June 24, 2013

Mr. Lemuel Walker
Clean Water Act ATP Coordinator
U.S. Environmental Protection Agency
Office of Science and Technology
Engineering and Analysis Division
1200 Pennsylvania Avenue, NW (4303T)
Washington, DC 20460

RE: Comments on Draft Methods 624B and 625B

Dear Mr. Walker:

Thank you for providing the Environmental Laboratory Advisory Board (ELAB) with the opportunity to provide a second round of comments on Methods 624B and 625B. The members were pleased to see that most of the Board's previously submitted comments have been incorporated in the current drafts.

While reviewing the methods, we noted that a couple of items seemed important from a more global perspective, given that they would apply equally to most, if not all, Part 136 methods. Rather than incorporating these into individual methods such as 624B and 625B, the ELAB members think that they could be included in a section of Part 136 that applies more generally.

These items are:

1. There is no requirement for verification that calculated method detection limits (MDLs) or reporting limits are accurate. ELAB understands that the U.S. Environmental Protection Agency (EPA) is considering an update to the Part 136 Appendix B MDL procedure and hopes that some sort of verification procedure could be included.
2. There is no description of the documentation required for or policies surrounding manual integration. Possibly this could be added to the QC sections at Part 136.7.

Sincerely,

Patsy Root
Chair, Environmental Laboratory Advisory Board

cc: Lara Phelps

General Comments – EPA Method 624 Revision B

Section 1.4 The MDLs referenced were determined 33 years ago and should be omitted from the document. Instead of MDLs, a list of typical ranges for quantitation limits could be included. If MDL tables are included, a note is needed explaining that they are just examples, and each laboratory will need to generate its own MDLs.

Section 5.2.2 Add language clarifying that the trap described in this section is just an example.

Section 6.5.5 A statement should be added indicating that acidification has been known to degrade 2-chloroethylvinyl ether, and if determination of this compound is needed, it should be performed on the analysis of an unpreserved sample vial.

Section 8.2.5 The data in **Table 7** are 24 years old. They were produced under conditions that have changed significantly since then. ELAB recommends that EPA publish Method 624B without new tables but should gather data that would allow updating the tables in a subsequent revision.

Section 8.3.3 See 8.2.5 above. **Table 8** in Section 8.3.3.2 is equally old and should be replaced with the suggested approach in Section 8.2.5.

Section 8.4 This section references a closing calibration. Typically, EPA GC/MS methods using internal standards do not require a closing calibration. Adding the requirement without detailed prior study of the potential impact would be a mistake. It is suggested that this requirement be removed and data gathered for consideration in a future revision

Section 10 This section is outdated. Although autosamplers are allowed (Section 10.3), it might be worth rewriting the detail according to more recent practice.

Section 11.2 There should be description of how the BFB is to be evaluated. We recommend specifying use of either the peak apex, or the average of the apex and the scans immediately prior to and immediately after the apex (total 3 scans). Background subtraction should also be used and should be a scan that is not part of the BFB peak, but is within 20 scans of the peak.

General Comments – EPA Method 625 Revision B

Section 1.1 This method was validated through an interlaboratory study conducted 29 years ago. Rather than rely on this statement alone, it would be appropriate to add that the fundamental chemistry principals originally developed for this method remain sound and continue to apply.

Section 1.4 The acceptance data in T**able 6** are dated. We recommend that EPA publish Method 625B without new tables but should gather data that would allow updating the tables in a subsequent revision.

Section 1.5 MDLs referenced were determined 33 years ago and should be omitted from the document. Instead of MDLs, a list of typical ranges for quantitation limits could be included. If MDL tables are included, a note is needed explaining that they are just examples and each laboratory will need to generate its own MDLs.

Section 7.2.1.1 There should be description of how the DFTPP is to be evaluated. ELAB recommends specifying use of either the peak apex, or the average of the apex and the scans immediately prior to and immediately after the apex (total 3 scans). Background subtraction should also be used and should be a scan that is not part of the DFTPP peak, but is within 20 scans of the peak.

Section 7.1 The data in **Tables 4 and 5** are 33 years old. The MDL and retention time data should be eliminated from the table.

Section 7.3 This section references a closing calibration. Typically, EPA GC/MS methods using internal standards do not require a closing calibration. Adding the requirement without detailed prior study of the potential impact would be a mistake. It is suggested that this requirement be removed and data gathered for consideration in a future revision

Section 7.3.1 The calibration acceptance criteria in **Tables 6 and 8** are expressed in terms of percent recovery rather than a percent difference from the initial calibration. For consistency with other EPA methods, calibration acceptance criteria should be expressed in terms of percent difference from the true value based on the initial calibration.

Section 8.3.3 The data in **Table 6** are dated and cannot possibly compare to real-world spiked sample data. Because matrix spike results are necessarily dependent on the sample matrix, ELAB suggests that this requirement be replaced by language instructing laboratories to develop their individual performance ranges for spiked samples.

Section 16.1 The data presented in **Table 7** are 29 years old. ELAB suggests that EPA generate new and more relevant data. The Board would be interested in assisting with this process.

Section 16.2 This chromatogram has little value and should be deleted.