Novel Applications of GC-AED for New Element Detection or Element Optimization with Gas Chromatography

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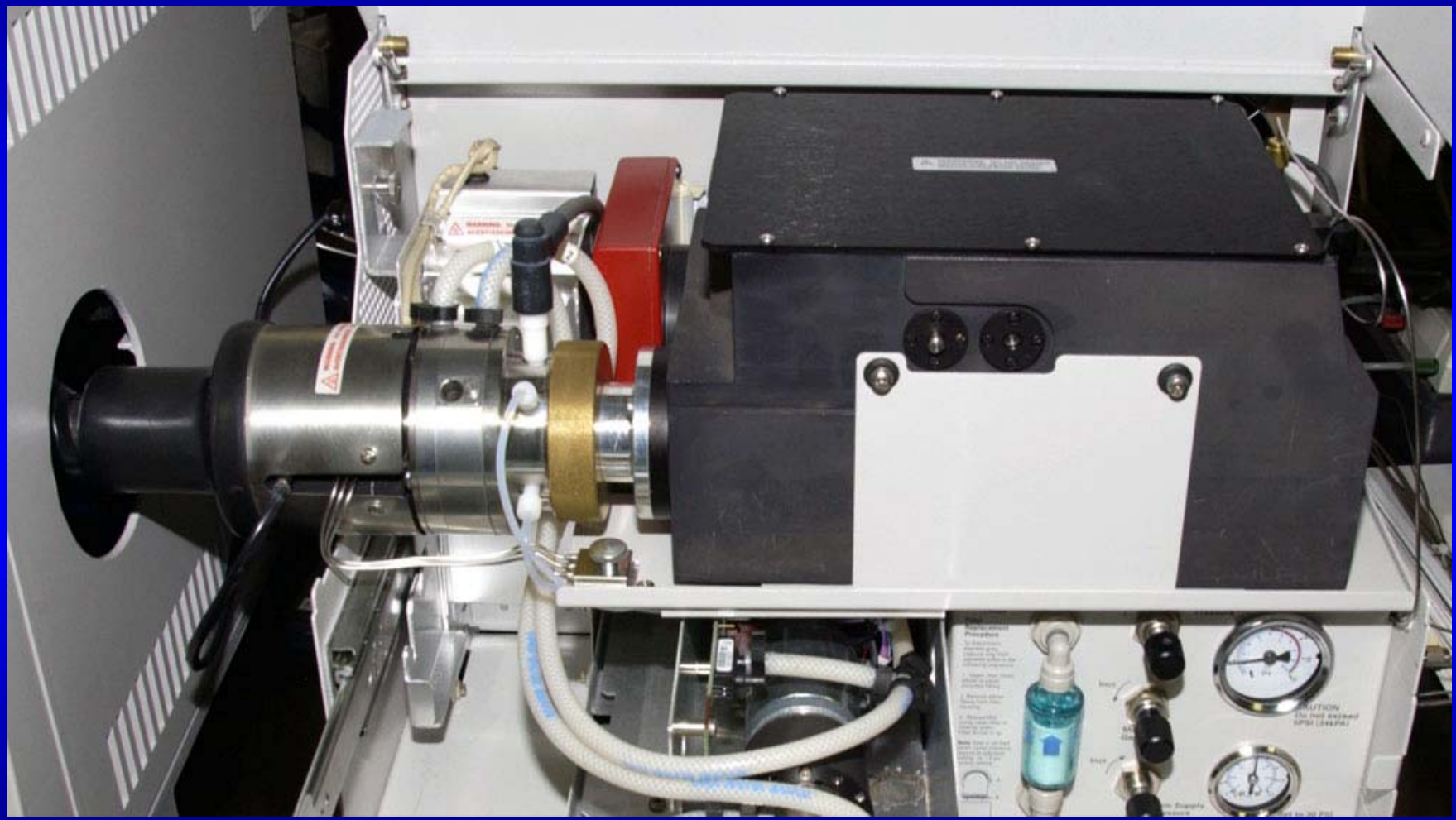
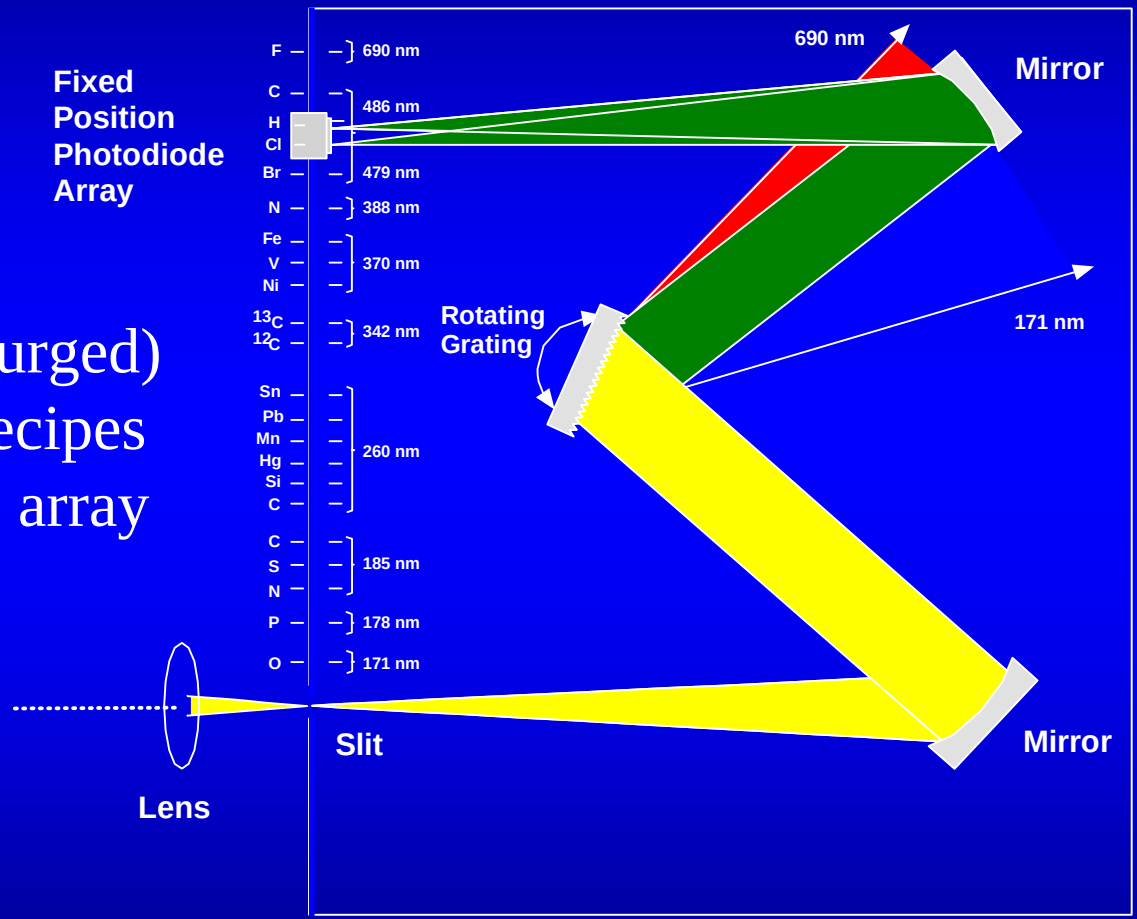
Agilent / JAS Atomic Emission Detector for Gas Chromatography



AED Spectrometer and Diode Array

- ~170 to 800 nm (N2 Purged)
- 329 Diodes Used in Recipes
- 20-26 nm imaged onto array



