

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

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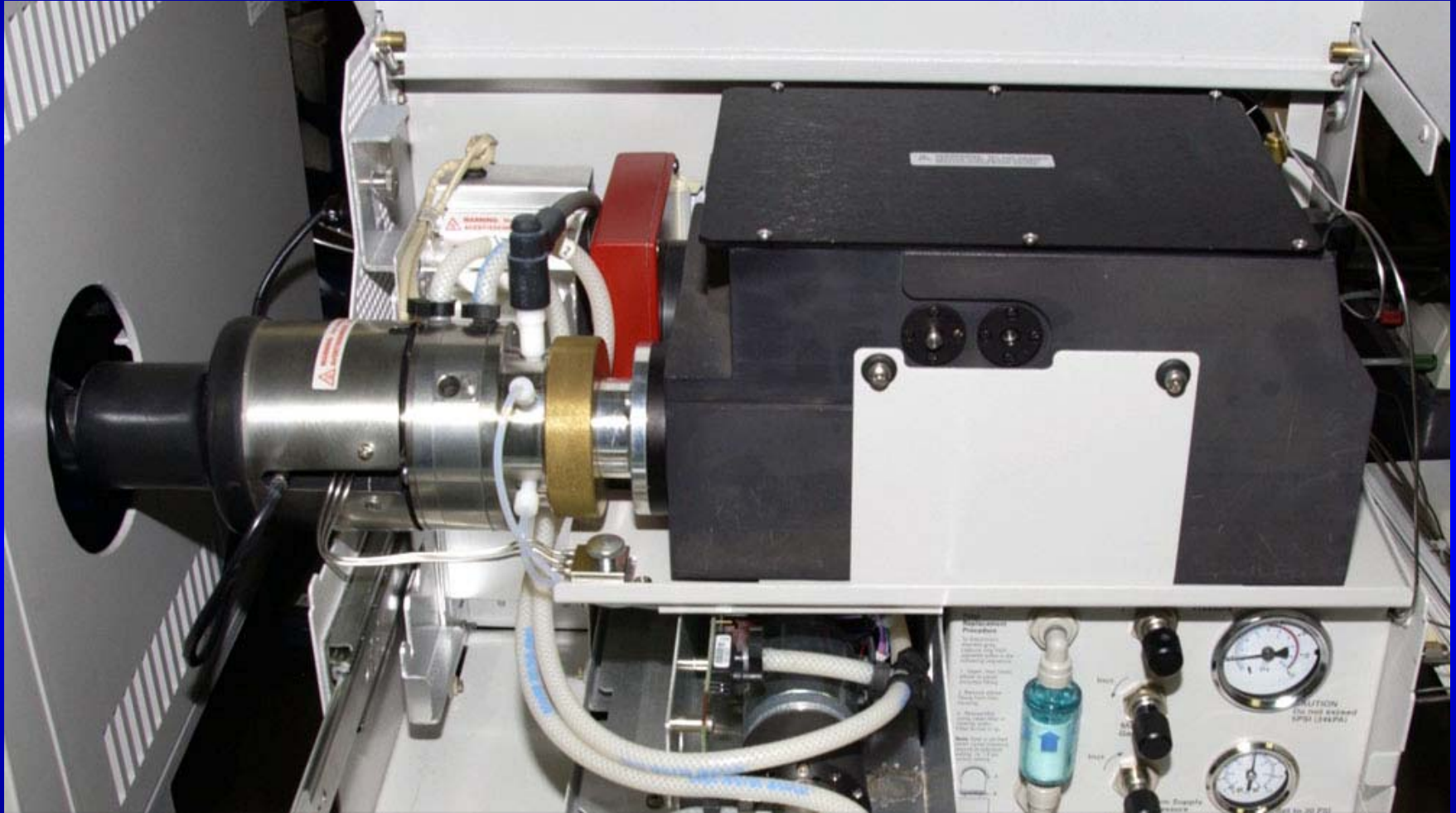
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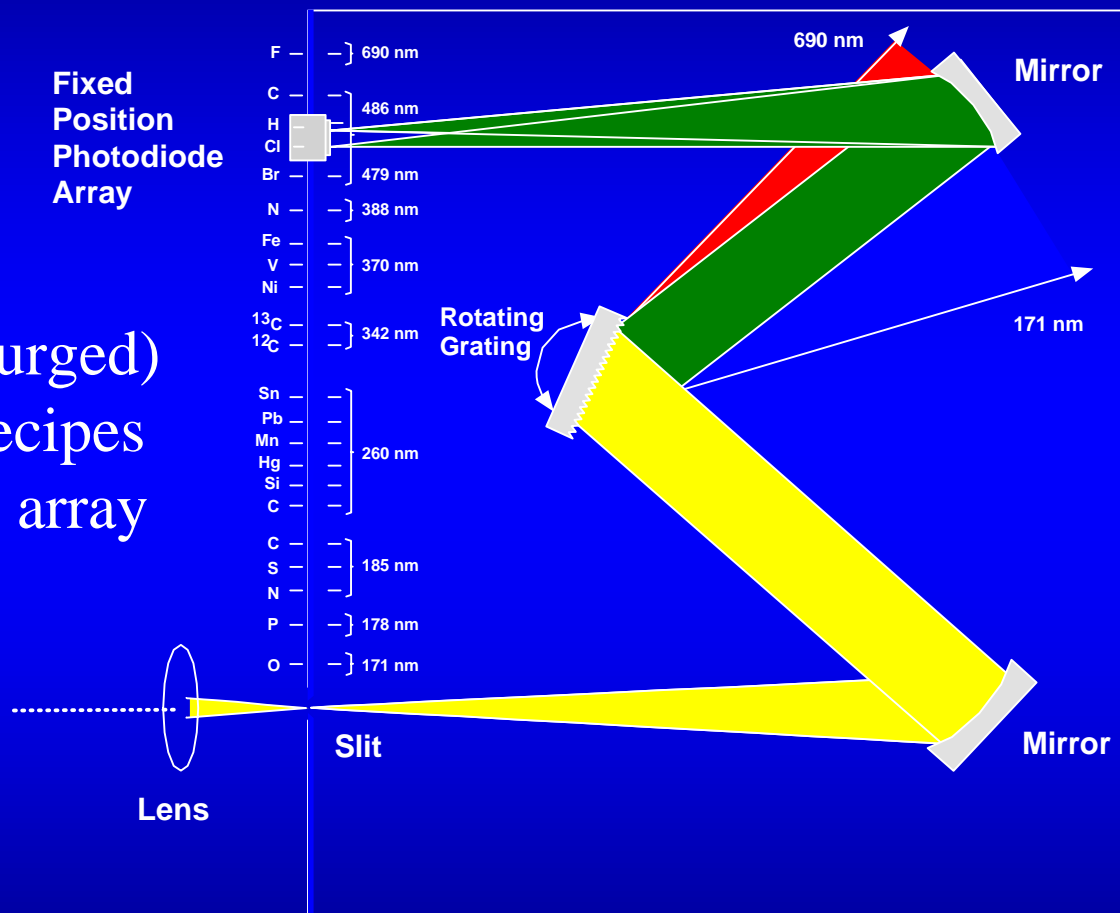
Agilent / JAS Atomic Emission Detector for Gas Chromatography



