Fast Analysis of Semi-Volatile Compounds: US EPA Method 8270

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Product Definition

Design a GC analysis for 8270 that increases laboratory sample throughput by:

- Decreasing analysis time
- Resolution of key analytes
- Utilizing run parameters that can be used for current mass specs including HP59XX.
- 106 compounds including internal standards and surrogates plus 30 chlorinated and organophosphorus pesticides

Classes of Compounds

8270 Calibration Mix #1

benzoic acid
4-chloro-3-methylphenol
2-chlorophenol
2,4-dichlorophenol
2,6-dichlorophenol
2,4-dimethylphenol
4,6-dinitro-2-methylphenol
2,4-dinitrophenol
dinoseb
2-methylphenol
3-methylphenol
4-methylphenol
2-nitrophenol
4-nitrophenol
pentachlorophenol
phenol
2,3,4,6-tetrachlorophenol
2,4,5-trichlorophenol
2,4,6-trichlorophenol

8270 Calibration Mix #2

aniline
benzidine
4-chloroaniline
3,3'-dichlorobenzidine
diphenylamine
3-nitroaniline
4-nitroaniline
N-nitrosodimethylamine
N-nitrosodi-n-propylamine
pyridine

Classes of Compounds

8270 Calibration Mix #3

aramite
bis (2-chloroethyl) ether
bis (2-chloroethoxy) methane
bis (2-chloroisopropyl) ether
4-bromophenyl phenyl ether
chlorobenzilate
2-chloronaphthalene
4-chlorophenyl phenyl ether
1,2-dichlorobenzene
1,3-dichlorobenzene
1,4-dichlorobenzene
1,3-dinitrobenzene
hexachlorobenzene
hexachlorobutadiene
hexachlorocyclopentadiene
hexachloroethane
hexachloropropene
isodrin
kepone
pentachlorobenzene
pentachloronitrobenzene
1,2,4,5-tetrachlorobenzene
1,2,4-trichlorobenzene

Classes of Compounds

8270 Calibration Mix #4

acetophenone
azobenzene
benzyl alcohol
bis (2-ethylhexyl) phthalate
butyl benzyl phthalate
dibenzofuran
diethyl phthalate
dimethyl phthalate
di-n-butyl phthalate
di-n-octyl phthalate
2,4-dinitrotoluene
2,6-dinitrotoluene
ethyl methanesulfonate
isophorone
isosafrole (cis & trans)
methyl methanesulfonate
1,4-naphthoquinone
nitrobenzene
4-nitroquinoline-1-oxide
phenacetin
safrole

Classes of Compounds

8270 Calibration Mix #5

acenaphthene
acenaphthylene
anthracene
benzo(a)pyrene
benzo(ghi)perylene
benzo(a)anthracene
benzo(b)fluoranthene
benzo(k)fluoranthene
chrysene
dibenz(a,h)anthracene
fluoranthene
fluorene
ideno(1,2,3-cd)pyrene
1-methylnaphthalene
naphthalene
3-methylcholanthrene
2-methylnaphthalene
phenanthrene
pyrene

Classes of Compounds

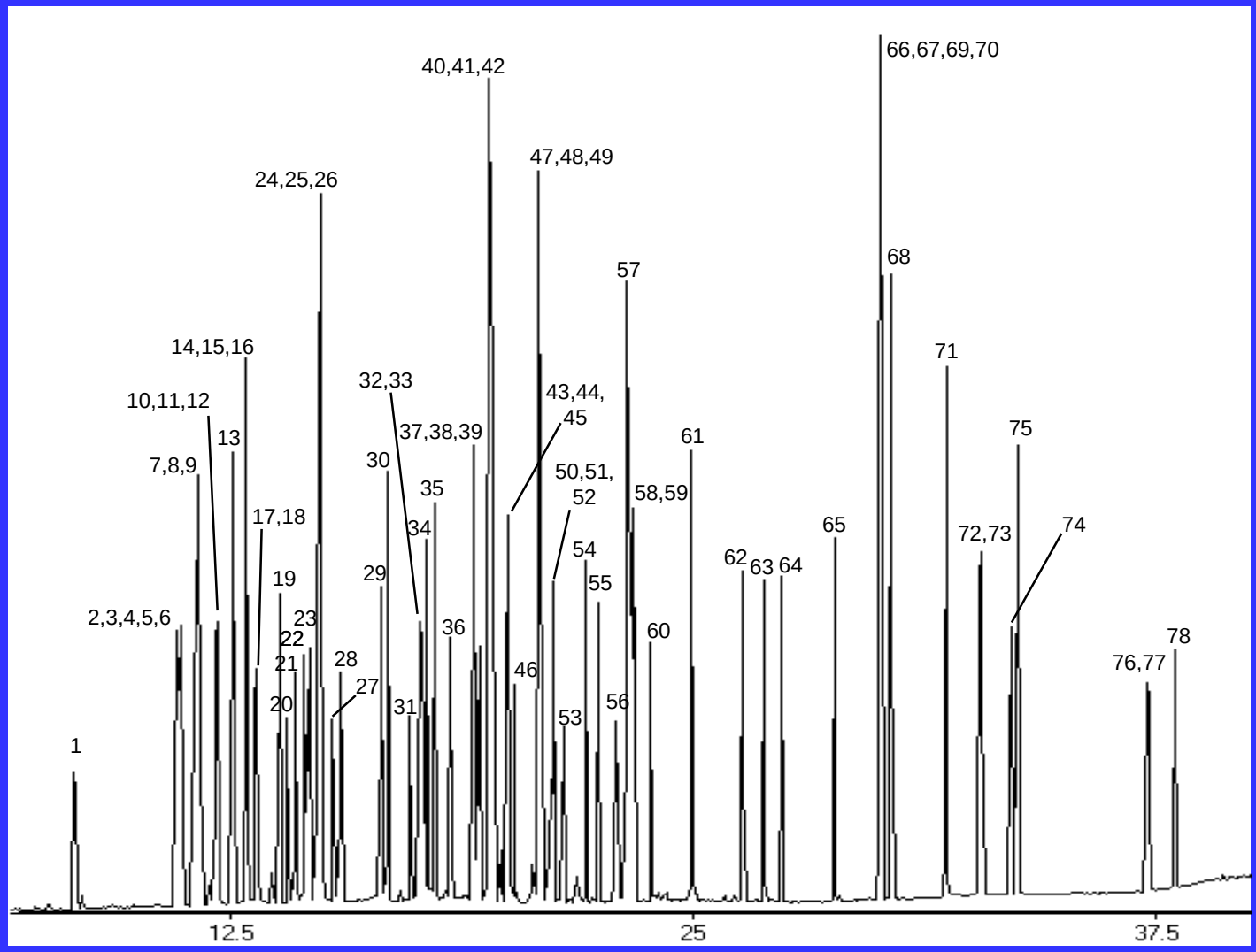
8270 Calibration Mix #6

diallate (cis & trans)
dimethoate
disulfoton
famphur
methyl parathion
parathion
phorate
pronamide
thionazine
0,0,0-triethyl phosphorothioate

Organochlorine Pesticide Mix AB #1

aldrin
a-BHC
a-chlordane
b-BHC
4,4'-DDD
4,4'-DDE
4,4'-DDT
d-BHC
dieldrin
endosulfan I
endosulfan II
endosulfan sulfate
endrin
endrin aldehyde
endrin ketone
g-BHC (lindane)
g-chlordane
heptachlor
heptachlor epoxide (B)
methoxychlor

Current Analysis



Analysis Conditions

30m, 0.25mm ID, 0.50µm Rtx-5MS (cat.# 12638)
2µL splitless injection. On-column concentration: 20ng.

Oven temp.:	40°C (hold 2 min.) to 300°C @ 10°C/min. (hold 4 min.), to 330°C @ 10°C/min. (hold 10 min.).
Inj. / det. temp.:	280°C / 300°C
Det. type:	MS
Carrier gas:	helium
Linear velocity:	32 cm/sec. set @ 40°C
Ionization:	EI
Splitless hold:	0.5 min.
Scan Range:	35-500AMU

Peak Identification

1. 2-fluorophenol (Surr)
2. phenol-d5 (Surr)
3. phenol
4. bis(2-chloroethyl)ether
5. 2-chlorophenol-d4 (Surr)
6. 2-chlorophenol
7. 1,3-dichlorobenzene
8. 1,4-dichlorobenzene-d4 (IS)
9. 1,4-dichlorobenzene
10. 1,2-dichlorobenzene-d4 (Surr)
11. 1,2-dichlorobenzene
12. 2-methylphenol
13. 2,2'-oxybis-(1-chloropropane)
14. 4-methylphenol
15. N-nitroso-di-n-propylamine
16. hexachloroethane
17. nitrobenzene-d5 (Surr)
18. nitrobenzene
19. isophorone
20. 2-nitrophenol
21. 2,4-dimethylphenol
22. bis(2-chloroethoxy)methane
23. 2,4-dichlorophenol
- 1,2,4-trichlorobenzene

25. napthalene-d8 (IS)
26. napthaler

27. 4-chloroaniline
28. hexachlorobutadiene
29. 4-chloro-3-methylphenol
30. 2-methylnaphthalene
31. hexachlorocyclopentadiene
32. 2,4,6-trichlorophenol
33. 2,4,5-trichlorophenol
34. 2-fluorobiphenyl (Surr)
35. 2-chloronaphthalene
36. 2-nitroaniline
37. dimethylphthalate
38. acenaphthylene
39. 2,6-dinitrotoluene
40. 3-nitroaniline
41. acenaphthene-d10 (IS)
42. acenaphthene
43. 2,4-dinitrophenol
44. 4-nitrophenol
45. dibenzofuran
46. 2,4-dinitrotoluene
47. diethylphthalate
- Fluorene
- 4-chlorophenyl-phenylether

50. 4-nitroaniline
51. 4,6-dinitro-2-methylphenol

53. 2,4,6-tribromophenol (Surr)
54. 4-bromophenyl-phenylether
55. hexachlorobenzene
56. pentachlorophenol
57. phenanthrene-d10 (IS)
58. phenanthrene
59. anthracene
60. carbazole
61. di-n-butylphthalate
62. fluoranthene
63. pyrene
64. terphenyl-d14 (Surr)
65. butylbenzylphthalate
66. 3,3-dichlorobenzidine
67. benzo(a)anthracene
68. bis(2-ethylhexyl)phthalate
69. chrysene-d12 (IS)
70. chrysene
71. di-n-octylphthalate
72. benzo(b)fluoranthene
73. benzo(k)fluoranthene
74. benzo(a)pyrene
75. perylene-d12 (IS)
76. indeno(1,2,3-cd)pyrene
77. dibenzo(a,h)anthracene

