The Analysis of Semi-Volatiles with Various Inlet Liner Deactivations

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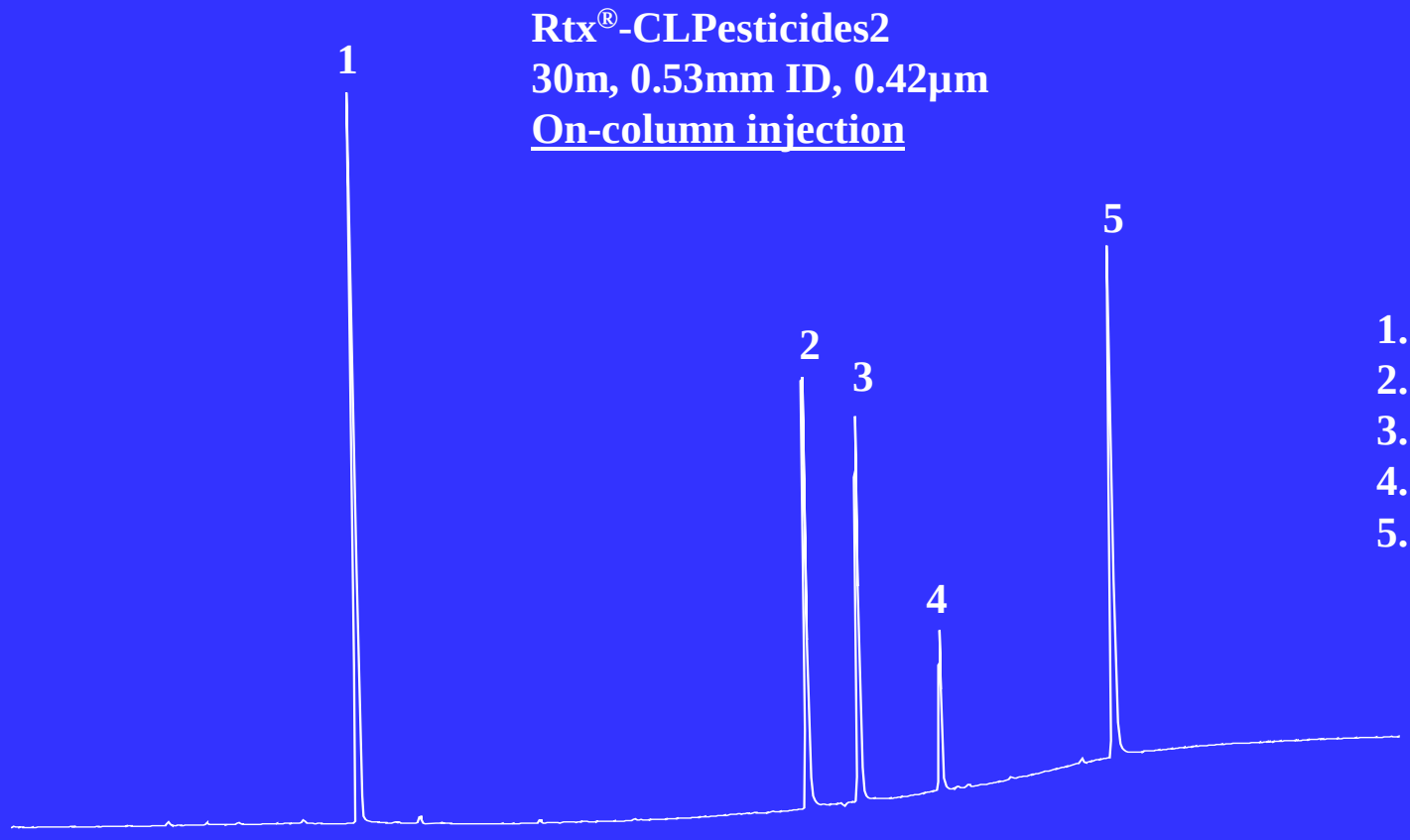
GC Inlet Liner Deactivation is Critical for Demanding Analyses

- Chlorinated pesticides
- Amines (basic compounds)
- Environmental semi-volatiles

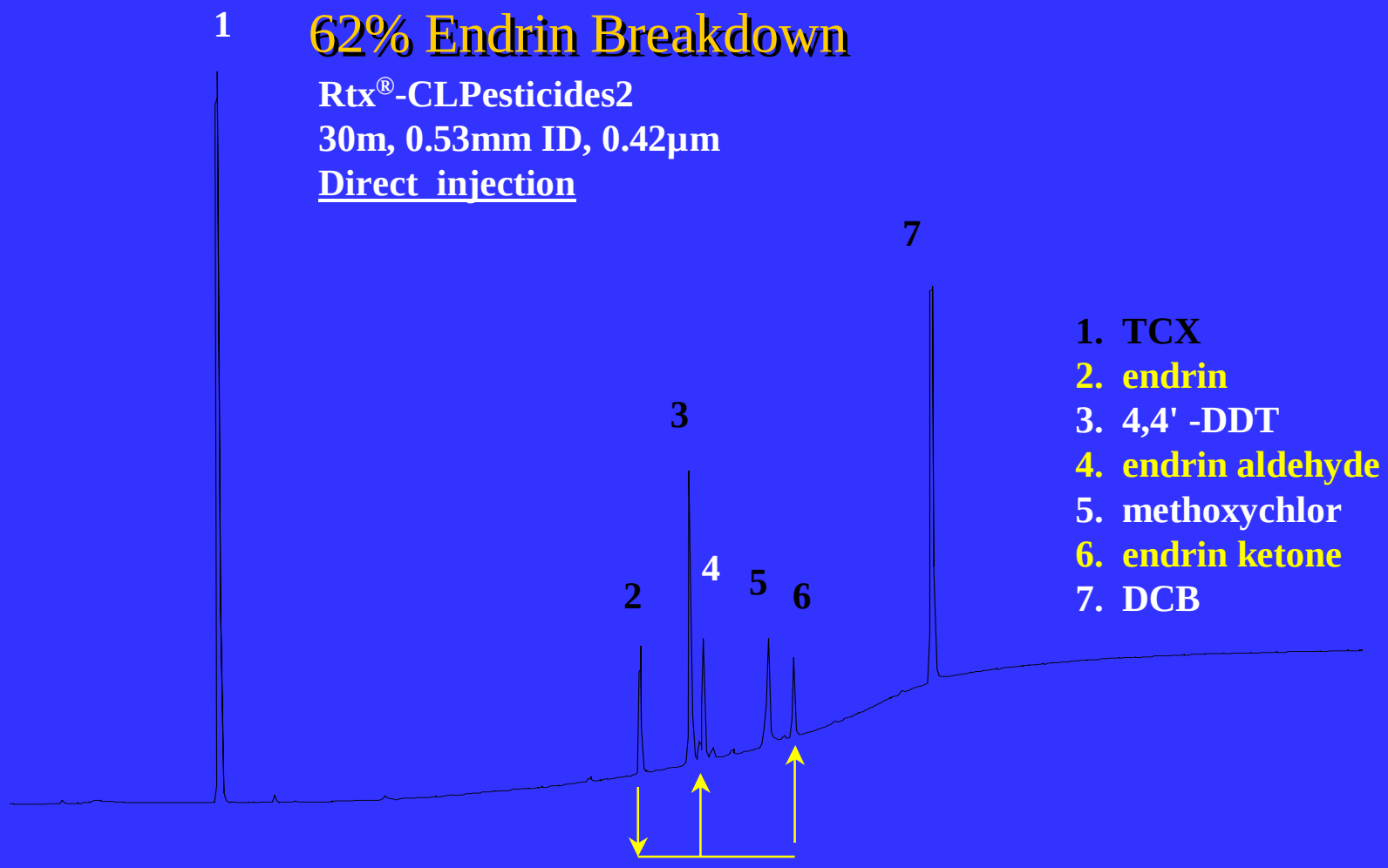
Criteria for Evaluating Inlet Liner Performance