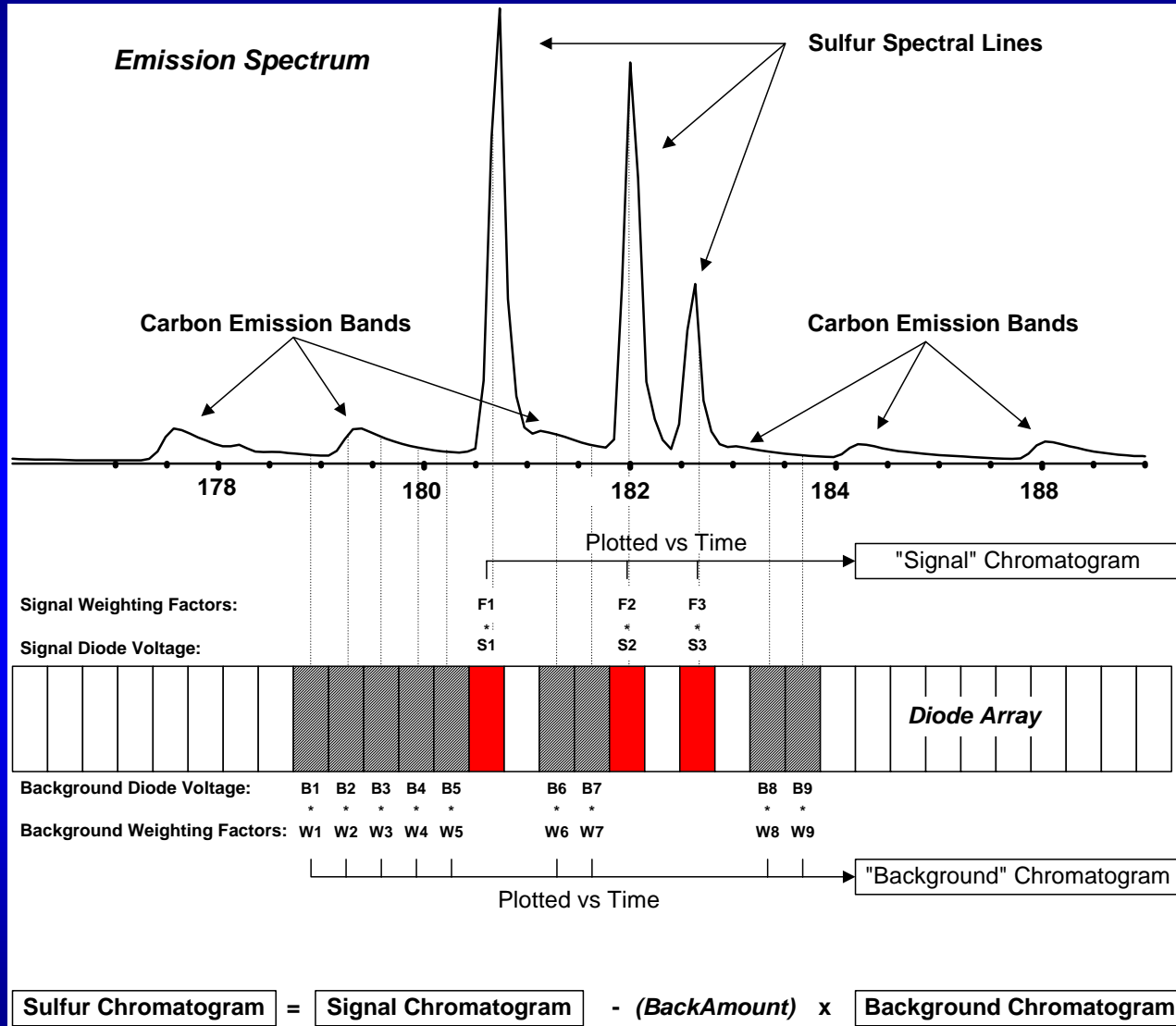
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AED Spectrometer and Diode Array

