referring to the Sample List, Analytical Methods List, and Data Comparison Tables here if this information is not provided elsewhere.

(2) The content and format of this section is mostly left up to the author, keeping in mind that the intent is to succinctly convey the overall results of the QA effort to the reader. Any major findings should be summarized here. State the possible effects upon the project sample data based upon: 1)a review of QA sample inspection results; 2)a comparison of QA sample data with project sample data; 3)a comparison of QC sample data with project sample data; 4)a review of primary and CMQAL QC data; and 5)a review of field QC data (*i.e.*, TB and EB results). Use the Data Evaluation Table in Chapter 3 for guidance in making this data review. State when a data review revealed no potential effects upon the project data. Also state when a complete data review could not be performed, *i.e.*, "A complete data review could not be performed because there were no QC or QA samples collected for pesticides." Potential effects on project data which might require immediate response or corrective action by the reader (*i.e.*, resampling) should be highlighted in some fashion (*i.e.*, bold print or underlined). Avoid, however, the use of strong adjectives to describe data with potential effects. The determination of data quality and usefulness lies solely with the district project chemist. The district project chemist is usually a district chemist, but may also be a HTRW-CX chemist or an CMQAL chemist when a district project chemist is not available. Do not use adjectives such as "invalid", "unacceptable", "suspicious", or "unreliable". The use of these or similar terms in the CQAR may be interpreted as contradictory by a regulator in the situation where a district project chemist determines that the data may be useful for project purposes, or may meet project DQOs in spite of weaknesses in laboratory or field QC measurements. For analogous reasons, avoid applying such terms as "valid", "acceptable", "reliable", *etc.* in describing data. The CQAR instead only should comment concerning the potential effects upon sensitivity (false negatives), precision (variability), accuracy (bias, false negatives, and false positives), representativeness, completeness (loss of data), and comparability (specified methods). Use statements such as, "The volatiles data may have an apparent negative bias because of improper preservation."; or, "The zinc values may contain false positives because of MB contamination."; or, "The explosives results were not corroborated by the method-required second column confirmation and may contain false positives."; or, "The low LCS recoveries for all semivolatile analytes may have caused some false negatives and probably a negative bias to detected analyte results. Any

positive semivolatile results should be considered as minimum values only."; or, "The disagreement between the field, QC, and QA sample results for metals may indicate sample inhomogeneity and a non-representative sample."; or, "The PCB results may be subject to false negatives because of elevated sample detection limits."; or, "The project data may not be legally defensible since the chains of custody were never signed in the field". Some indication of what portion of the data was affected should be given, *i.e.*, "EB contamination may indicate false positives and/or high bias in five of the nine sample results for mercury."

c. Sample List. List all QC, QA, and corresponding project samples with descriptive information including matrices, sample dates, field IDs, and laboratory IDs. A comprehensive list of all project samples is not required. Only those project samples which are part of a QC/QA/project sample set will be listed. This may not be necessary if there is only one set of QC/QA samples, or if there is relatively little data for each sample (*i.e.*, if the only analysis performed was for lead). However, where there is a large amount of data on multiple samples, a sample list is highly recommended to aid the reader in grasping what data is available to examine.

d. Analytical Methods List. This information can be presented in tabular form and at a minimum should specify the analytical method numbers and preferably (if known) the preparation method numbers as well. Note that this information may alternatively be provided in the data comparison tables.

e. Review of QA Sample Data. One of the purposes of this section is to assure the reader of the quality of the QA sample results, since the QA sample results will be the benchmark against which the project sample results will be judged. A second purpose is to evaluate the sample handling of the QA samples, since that has implications on how the project samples may have been handled.

(1) Review of QA Laboratory Quality Control Data. At a minimum, the following laboratory QC data should be reviewed: holding times, methods utilized, the results for MBs, LCS/LCSDs, MS/MSDs, matrix duplicates, and surrogates (see also Paragraph 4-4.g(1) below). This may be accomplished through tables summarizing laboratory QC data, or through descriptive statements such as, "The data package from XYD laboratory was complete with all required QC information. All MBs were free from contamination. All analyses were performed using specified methods within proper holding times. The majority of the duplicates, RPDs, laboratory control, surrogate, and MS recoveries were within laboratory control limits with the following exceptions..." Any excursions beyond laboratory control limits could then be listed. Since the QA data should be of high quality to begin with (implying that excursions should be few), it may be more efficient to just list the deviations from acceptable limits, rather than to tabulate all of the QC data in some kind of statistical format. The actual evaluation criteria could be the laboratory's own control limits, or could be set by the project DQOs. Project DQO evaluation

criteria sometimes may include USACE validation guidelines, or EPA national or regional functional guidelines, depending upon regulator requirements. See the Data Evaluation Table in Chapter 3 for general guidelines on evaluating data.

