

# ***FAQs: Quality Systems Manual (QSM) Version 5.0***

## **General**

### **1. Why was "client" changed to "customer" and "documents" to "records" throughout the QSM?**

The changes were due to ISO requirements, but in general use client/customer and document/record are analogous. The changes do not require laboratory action.

### **2. We still get DoD projects where the project does not specify risk drivers and we are analyzing a full analyte list for the project. Can there be some guidelines in the QSM that when a project QAPP is being written risk drivers are identified?**

The QSM is a laboratory manual, and was not intended to provide guidance for establishing risk drivers by project managers. This type of information should be a part of the pre-planning process and incorporated into a quality assurance project plan (QAPP).

### **3. If our on-site laboratory assessment occurs at the same time as QSM 5.0 is approved, then which QSM will our lab be assessed? When should laboratories implement the changes to the QSM?**

The Accreditation Bodies (ABs) will begin assessing QSM version 5.0 six months after the final signed document is released (January 2014). Laboratories should start to implement the changes as soon as practical; whenever your laboratory becomes accredited to version 5.0 will be due to the scheduling of assessments by your AB. The QSM preface states that version 4.2 and version 5.0 are considered equivalent until such time as your AB is able to schedule you for version 5.0 accreditation.

## **Module 1: Proficiency Testing**

### **1. What is the difference between PT providers described in the ISO-17043 and ISO-17025?**

ISO-17043 is specific to PT providers. PT providers must be accredited under ISO-17043 for the purpose of DoD-ELAP. ISO17025 is specific to laboratories.

### **2. Module 1, Section 2.1.1 allows one PT for those who combine SOPs (Standard Operating Procedures) for methods (such as 624 and 8260). Why would separate SOPs require separate PTs? Some contractual programs require separate SOPs.**

Generally, when the two methods are combined into one SOP, the more stringent QC requirements from both are in the SOP. When there are separate SOPs, the QC requirements for the separate methods are generally different, thus requiring two PTs. However, if the QC requirements for two separate SOPs are the same, one PT will be acceptable.

### **3. If an analyte is not available as a PT sample, then can the laboratory perform an LCS Study (4 replicates) to demonstrate proficiency?**

Yes. Module 1, Section 2.1.4 states "When PT samples for an analyte-matrix-method combination cannot be obtained from a PT provider...and is required...Other measures as outlined in the appropriate TNI Standard Test Modules must be performed to satisfy the PT requirement."

## **Module 2: Quality Systems**

**1. The definition of a calibration range in Section 3.1 states: "For metals analysis with a single-point calibration, the low-level calibration check standard and the high standard establish the linear calibration range, which lies within the linear dynamic range." This is not a correct statement for ICP determinations using single point standardization. The calibration standard is at a fixed concentration but not at the limit of the linear range. The linear range is established and maintained on an annual basis through the determination and confirmation of a high level standard at a point within a 10% recovery range from the defined linear calibration.**

The QSM makes a distinction between the "linear dynamic range" and the "linear calibration range". The linear calibration range lies within the linear dynamic range. Raw values that are greater than the linear calibration range must be diluted to within the linear calibration range unless a high level check standard that is greater than the raw value (and still within the linear dynamic range) was analyzed and met acceptance criteria. The instrument calibration based on the original high standard is still used for the analyte quantitation.

**2. In section 4.5.6, if a laboratory uses an external firm for data review, it is stated that they must comply with the QSM and the primary lab's quality systems, as applicable. That seems to imply that the external data reviews do not have to be DoD ELAP accredited. Is this correct? How about the case where a non-DoD lab in a network prepares trip blanks and methanol?**

Yes. However, all subcontracted or outsourced management systems elements (such as data review or trip blank preparation) or outsourced personnel must comply with the laboratory's overall management system, must comply with the requirements of this standard as applicable to them, and are subject to review/approval by DoD/DOE customer.

**3. In section 4.7.1, is the last promulgated method considered obsolete if a draft or new method is posted on SW-846 Online? For instance, 5035A is draft and available as a new method.**

No. While the latest method(s) should be incorporated in most cases, the method that is required by contract or regulation shall drive laboratory practices.