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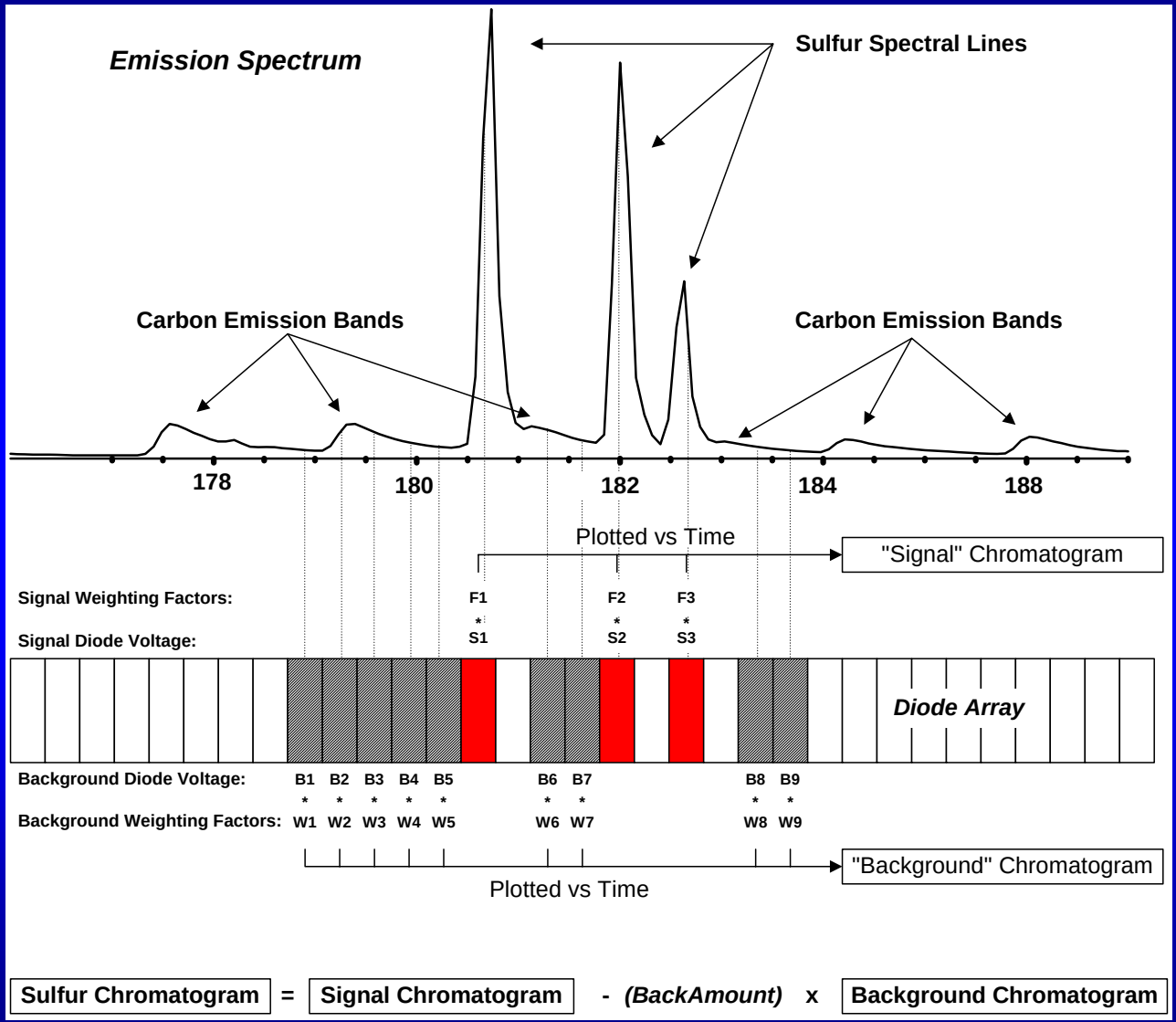
AED Signal Generation



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G2350A AED Standard* Recipes

Carbon	Hydrogen	Nitrogen	Oxygen	Sulfur	Chlorine
179, 193, 248, 264, 496, 834	486, 656	174, 388	171	181	479, 837
Bromine	**Fluorine**	**Iodine**	**Phosphorus**	**Boron**	**Selenium**
478, 827	690	183, 206	178, 186	250	196
Arsenic	**Germanium**	**Lead**	**Manganese**	**Mercury**	**Silicon**
189	265	261, 406	259	254	252
Tin	**Iron**	**Nickel**	**Vanadium**	**Antimony**	**Tellurium**
271, 301, 303, 326	302	301	292	218	208

*** Excludes stable isotope recipes**

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Why are Custom Recipes Needed?

- Add new element capability
- Extend existing element to alternate wavelength – compatibility with other recipes
- Re-optimize for sensitivity / selectivity / linearity
- Different background/matrix
  - All of the “normal” AED recipes were optimized for selectivity against ***carbon*** interference
  - Water in headspace GC
  - Silicon and Oxygen in Siloxanes
  - Nitrogen and/or oxygen in gas analysis