- Breakdown of endrin and DDT
- Linear response for acids and bases
- Short conditioning times and low bleed
- Thermal stability
- Resistance to sample degradation

Pesticide Breakdown, On-Column



Raw Glass Inlet Liner



Types of Liner Deactivation Chemistry

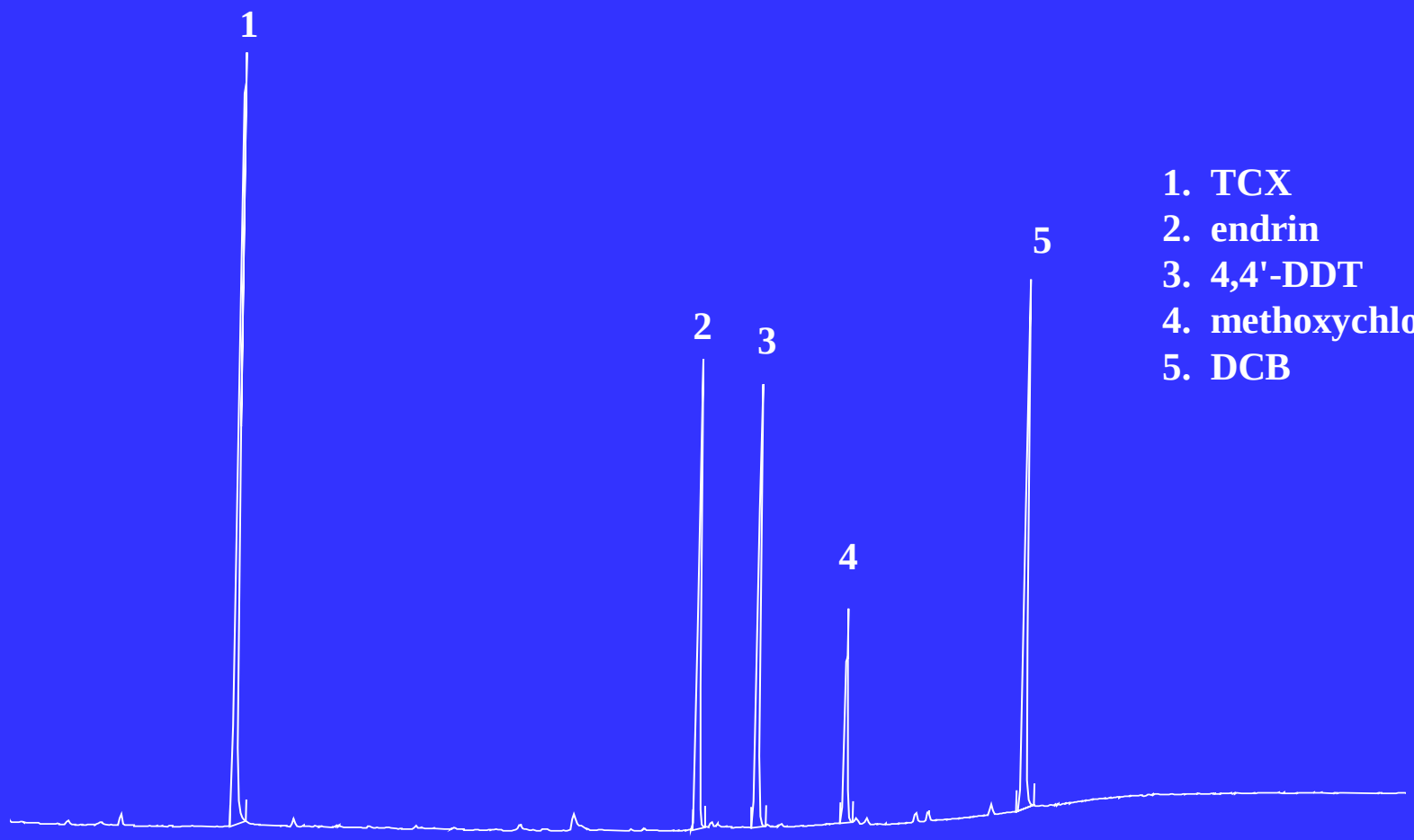
- Pinpoint
- Polymeric
- Base or amine
- Siltek™ Deactivation

Siltek™ Deactivation

- Inertness for acids, bases, neutrals, pesticides (developed for pesticides analysis)
- Low bleed
- Thermal stability
- Durability (acids, bases, water)
- Regeneration (solvent sonication: explosives analysis and customer feedback)
- Developing Siltek II for SemiVols+