Fast Analysis Concerns

- Flow rate considerations for diffusion pumps (<1.3 mL/min)
- Closely eluting compounds w/same quantitation ions
  - phenol / aniline / bis(2-chloroethyl)ether
  - 1,3- & 1,4-dichlorobenzene
  - 2- & 1-methylnaphthalene
  - 2,4,6- & 2,4,5-trichlorobenzenes
  - phenanthrene / anthracene
  - benz(a)anthracene / chrysene
  - benzo(b)fluoranthrene / benzo(k)fluoranthrene

Reducing Analysis Time

- Important criteria to reducing run times
  - initial hold time
    - resolve early eluting compounds
  - eluting compounds on ramp rate vs isothermal
  - fast ramp rate through non critical areas

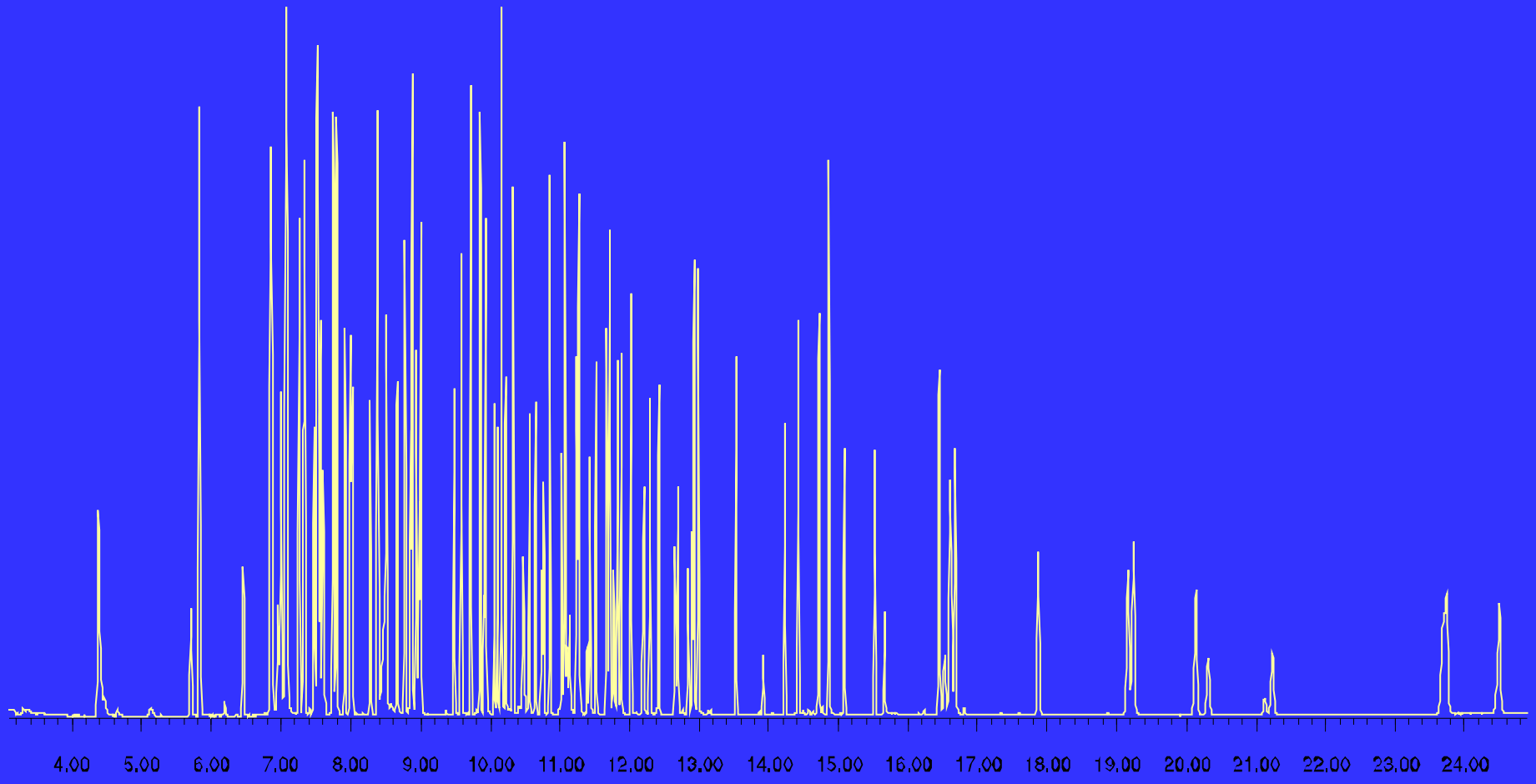
Initial Work

- Constant flow
- Maximize ramp rate
  - Start out with fast ramp rate and work backwards
  - Adjust oven temperature program so all compounds after initial hold elute on ramp rate
- Overload of high standard
  - Testing performed with 160ng due to potential overload causing coelution of closely eluting compounds

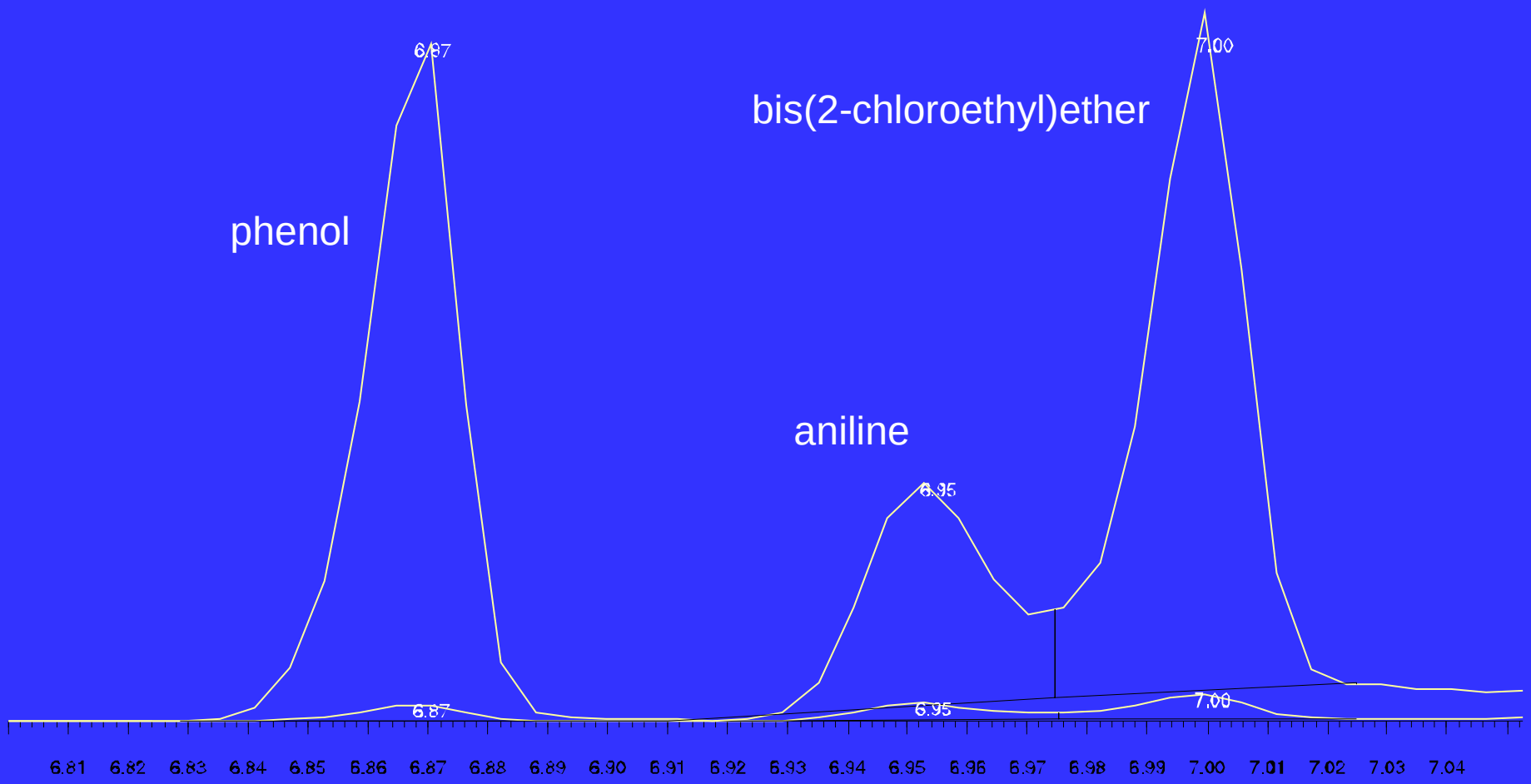
Resulting Run Conditions

- Constant flow rate @ 1.0 mL/min
- Temperature program:
  - 40°C (2 min)
  - 20°C/min
  - 290°C (0 min)
  - 2°C/min
  - 303°C (0 min)
  - 6°C/min
  - 330°C (1 min)

Rtx-5Sil MS
(30m x 0.25mm ID, 0.5µm film)



Separation of Critical Pairs
Rtx-5Sil MS (30m x 0.25mm, 0.5µm film)