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



AED Signal Generation



G2350A AED Standard* Recipes

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



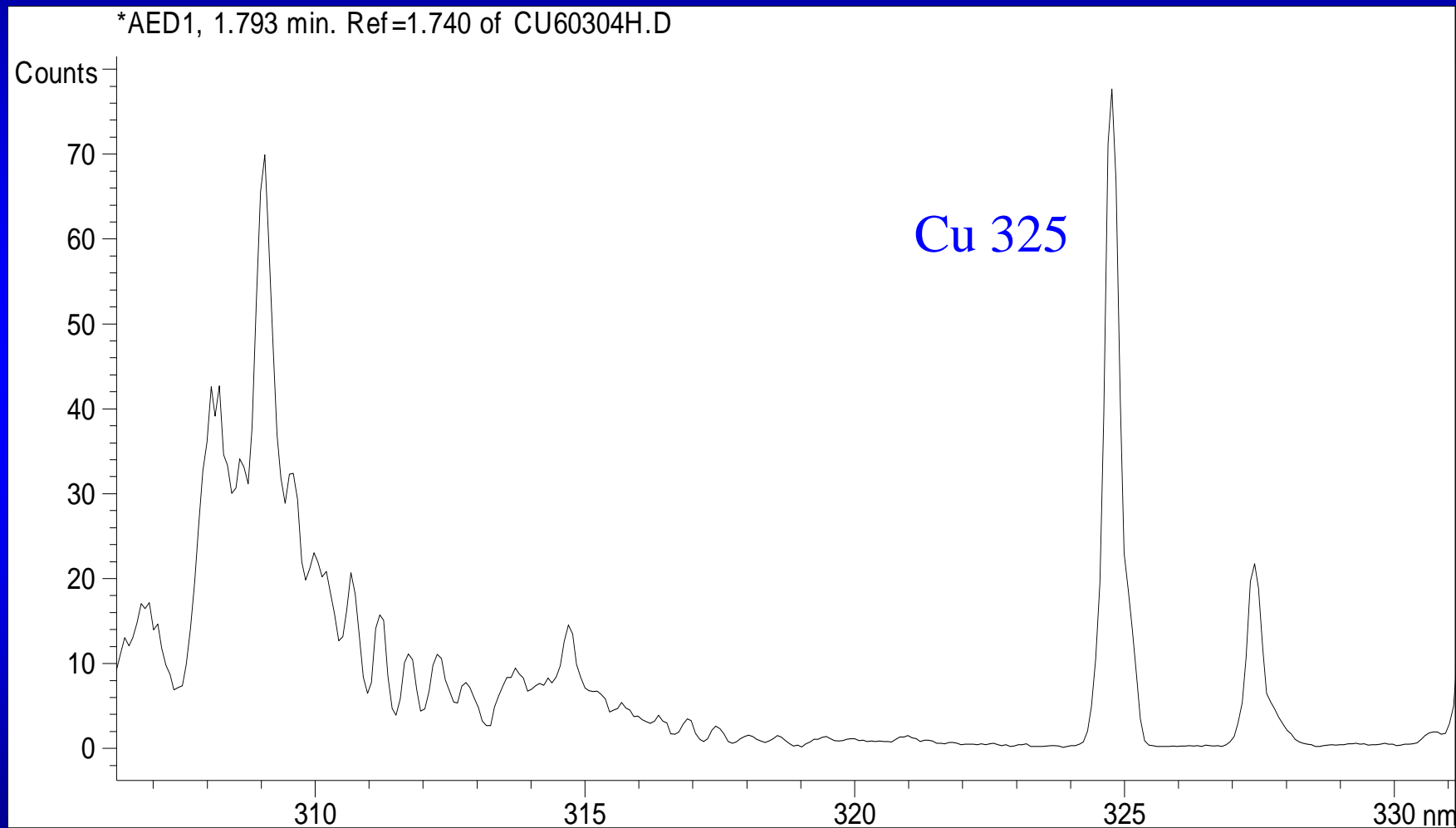
Example Custom Recipe

Copper 325

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

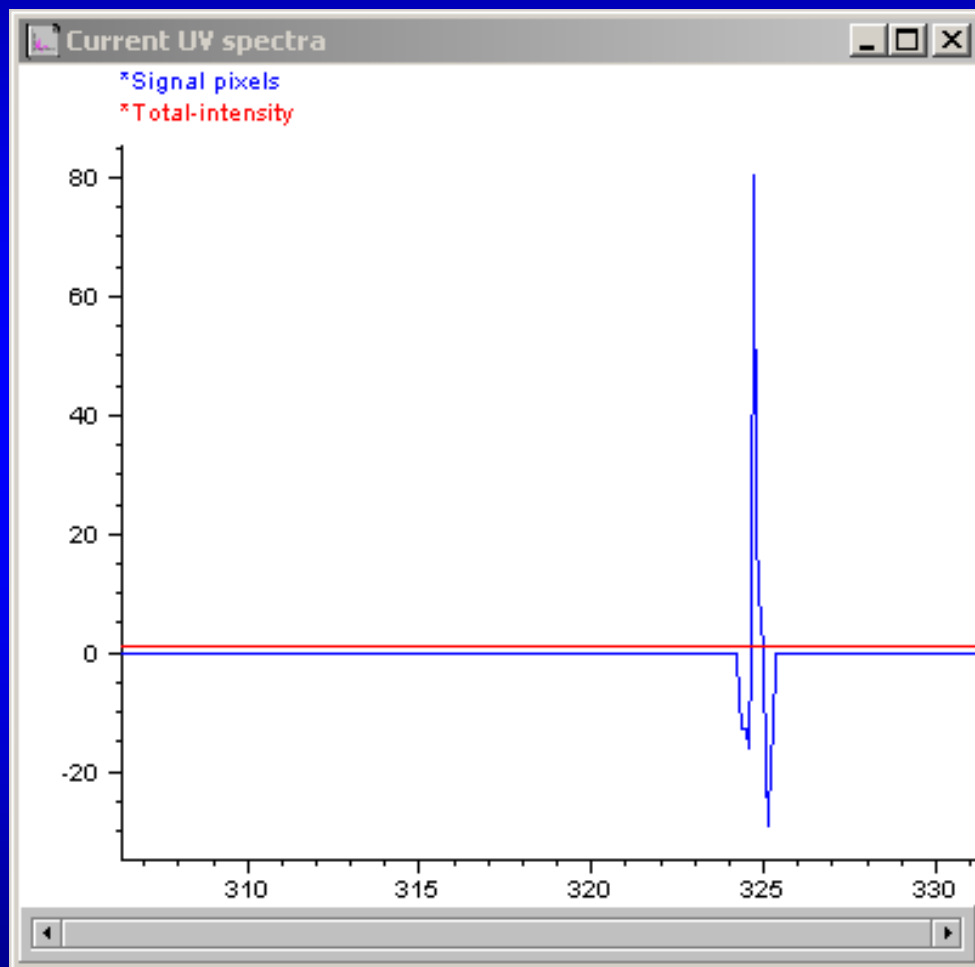


Spectrum at Cu TFA Ret. Time



Create the Recipe

Select and Optimize Signal and Background Diodes

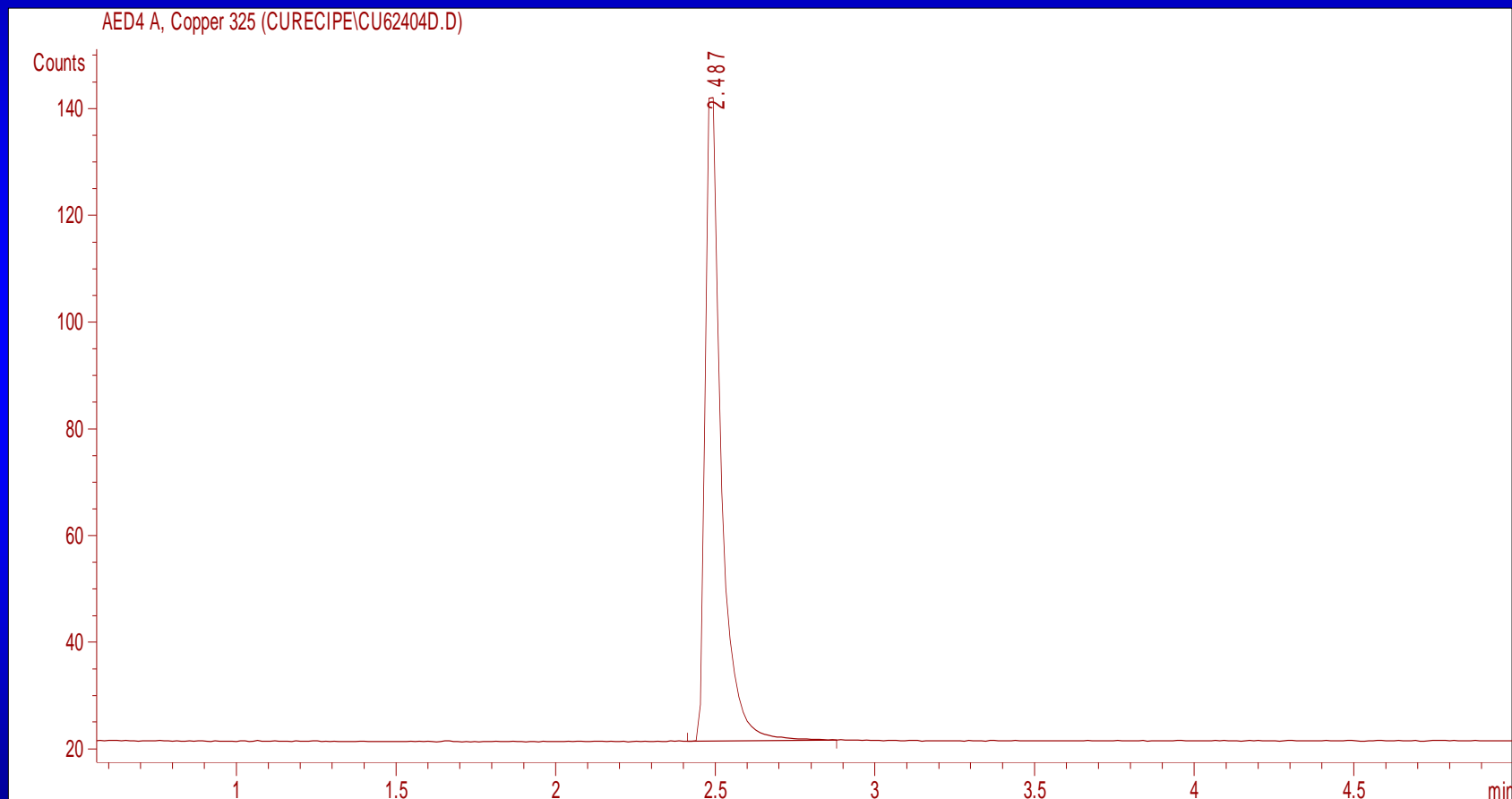


Pixel	Wavelength	Factor
234	324.012	0.00
235	324.088	0.00
236	324.163	0.00
237	324.239	0.00
238	324.314	-7.61
239	324.390	-12.65
240	324.465	-12.65
241	324.541	-16.25
242	324.616	0.00
243	324.692	80.46
244	324.767	22.30
245	324.843	9.26
246	324.918	6.35
247	324.994	0.00
248	325.069	-19.61
249	325.145	-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



