Modifications in the Extraction of Water Samples for Method 625
Method 625: The Extraction

- Currently, samples are extracted by either separatory funnel or CLLE.

- What's more achievable to a lab – “greenness” or time?
  - Sep funnel = time
  - CLLE = green
Why Not Separatory Funnel?

• Separatory funnel *much* faster, but…
  • Uses large volume of CH$_2$Cl$_2$:
    (3 x 60 mL acid extract + 3 x 60 mL base extract = **360 mL** solvent)
  • Could form emulsions difficult to separate
  • For our laboratory, 625 projects are infrequent.
Pros/Cons of CLLE in Method 625

- CH$_2$Cl$_2$ volume reduced to slightly > 100 mL
- Design of apparatus means no emulsions or drying of extract
- But… total extraction process is over 48 hours in length
  - Could factor in low recovery of some analytes
  - Experience with Method 3520 show that 24 hours is not necessary
- Method 625.1 to the rescue!
Updates in Method 625.1

- Should be promulgated later in 2015-16, includes SPE as alternate extraction technique

- From Sections 10.3.4 and 10.3.5 in 625.1: “…extract for 18 – 24 hours. A longer or shorter extraction time may be used if all QC acceptance criteria are met.”

- DOC: 6 hours @ pH 2, 6+ hours @ pH 11, completed Feb. 2015

- MDL study: Same conditions as DOC, completed April 2015
Method 625.1 – SPE Capable

- Our lab has SPE modules capable for Method 625.1
- However, they are exclusively used for Method 525.2 (drinking water) at the moment.
All analytes meet 625 acceptance criteria limits.

- Standard deviation for all analytes no greater than 12.3 µg/L
- All RSD values 22.8% or less

For reference, the lowest standard deviation listed for any analyte is 13.0 µg/L

Analyte list based on TTO (Total Toxic Organics)
MDL Study for Method 625 – Part 1

• Originally performed along with DOC in February, spiked at 1.0 µg/L

• Most analytes passed the stringent MDL requirements
  • The few that failed had MDLs < 0.1 x MRL
  • 40 CFR Part 136, Appendix B stipulates 0.1X MRL ≤ MDL ≤ MRL
Most analytes on TTO list have MRL ≥ 10 µg/L

Our MRLs set to either 5 or 10 µg/L, depending on MRLs listed for TTOs.

Repeated study in April

• Analytes spiked at 10 µg/L
• This time, all passed MDL requirements
Conclusions

• Our extraction technique results in less solvent and time used

• DOC/MDL data show consistent recoveries
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