(2) Review of QA Sample Handling. Review of sample handling is performed at sample log-in and includes looking for correct sample containers, sampling procedures, sample preservation (*i.e.*, temperature, pH, *etc.*), packaging, labeling, and chain of custody procedures. Deficiencies noticed on QA samples at the QA laboratory imply that the project samples possessed similar deficiencies upon arrival at the primary laboratory. The QA laboratory should notify the district project chemist or TM of any serious deficiencies upon arrival. The project POC should be apprised of the implications of the deficiencies when notified, and asked for a decision on whether to proceed with the analyses. If the samples are analyzed in spite of the deficiencies, then the possible effects upon the QA and project sample data should be discussed in this section, highlighting any potential negative effects upon the data.

f. Data Comparison Tables. These tables compare the project, QC, and QA sample results in a matrix-type presentation. The header information should include project, sample, and analysis information, including facility name, project feature, sample date, field and laboratory ID numbers, sample description, method numbers, dates analyzed, dilution factors percent moisture, and concentration units. The primary and QA laboratories should be identified here as well. The body of the table should list any detected analytes, estimated detection limits (DLs) and quantitation limits (QLs) for detected analytes and a range of DLs and QLs for non-detected analytes from both the primary and QA laboratories; results from the project, QC, and QA samples, including the number of tentatively identified compounds (TICs) and the sum of the TIC concentrations; and an indication of agreement or disagreement in the data. A separate page detailing the agreement criteria and explaining any qualifiers used in the tables (*i.e.*, <, J, U, B, *etc.*) should be attached. Sensitivity (*i.e.*, DLs and RLs) should be evaluated only to verify that project-specific DQOs were satisfied. The agreement criteria shall be as shown in Table 4-1.

**Table 4-1**  
**Criteria for Comparing Field**  
**QC and QA Sample Data**  
(see text)

| <b>Matrix</b>  | <b>Parameter</b>                       | <b>Disagreement</b>                    | <b>Major Disagreement</b>               |
|----------------|--|--|---|
| All            | All                                    | >5x difference when one result is < DL | >10x difference when one result is < DL |
| All            | All                                    | >3x difference when one result is < RL | >5x difference when one result is < RL  |
| Water          | All except TPH                         | >2x difference                         | > 3x difference                         |
| Soil           | All except metals, VOCs, BTEX, and TPH | >4x difference                         | >5x difference                          |
| Soil           | Metals                                 | >2x difference                         | >3x difference                          |
| Water and Soil | TPH                                    | Arbitrary (suggest >3x difference)     | Arbitrary (suggest >5x difference)      |
| Soil           | VOCs and BTEX                          | Arbitrary (suggest >5x difference)     | Arbitrary (suggest >10x difference)     |

Reference: CRREL Special Report No. 96-9, "Comparison Criteria for Environmental Chemical Analyses of Split Samples Sent to Different Laboratories - Corps of Engineers Archived Data", Grant, C.G., Jenkins, T.F., and Mudambi, A.R., USACE Cold Regions & Environmental Research Laboratory, Hanover NH, May 1996.

The above criteria shall be applied when comparing field and QC sample pair data, as well as when comparing project and QA sample pair data. With the exceptions of volatile organic compounds (VOCs) in soil; and benzene, toluene, ethyl benzene, and xylenes (BTEX) in soil; and of total petroleum hydrocarbons (TPH) in either water or soil, the above criteria shall be used for all CQAR data comparisons. There is no definitive data for establishing comparison criteria for TPH (in water or soils) because of the wide variety of method modifications used by laboratories in the SW-846 8015M method ("M" is for "Modified"). The same is true for VOC and BTEX in soils because of the large error introduced during the conventional sample handling process. Result pairs are considered to disagree whether they are in the "Disagreement" or "Major Disagreement" category.

g. Review of Project Sample Data. This is the section the reader will refer to when seeking more details after reading the cover memorandum or the Executive Summary.