**4. In section 5.3.3, are storage blanks analyzed for volatile organic compounds only or is the analyte list larger?**

Storage Blanks are analyzed for VOCs (including GRO) only.

## **Module 4: Chemical Testing**

### **1. In sections 1.5.2.1 and 1.5.2.2 why are LOD and LOQs required for surrogates?**

The LOD and LOQ are evaluated for surrogates in order to determine the point at which a surrogate is "diluted out" and therefore not evaluated for percent recovery. While not necessarily important from the laboratory's point of view, this evaluation can be very important during data validation and in evaluating data usability.

### **2. In section 1.5.2.1.b) please clarify if MDLs are still required annually.**

No. MDLs (DLs) are not required annually by DoD ELAP. An MDL study as defined by the USEPA may be one method of determining the DL, as stated in Module 4, Section 1.5.2.1 b), which is then used to establish the LOD and LOQ. Other methodologies may be used, as noted in the cited section.

### **3. In section 1.5.2.1.f.ii), what are the acceptance criteria for passing LOQ verification? The requirement identifies that the LOQ must be verified quarterly. Where can we find the specifications for acceptance of the LOQ verification?**

The laboratory's stated precision and bias at the LOQ is verified quarterly. The acceptance of this verification is based solely on the needs of the client, i.e. whether or not the verified precision and bias at the LOQ will meet project goals. The verification of the LOQ is included in Section 1.5.2.2 c). The QSM states "The laboratory procedure for establishing the LOQ must empirically demonstrate precision and bias at the LOQ for each suite of analyte-matrix-method, including surrogates. The LOQ and associated precision and bias must meet client requirements and must be reported."

### **4. In section 1.5.2.1.g), if a lab decides to not perform quarterly LOD/LOQs for a particular method and uses the 1 per batch approach, can the lab switch back to quarterly and is there a time frame limit to making this change? For the LOD/LOQ frequency criteria for infrequent methods, does the per batch basis mean per batch of DoD samples?**

Yes. The lab may switch from a one per batch approach to a quarterly basis provided all records and documents relating to the LOD/LOQ are current and accurately reflect the laboratory practice at the time the analysis is performed. There is no timeframe to making the change. The frequency criteria in the QSM apply to DoD/DOE samples only.

### **5. In section 1.7.1.1.d), if the ICV fails and the lab has analyzed a successful CCV and LCS next in the analysis run, can the lab CHANGE the CCV and LCS to the new ICV and CCV (renamed) with another LCS later to avoid reanalysis?**

No. It must first be determined whether the problem lies with the ICV or with the calibration standards. If the problem is with the calibration standards, then a new ICAL would be required. In addition, for most methods, the LCS undergoes an extraction/digestion, therefore even though it may be from a second source, an added element of uncertainty exists and may not be used as an ICV or CCV, unless the sample preparation is part of the sample analysis procedures (such as Method 8260).

**6. Can a secondary standard be used to verify a calibration from the same vendor or does it have to be from a different vendor? The two standards are prepared in separate lots. Is that sufficient?**

Yes. QSM Section 1.7.1.1 d) allows the use of a standard from a second lot obtained from the same manufacturer (independently prepared from different source materials).

**7. Please clarify the following scenarios: during automatic runs if you fail a CCV followed by 10 samples, are you allowed to inject 2 consecutive CCVs to acceptably report these 10 samples without qualification? If the ending CCV fails, how much time can lapse before the lab analyzes two additional CCVs (analytical sequences sometimes run overnight and finish very early in the morning)? What if your run time for two additional CCVs is too long to analyze 2 within an hour (for example, a radiochemistry analyte that requires a count time longer than one hour)?**

Section 1.7.2. i) of the QSM specifies that a rerun for a failed CCV must be started immediately (i.e. within one hour). This means that the rerun of the first CCV after the failure must START within one hour regardless of the run times of two consecutive CCVs. No samples can be analyzed in between the failing CCV and the two additional CCVs.

