

US EPA ARCHIVE DOCUMENT



September, 19, 2018

Dr. Jennifer Orme-Zavaleta
EPA Science Advisor
United States Environmental Protection Agency
Ariel Rios Building (MD 4101M)
1200 Pennsylvania Avenue, N.W.
Washington, DC 20460

RE: User-Generated Mass Spectral Libraries and Tuning Criteria

Dear Dr. Orme-Zavaleta:

The Environmental Laboratory Advisory Board (ELAB or Board), a standing Federal Advisory Committee Act board that advises the U.S. Environmental Protection Agency (EPA or Agency), was asked to review instrument performance criteria in existing EPA methods from Office of Water and the Office of Land and Emergency Management. In response to comments from EPA representatives to a previous draft, we offer this letter of request. The substitution of laboratory-generated libraries/relaxed tuning criteria could be applicable for quantitative analysis including; 624.1, 625.1, 8260 and 8270, and others that require verification against National Institute of Standards and Technology (NIST) library spectra or require strict instrument tuning rules. The evolution of analytical platforms (e.g. triple-quadrupole, ion trap, and time of flight mass spectrometers) has provided significant advances in both selectivity and sensitivity. ELAB recognizes there are scientific benefits to be gained by having the EPA methods evolve to benefit from these advantages. Unfortunately, the current validation of EPA method instrument performance criteria against NIST spectra results in having to detune from optimized parameters, reducing the instrument's performance. The detuning limits the instrument's performance just to meet the EPA method specific criteria. The topic was first addressed at ELABs August 2016 annual face-to-face meeting with the general public with additional comments received at the face to face meeting in August 2017. The comments were received from stakeholders representing; Test America, Advanced Systems Inc., Askew Scientific Consulting, David Friedman Consulting and Eurofins Lancaster Laboratories Environmental as well as Mr. Larry Adrian from EPA Region 7. There was an observation that the analytical community would benefit from being able to use the full capacity of laboratory instrumentation and in not being required to meet tuning criteria that do not allow instruments to be run optimally. It was also pointed out in the discussion that this topic goes well beyond a platform issue to include whether identification with a user-generated library would be acceptable to EPA, regardless of platform. The tuning required in the existing methods forces users to bypass advancements in sensitivity afforded by modern spectrometers, in favor of meeting the method specifications.

ELAB asks if EPA is willing to consider increasing the flexibility of a method by allowing alternative tuning criteria and to clarify under what conditions would a user-generated library be acceptable? Could a set of minimum validation criteria for mass spectra be developed to be used by those who wish to generate their own library as well as for those who would like to continue to use the NIST library and would EPA be amenable to accepting these criteria?

Quantitative analysis is more appropriate for the application of user-generated, instrument specific spectral libraries. Qualitative analysis – the identification of non-target unknowns – is better suited for the use of the NIST libraries given their breadth and the way they were created.

If EPA were willing to accept alternative tuning criteria and user-generated libraries, ELAB could build a compelling case for establishing suitable validation criteria. Those validation criteria would include:

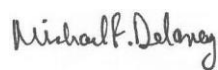
1. The requirement that fundamental criteria would need to be met, presenting the science and reasoning behind the process of how a user-based library could be validated.
2. Initially limiting the scope of the application to quantitative analyses that do not involve unknown identification and GC/MS applications only. It would require the need for a skilled GC/MS operator.
3. To improve the flexibility without sacrificing quality in the existing methodology by using a standard approach in determining the validation criteria and demonstrating why those criteria were selected. These could either be established by EPA; specifying how laboratories set their criteria or the Agency could specify the criteria directly. This might include the criteria NIST uses to include a spectrum in its own library.
4. Use of an across the board standard (s) reference material (e.g. cholesterol), for evaluating performance that should transcend platform or matrix and demonstrate that a minimum instrument performance is met before a spectrum can be added to a user generated library.
5. Validation protocols that would be independent of methods, programs platforms and matrices.

The overall goal is to leverage the improvements in available instrument technology. This requires a more performance-based approach to tuning criteria, which inherently requires an increase in method flexibility. This topic generated significant discussion at the face-to-face meeting, demonstrating the interest among the community of environmental analysts. It was also observed that those who prefer to work within the existing requirements of a NIST library would not be required to change. ELAB recommends that EPA consider the evaluation of possible validation criteria for user generated libraries allowing for alternatives to the existing tuning criteria to meet NIST spectra matching.

ELAB appreciates the opportunity to provide this information in support of performance-based approaches that meet Agency's program goals.

Please let us know if you would like ELAB to perform additional review of this topic.

Respectfully,



Michael F. Delaney, Ph.D.
Chair, Environmental Laboratory Advisory Board

cc: ELAB Board
Thomas O'Farrell, ELAB Designated Federal Official