

EPA Method 625.1 - A Summary of the Changes in the Newly Promulgated GC/MS Method for Semi-Volatile Organics in Wastewater

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AKA =

**11+ things you need to know about the revised
method EPA 625.1**



REVISED → no new Inter-lab study

Method data still based on 1984 Study

- **Priority pollutants**
- **LLE**
- **Packed Columns**
- **Separate Acids and B/N → special columns**
- **3 point calibration**
- **Full scan MS**

Method 625 will be Method 625.1

- Table 1 → 38 base neutral
- Table 2 → 11 acid extractable
- **Each Table lists MDL and ML**
 - ML = 3 times MDL
 - MDL = 1984 MDL

EPA METHOD STUDY 30,

METHOD 625 --

BASE/NEUTRALS, ACIDS AND PESTICIDES

by

Radian Corporation

P. O. Box 9948

Austin, Texas 78766

Contract No. CI-68-03-3102

Table 3 list 316 analytes

- **13 are priority pollutant pesticides and PCB's**
 - These have MDL and ML data
- **303 have no MDL or ML data**
 - YOU must establish your own

BRING YOUR OWN

MDL

“Cutting edge” – You can now use Capillary columns

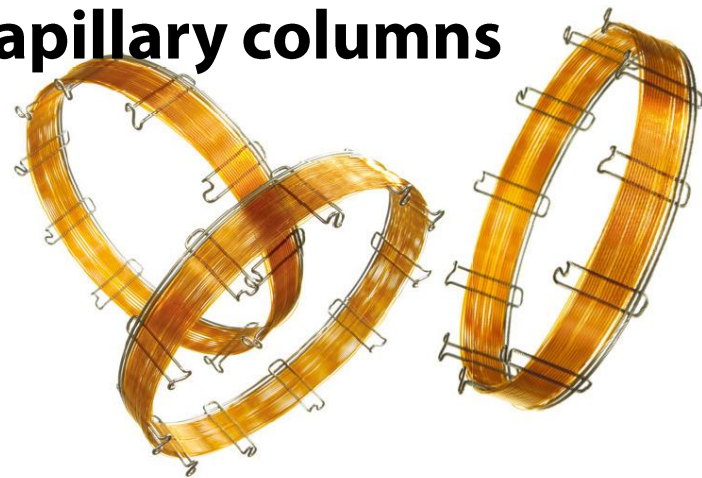
- **Capillary columns in text**

- Examples given

- **EI and CI in Table 4 for priority pollutants**

- Includes quant and secondary ions
- Retention times (elution order)

- **No Quant ion, secondary ion, or retention time data for the 303 new analytes in Table 3**



$$\text{RSE} \leq 35\%$$

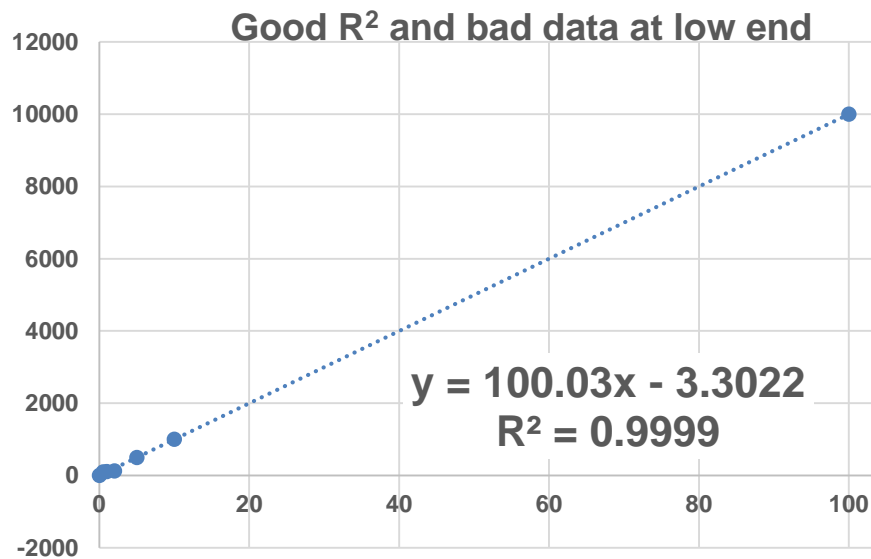


Table 8 provides 38 surrogates or internal standards

- No quant ions or secondary ions
- No retention times
- Internal Standard response 50 – 200%
- CCV = second source standard
- Method Study 30 → no correlation of SS with analytes found

Solid Phase Extraction is allowed

- Individual lab or Vendor MUST validate Table 1 and Table 2
 - Spiked MS/MSD complete list, 4 IDC, 1 PT
 - Up to 9 matrices, depending
 - MDL (lab must do)
 - Must fortify with surrogates
 - Must meet 625 criteria for Table 1&2, or 60 – 140% for Table 3

<https://www.epa.gov/cwa-methods/alternate-test-procedures>

100 – 1000 ml sample size

- Smaller sample volume = better for SPE
- Extract less means use less reagent
- New instruments can detect lower



One calibration Standard must be at ML

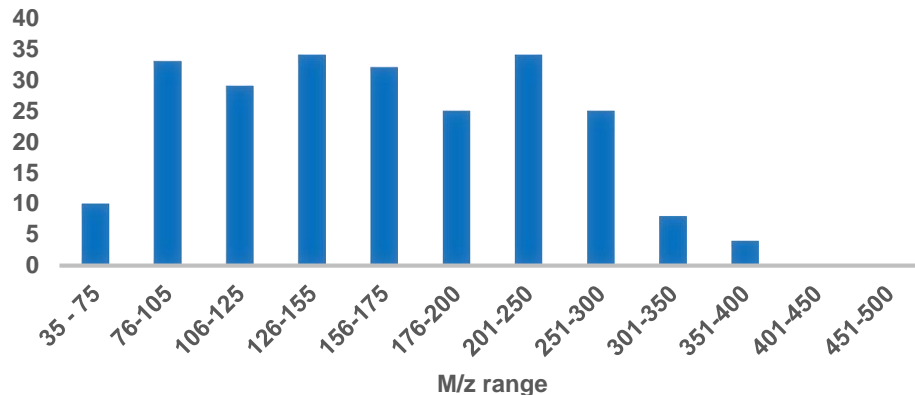
- Or as specified in a permit
- Or your own as long as lower than Table 1 or Table 2 ML
- Table 3 has no ML (develop your own)



DFTPP tune criteria more flexible, by footnote

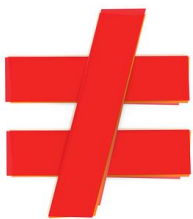
- Adds TOF criteria as Table 9B
- TOF criteria wider
- 442 can be base peak

QUANT AND SECONDARY MASS METHOD 8270



Can do Selected Ion Monitoring (SIM)

- No qualitative ion criteria for SIM
- Do not need to tune (for SIM) → assumes SIM for all analytes

SIM  **MRM**

Can use Hydrogen as a carrier gas

- No tune criteria
- Must meet method QC acceptance criteria



New Quality Control Criteria

- **No closing CCV**
- **Limits is Table 6 (Q) as broad as 13 – 200%**
- **5 – point calibration curve → minimum**
- **DOC per lab, not analyst**



Matrix spikes

- **Data user chooses sample to spike**
- **If spike fails that analyte cannot be reported**

Sample injection

- **split**
- **Splitless**
- **Large volume injector**
- **On-column injection**
- **Use whatever volume works**
 - Must be same for samples as standards





Thank You

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