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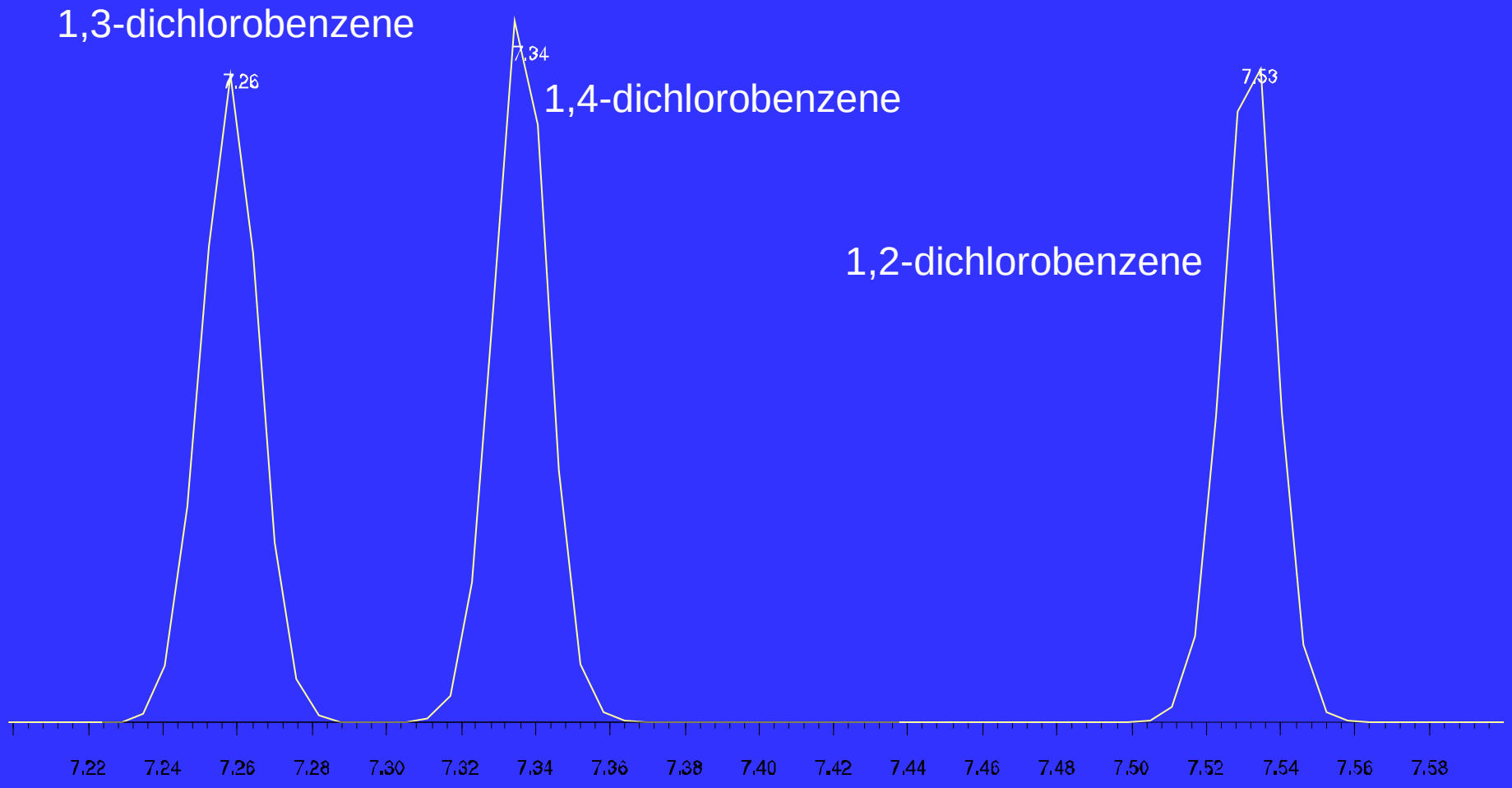

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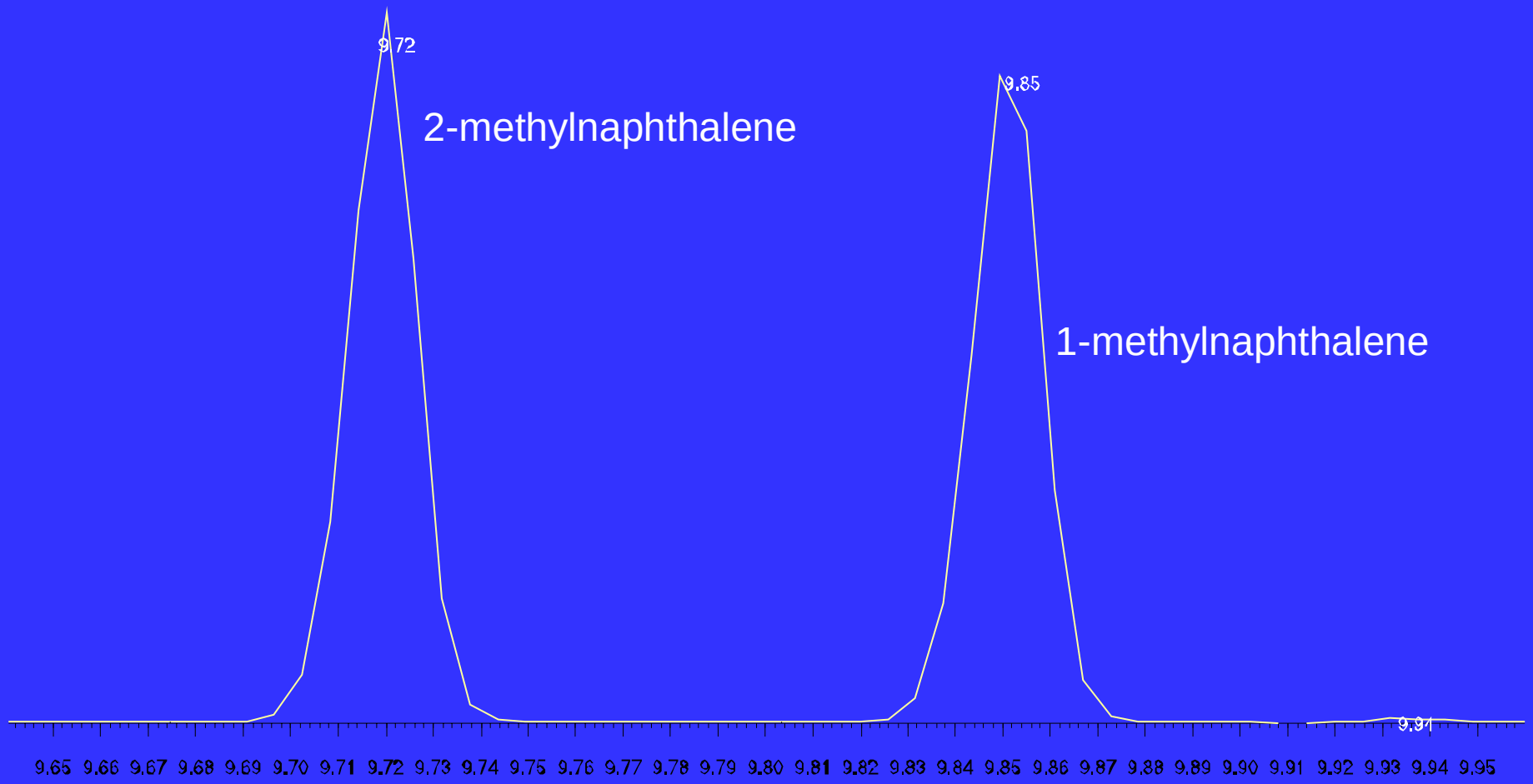

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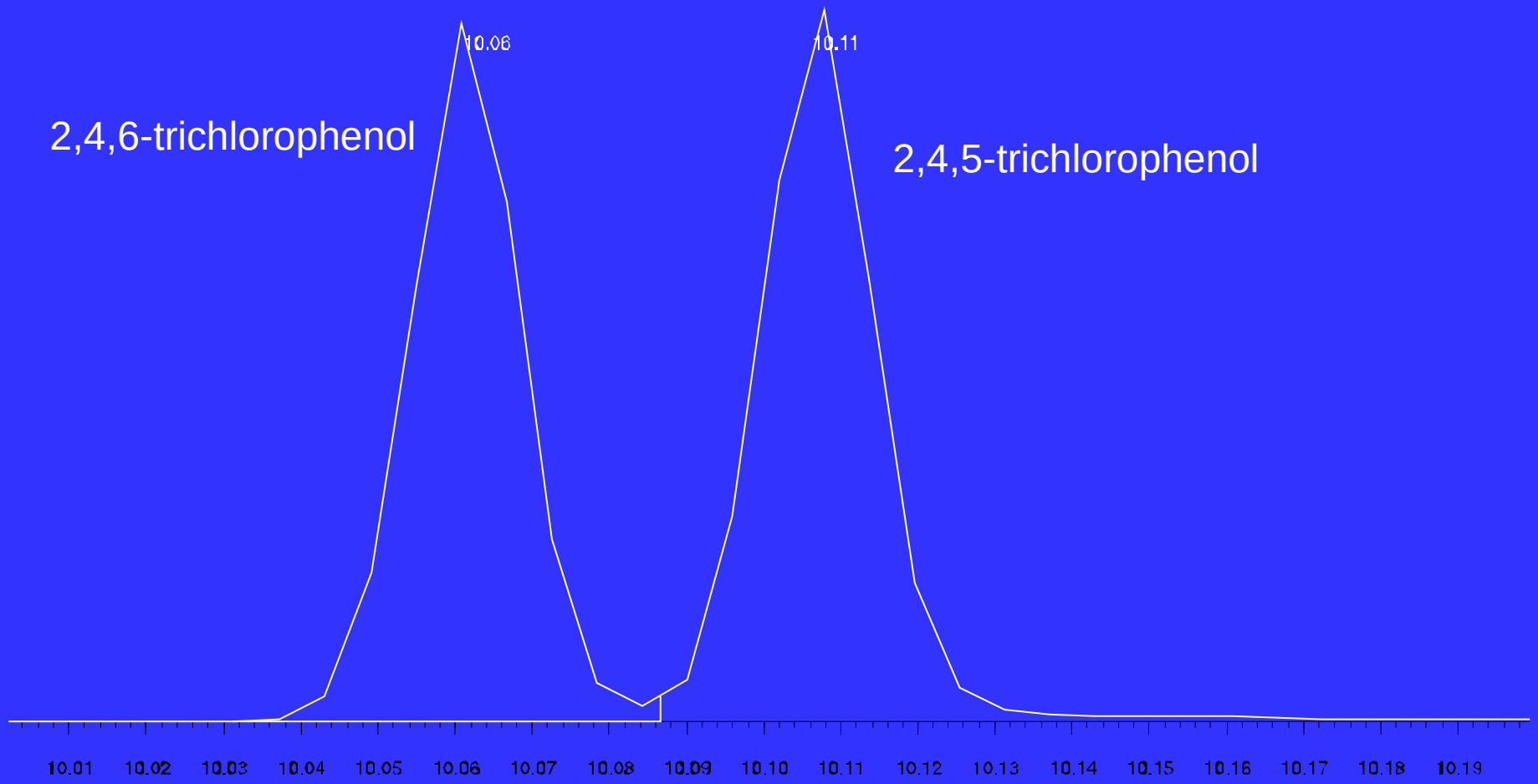