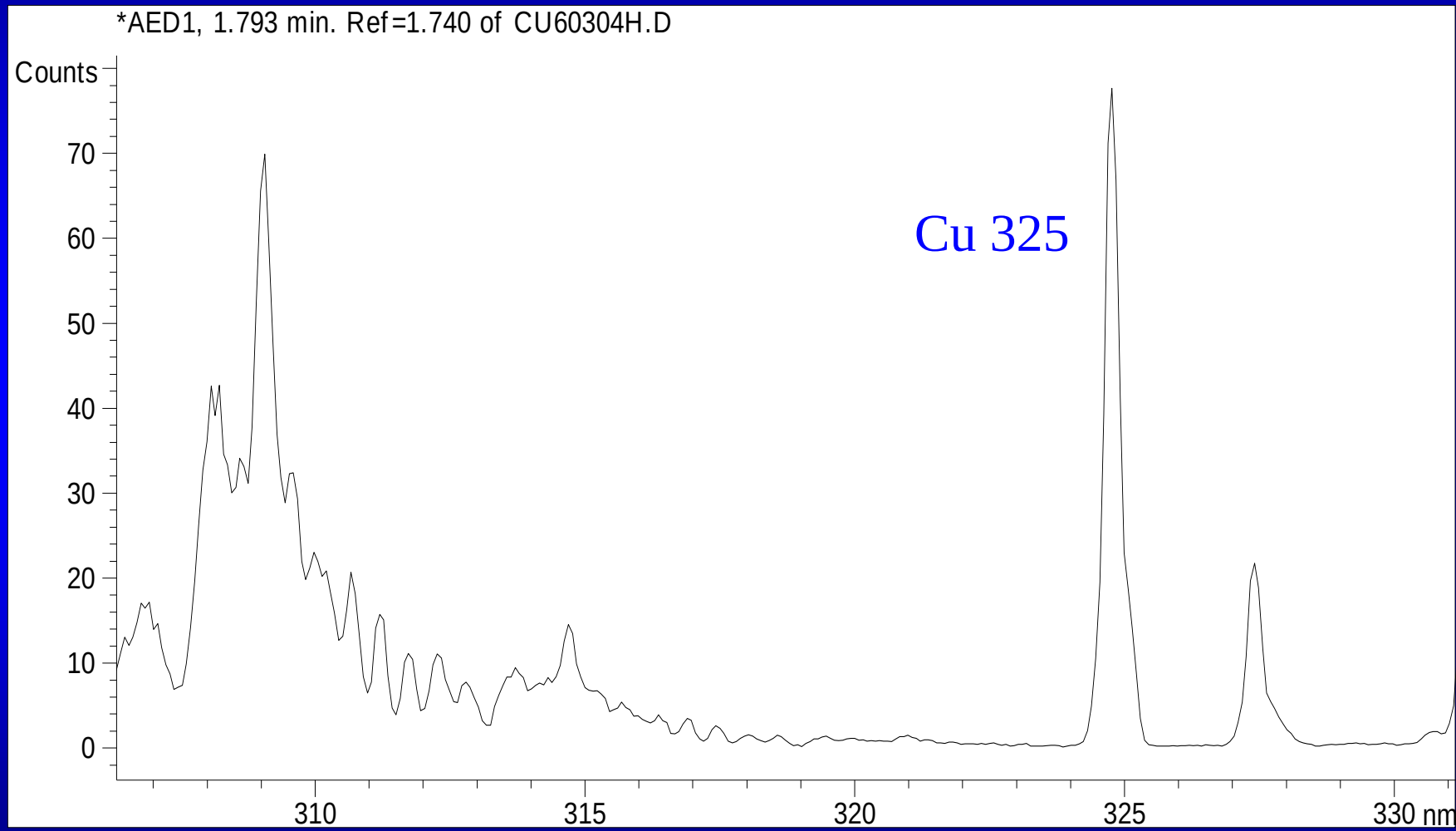
AED Recipe Creation

- HP 5921A AED
  - Simple recipes
  - Custom recipes could be created by the end user
  - Included as Part of AED “Pascal” ChemStation
- G2350A AED
  - Much more sophisticated recipe creation
  - Undocumented “Windows” GC ChemStation Recipe Creation Macro
  - Not distributed to end users

Example Custom Recipe
Copper 325

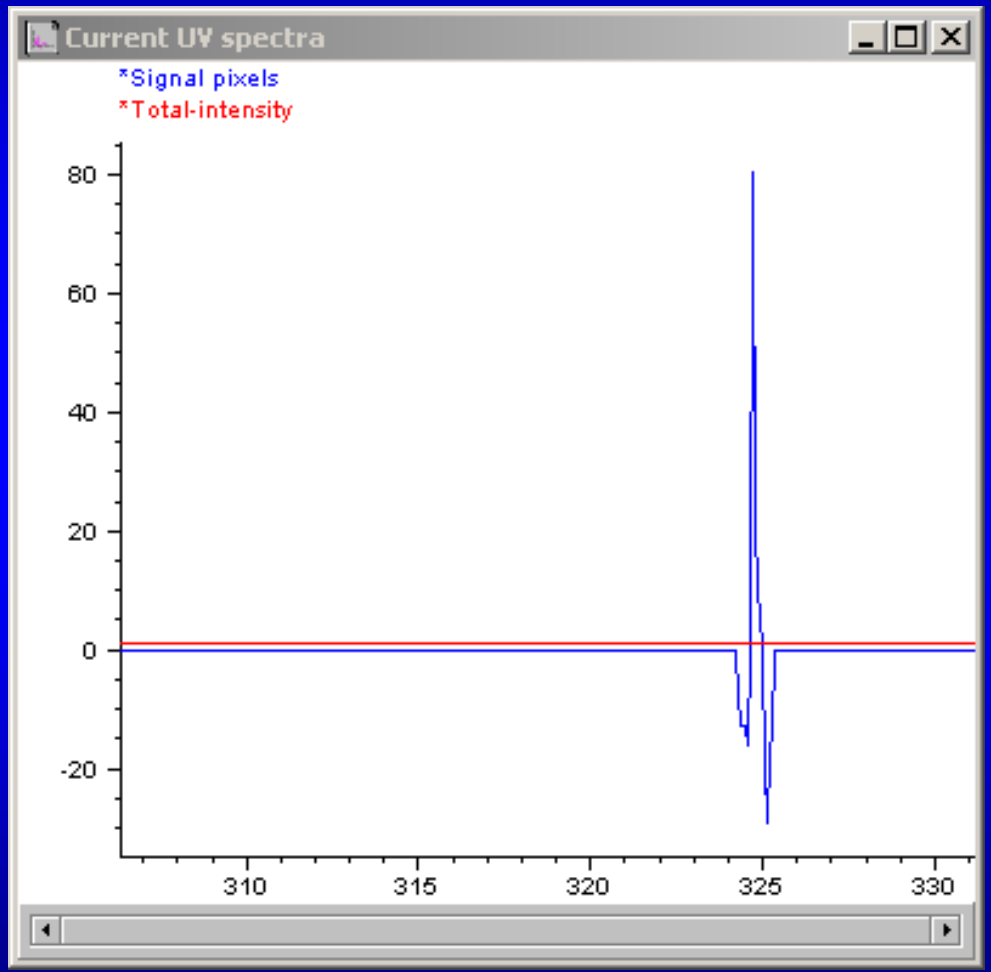
- Used Cu (II) Trifluoroacetoacetonate metal complex as the element standard
- Used n-Paraffin mix (C10, C12, C14, C16) for interference peaks
- Selectivity optimized against Carbon interference (Hydrocarbon Application)

Spectrum at Cu TFA Ret. Time



Create the Recipe

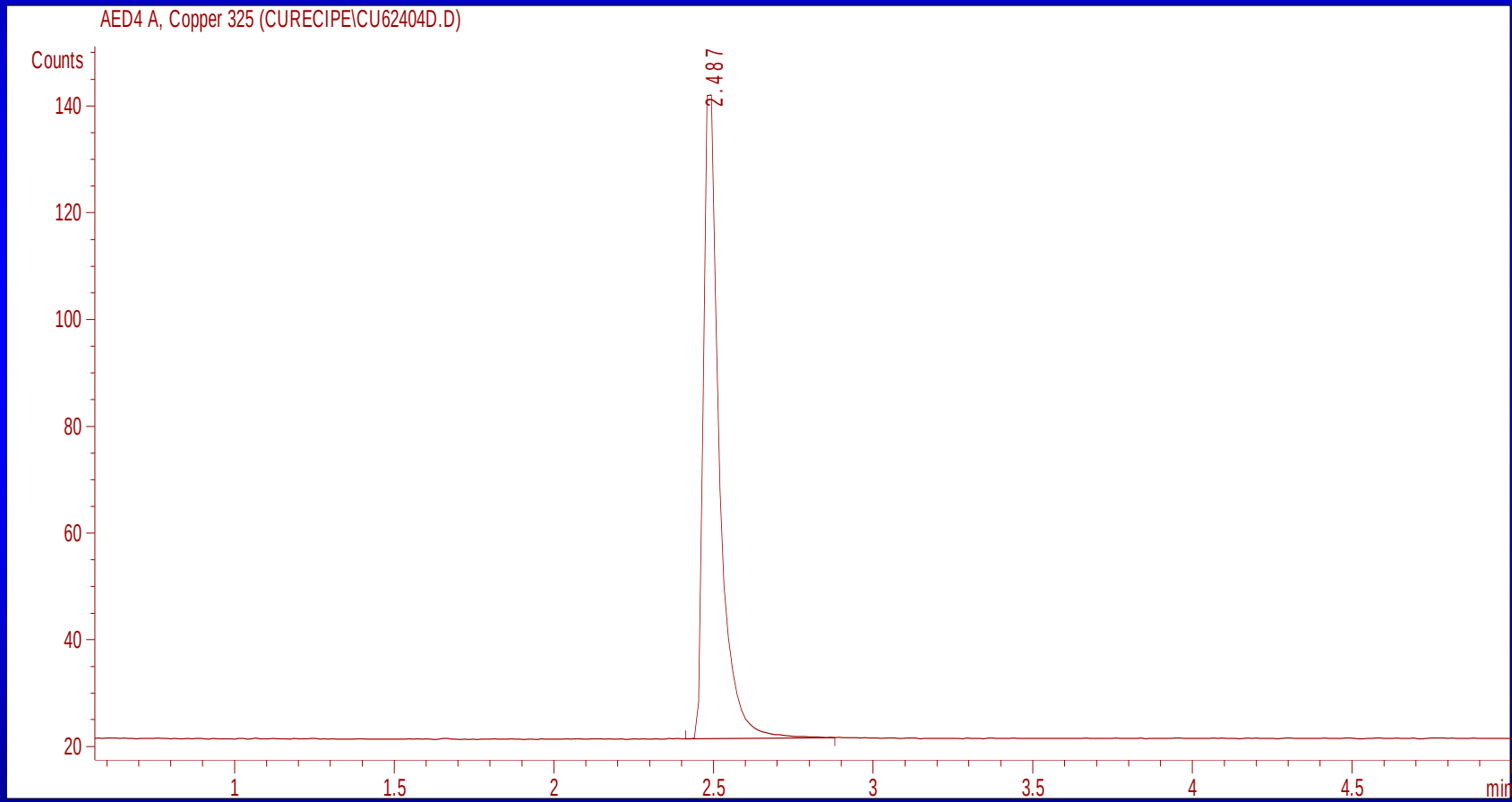
Select and Optimize Signal and Background Diodes