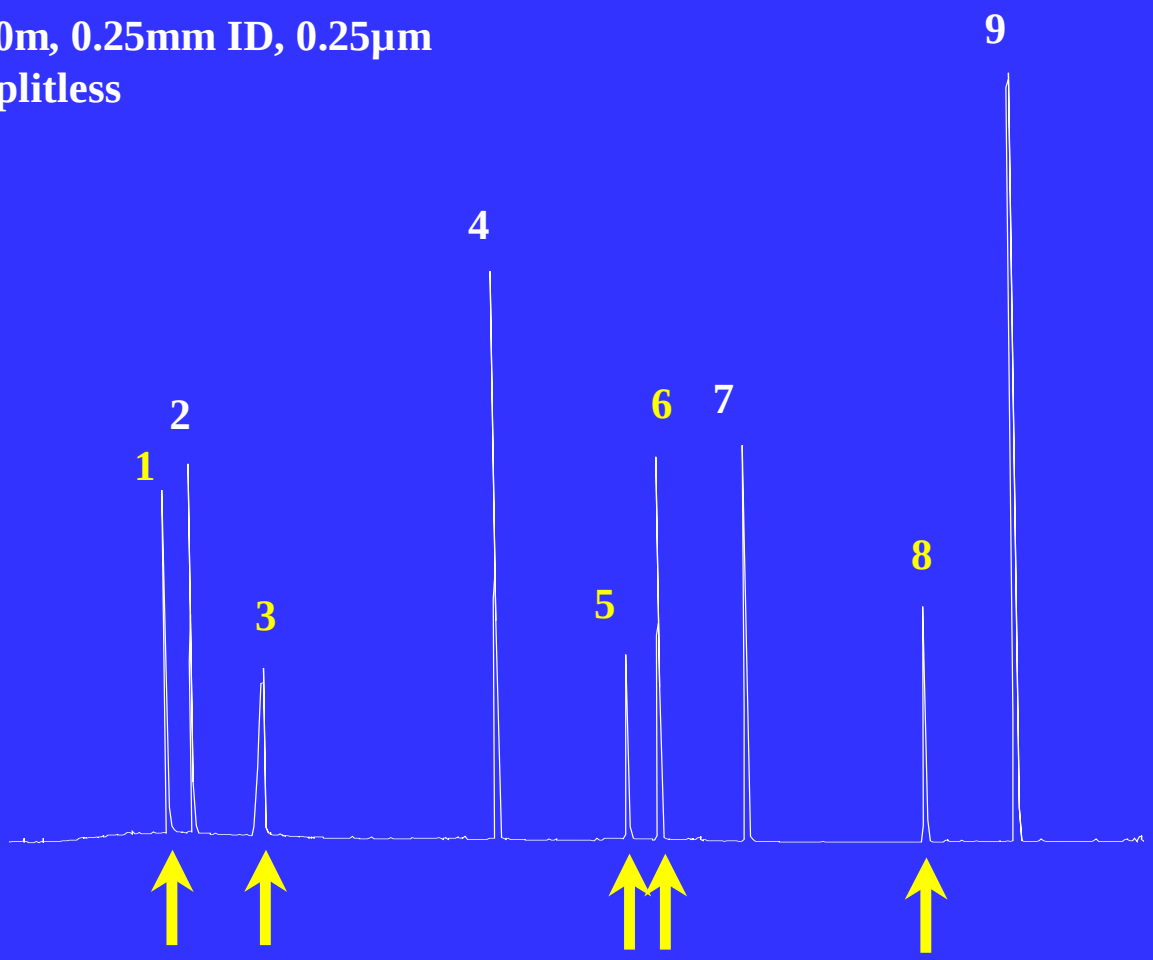
Siltek™ Inertness

1.5% Endrin Breakdown



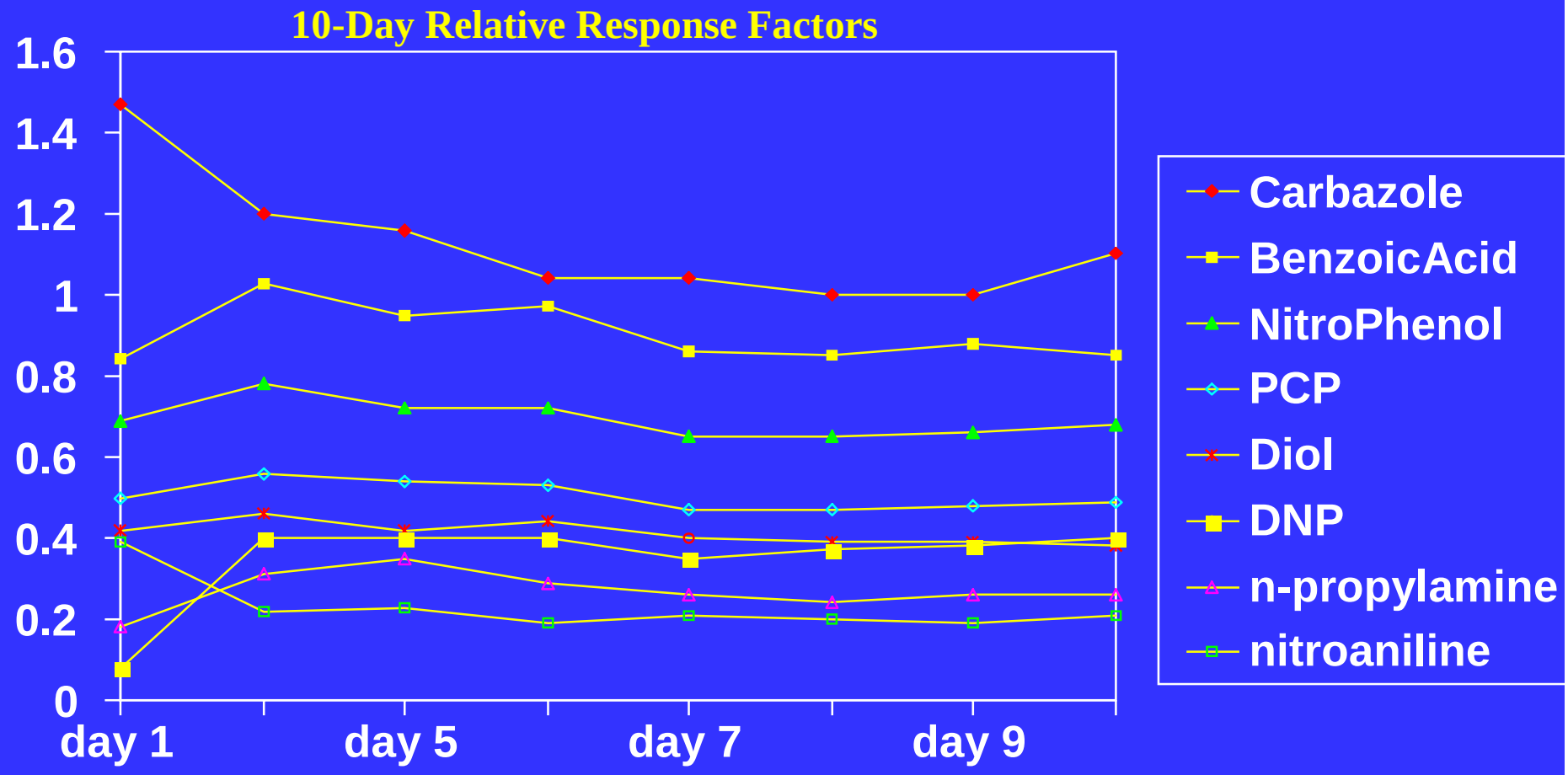
Acid Response Splitless XTI Injection

Rtx®-5
30m, 0.25mm ID, 0.25µm
Splitless



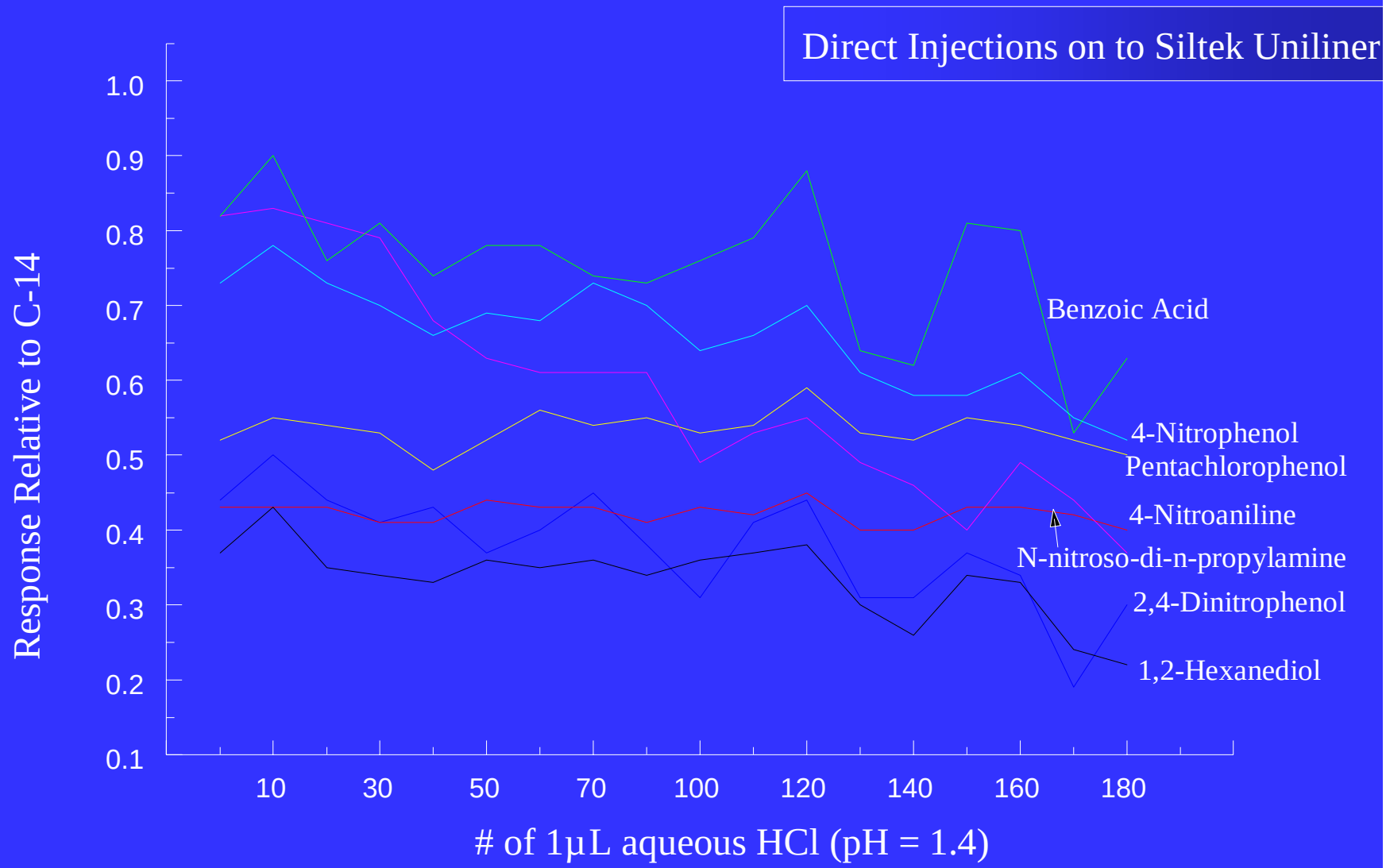
1. **1,2-hexanediol**
2. **nitro-di-n-propylamine**
3. **benzoic acid**
4. **C14**
