

Operation of Markes-Agilent AutoGC-FID in 2018 at NJ PAMS

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Rutgers University Photochemical Assessment Monitoring Station (PAMS)



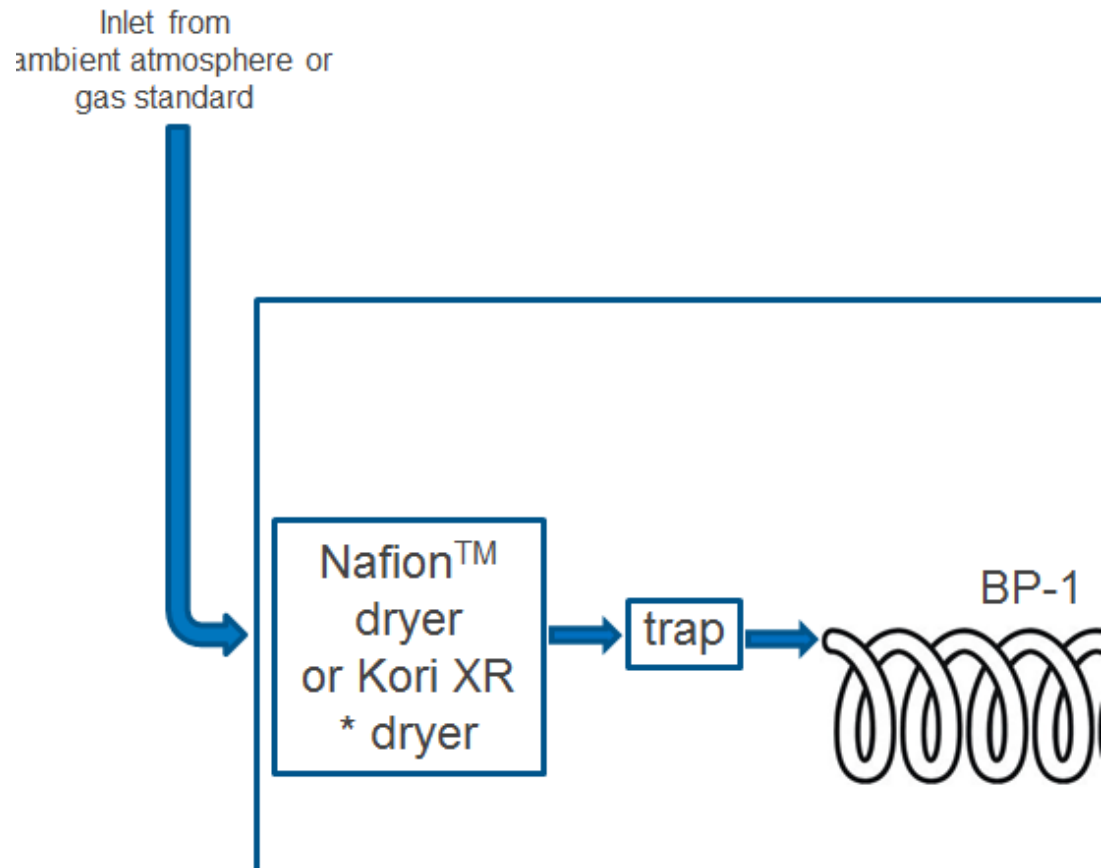
Markes-Agilent AutoGC-FID

- Sample Preconcentrator – Markes
- Gas Chromatograph-Flame Ionization Detector – Agilent
- NJ was early adopter - system delivered March 2018
- Regular data collection started June 2018
- QA/QC not fully implemented