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

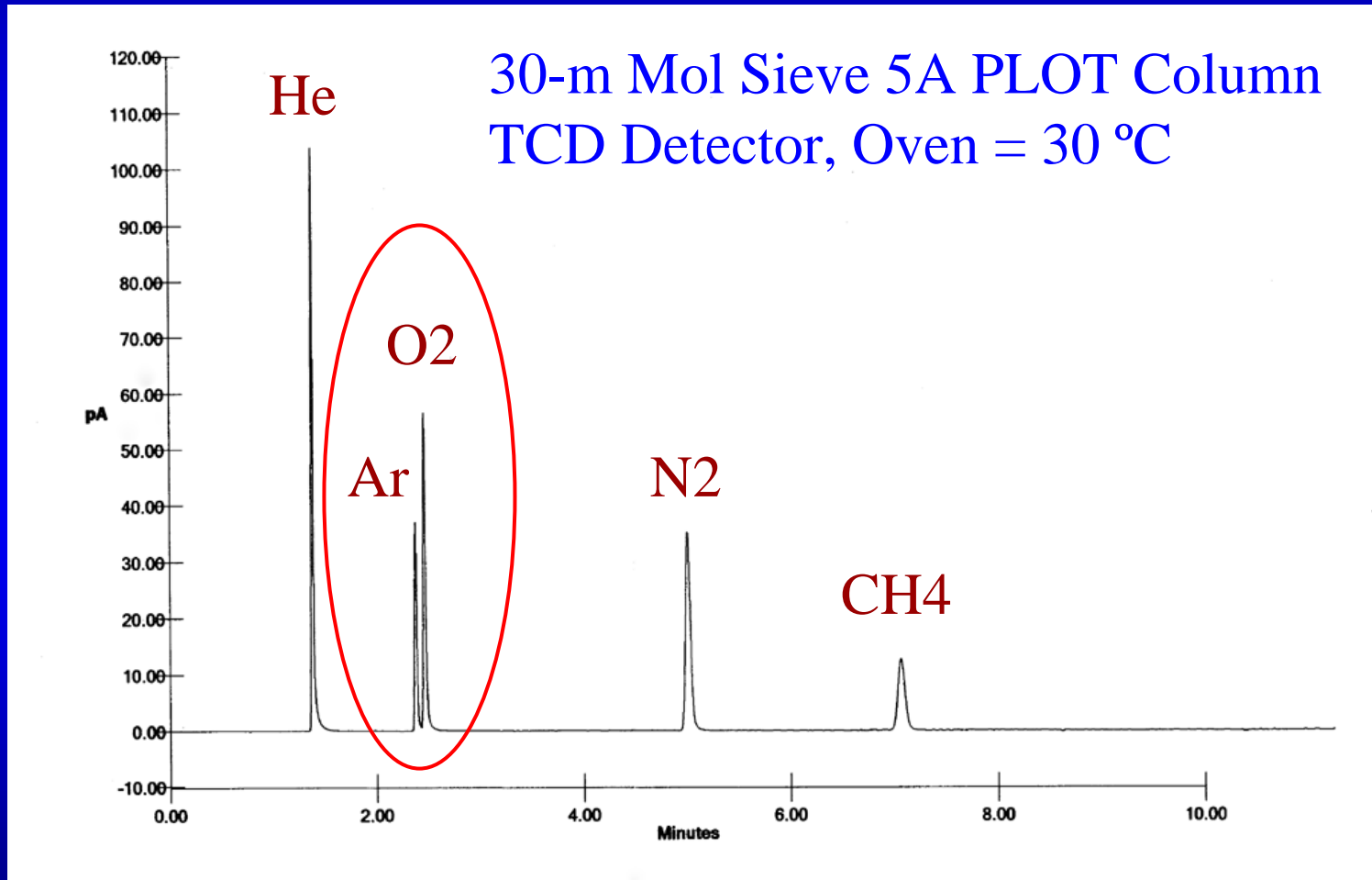


Determination of Ar in Samples Containing Oxygen

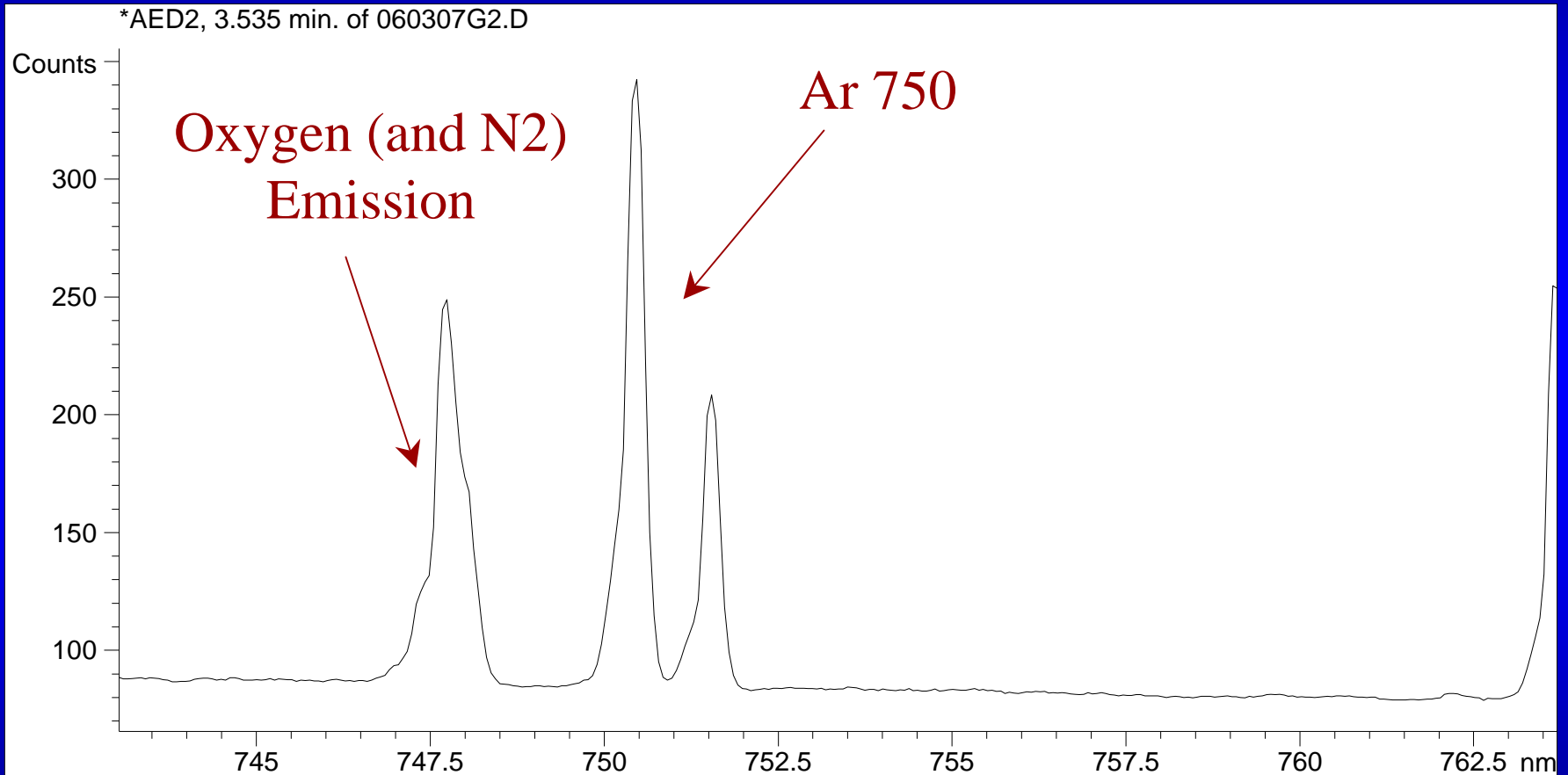
- Often done using non-selective detection with a TCD or PDID
 - Difficult GC separation on a Mol Sieve 5A column
 - May require sub-ambient oven temperature
- Alternative is to use the AED with Ar 750 recipe
 - Ar does not have to be separated from O₂
 - Use Oxygen reagent gas to reduce the impact of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