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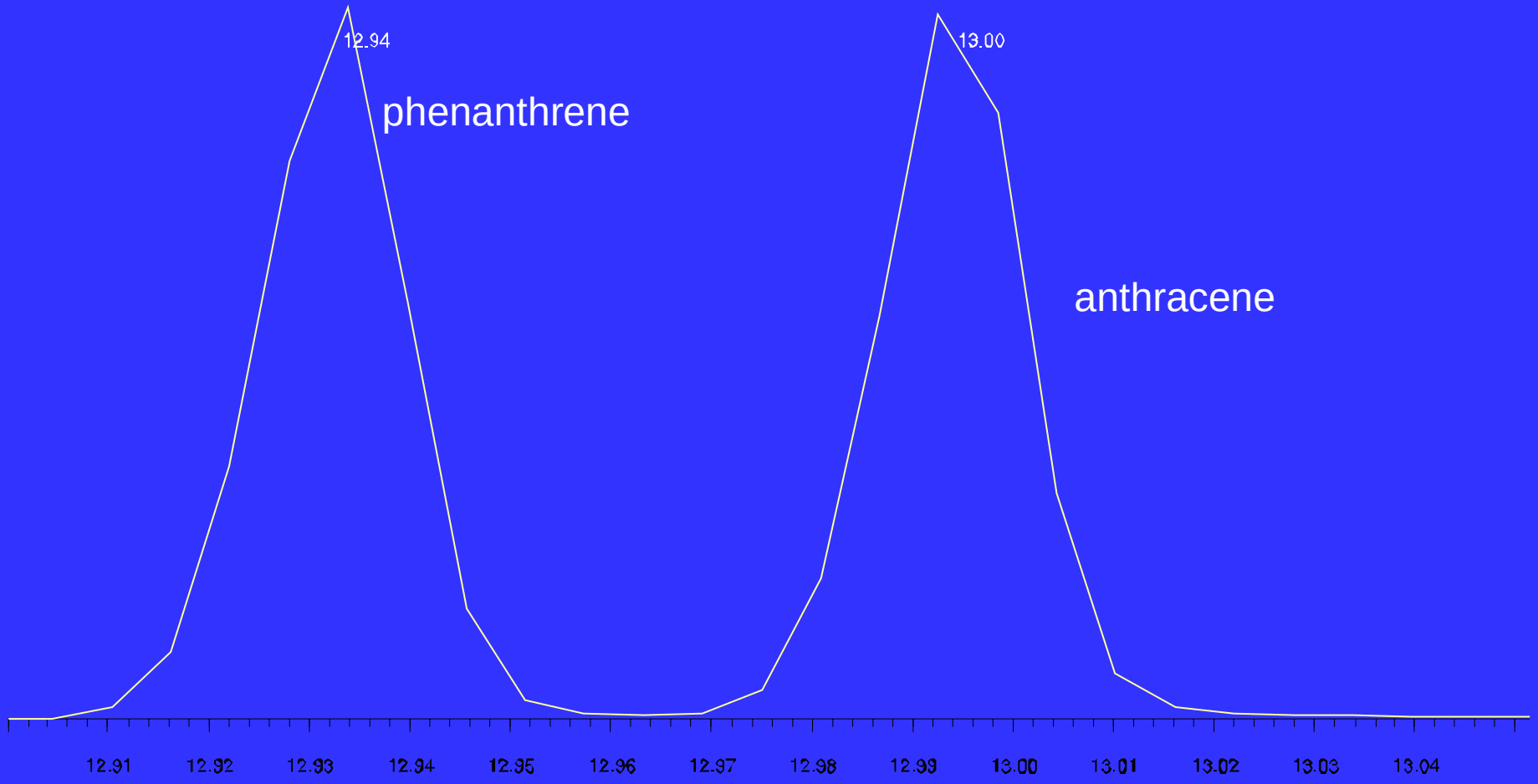

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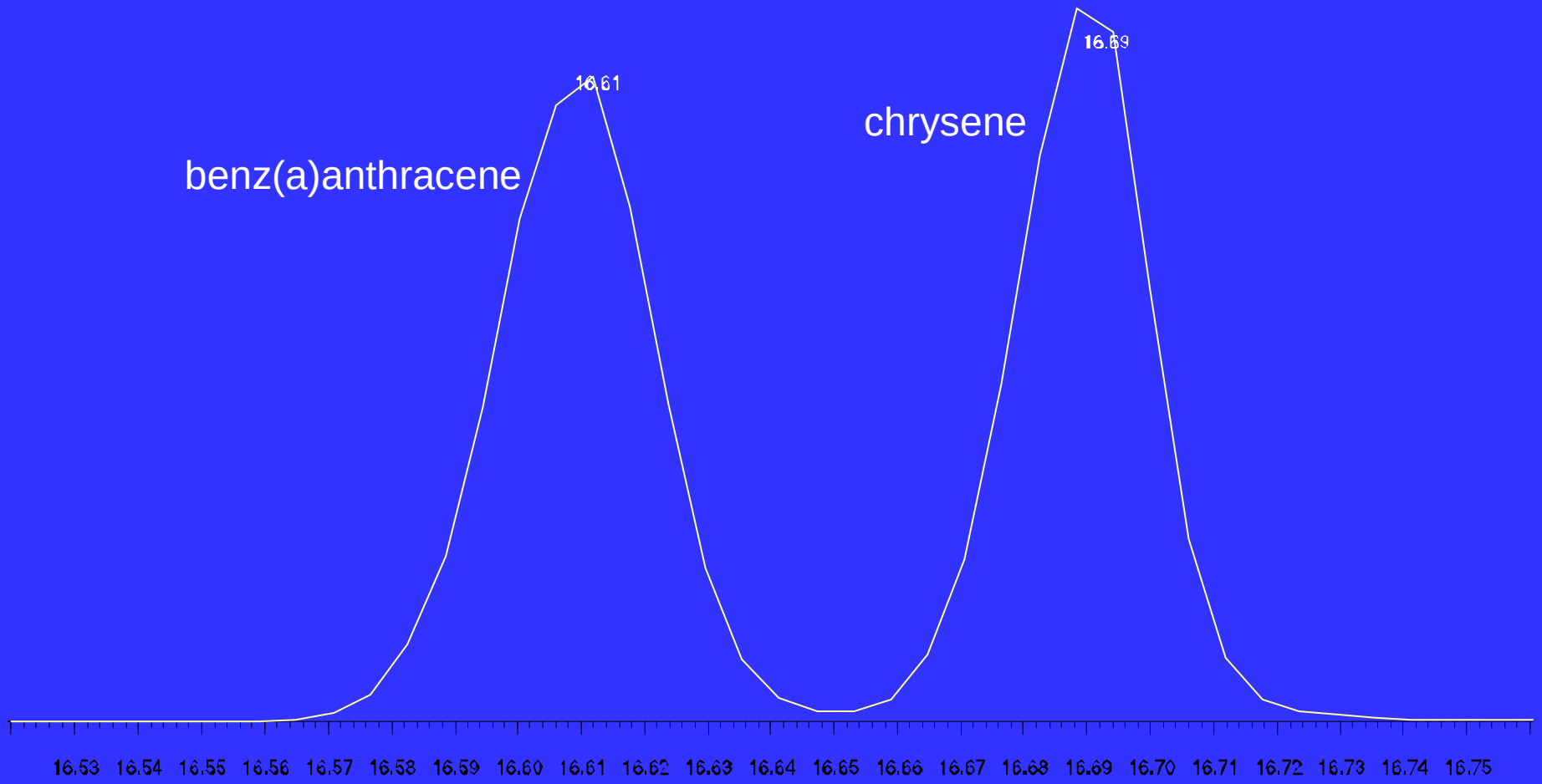

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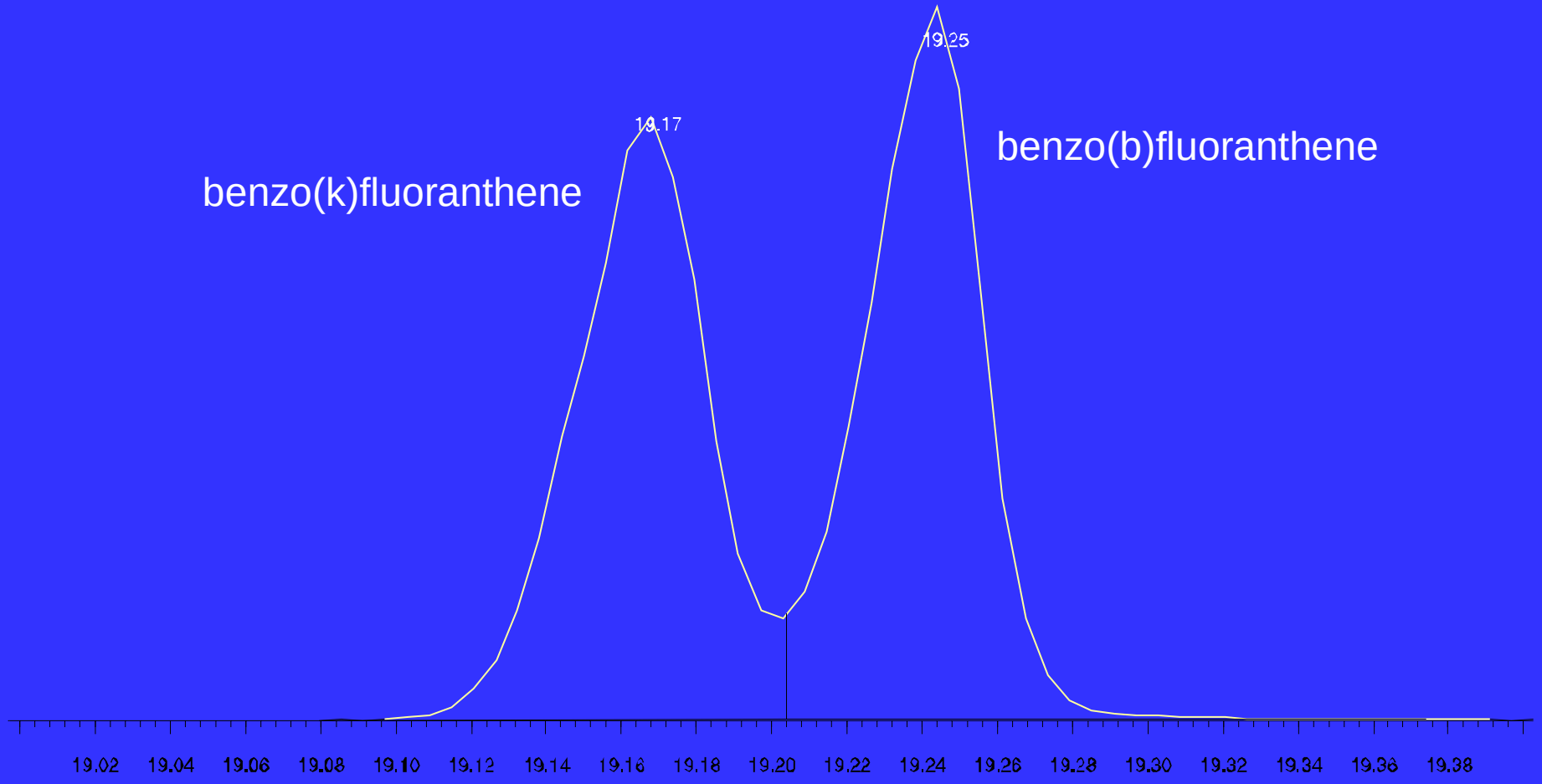