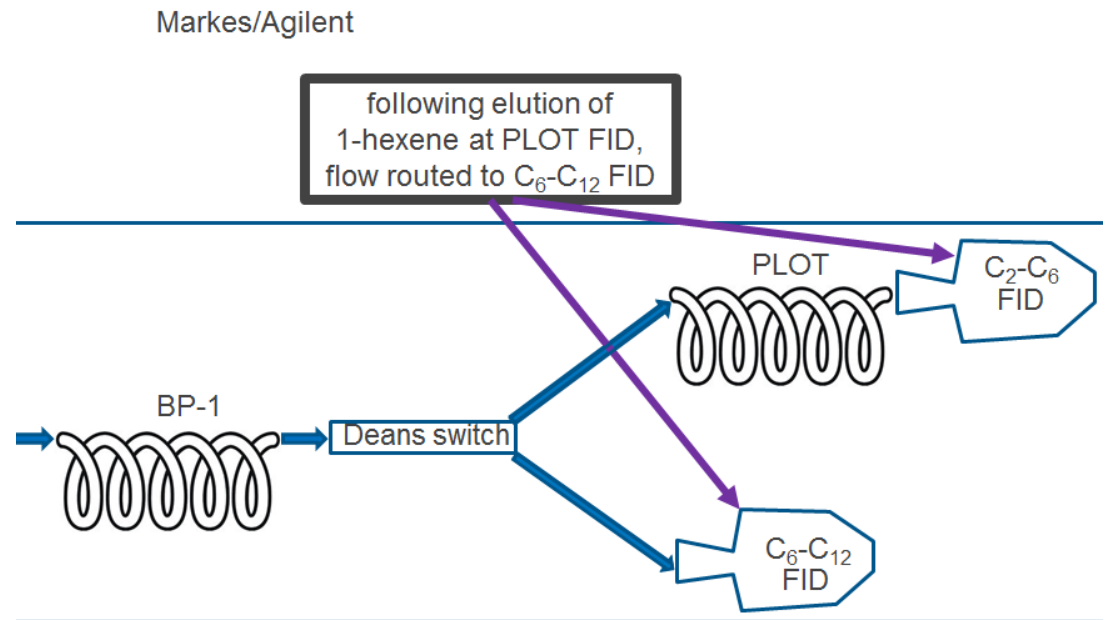
Markes Unity Air Server with Kori-XR

- Ambient sampling inlet
- Directs sample air into Kori-XR to remove water by freezing
- Directs dried sample air into cooled sorbent to trap VOCs
- Stops flow, heats trap to release VOCs
- Backflush (reverse flow) with carrier gas to direct VOCs to GC



Agilent 7890 GC

- Receives pre-concentrated sample air from Markes
- Directs flow into BP1 column to separate C₆-C₁₂
- Directs flow into Dean's switch to divide C₆-C₁₂ and lighter Cs
- C₆-C₁₂ directed to FID1
- Lighter Cs goes to PLOT column to separate C₂-C₆
- C₂-C₆ directed to FID2



Markes-Agilent Issues

- One PC, separate software, no integration between software
- Blanks and standards can be identified in Markes software, but they are treated as routine samples in Agilent software
- Need to manually rename sample folders created by Agilent software to identify QC data
- PAMS TAD recommends staggering schedule of QC checks, need extra effort to rename sample folders
- NJ decided to use H₂ as carrier and fuel gas, so larger capacity H₂ generator was needed

Markes Issues

- Operator can set start time for 1st hour of a sequence, but subsequent sample start times are based on an estimate of the GC cycle time
- Actual sample start times will drift and need to be corrected 1-2 times/week, but the frequency can vary, as often as everyday
- Desorption duration needs to be long enough to remove water in the Kori
- Need to position the sample inlet close to the manifold as possible to reduce stale air in the sample line
- Training needs to be targeted for PAMS operation

Agilent Issues – GC-FID

- Columns – original GC columns supplied by Agilent had problems resolving components
 - replaced with different columns, but results were still not ideal
 - will change test a third set of columns 2019
- H₂ sensor – since autoGC-FID uses H₂ as a carrier, we installed the H₂ safety sensor
 - needs a minimum amount of flow
 - At flows below the minimum, sensor will shut down GC

Agilent Issues - Software

- Software installation is very complicated
- Many different software products and versions – Open Lab, Chem Station, EZ-Chrome, Mass Hunter, Open Lab v2.3
- Each product has different capabilities and limitations – some products do not quantify unknowns
- In Open Lab v1, method file and data files are linked – any attempt to revise peaks and baseline of an individual chromatogram will result in revising the method file and applying revisions to all chromatograms

Agilent Issues – Software (continued)

- PAMS TAD 9.3.3.1: Manual Integration of Chromatographic Peaks – due to differences in peak shape, coeluting peaks and baseline noise, may need to be manually correct peak integration
- Need the capability to apply and save changes to individual chromatogram data files without changing the method file
- Need the capability to generate report files from the data files which can be used for reviewing and validating data and for generating AQS transactions
- Training needs to be targeted for PAMS application, Agilent software support was slow to respond and unfocused

NJDEP QA/QC in 2018

- Weekly blanks using dedicated canisters filled with N₂ obtained from the headspace of liquid N₂ tank
- Weekly checks of concentrations and retention times using PAMS Retention Time Standard (RTS)
- Monthly checks of 10% of the PAMS RTS concentrations
 - Markes has a setting for injection of 80 cc instead of 800 cc of the PAMS RTS
- Results
 - Blanks: 2.92 to 14.88 total ppbC
 - Recovery: 74.9% to 138.9% (w/o acetylene, n-dodecane, α -pinene & 1,2,3-trimethylbenzene)

QA/QC Required in 2019

ACTIVITY	FREQUENCY	STANDARD	DETAILS
ICAL, Initial Calibration	Before & After season	ICAL standard, benzene and propane	3 points, 1-25 ppbC
System blank	Daily	Nitrogen	< MDL or 0.5 ppbC
Secondary Source Calib. Verification SSCV	Weekly	can use PAMS Retention Time Standard	$\pm 30\%$ of concentrations of benzene and propane
Continuing Calibration Verification, CCV	Daily	Can use PAMS Retention Time Standard	$\pm 30\%$ of concentrations of target compounds
Retention Time Standard	Weekly	Can use PAMS Retention Time Standard	Within established retention time windows
Precision check	Weekly, replicate of CCV	Can use PAMS Retention Time Standard	Abs. Rel. % diff $\leq 25\%$ for each target

Other Issues

- NJ could use technical support from the Regional EPA office
- NJ paid for additional training from Markes and Agilent separately after determining that the training that came with equipment was not targeted to PAMS operation
- NJ will use ERG, EPA contract laboratory to analyze for PAMS carbonyls, an extra \$20,000 annually
- Overall, good data in 2018, but acetylene, α -pinene recovery not better than older autoGC-FID
- NJ waiting on software update, more focused training

Acknowledgements, Discussion

- Thanks to Pete Furdyna, NYSDEC
- NJ PAMS operator: Joshua Ray
- NJ PAMS data reviewer: Rudy Zsolway
- Questions
- Comments