5. **2,4-dinitrophenol**
6. **nitrophenol**
7. **nitroaniline**
8. **pentachlorophenol**
9. **carbazole**

Thermal Stability at 330°C

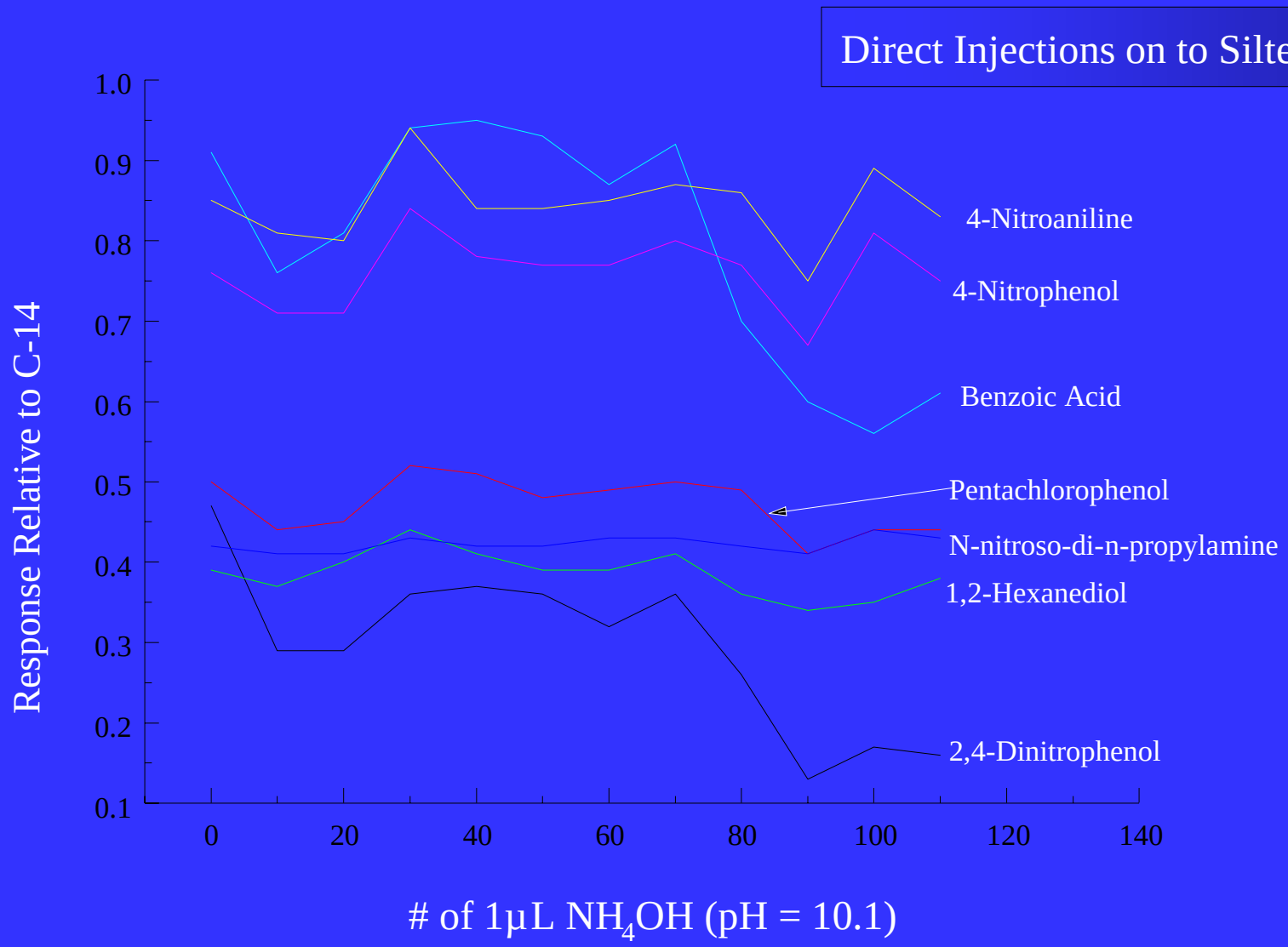


XTI-5, 30m, 0.25mm ID, 0.5µm, splitless Siltek™ liner; Inj.: 330°C; Det.: 330°C;
Oven temp.: 40°C (hold 2 min.) to 190°C @ 6°C/min to 330°C @ 30°C/min. (hold 10 min.)