Pixel	Wavelength	Factor
234	324.012	0.00
235	324.088	0.00
236	324.163	0.00
237	324.239	0.00
238	324.314	-7.61
239	324.390	-12.65
240	324.465	-12.65
241	324.541	-16.25
242	324.616	0.00
243	324.692	80.46
244	324.767	22.30
245	324.843	9.26
246	324.918	6.35
247	324.994	0.00
248	325.069	-19.61
249	325.145	-29.21
250	325.220	-17.43
251	325.296	-13.55
252	325.371	0.00
253	325.447	0.00
254	325.522	0.00
255	325.598	0.00

Copper 325 Chromatogram

Cu TFA Standard



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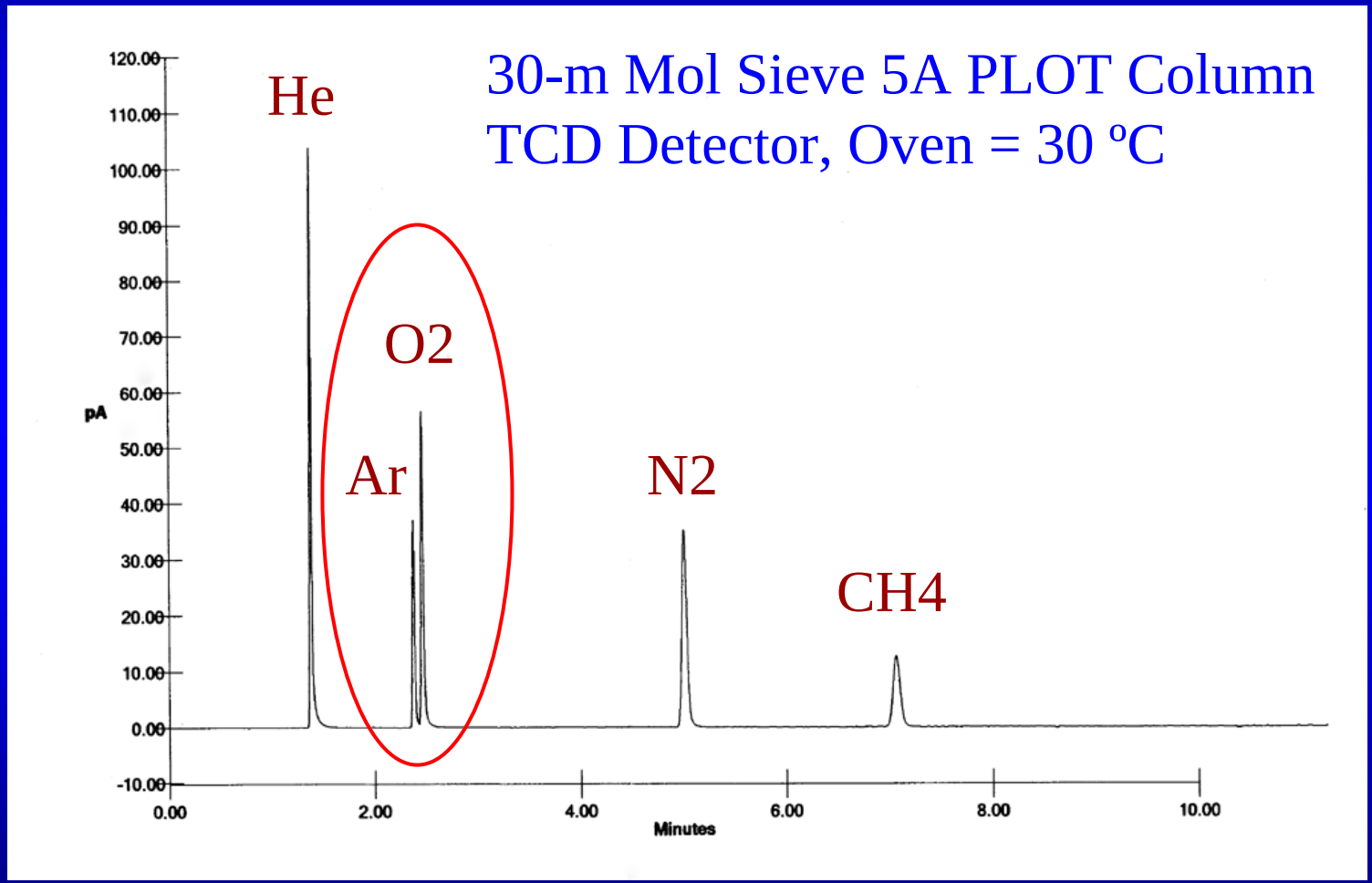
Nobel Gas Applications

- Custom Noble Gas Recipes
  - Xenon 484
  - Argon 750
  - Krypton 760
  - Nitrogen 747
    - can be run with Ar 750 and Kr 760
- Selectivity optimized against Oxygen/Nitrogen interference, *not* Carbon