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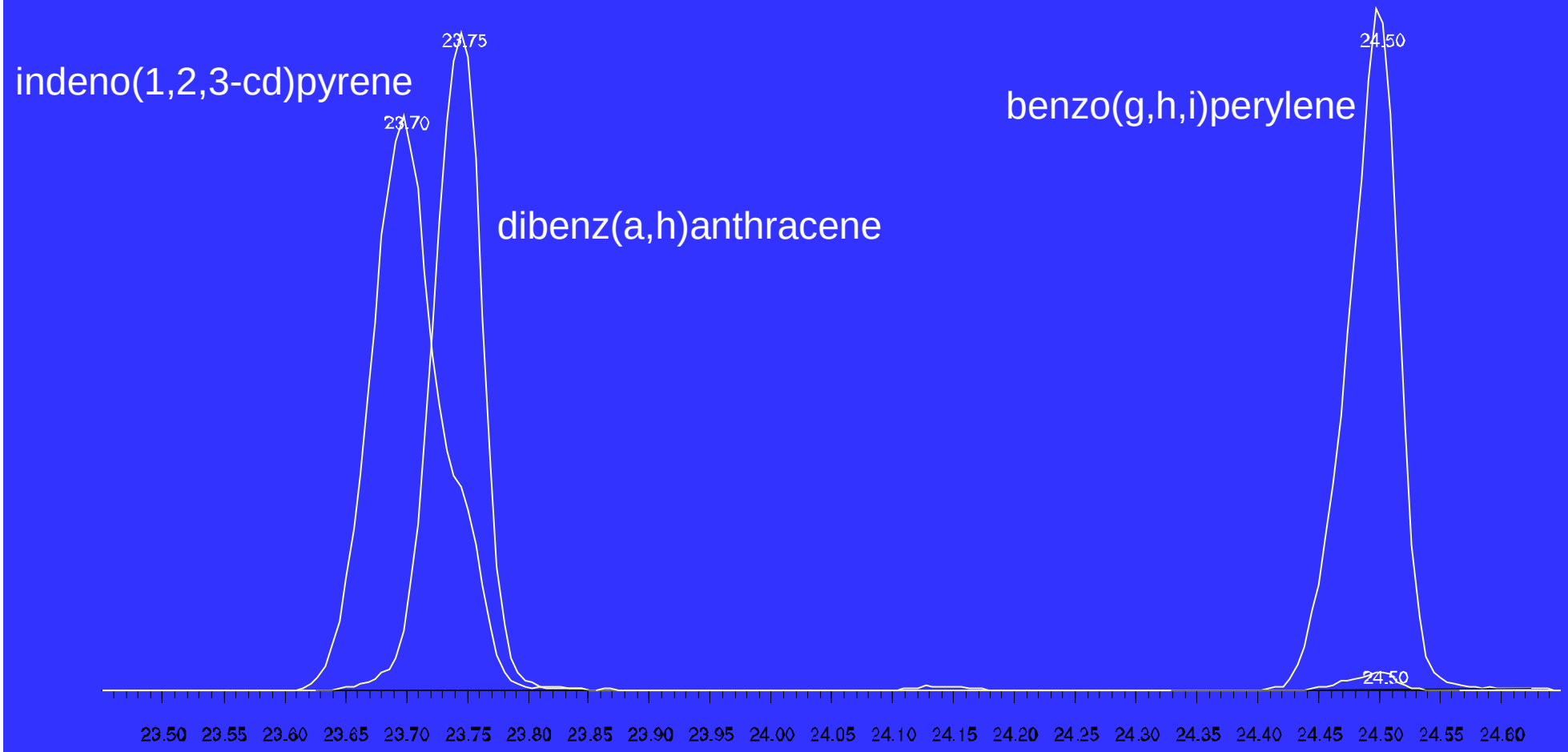
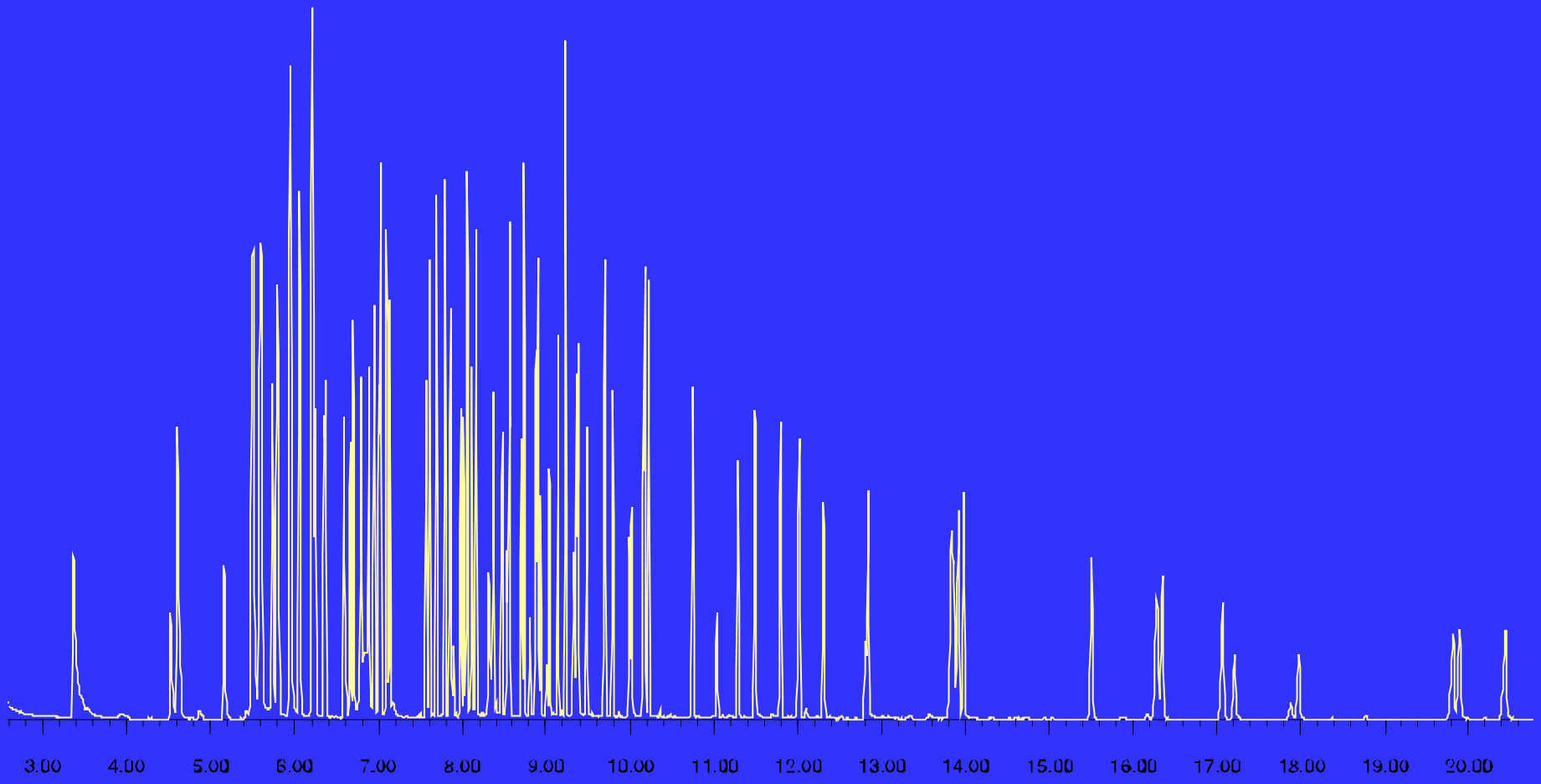


Can We Go Faster?

- Thinner film columns
- Shorter columns
- Smaller ID

Rtx-5Sil MS
(30m x 0.25mm ID, 0.25µm film)



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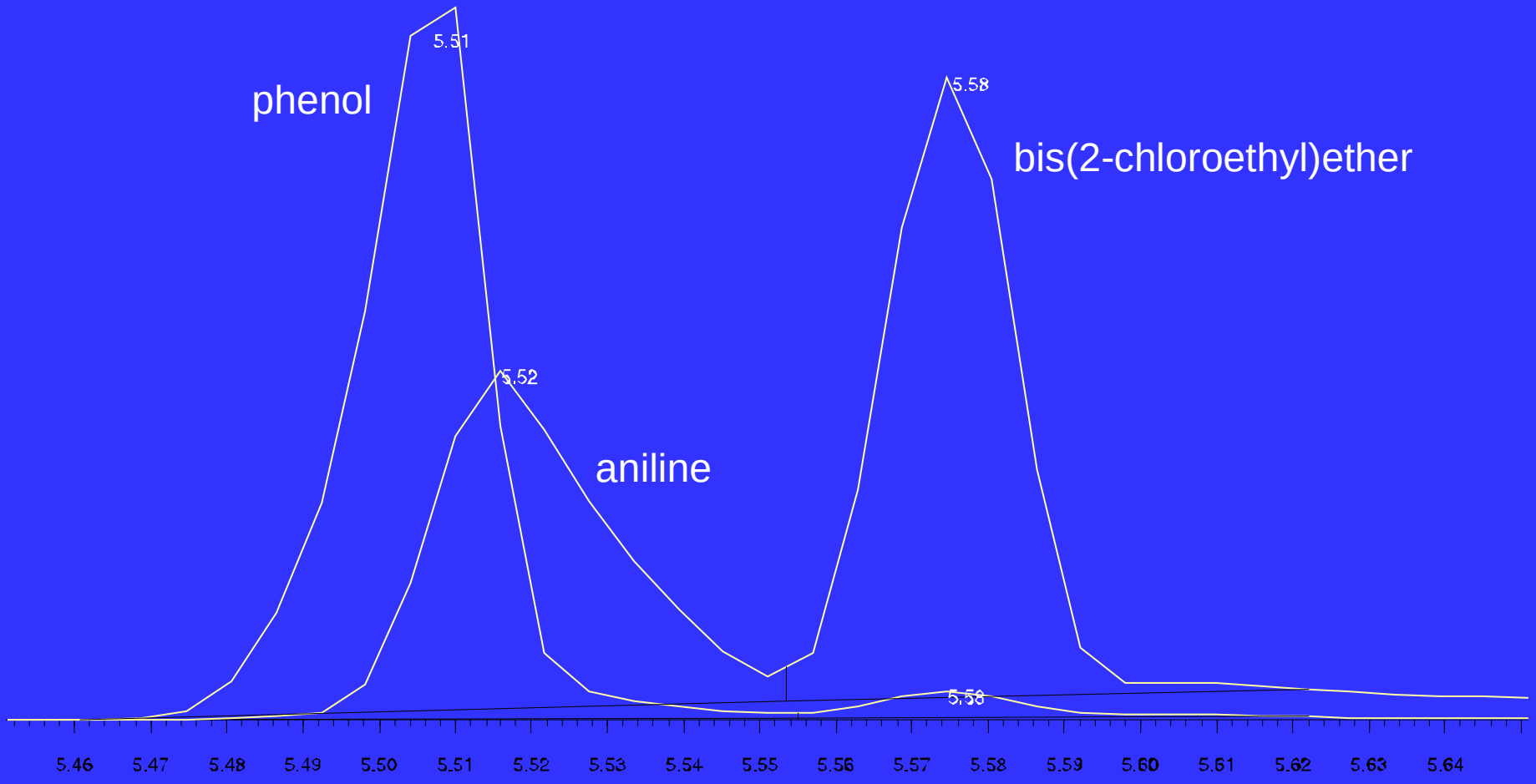