Siltek Sleeve Resistance to Acidic Matrix



Siltek Sleeve Resistance to Basic Matrix



Chromatographic System

- Agilent 6890 GC
- Agilent 5973 GC/MS
- Restek Rtx-5Sil MS 30m x 0.28mm x 0.5um
- Constant flow rate @ 1.0 mL/min
- Temperature program:
  - 40°C (2 min); 20°C/min to 290°C (0 min); 2°C/min to 303°C (0 min); 6°C/min to 330°C (1 min)
- Splitless hold time 0.4min
- Single gooseneck 4mm ID liners

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
2-nitroaniline
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

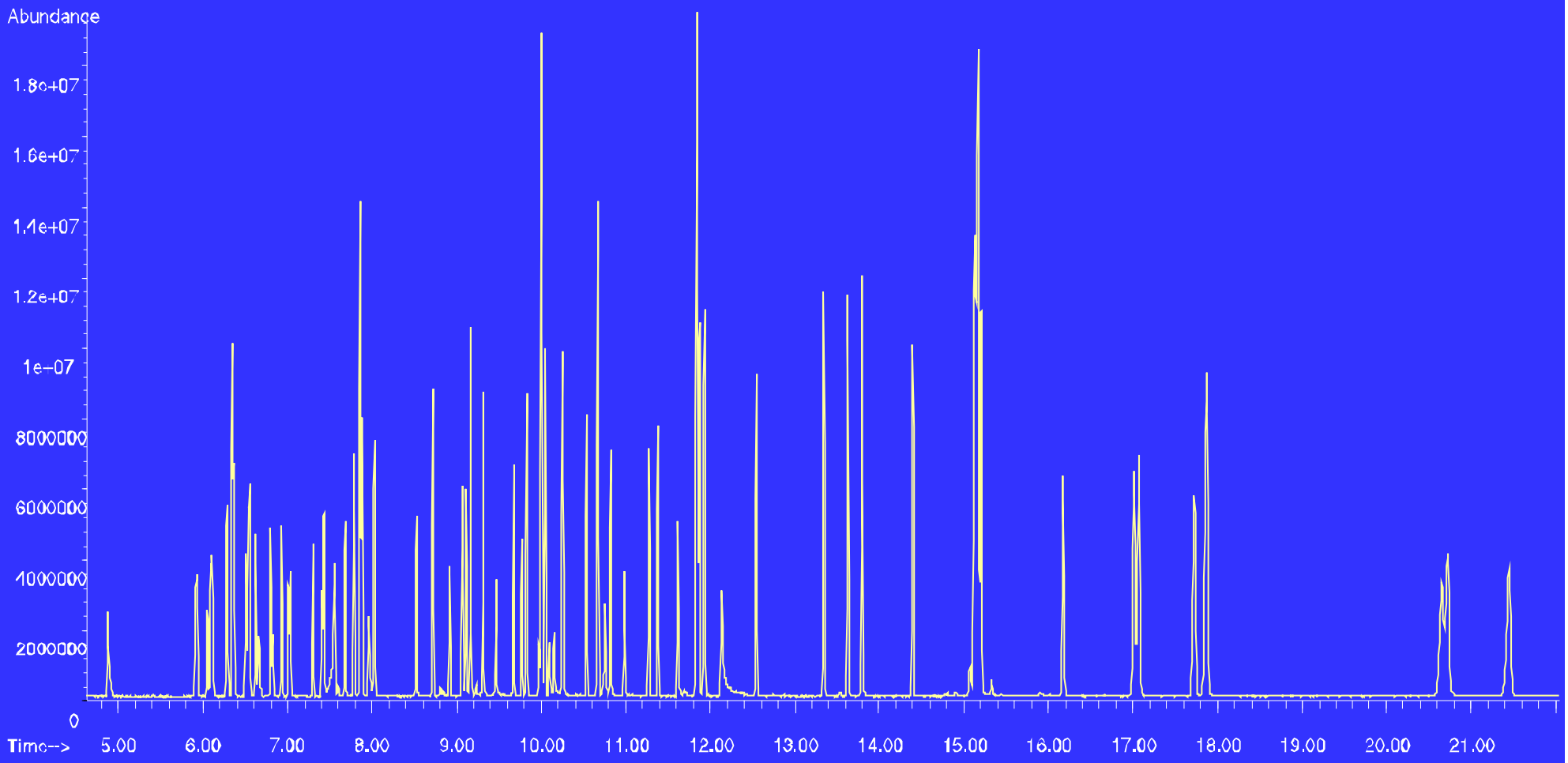
aldrin
a-BHC
a-chlordane
b-BHC
4,4'-DDD
4,4'-DDE
4,4'-DDT
d-BHC
dieldrin
endosulfan I
endosulfan II
endosulfan sulfate
endrin
endrin aldehyde
endrin ketone
g-BHC (lindane)
g-chlordane
heptachlor
heptachlor epoxide (B)
methoxychlor

Criteria for Trace Analysis
EPA Method 8270C

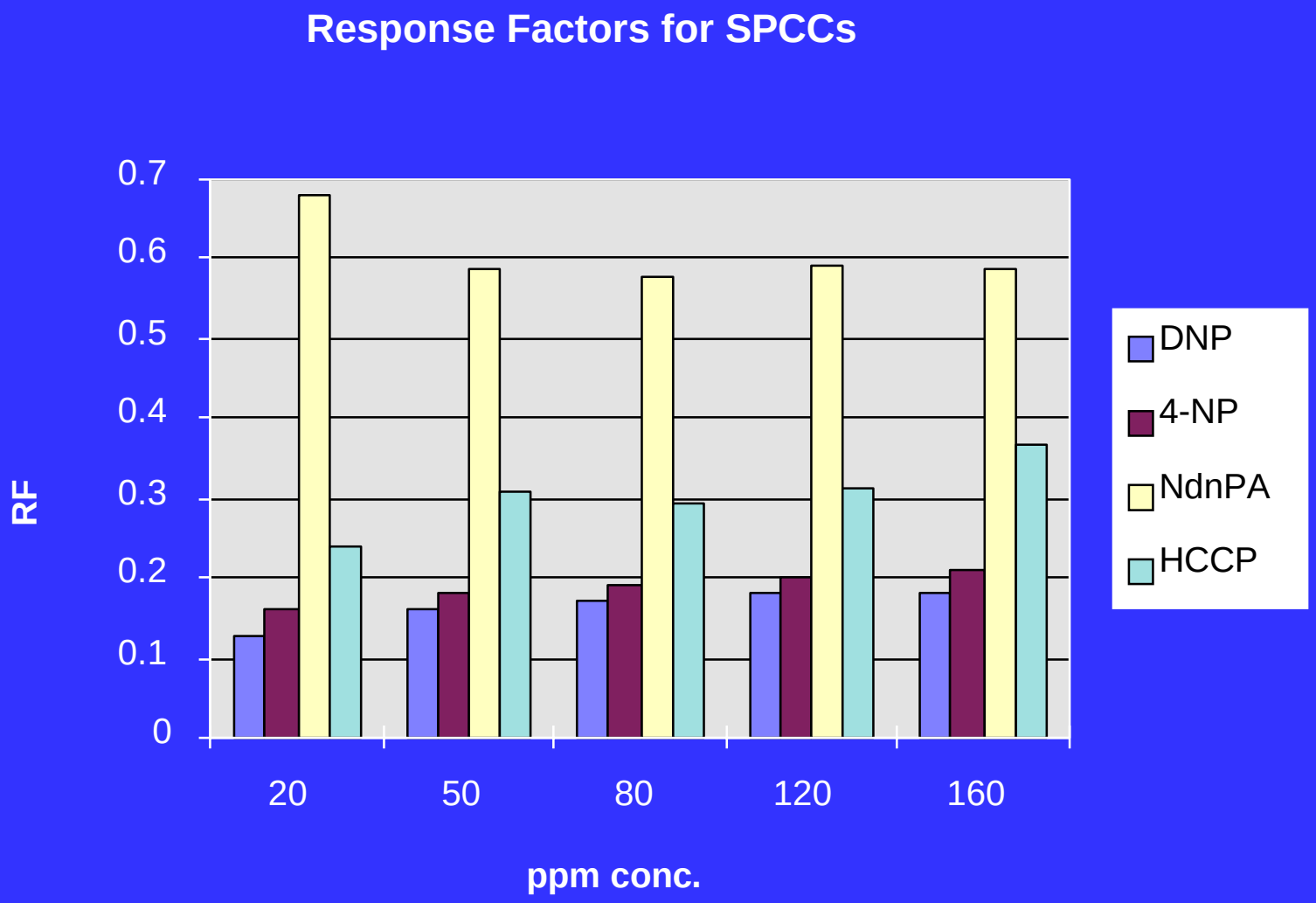
- Linearity of response relative to ISTD 20 - 160ng/µL
- Minimum relative response factor (RRF) and % relative standard deviation (RSD)