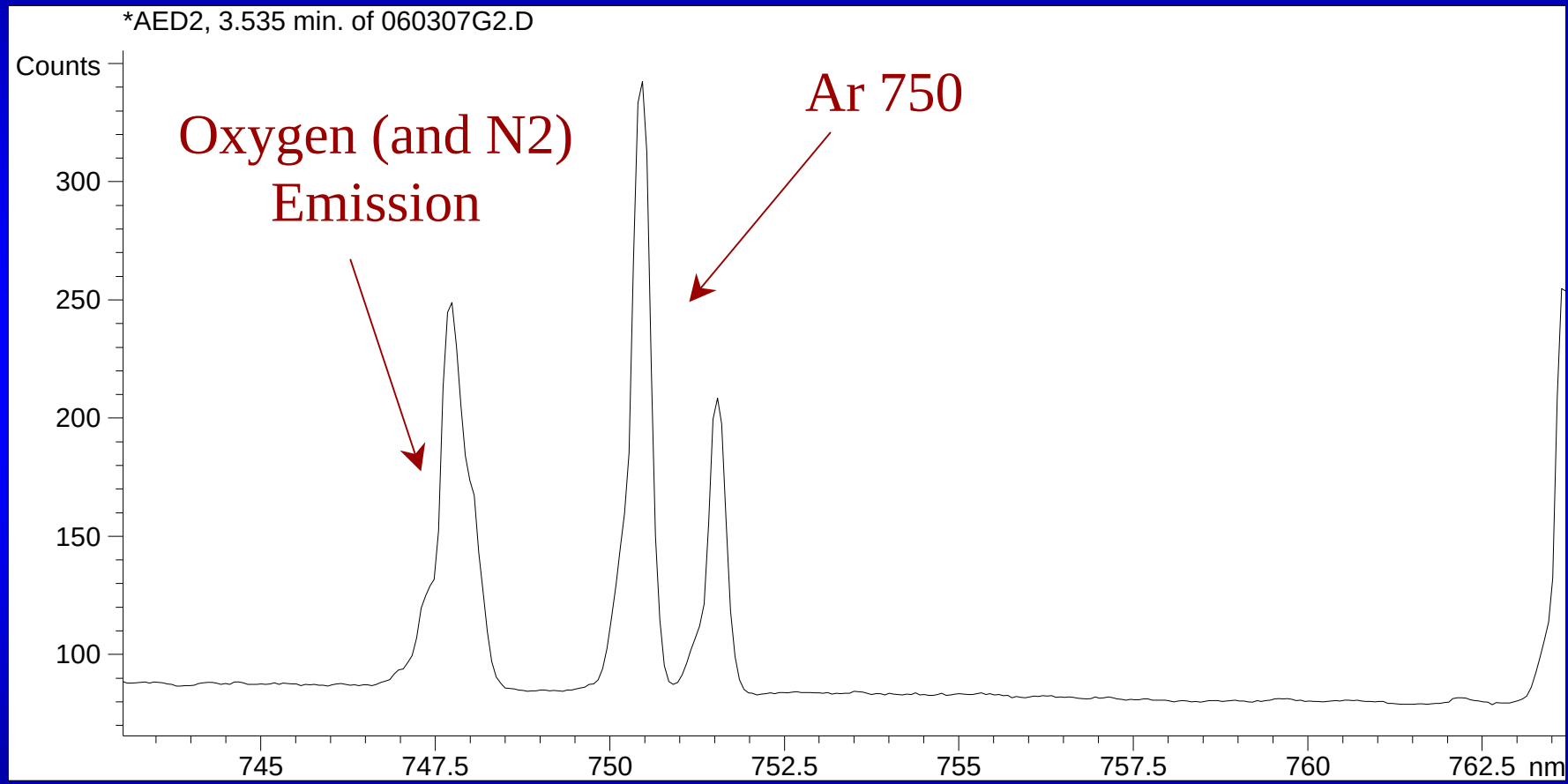
Determination of Ar in Samples Containing Oxygen

- Often done using non-selective detection with a TCD or PDID
  - Difficult GC separation on a Mol Sieve 5A column
  - May require sub-ambient oven temperature
- Alternative is to use the AED with Ar 750 recipe
  - Ar does not have to be separated from O2
  - Use Oxygen reagent gas to reduce the impact of the Oxygen sample matrix