**8. For section 1.7.2 i) do all target analytes in both CCVs need to pass? For example, if CCV1 failed compounds A and B and CCV2 failed C and D, did the 2 CCVs just failed, which then requires re-analysis of all associated samples?**

The target analyte(s) driving the re-analysis of the failed CCV must pass the two reruns. If other analytes are out in the re-analyses, the samples were still bracketed by a passing CCV (for those analytes) proving that the system was in control at the time the samples were analyzed.

**9. What is the corrective action for END RUN CCV 50% criteria failure (GC/MS Methods)?**

The corrective actions for an end of run GC/MS CCV infraction is the same as the corrective actions for any other CCV. There are two options: Recalibrate and re-analyze all affected samples, or immediately analyze two consecutive CCVs, and if both pass, the samples may be reported without re-analysis.

**10. If multiple LCS (mixes) are prepared in order to avoid compound interferences are any failures allowed, as long as recoveries are within marginal exceedances? If so, is the number of allowable failures based on the total number of target analytes or the number of analytes spiked in a particular LCS injection? Do the LCS spiking and acceptance criteria outlined in the previous question also apply to MS and MSDs?**

Section 1.7.3.2.3 h) states that "Sporadic marginal exceedances are allowed for those analytes outside the 3 standard deviation control limits but still within 4 standard deviations." The number of allowed exceedances should follow guidance in the TNI standard. The number of exceedances allowed is based on the total number of reported analytes, not the number of analytes per LCS mixture.

Yes, the LCS spiking and acceptance criteria also apply to the MS and MSD.

**11. If we are requested to analyze samples with a modified method, such as a low-level 8270 or 8270 SIM, for a DoD project, are we to use in-house LCS limits for both LCS and MS/MSD? We have calculated in-house limits and the QSM directs us to use them, but we want to clarify that the LCS limits are to be used for the MS/MSD as is required for Appendix B Table methods.**

Method 8270 SIM is covered in the LCS tables in Appendix C. For a method or analyte not listed in Appendix C or a modified method that is significantly different from the standard version, in-house LCS limits shall be used. The in-house LCS limits apply to the MS and MSD as well.

### **Appendix A: Reporting Requirements**

**1. Appendix A indicates that DL, LOD, and LOQ must be included in the report or within the body of the report. Can the LAB not report the LOD if the client instructs? Does this directive supersede the QSM requirement?**

Client instructions supersede QSM reporting requirements. The laboratory would not be required to report the LOD if directed by the client.

**2. Why do you need to report both LOD and MDL (DL)? Shouldn't it be one or the other?**

The MDL (DL) is included largely for risk assessment purposes. According to QSM requirements, any analytes detected between the DL (or MDL) and LOQ shall be reported as estimated results; any analytes not detected shall be reported as less than the LOD (or LOD U). It is correct that for many reports either MDL (DL) or LOD is sufficient. Report contents may be specified by any project, and project-specific reporting directives may be used in lieu of QSM reporting requirements.

### **Appendix B: Quality Control Tables**

**1. Other Quality Manuals had the calibration correlation coefficient as r. Now Appendix B Tables shows it should be r<sup>2</sup>?**

The acceptance criterion for instrument calibrations in QSM Version 5.0 is coefficient of determination ( $r^2$ ) <sup>3</sup> 0.99 for chemical analyses and correlation coefficient (r) <sup>3</sup> 0.995 for radiochemical analyses. They are essentially the same. The guidance in Appendix B Tables is current and should be followed.

**2. Throughout Appendix B Tables for MS/MSD recoveries, one is to meet the LCS requirements for the analyte in Appendix C. Why is a clean matrix (LCS) the same as the more complex matrix in terms of percent recovery?**

Matrix Spike analyses are considered a quality indicator. MS/MSD recoveries from different sites are not comparable due to different matrices (e.g., the matrix from an estuary sediment sample is different from the matrix of an arid alkaline soil sample). The LCS is the control against which a specific matrix can be compared (i.e., a passing LCS and a failing MS may indicate a matrix interference).