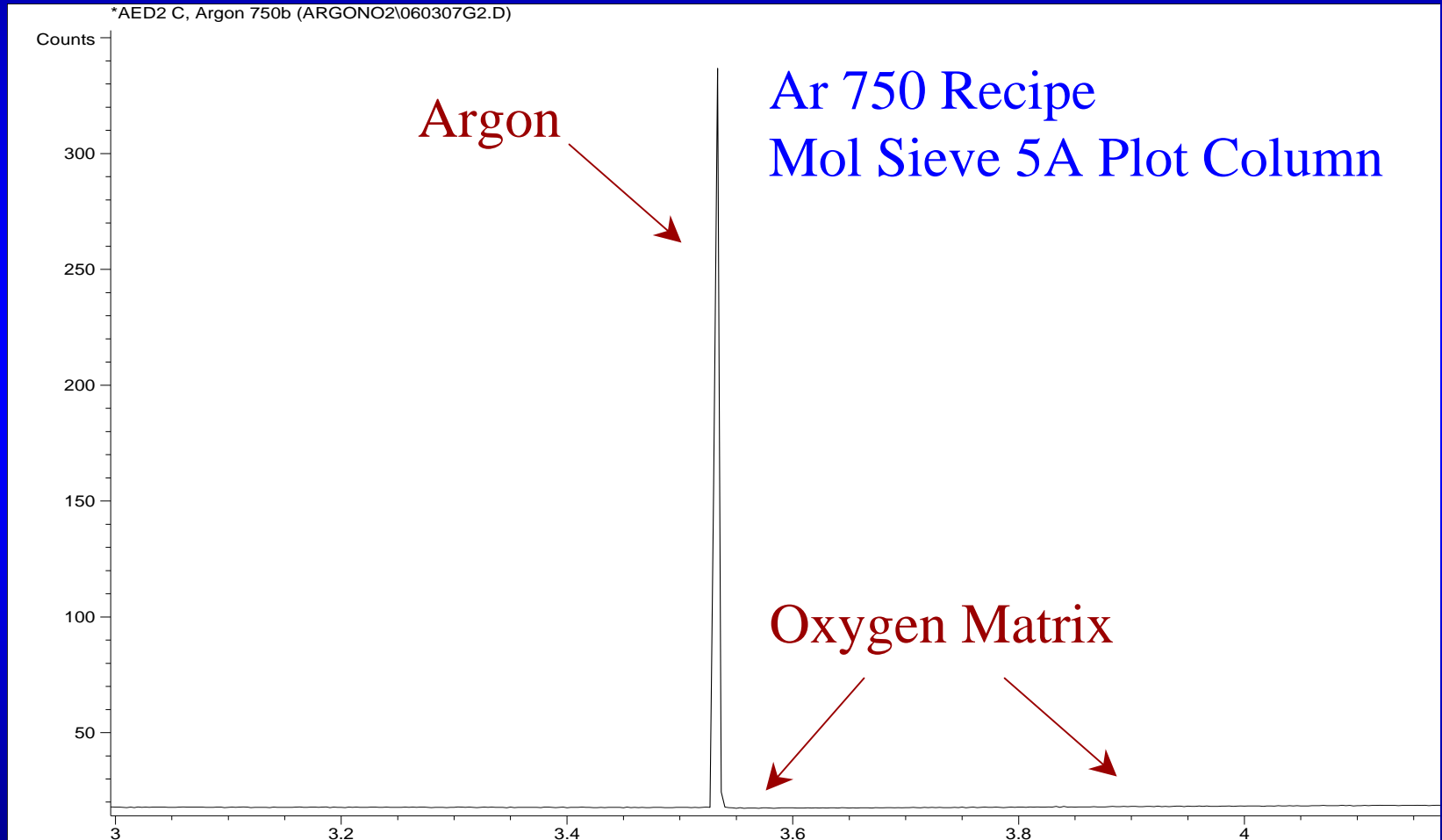
Example Mol Sieve 5A Chromatogram



Argon Spectrum



Determination of Argon in Oxygen with AED



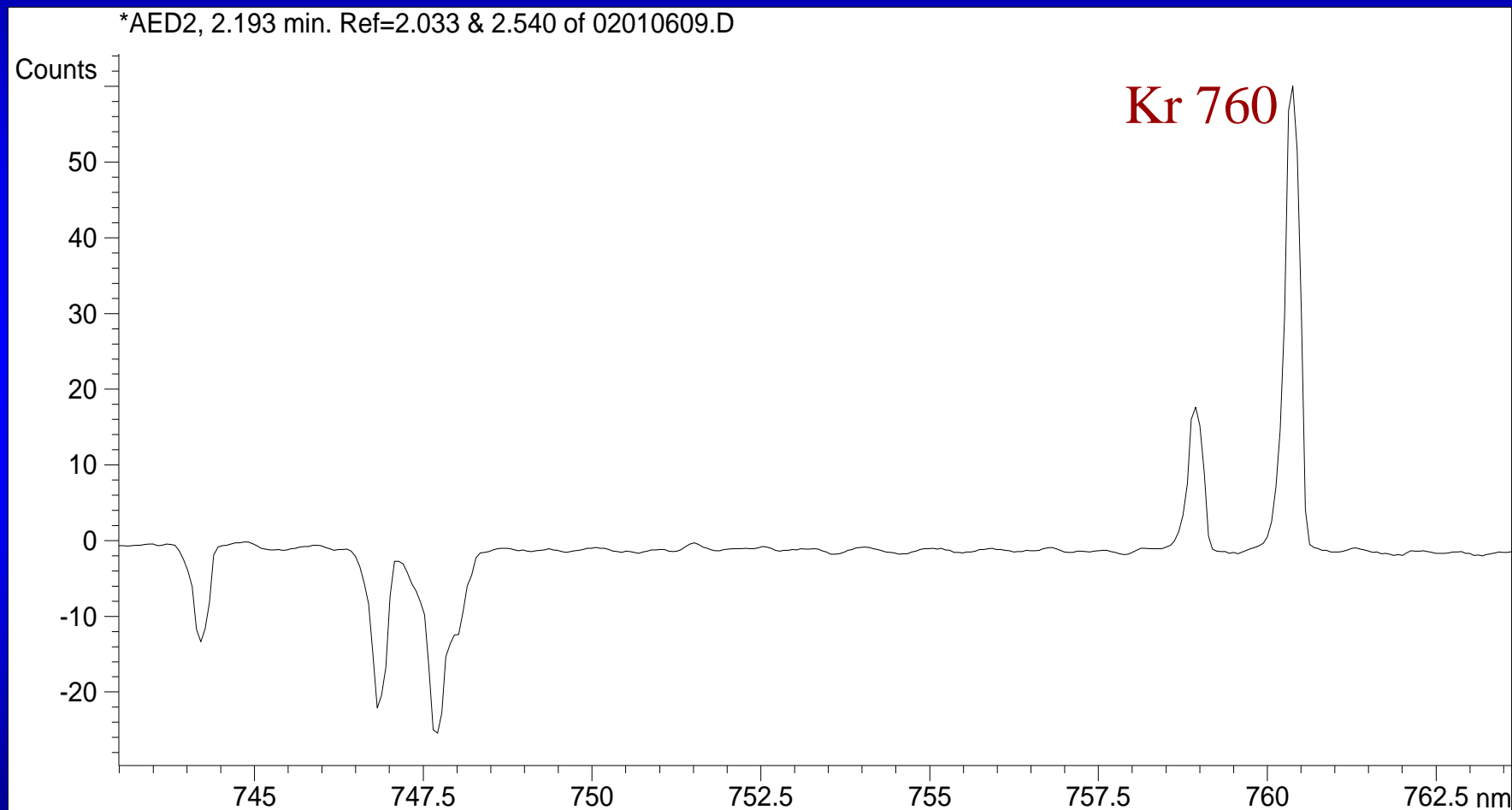
Distillation Application

Determination of ppm Xe and Kr in O₂

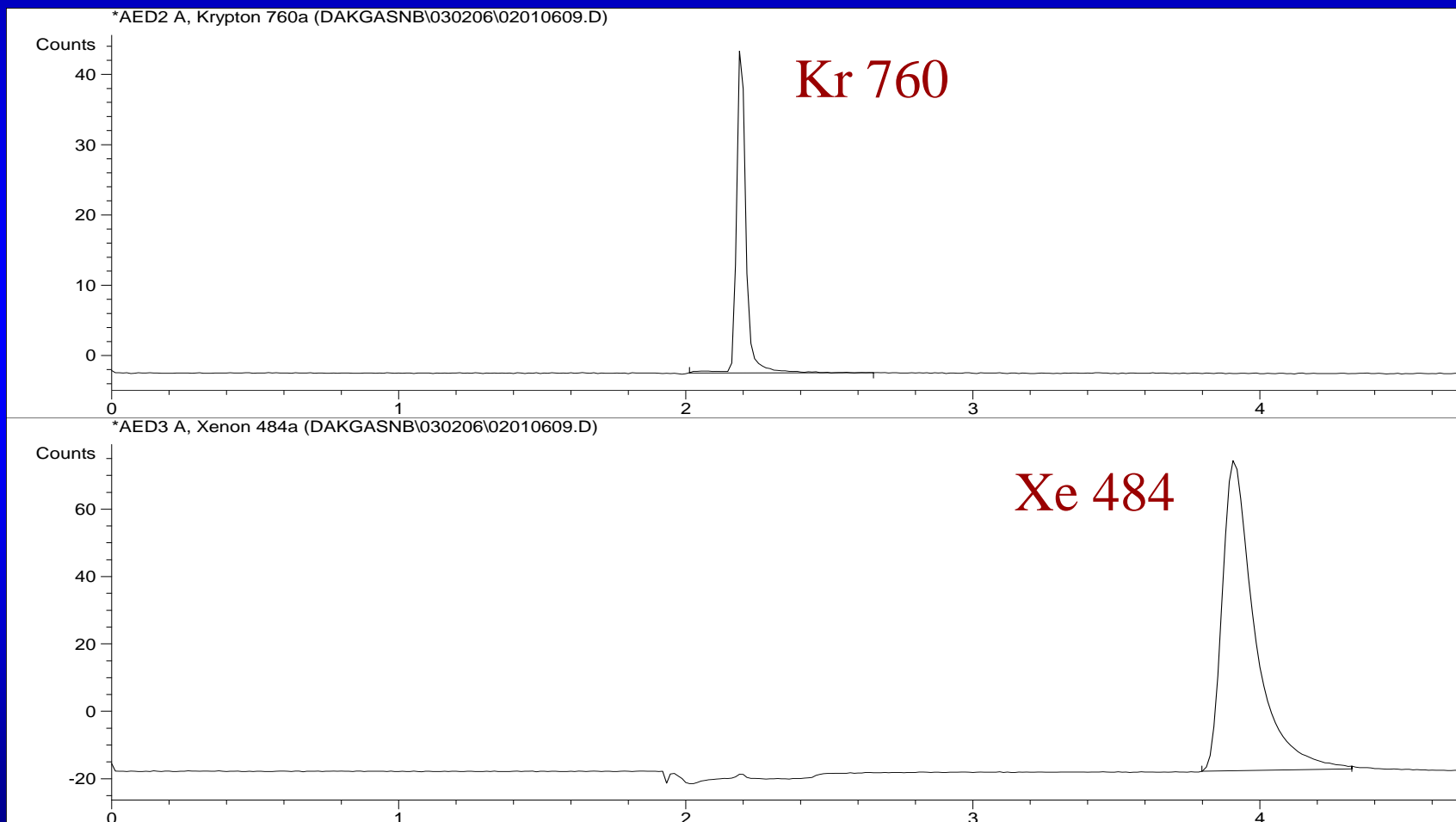
- Distillation Process
 - Separation of Xenon and Krypton from Oxygen and Argon
 - Oxygen and Argon – overhead
 - Xenon and Krypton - bottoms
 - Xenon and Krypton sold as product
- Kr / O₂ separation in the distillation column is critical
 - Requires determination of ppm levels of Kr and Xe in O₂