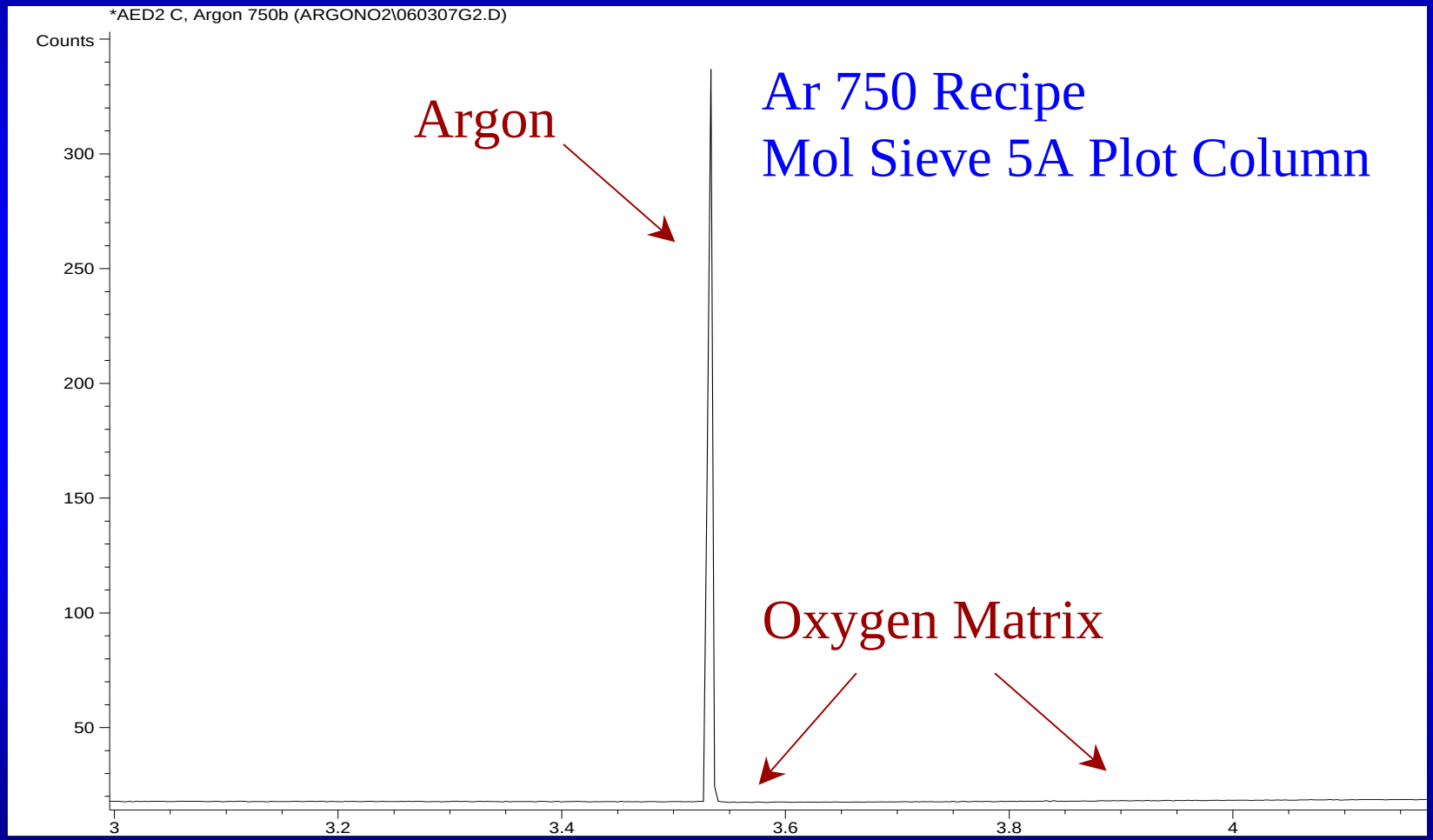
Example Mol Sieve 5A Chromatogram



Argon Spectrum



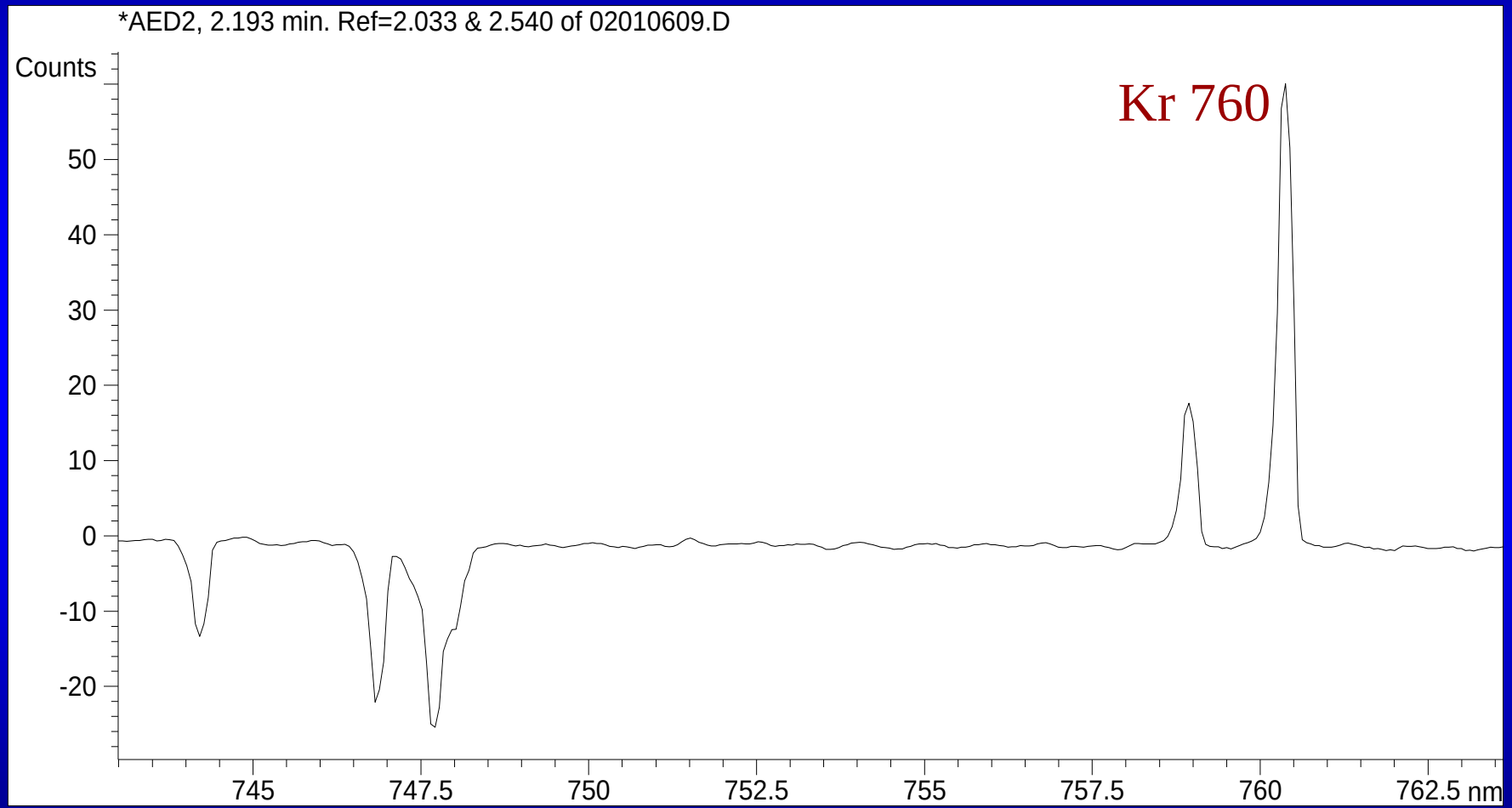
Determination of Argon in Oxygen with AED



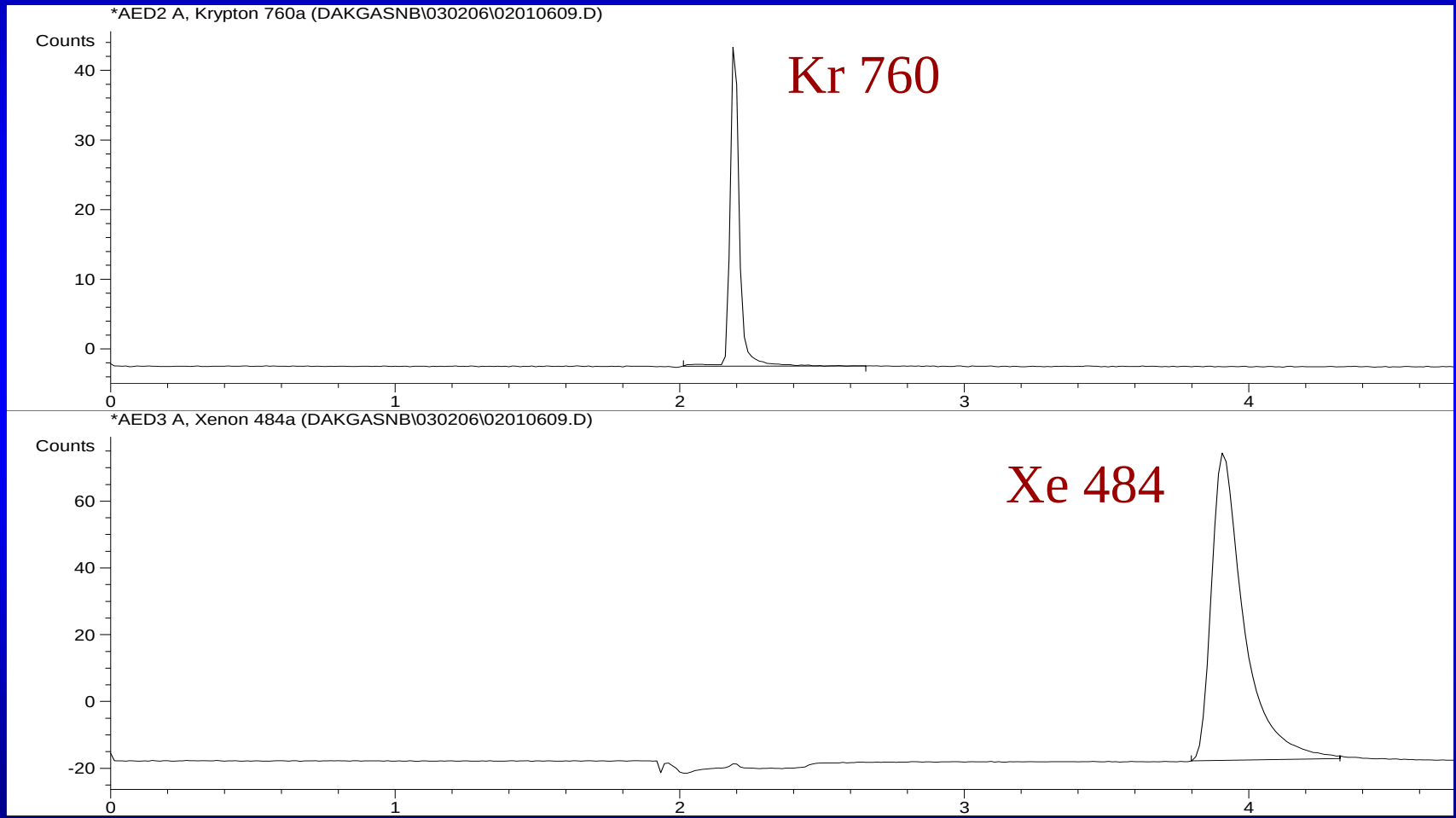
Distillation Application
Determination of ppm Xe and Kr in O2

- Distillation Process
  - Separation of Xenon and Krypton from Oxygen and Argon
    - Oxygen and Argon – overhead
    - Xenon and Krypton - bottoms
  - Xenon and Krypton sold as product
- Kr / O2 separation in the distillation column is critical
  - Requires determination of ppm levels of Kr and Xe in O2