	Min. RRF	Min. %RSD
2,4-dinitrophenol	0.050	15
4-nitrophenol	0.050	15
N-nitroso-di-n-propylamine	0.050	15
hexachlorocyclopentadiene	0.050	15

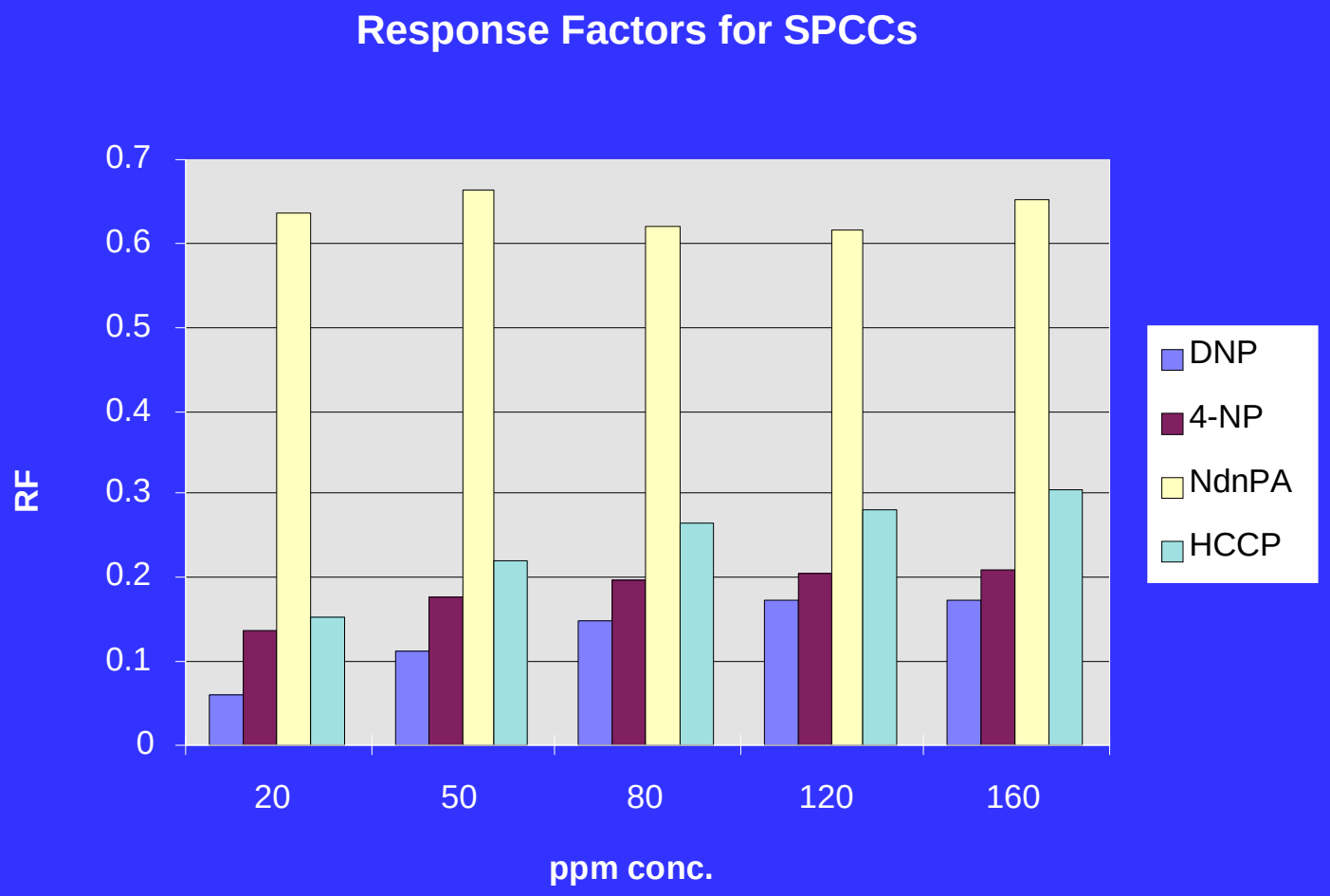
Sample Chromatogram



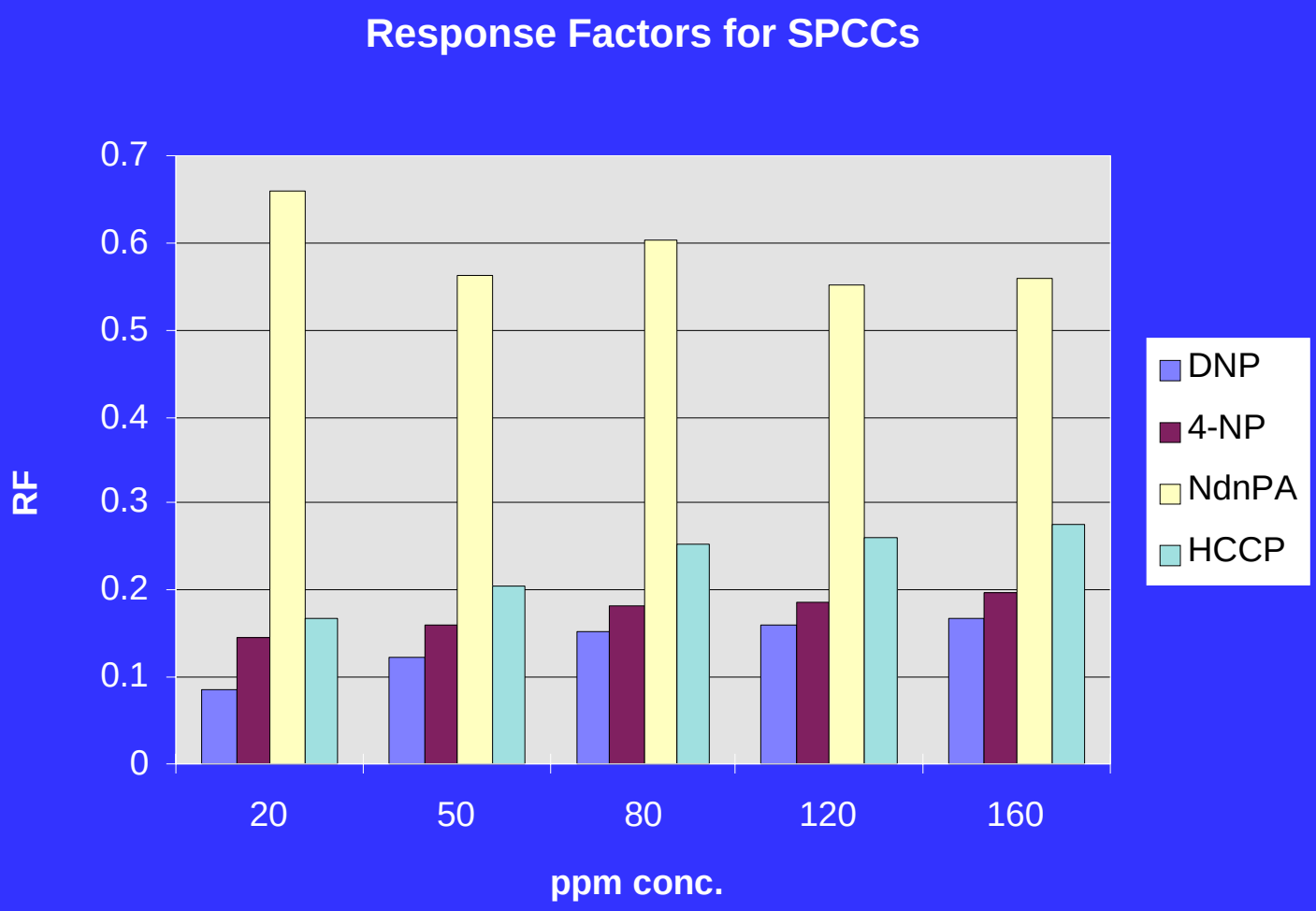
Standard Liner Response



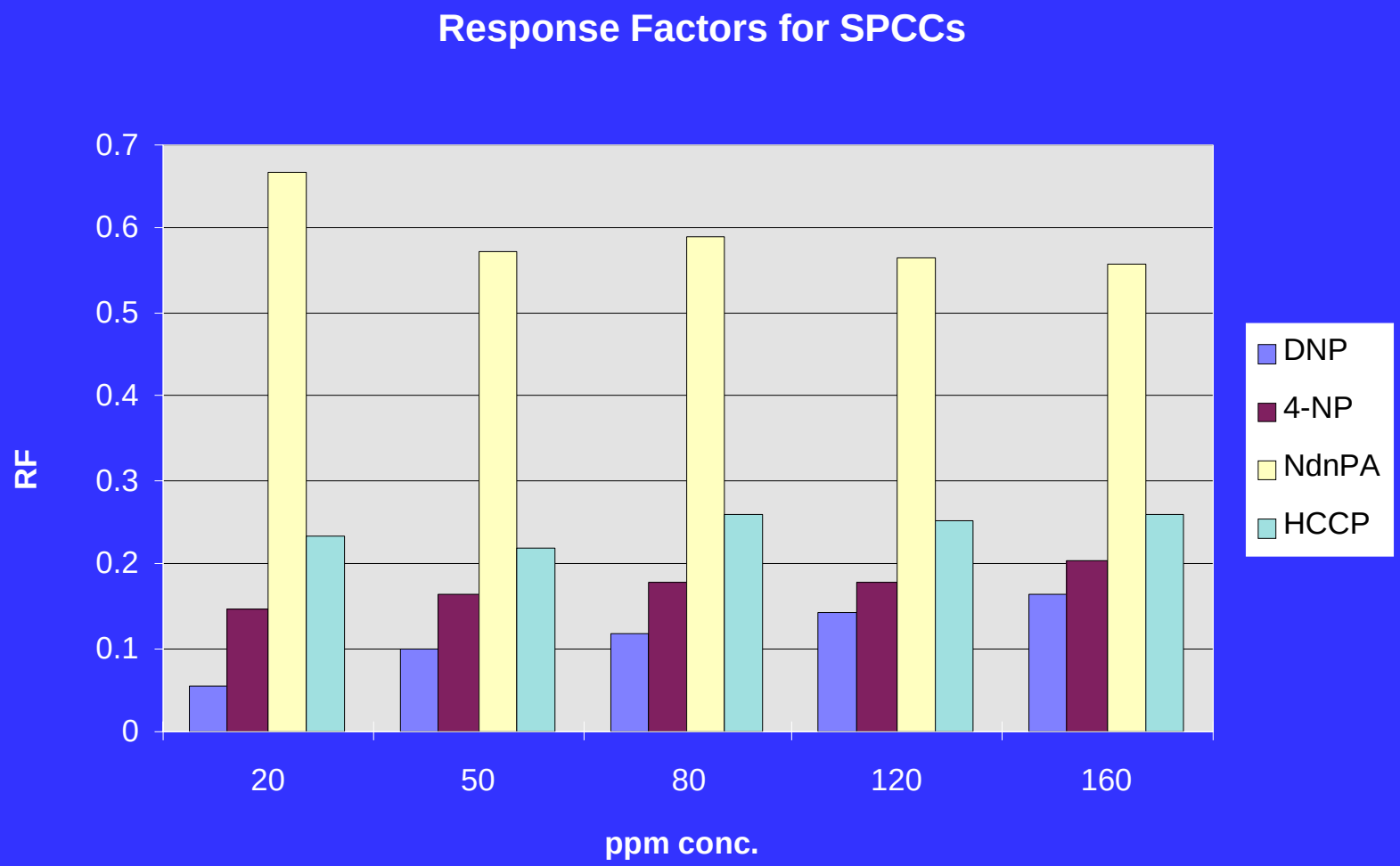
Standard Siltek Liner Response



Experimental Siltek-A Response



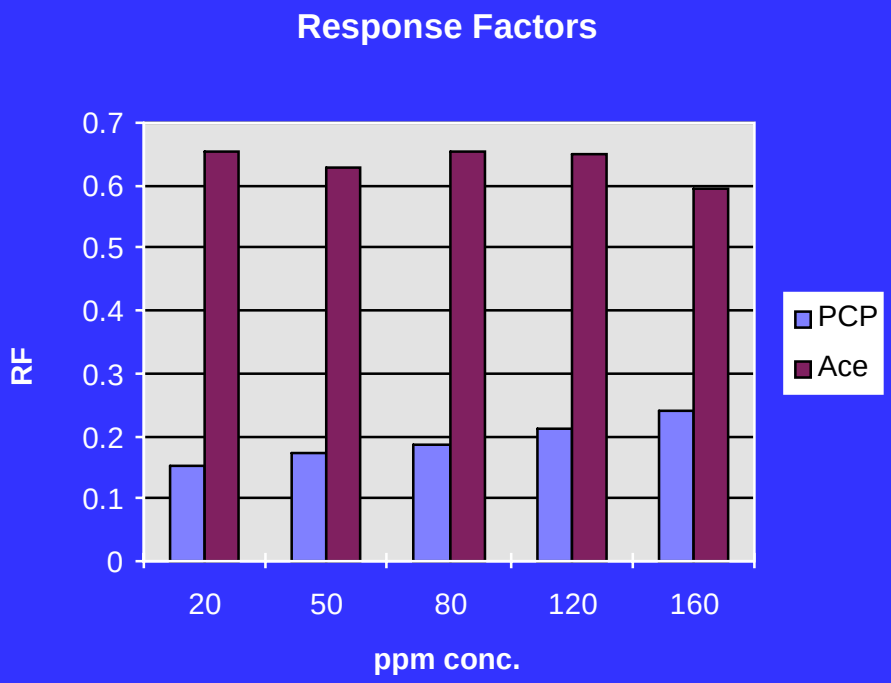