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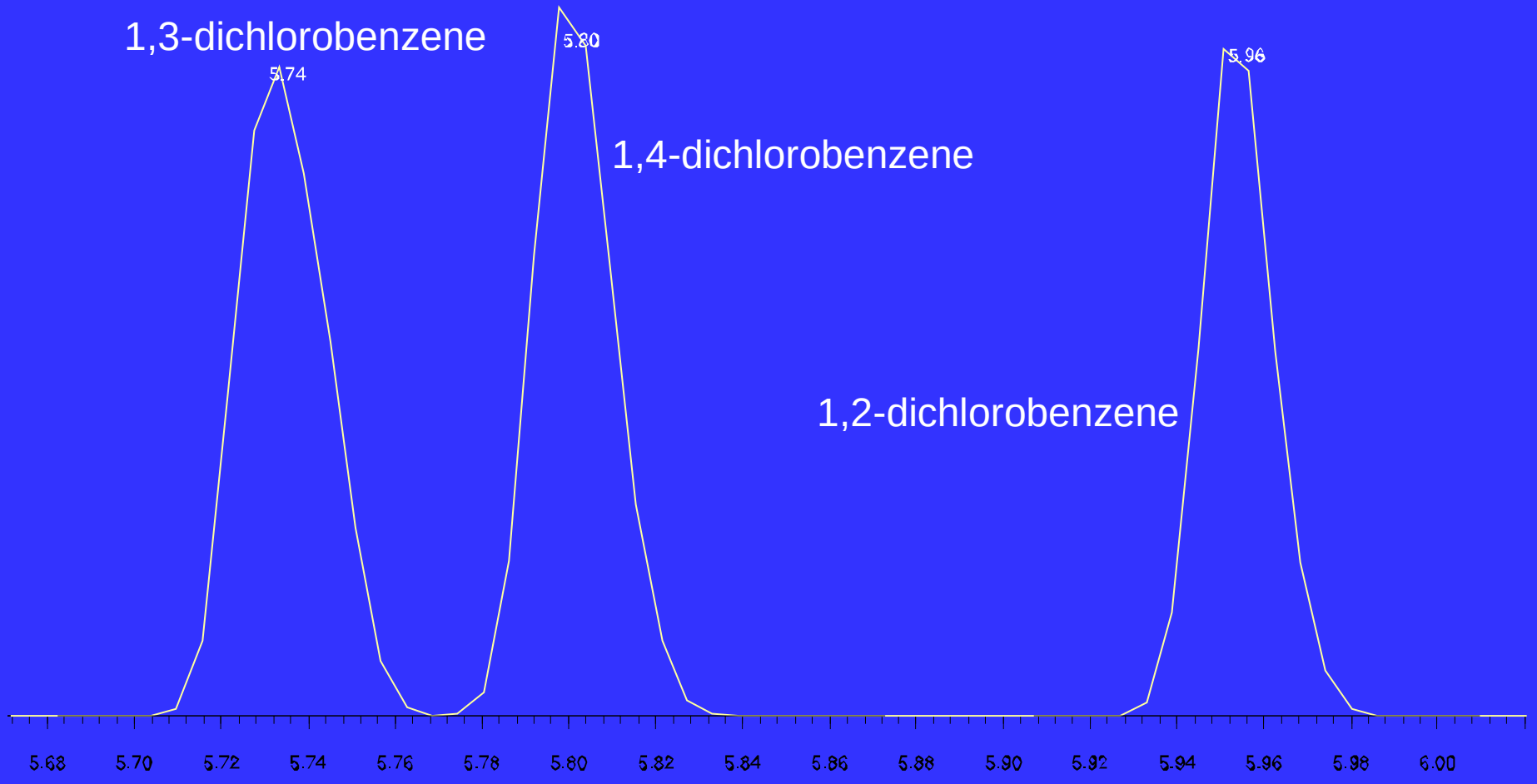

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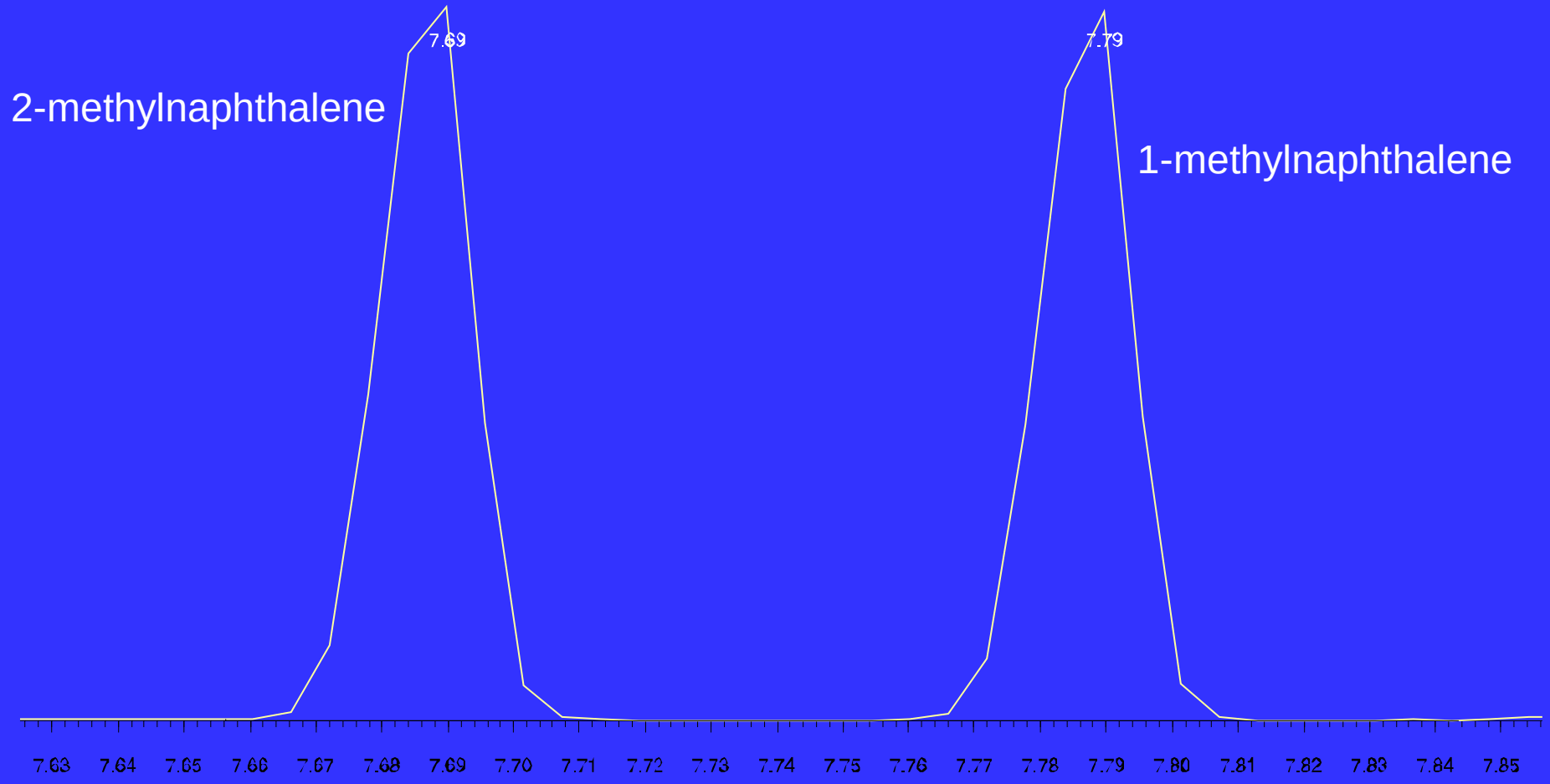

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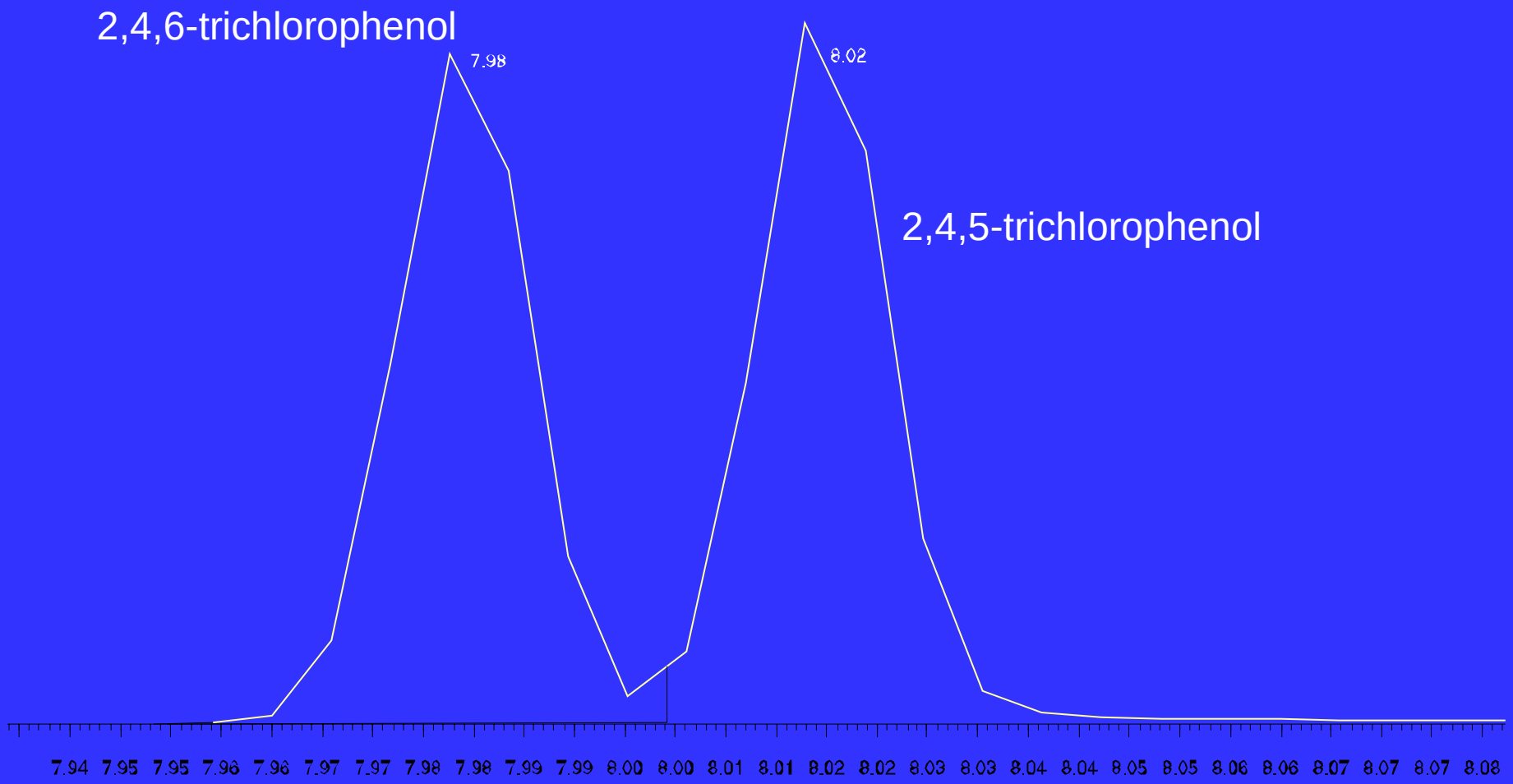