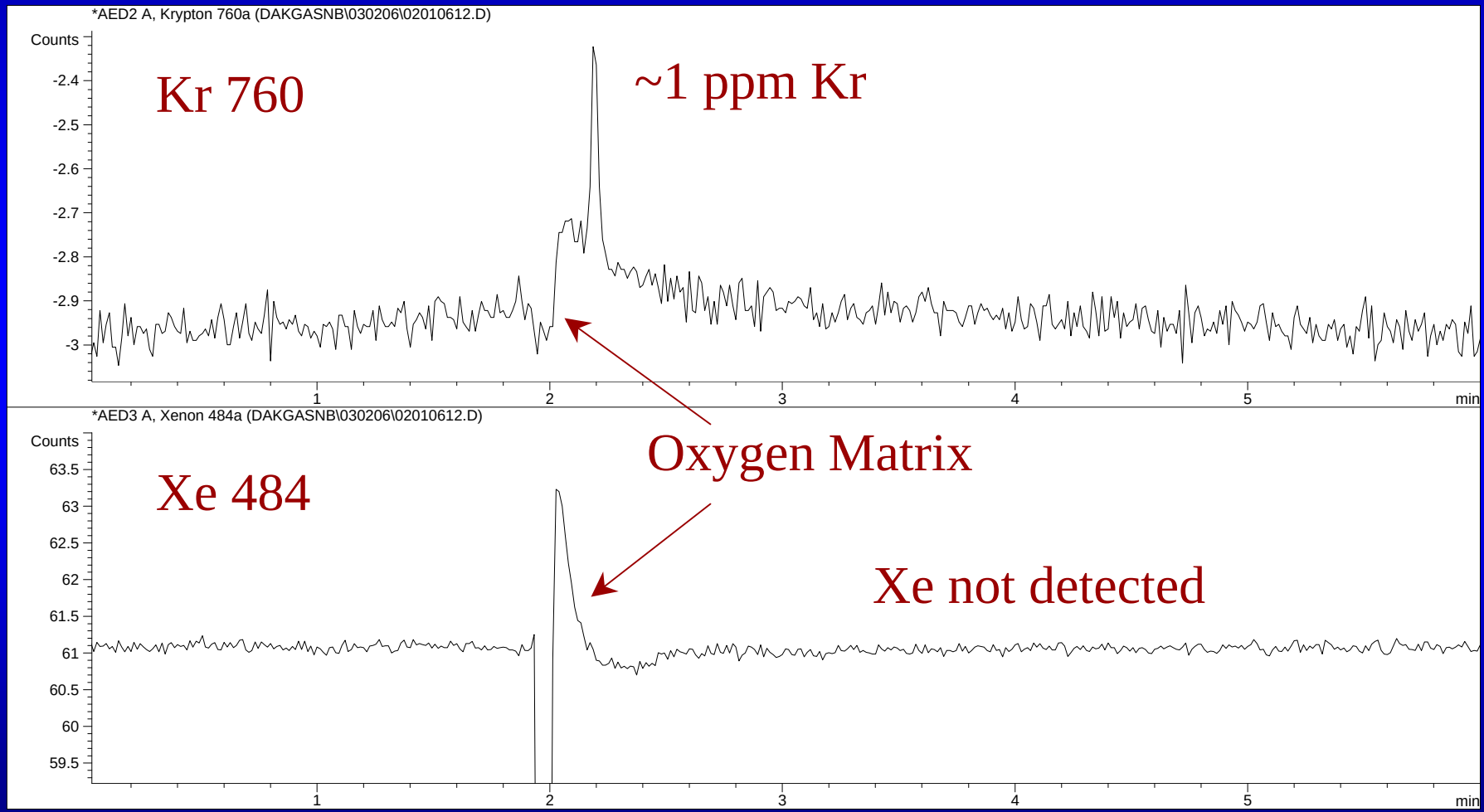
Example Spectrum - Krypton



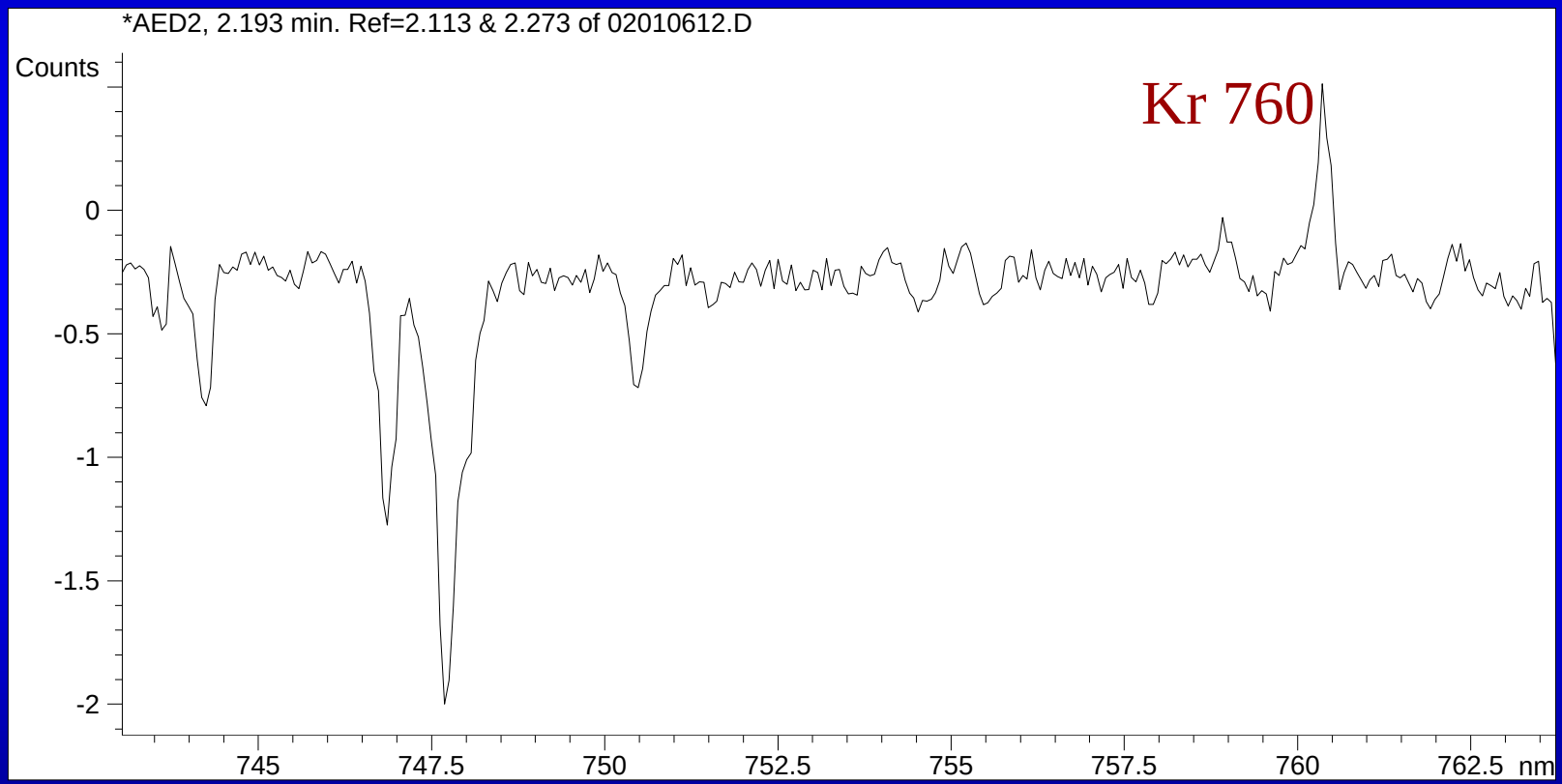
100 ppm Xe and Kr in O2 Standard Chromatograms