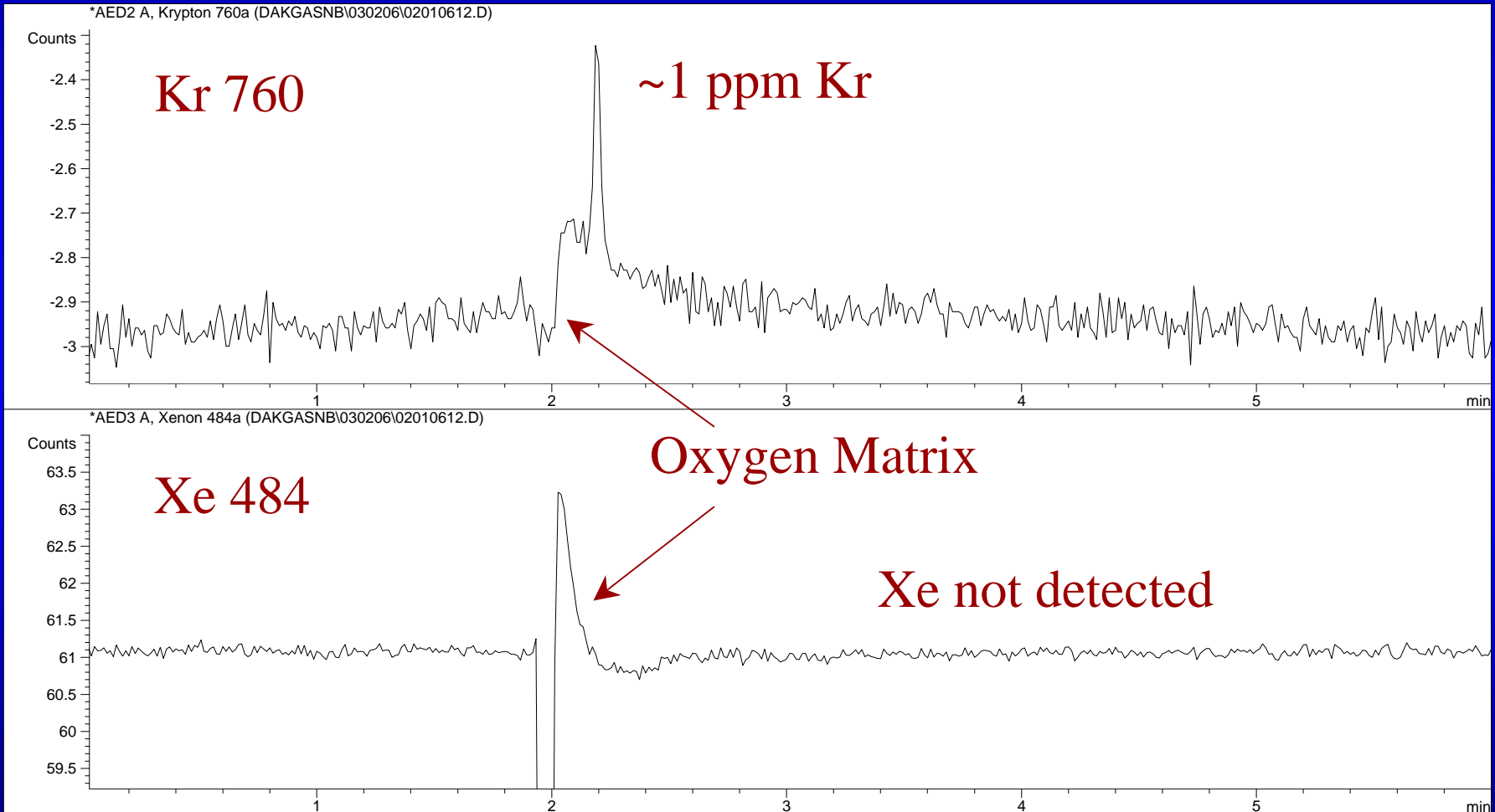
Example Spectrum - Krypton



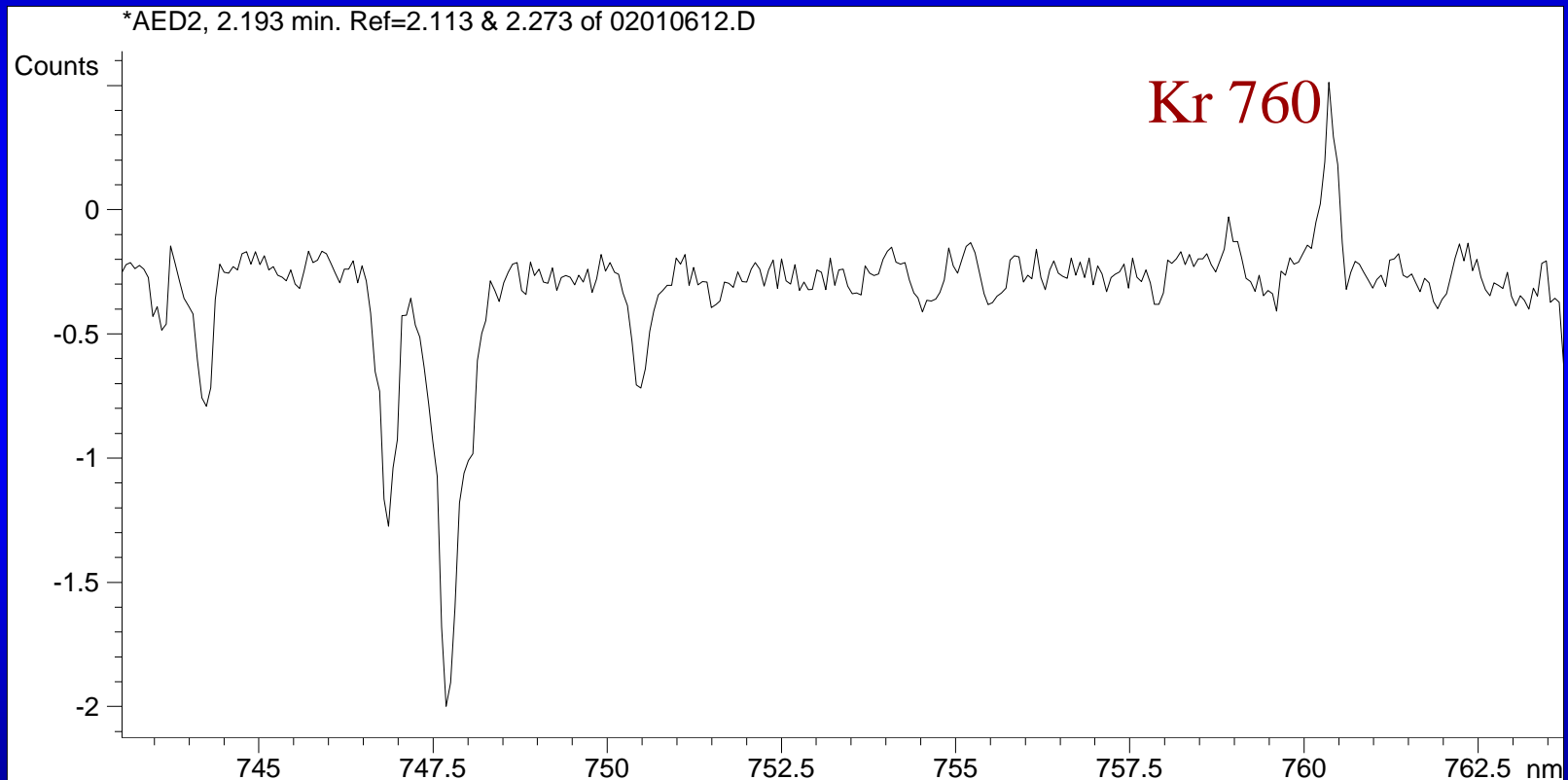
100 ppm Xe and Kr in O₂ Standard Chromatograms