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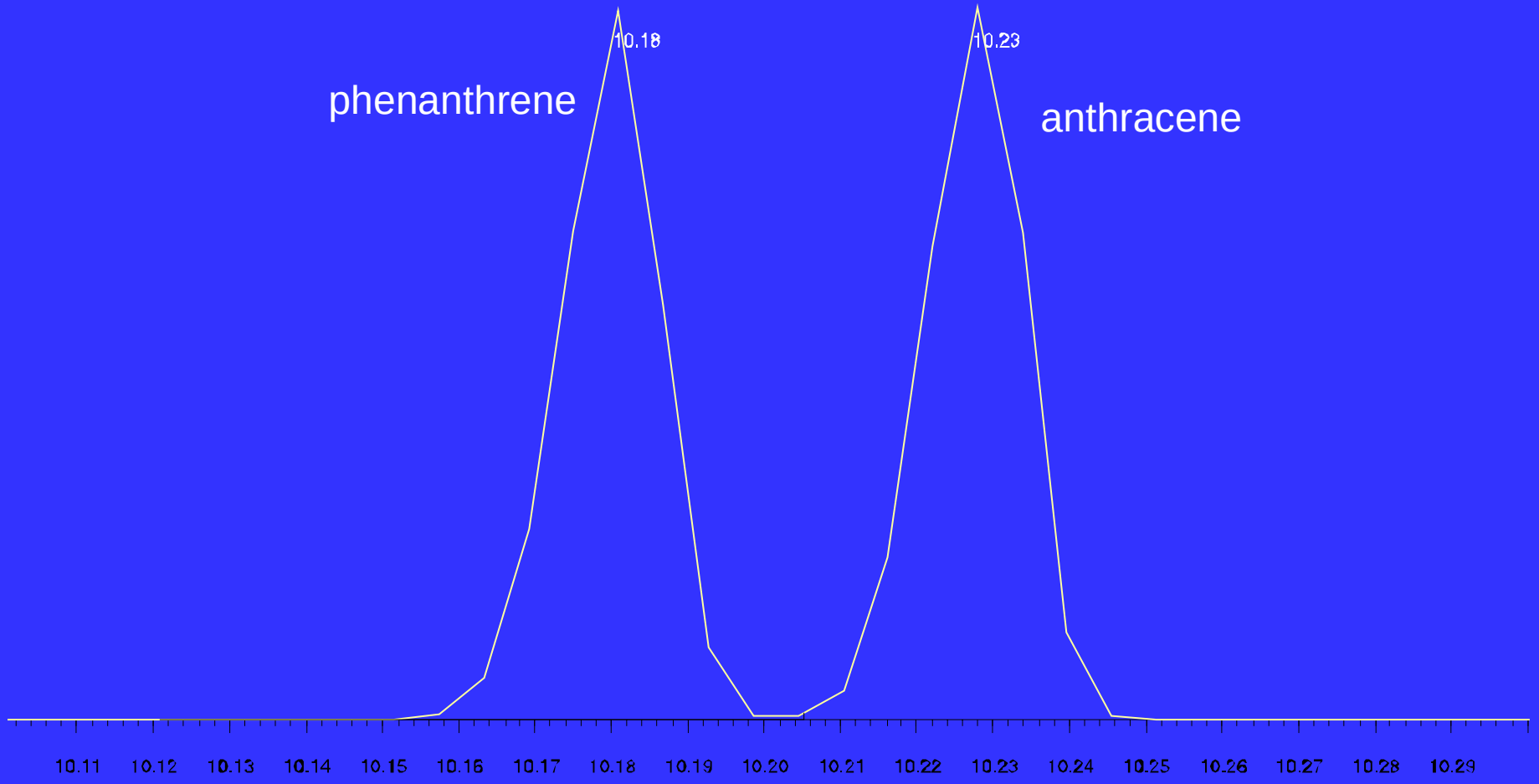

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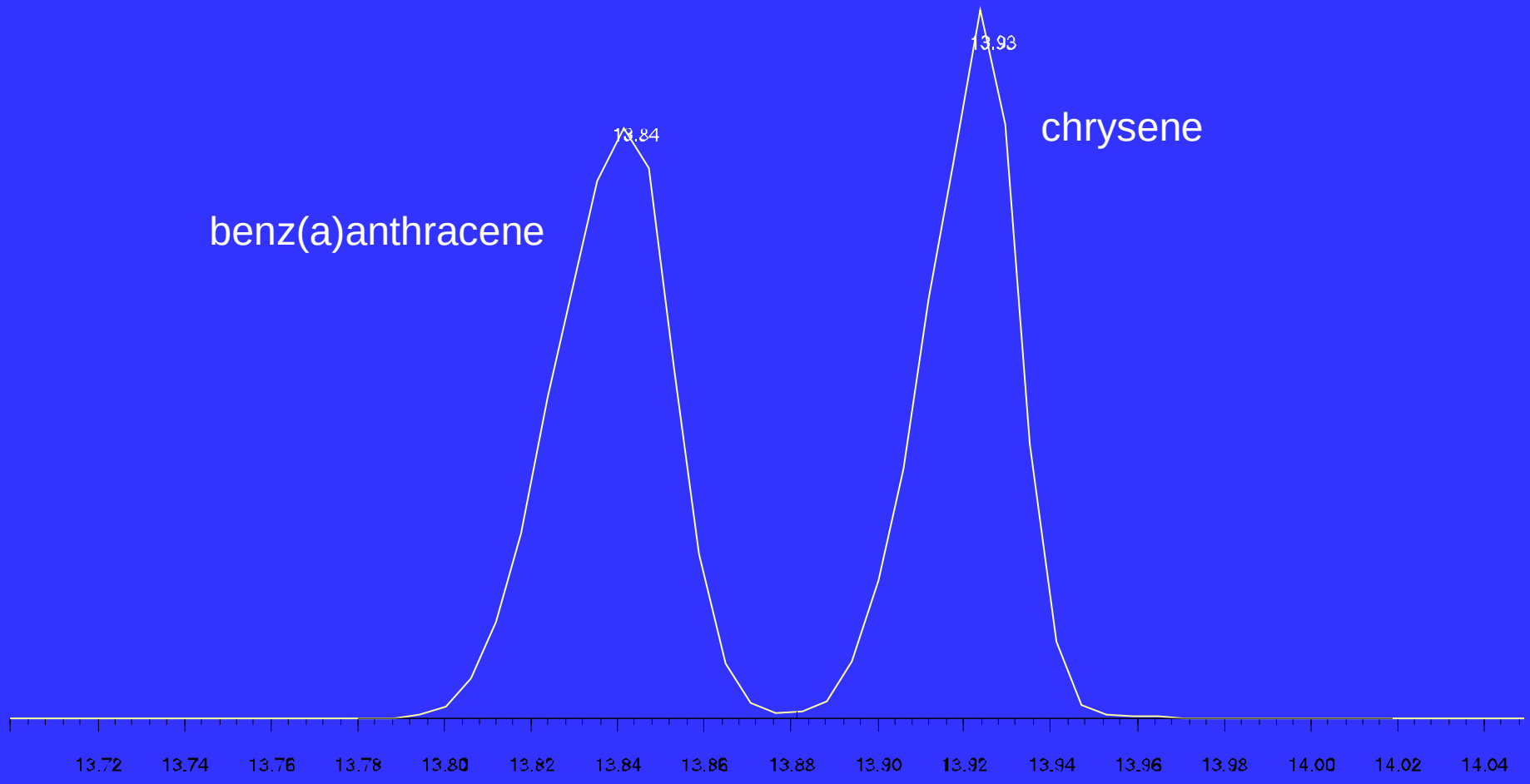