Distillation Overhead Sample Chromatograms



Spectrum of Krypton Peak in Overhead Sample



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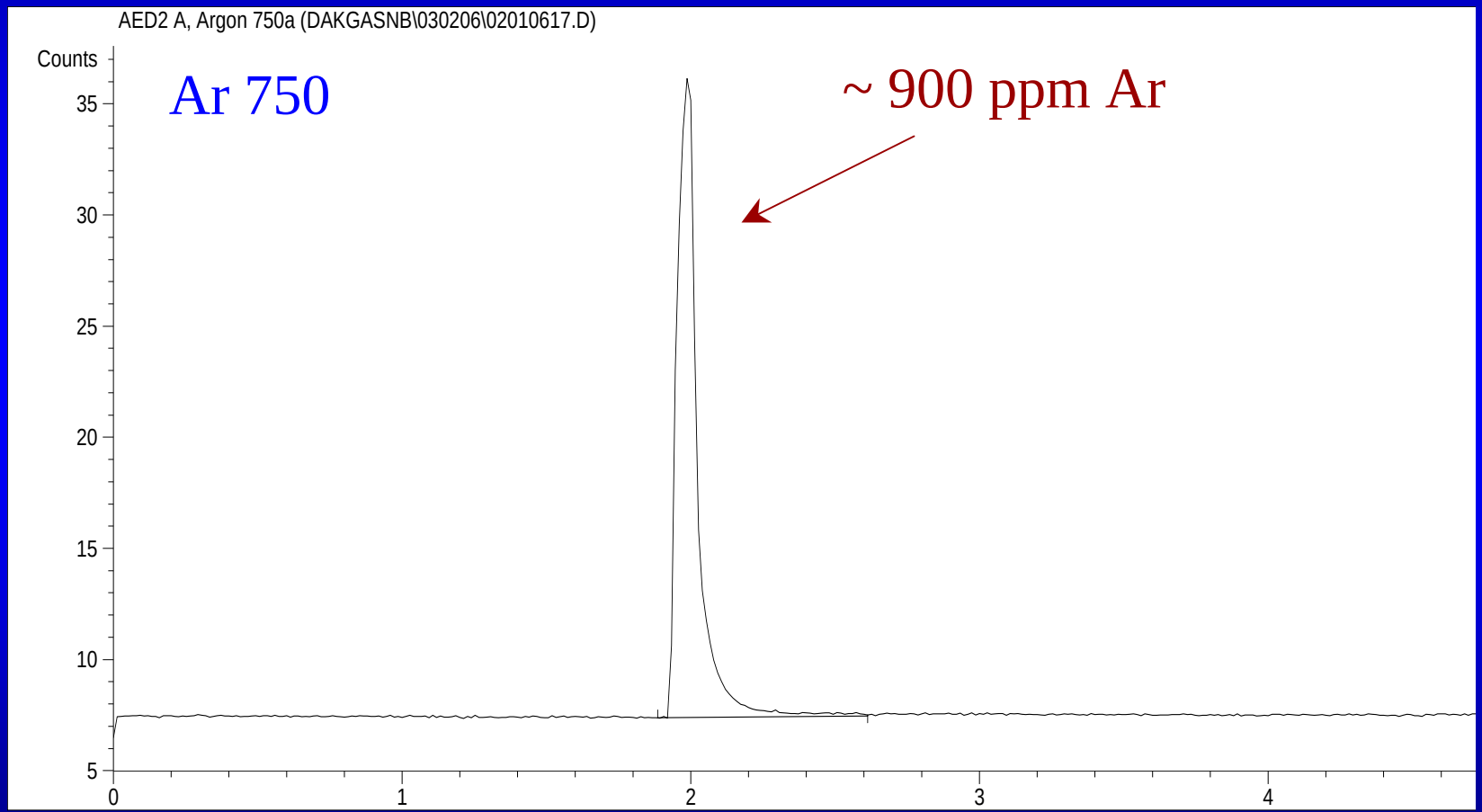
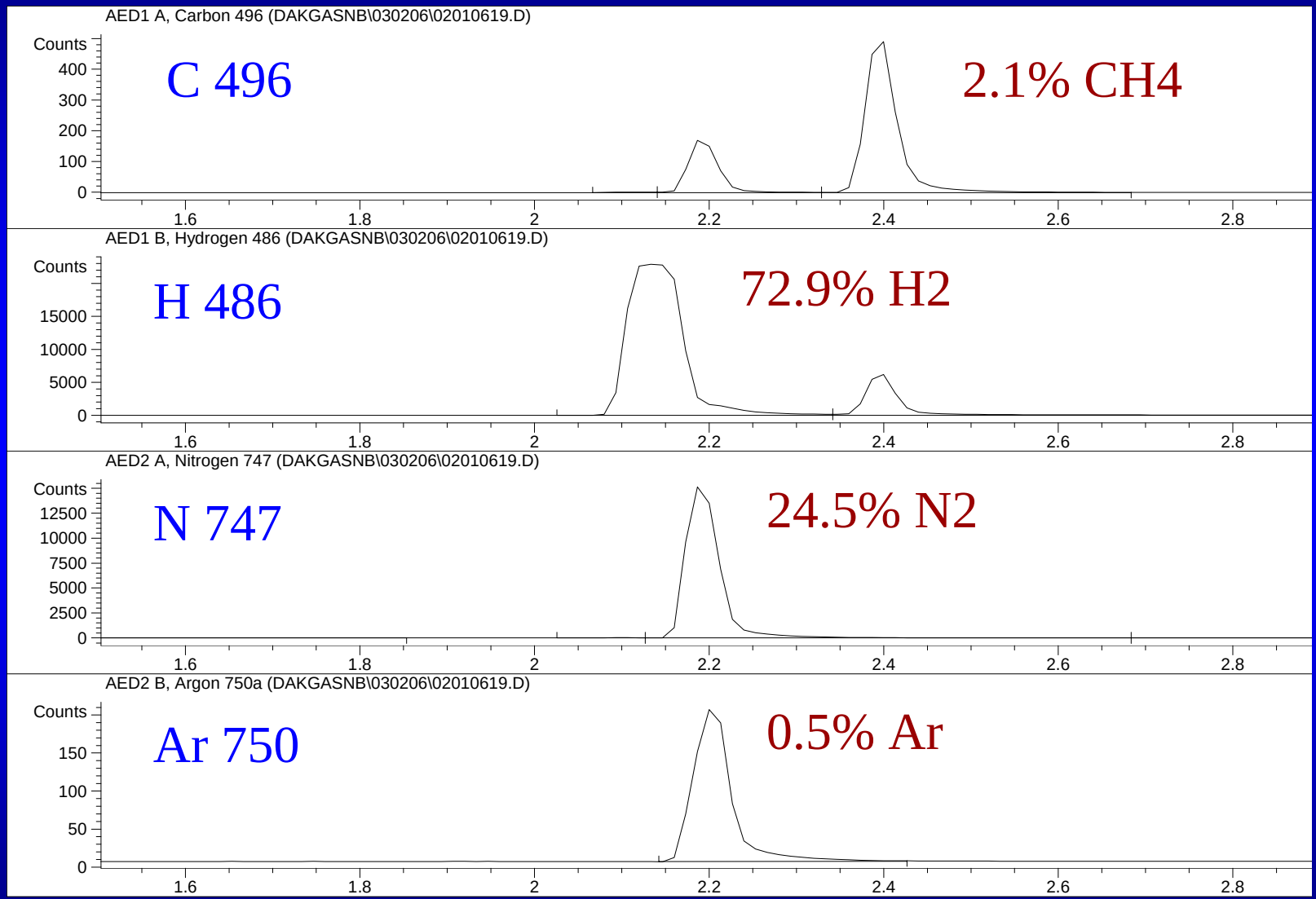
Distillation Overhead Sample Chromatogram



Fast Analysis of Ammonia Plant Process Streams for H2, Ar, N2, CH4

- Selectivity of the AED allows determination of Ar in the presence of co-eluting N2, O2, and other components.

Fast Analysis with Carboxen Column



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Conclusion

The ability to create custom recipes allows the AED to be extended into new application areas and can improve the performance of existing applications significantly.

Acknowledgements

- Dr. Peter Uden, University of Massachusetts
- Bruce Quimby and Paul Dryden, Agilent Technologies
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