

HASQARD Focus Group

Meeting Minutes

February 21, 2012

The meeting was called to order by Huei Meznarich, HASQARD Focus Group Chair at 2:02 PM on February 21, 2012 in Conference Room 308 at 2420 Stevens.

Those attending were: Huei Meznarich (Chair), Cliff Watkins (Secretary), Lynn Albin, Taffy Almeida, Courtney Blanchard, Glen Clark, Scot Fitzgerald, Shannan Johnson, Kris Kuhl-Klinger, Larry Markel, Karl Pool, Steve Smith, Cindy Taylor, Amanda Tuttle, Sam Vega, Rick Warriner, Rich Weiss and Eric Wyse.

- I. Huei Meznarich requested comments on the minutes from the January 17, 2012 meeting. No HASQARD Focus Group members present stated any comments on the January meeting minutes and, after hearing no objections, the minutes were approved.
- II. The Status of the preparations of Revision 4 for Volumes 1, 2 and 3 were discussed.
 - a. The Status of the review for Volume 2 was discussed. Chris Sutton was not present for the meeting. Scot Fitzgerald reported in Chris' absence that the comments received are being addressed. Scot believes it will take at least another month to address the comments received.
 - b. Steve Smith reported that the QA Group has completed a red-line of Volume 1 to address the QSAS deviations from HASQARD. That red-line has been presented to the HASQARD Focus Group. The subcommittee then looked at the corrective actions and assessments sections. The subcommittee has recently received comments on the procedures section and will be adding language to make it clear under what circumstances a procedure or method may be changed. This latest revision should be completed in two or three weeks. Huei Meznarich will add the revision of the procedures section to the schedule since this was in addition to the original scope of the effort.
- III. HASQARD Volume 4, Revision 4 Proposals

Continuing with the process begun at the November Focus Group meeting, the Secretary projected the Word file containing the combined set of proposed revisions to Volume 4 of HASQARD as provided by the organic analysis, inorganic analysis, radiochemistry and quality assurance (QA) subcommittees on a screen for all to view. The Secretary used the software to revise as

necessary as the Focus Group started discussing proposed revisions from the point they left off at the January meeting, the beginning of Section 4.4.1.

Prior to discussing Section 4.4.1, Rich Weiss thought it would be helpful to skip to Section 5.3.1 to make sure everyone was in agreement on the terminology used for reporting uncertainty. The concern was that the term uncertainty is meant to represent total propagated uncertainty rather than a single component of the total propagated uncertainty (e.g. counting uncertainty). The Focus Group felt like the language used in this section was adequate as it is currently presented.

Returning to Section 4.4.1, the HASQARD Focus Group agreed to strike a sentence from the following paragraph as shown:

~~“Calibration procedures and frequency shall be established by the laboratory and shall consider the manufacturer’s recommendations and the requirements specified in this section. Manufacturer recommendations may include the number of standards employed, standard (activity) levels, energy ranges, voltages, count times, reagents, or other parameters. In addition the American National Standards Institute (ANSI) standards N42.14, N42.25, & N42.15 provide guidance for the calibration and operation of radioanalytical detector systems. These guidance documents should be evaluated for applicability or incorporation into laboratory procedures.”~~

The Focus Group determined that the sentence order in another paragraph of Section 4.4.1 should be revised as shown:

The isotope(s) used in calibration shall have relevance to the emission type and energy of the analyte to be determined. Ideally, detectors should be calibrated using pure materials of the same isotopes as might be encountered in samples, since the energy of the emitted radiation strongly affects the efficiency of both beta and gamma detectors. ~~The isotope(s) used in calibration shall have relevance to the emission type and energy of the analyte to be determined.~~ For example, a ⁹⁹Tc standard is the optimum choice for calibrating a scintillation counter used for ⁹⁹Tc analysis; ¹⁴C is acceptable for the LSC calibration for ⁷⁹Se as there is no standard source of ⁷⁹Se available and the β⁻ energies are comparable.

The last sentence of the paragraph concerning use of software corrections for calculations of activities of gamma analysis samples in counted in geometries other than those for which a valid calibration is obtained was changed to say:

“Whenever software geometry corrections are applied, the client shall be notified; to either concur with the approach or agree on an alternative.”

The paragraph in Section 4.4.1 concerning control of interferences was changed to say:

“Interferences are often a fundamental problem in counting and can occur during calibration and analysis. Examples of interferences are α/β cross-talk for gas proportional counting, coincident summing, dead time or pulse pileup of gamma spectral photons and peak tailing in alpha spectrometry. Potential interferences shall be evaluated. Processes to control or manage interferences shall be documented as part of the calibration and analysis procedures. Software tools for interference corrections and or peak tailing are typically part of the software associated with gamma and alpha spectral counting systems. The use and application of these tools is dependant upon the cognizant scientist. Software used for interference corrections is subject to the verification and documentation requirements in Section 5.2 and Volume 1, Section 7.0.”

In several tables in Section 4.4.1, the term “Initial use” was replaced with “Initial start-up” when describing the calibration frequency requirements.

In the tables specifying requirements for frequency for background checks and counter control or control standard checks, the phrase “...or after analytical run whichever is longer” was replaced with “...or after analytical run if it is longer.”

In Table 4-5, the Focus Group discussed the calibration requirements for cyanide determinations by manual and semiautomated spectro-photometric methods. The current wording for the frequency requirement is: “Before each new analytical run with the following exception: for dedicated instruments, when six concentrations are used in the calibration, the calibration is valid as long as calibration verification acceptability is demonstrated or for up to 90 days.” The Focus Group could not come to agreement on what is meant by “dedicated instrument.” Also, without the presence of a member of the Inorganic subcommittee that remembered where this exception is coming from, the question was tabled for resolution at an upcoming Focus Group meeting.

As the Focus Group began looking at Section 5.0 and 5.1, the subject of laboratory records was discussed at length. Of specific interest was a discussion on the use of laboratory logbooks as records. The use of three-ring binders, bench sheets and bound logbooks were discussed and the level of rigor that must or should be applied to generate an adequate record. The need

to sign a review verification on every page of a logbook was questioned. The group concurred that when a chronological order of events over a significant number of entries is required (e.g., a shift log), a bound notebook is necessary. But, when sample preparations are being documented, the need for a bound logbook may be questionable. The Focus Group agreed that there is a need to clarify the terms “notebook” versus “logbook” and ensure that the terms are accurately used in HASQARD. The Focus Group believes Section 5.1 should be removed from Volume 4 and this material should be contained in Volume 1 only.

Section 5.2.1 was discussed in detail. The section discusses significant figures and sites an ASTM standard for the method for determining and reporting significant figures. Because the section specifies no requirements, the value of the section was debated. The Section was retained to say:

“Significant figures reflect the limits of the particular analysis method. Basic rules for significant figures and for calculating values and retaining the number of significant figures are provided in American Society for Testing and Materials (ASTM) E-29, *Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications*.

Reported values should contain only the appropriate number of significant figures. Recognizing that vendor-supplied software may not meet the general rules for significant figures, the laboratory should work with the client to determine the best way to report results, based on the project needs.”

Huei Meznarich took the action to determine if the ASTM E-29 Standard was still applicable and valid.

After discussing Section 5.2.2, the Secretary noted the time for closure of the meeting was at hand. Therefore, the Chair stated that rather than start into Section 5.2.3 the meeting should be adjourned.

Hearing no additional new business, and no objections to the proposal to adjourn, the meeting was adjourned at 4:25 PM. The next meeting is scheduled for March 20, 2012 at 2:00 PM in 2420 Stevens, Room 308.