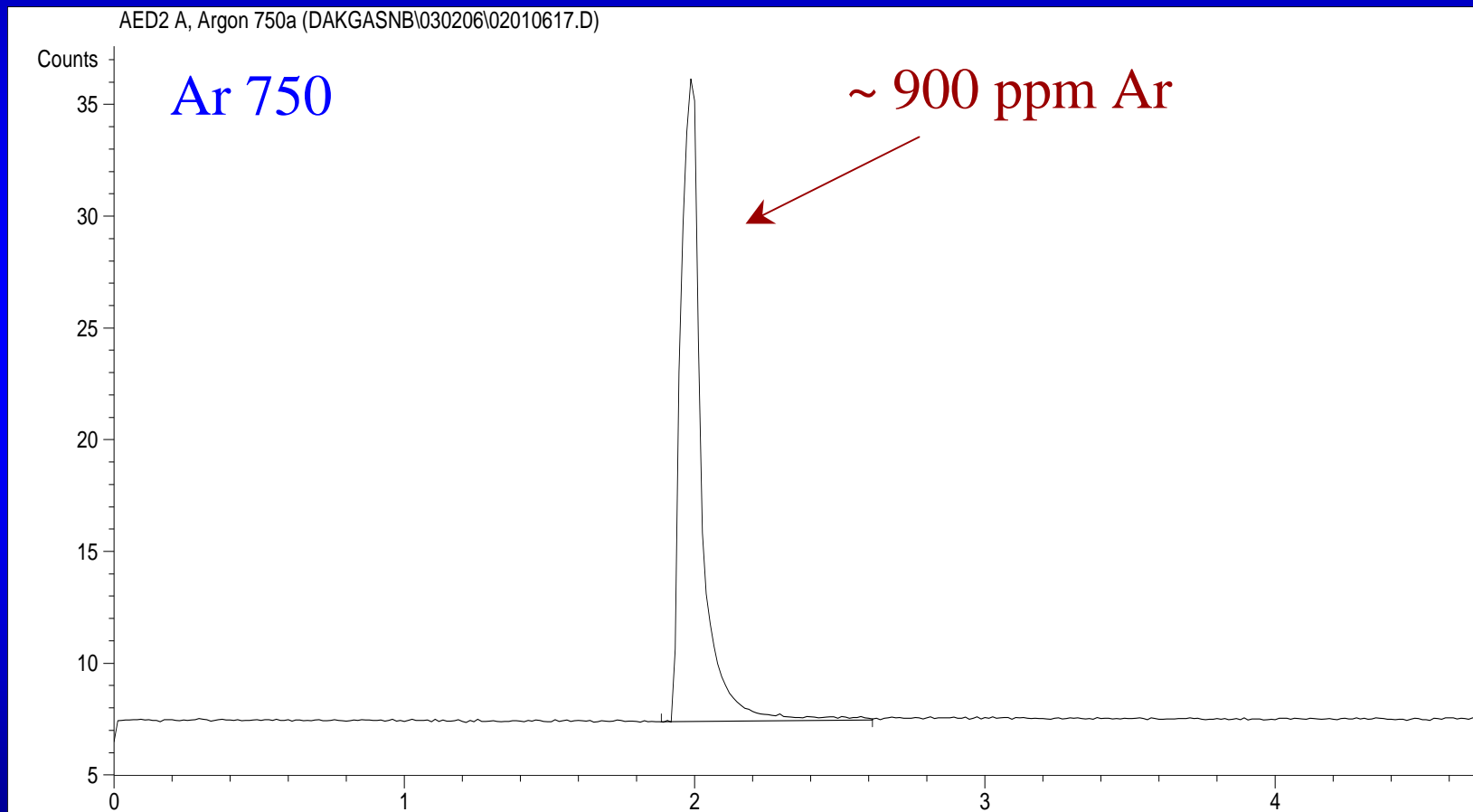
Distillation Overhead Sample Chromatograms



Spectrum of Krypton Peak in Overhead Sample



Distillation Overhead Sample Chromatogram

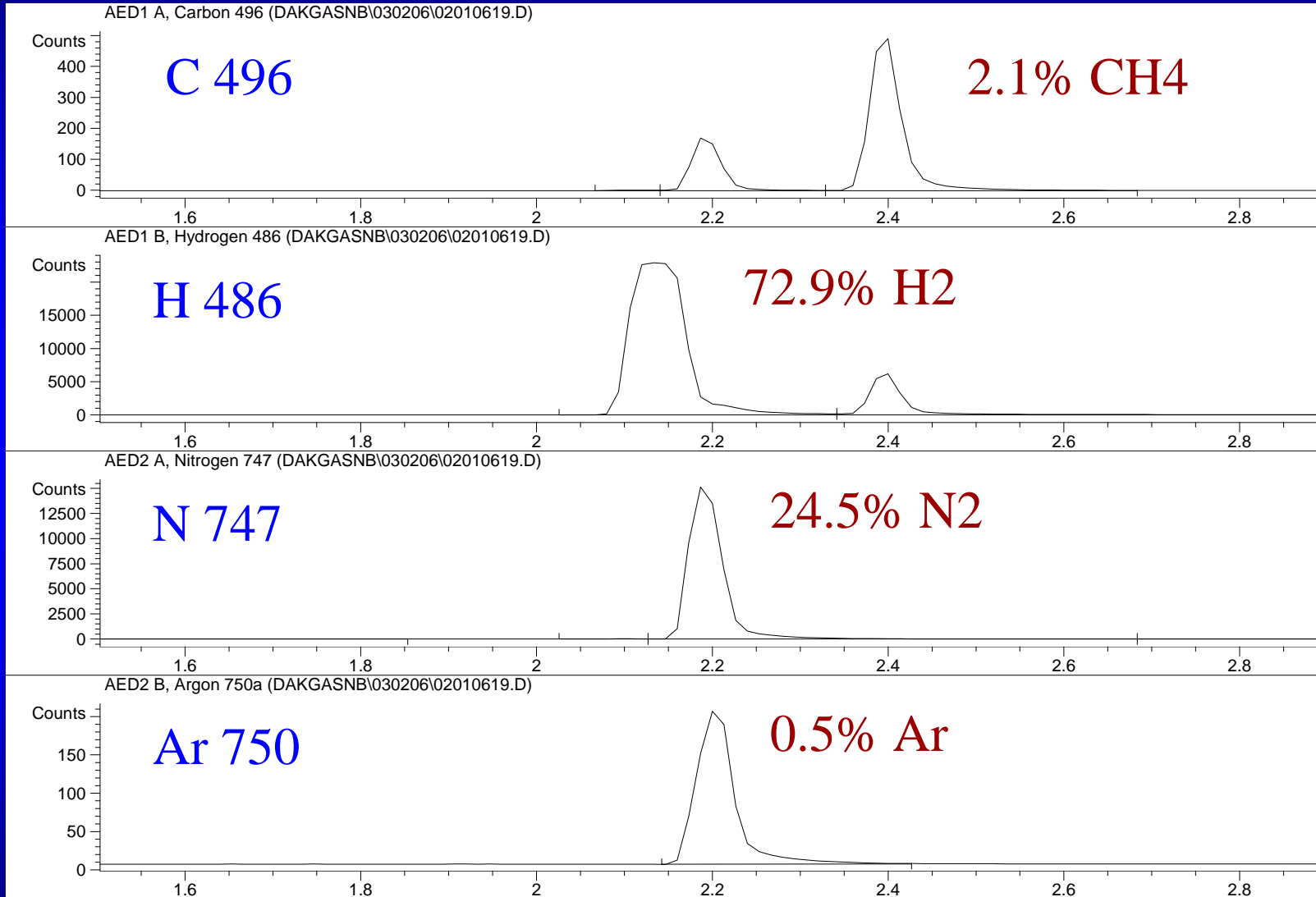


Fast Analysis of Ammonia Plant Process Streams for H₂, Ar, N₂, CH₄

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



Fast Analysis with Carboxen Column



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
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