**3. When we try to use alternate calibration options for GC/MS methods like 8260 and 8270 using DoD criteria we get positive results in the Method Blank (MB) for same compound or ND for sample with slightly higher concentration than curve. Unless we are allowed**

**using forced through zero for some of these situations, it is not easy to use alternate calibration options. We need more clarifications on these issues.**

If the calibration is scientifically valid (e.g., the intercept is statistically not different from 0) and the reason is justifiable and documented, forcing through zero is allowed, as are weighted calibrations. However, forcing thru zero should not be used to treat the MB results exclusively; otherwise, biased results will be obtained, especially at low concentrations.

**4. When ICV is as stringent as CCV, do you require the ICV to be a second source as is required in those methods where the ICV had different criteria for passing?**

Yes, the ICV is a second source verification that reveals potential bias on the primary source calibration standard. For QSM version 5.0, the ICV is as stringent as the CCV.

**5. Table 1 - Confirmation of positive results (second column) says “All positive results must be confirmed, but if the secondary column RPD is > 40%, flag with a J, discuss in narrative and go ahead and report primary column. However, if the second column shows no recovery, still report the primary column result with a J qualifier? We believe this would not be a confirmation.**

According to the SW-846 methodology, if RPD > 40%, the detection is not confirmed or the analyte is not detected. In the stated situation, the results would not be confirmed, so a J flag would be inappropriate.

**6. Table 4 - ICV: The acceptance criteria are  $\pm 20\%$ . The determinative method, 8270D, allows for  $\pm 30\%$ . Which value would be used?**

The acceptance criteria in the QSM tables supersede the acceptance criteria in the methods.

**7. Table 4 - For GC/MS, there is a new requirement to analyze a CCV at the end of each run. Does this mean at the end of each 12-hour window or at the end of each batch (20 samples)?**

Both. A CCV shall be analyzed at the end of each batch, but can also be used as the opening CCV for the next 12-hour window if all the criteria for a continuing CCV is met.

**8. Table 4 - Organic GC/MS methods are required to have an "end of run" CCV at  $\pm 50\%$  (does this standard need to be within tune?)**

Yes. The “end of run” CCV analysis shall be started within the tune window.

**9. Table 4 - If multiple CCVs are analyzed to avoid interferences, do they all have to be injected within the same tune window as the samples? Are any exceptions allowed for documented poor responders?**

Assuming that the first CCV was analyzed within the 12 hour tune window, and the subsequent CCVs were performed consecutively, then the subsequent CCVs do not have to fall within the 12 hour tune window. There are no exceptions for poor responders.

**10. Table 4 - End of Run CCV for GC/MS methods - does 50% applies to all analytes for multiple analyte methods? Can a representative analyte list be used for the end of analytical batch CCV?**

Yes, the 50% acceptance criterion applies to all analytes. The end of batch CCV must contain all target analytes.

**11. Table 4 - Internal standards: Has the retention time window referenced to the midpoint standard of the ICAL been changed to  $\pm 10$  seconds from  $\pm 30$  seconds?**

Yes. The retention time window has been changed from  $\pm 30$  seconds to  $\pm 10$  seconds in QSM version 5.0.

**12. Table 8 – For ICP the Low-level Calibration Check Standard (Low-level ICV) says to report analytes within  $\pm 20\%$ , but the comments state the Low-level calibration check standard should be less than or equal to the Limit of Quantitation (LOQ). That would place the Low-level ICV below the LOQ at times and thus unreasonable to expect quantitation at  $\pm 20\%$  below the LOQ?**

Correct, however, LOQs must lie within the calibration range and would be expected to have a LLICV within  $\pm 20\%$ . If not, the LOQs might be set too low and need to be increased.

**Appendix C: LCS Control Limits**

**1. Can we please get a complete list of all analytes in the LCS study?**

Yes. A full report of the LCS study is now available on the DENIX website.

**2. Method 1668 is addressed in the LCS tables; however, there is not a QC table for PCBs by HRGC/HRMS?**

The LCS study was performed after a data call to laboratories and included all method and analyte combinations that were received and provided enough data to perform the applicable statistical analysis. The QC Tables represented the typical/routine SW-846 methods encountered by DoD/DOE projects so there are some methods that have corresponding LCS limits but do not have their own QC table.