2018 NEMC
Resolving Method Differences for Volatile Organic by GC/MS with Best Practices

Best Practices for Quality Control

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Volatiles by GC/MS EPA Methodology

* ORCR (Streamlined Approval Process for SW-846)
  * 8260B, 8260D (just finalized in July)
* OW (Part 136)
  * 624.1 (When will NELAP ABs offer?)
* OGWDW (Part 141.24)
  * 524.2, 524.3

524.2, 8260B and now 624.1 are the current work horses!

Will your lab move to 8260D?

Not even taking into consideration TO-15, CLP SOW for Organics, etc.

Underlying Questions and Driving Forces of Quality Control (QC)

It get's complicated!

* What regulatory program and accreditation requirements apply?
* What are the measurement quality objectives for the method?
* Has the data quality objective process been used for a QAPP?
* Definitions for preparation batch and analytical batch?
* What are the quality control measures?
* Is it a "may," "should" or a "shall" in test method?
* What is the minimum frequency of the QC?
* What are the QC acceptance criteria or limits?
* Are statistically derived control limits or set method limits used?
* What corrective action is called for and is root cause analysis utilized?
* If QC outside limits does this trigger a resample, reanalysis or qualification of associated test results?
* Can you qualify and then report the associated test results? Ask your state.

Is it overly complicated?
You Decide

* Initial Demonstration of Capability or Proficiency:
  * different number of spikes
  * spike levels vary
  * source of standard, same or different from ICAL standards
  * acceptance criteria varies from method to method
* Tune:
  * BFB
  * varying concentrations can be used
  * additional masses (524.2 vs. 8260B and 624.1)
  * prior to calibration/analysis, but frequency varies (Every 12 hrs. or 24 hrs., 524.3 daily analysis not required)
* MDL:
  * OGWDW Memo on OW's MDL clouds situation

Is it overly complicated?
You Decide

* Blank, Method Blank, Laboratory Reagent Blank
  * Frequency - with each analysis batch and if samples exceed ICAL range; prior to each 12 hour shift (one per tune) and after sample with carryover; before processing samples or each group of 20 samples on same instrument during same analytical shift.
  * Criteria - < ½ the MRL; >MDL or > 1/3 compliance limit or > 1/10 conc. in sample in 12 hour shift, whichever is greater; < LLOQ

Do you feel SW-846 overuses the word "should?"

Is it overly complicated?
You Decide

* Laboratory Control Sample, Laboratory Fortified Blank
  * Spike with – all or some (same as in MS).
  * LCS = CCV for VOA
  * Frequency – at beginning of analytical batch, every ten samples and at end; prior to field samples and during 12 hour shift (one per tune); in each analytical batch.
  * Criteria - ±30%; ±3 SD, Table 7 lower and upper limits or interim 60-140%; 70-130% as interim then ±3 SD (what about marginal exceedances and <10% recovery)
  * 624.1 basically indicates if first LCSs out then make a fresh LCS and reanalyze for analytes that fail. Contrary to TNI! (See 624.1, Sections 8.4.3 and 8.4.4)

Is it overly complicated?
You Decide

* Matrix Spike, LFMS
  * LFMS data collected from each drinking water source; 5% for each discharge being monitored.
  * Frequency - Spike each analysis batch; at least 5% of samples or one per extraction batch; at least one matrix spike in each analytical batch.
  * Spike analytes of interest and rotate spike conc.
  * Spiking conc. greater than or equal to native background conc.; spike at regulatory concentration limit or 1 to 5 times higher than background conc.; Spike with LCS analytes at same conc. as in LCS
  * Criteria – ±50% near MRL, ±30%; QC limits Table 7; Statistical Control Limits.

Is it overly complicated?
You Decide

* Surrogates – 70%-130% take CA and reanalyze; in-house limits if not in Table 7, 60%-140% as interim criteria for surrogates not listed in Table 5; control limits developed by laboratory. Why should a surrogate fail in a blank or LCS?
* Internal Standards - ±30%; 50%-200%.
* Other:
  * Methods don't always adequately address: corrective action, use of trend analysis, have realistic method based limits (to wide).
  * Can data be qualified and is it allowed to be reported. New 600 series method language rather controversial in this regard and needing clarification! Can state or EPA even accept appropriately qualified data into database?

Q: How many labs will need to have separate SOPs for 524.2 or 524.3, 8260B or D and 624.1 due to differing QC requirements, even for water matrix samples? Q: Are you having to go with most stringent QC approach to have one or two SOPs for GC/MS VOAs? Show of hands.

Hot off the Press
8260D Finalized 7/12/18

Section 9: This section was completely updated and reorganized. New language and references were added from Method 8000. Sections on IDP (Sec. 9.4), blank language (Secs. 9.5 through 9.5.4), and LLOQ (Sec. 9.9) were updated and expanded. Significant revisions/additions were made to the blank section adding clarifying information about concentrations allowed in blanks (one half LLOQ), how blank concentration relates to sample concentration (<1/10), and some guidance for qualifying data. Information was added about the required frequency of LLOQ check standards (Sec. 9.9.1.2).

Is this a step in the right direction? 

Does this help further **harmonize** the GC/MS VOA methods?

You Decide.

Best Practices
Suggested Approach

1. TNI Standard, V1M4 - TNI CEC **must be the driver and change agent** for adopting best practices in its standard language.
2. EPA Program Offices **must effectively** coordinate **together** and adopt best practices for QC terminology, measures, frequency, criteria, etc. aka Harmonization!
3. Laboratories **must** incorporate, to extent possible, best practices in their test method SOPs for QC.
4. Accreditation Bodies **must** take into account best practices for QC, where they can, and look at QC from a performance based perspective.

We All Can Do Better!

Audience Discussion/Feedback

* What is your top rated conflicting method QC that you feel has no legitimate basis, causes you unnecessary problems, does not improve data quality and results in lessened operational efficiency?
* What would you like to see done next?
* What actions should TNI, ELAB and USEPA take to address laboratory concerns of this nature?
* Please send any after thoughts to:
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THANK YOU! - Special thanks to John Briney at PDC Labs.