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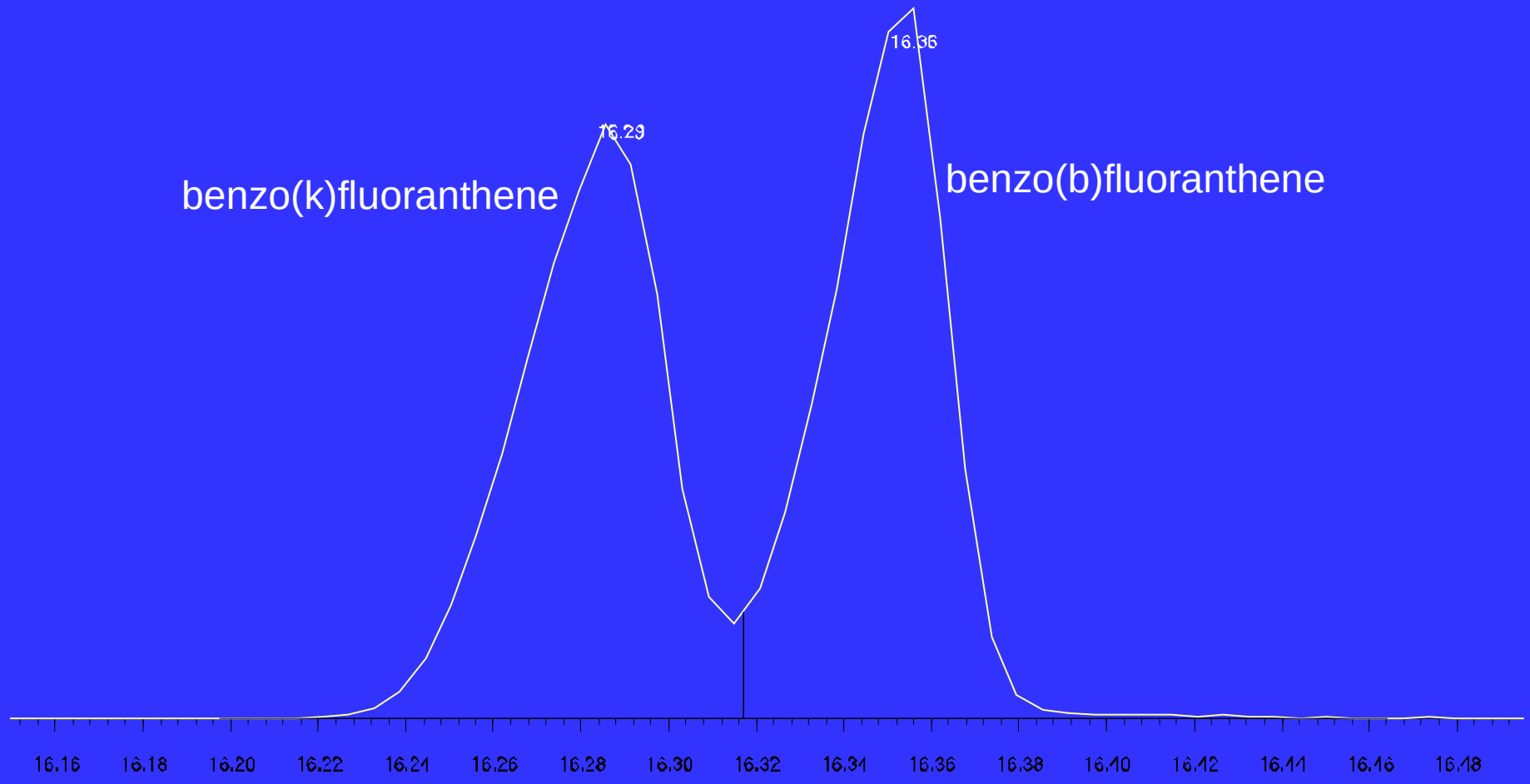

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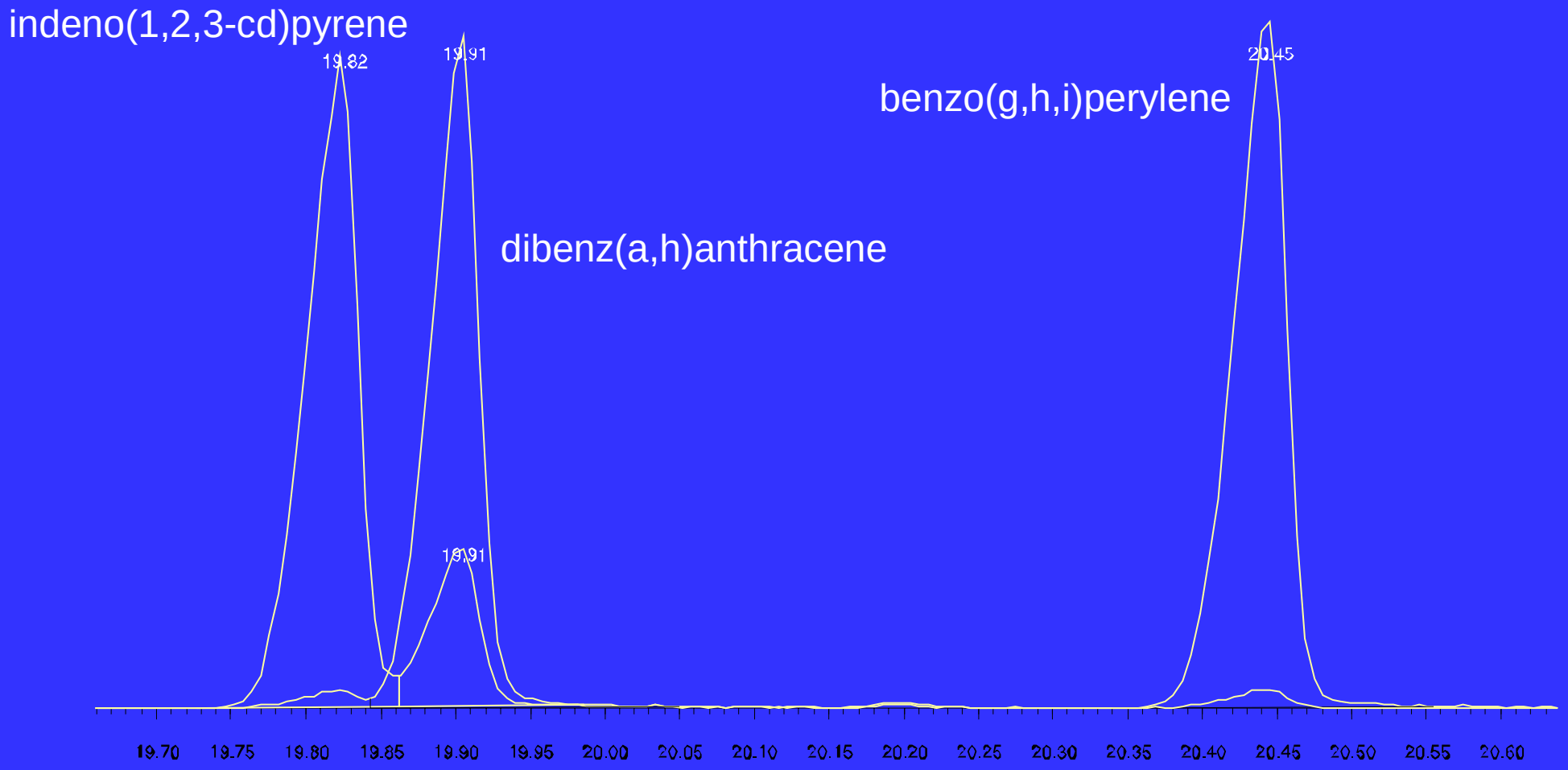
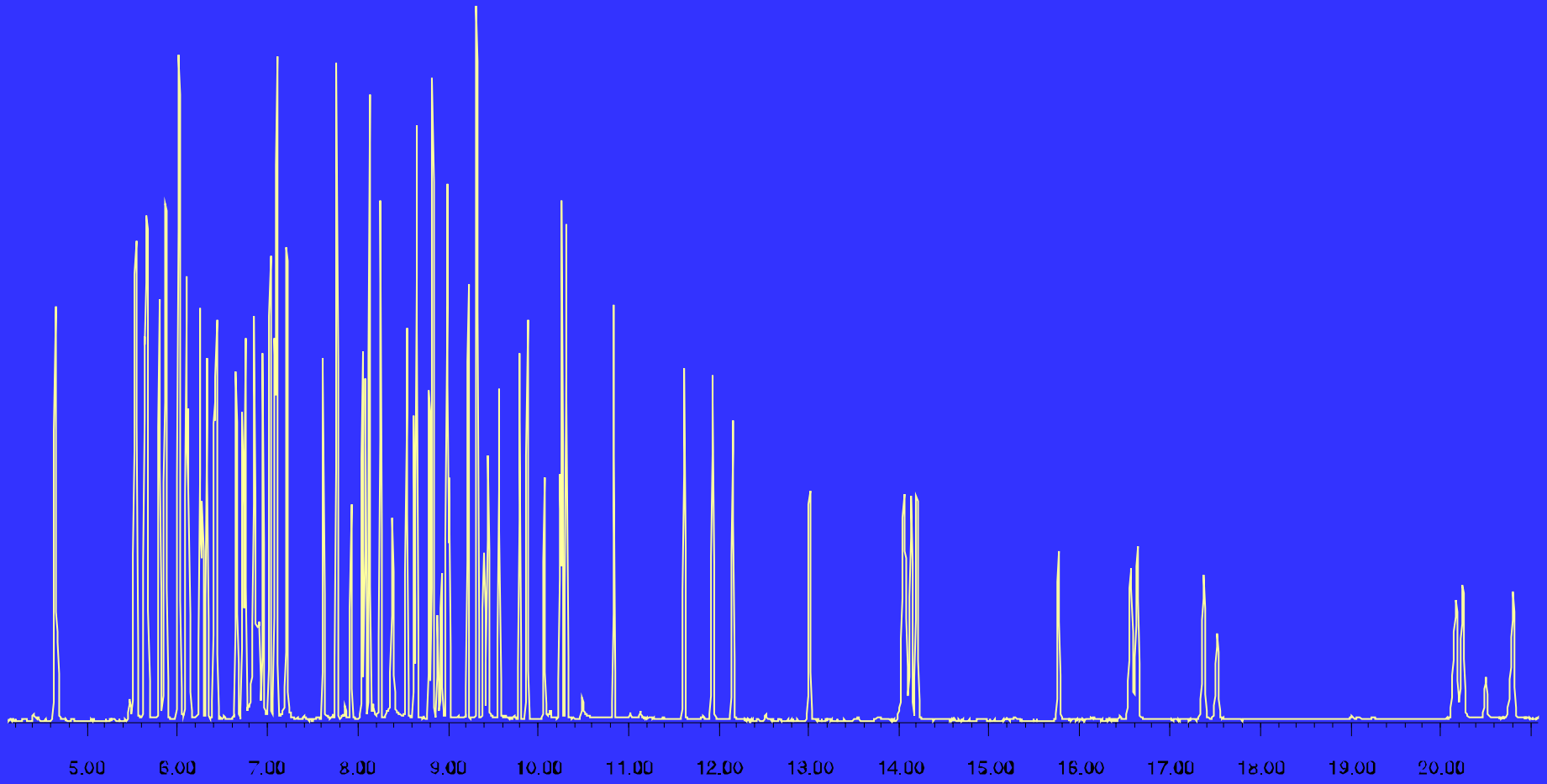


Rtx-5 MS Chromatogram
(30m x 0.25 mm ID, 0.25µm film)



Conclusion

- Faster analysis can be accomplished for 8270
- Keep compounds eluting on the temperature program ramp rate
- Faster elution of PAHs helps with column capacity due to lower k value