(1) Review of Primary Laboratory Quality Control Data. At a minimum, the laboratory QC data for the project and QC samples which correspond to the QA samples shall be reviewed. Some districts may arrange with the CMQAL to review the QC data for all of the project samples, although that is not required content for a CQAR. The laboratory QC data for project sample results should be examined in a manner similar to that used for the QA sample data (paragraph 4-4.e(1), above, and Table 3-1 in Chapter 3. Observed weaknesses in laboratory QC data may undermine the credibility of project sample data, even before comparison with the QA sample results. Missing QC data is always a deficiency, and will automatically injure data credibility by presenting the data in an unsupported manner. Samples prepared or analyzed outside of holding time may promote false negatives and give a negative bias to the associated data. Data sets without the required frequency of laboratory QC samples may have undefined data quality, although some explanation may be required. For example, sample results from a data set without a MB may be subject to false positives, but any samples in that same data set with undetectable levels of analyte would be unaffected (assuming LCS/LCSD recoveries were acceptable). Serious matrix effects may cause the data to fail project DQOs, making it unusable for project purposes. High RPDs in the project sample/matrix duplicate and MS/MSD pairs indicate inhomogeneity in the sample matrix, which would imply high variability (*i.e.*, low precision) in the project sample results. Some samples defy homogenization attempts; *i.e.*, sludges, clayey soils or sediments, multiphasic samples, and samples with macroscopic particles of analytes such as explosives and metals. High sample inhomogeneity can result in a determination that the samples were non-representative, making the associated analytical data unusable for project purposes. Determine if the primary laboratory possessed a current HTRW-CX validation when the analyses occurred, and if the project DQOs required that the project laboratories be validated. Data generated by an invalidated laboratory can adversely affect sensitivity, as well as all of the PARCCS parameters (precision, accuracy, representativeness, comparability, completeness, and sensitivity), making evaluation of its quality difficult. The above techniques also may be applied to QA sample data. Provide as much discussion as

necessary to fully explain the implications of out-of-control laboratory QC data upon the project sample results.

(2) Review of Field Quality Control Data. Any detectable analyte concentrations in the EB and/or TB should be commented on and their implications explained. There may be field notes provided separately or as part of the chains of custody which may yield clues concerning out-of-control QC data. Provide as much discussion as necessary to fully explain the implications of out-of-control field QC data upon the project sample results.

(3) Comparison with QA Sample Data. The availability of QA sample data provides more information for the data evaluator to further qualify the project sample data. QA sample data can reveal defective project sample data even when laboratory QC data are all in control. On the other hand, the confirming analysis of QA samples by an independent QA laboratory can provide evidence supporting the useability of project data that may otherwise have been questioned because of out-of-control laboratory QC data. QA sample data that does not agree with either the project sample or QC sample data should be discussed in detail in this section. When a data disagreement is observed, every attempt should be made to explain or to reconcile the disagreement. Verify at the outset that the data being compared all originated from splits (or co-located replicates in the case of volatiles) of the same sample. Do this by comparing sample descriptions, laboratory and field ID numbers, and the results from other analytes. Where feasible, both laboratories should be asked to check their results. Although there is the presumption that QA sample data in general is of higher quality, that may not always be the case. Where there is a disagreement involving the QA sample data, both data sets should be evaluated to ascertain if either has any weaknesses in its supporting laboratory QC data (*i.e.*, missing or out-of-control data). If the QA laboratory QC data is all present and in control, then the QA sample data is to be considered the "more correct", regardless of the status of the primary laboratory QC data. If the primary laboratory QC data is deficient, but the QA data agrees with the project sample results, then the QA data can be used to confirm the project data. These discussions all assume a single analyte perspective, *i.e.*, an out-of-control analyte will not affect the evaluation of another analyte that is in control, even if analyzed by the same laboratory. There is always the possibility that differences between the QA and project data could be due to sample inhomogeneity, and the temptation might exist to assign data discrepancies to this effect. The data evaluator is cautioned to use this explanation only as a last resort, or when supporting information is available, *i.e.*, when all three sample results (*i.e.*, project, QC, and QA) disagree, or when data from other parameters is also highly variable.

h. Sample Handling Documentation. This section should contain copies of all documentation related to sample handling and the QA inspection process, *i.e.*, chains of custody, cooler receipt forms, notices of deficiency, and documentation of any other communications (written or oral) with USACE or contractor POCs on correction of sample handling deficiencies.

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i. Project Specific Concerns. This section should address all communications between the district, field offices, prime contractor, and the CMQAL. Examples of this may be a request from the district for lower detection limits, quick turnaround analysis, or other requests or comments of an unusual nature (*i.e.*, outside the boundaries of the pre-established project DQOs). This section should also address anything that may have improved the chemistry aspects of the project (*i.e.*, use of a USACE-validated laboratory, more appropriate methods, more QC and QA samples, faster turnaround of QA sample results, more field oversight, *etc.*).