Experimental Siltek-B Response



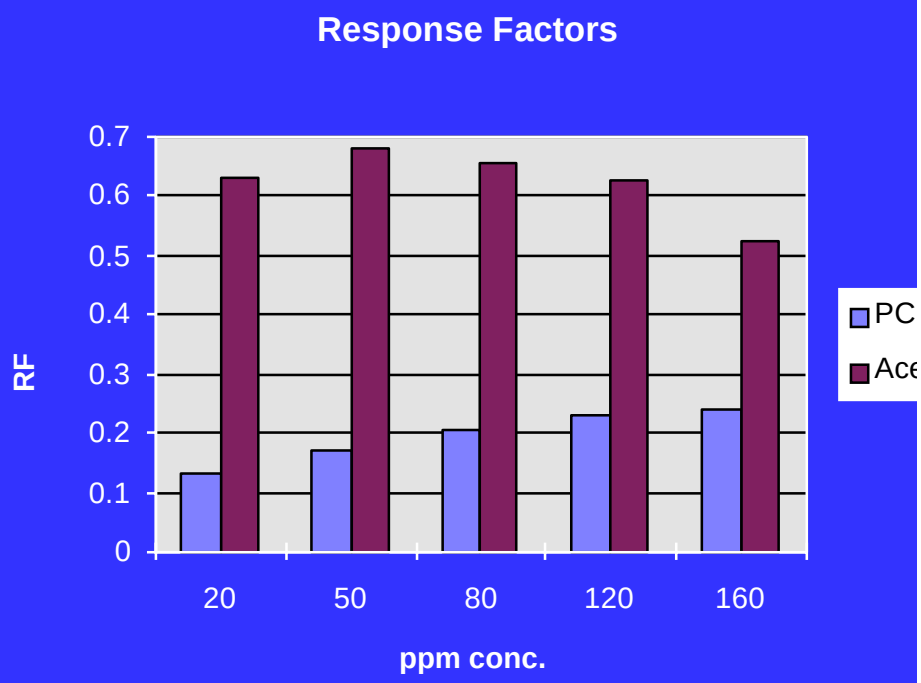
Relative Standard Deviation Comparison

	Standard	Siltek	Siltek A	Siltek B
DNP	13.6	35.1	24.7	36.1
4-NP	9.0	16.2	12.4	12.1
NdnPA	7.0	3.2	7.6	7.6
HCCP	14.8	24.5	19.1	7.4

Results of Other Test Probes - Standard Liner and Siltek Liner

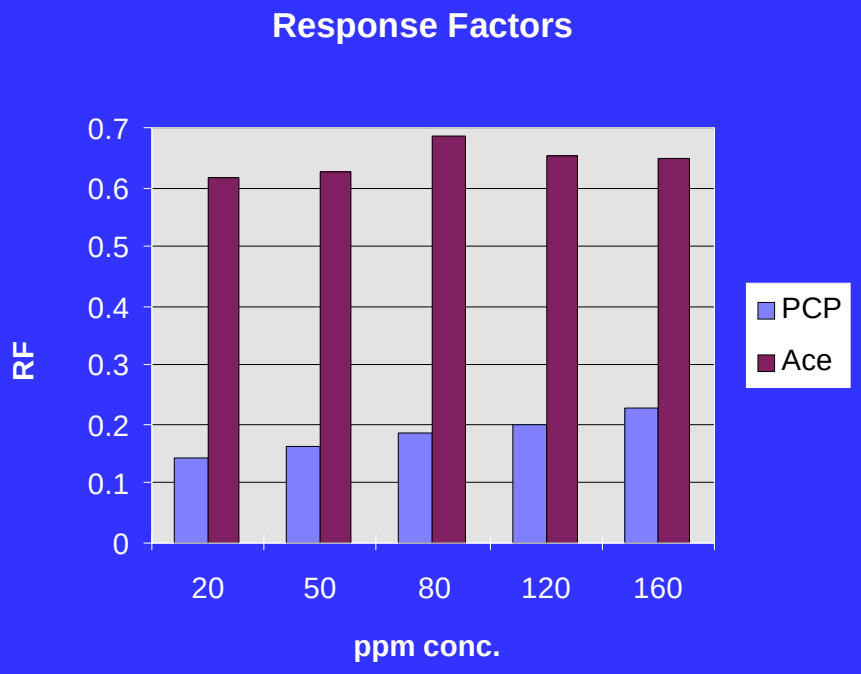


Standard

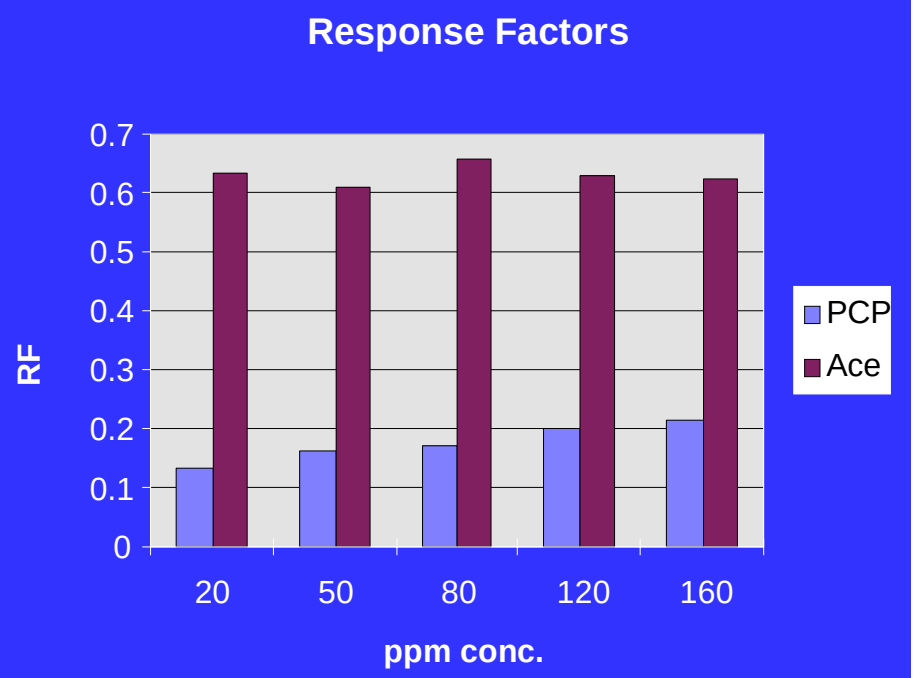


Siltek

Results of Other Test Probes - Siltek A & B



Siltek A



Siltek B

Other Test Probes: RSDs

	Standard	Siltek	Siltek A	Siltek B
PCP	17.8	22.3	18.0	17.9
Acenapthylene	4.0	9.7	4.3	2.8

Conclusions

- Current Siltek™ products allow a wide variety of analyses, except at acidic and basic extremes
  - standard liners for acidic (i.e., semivols)
  - base-deactivated for amines (ethanolamines)
  - Siltek for pesticides, explosives, sulfurs, volatiles, petrochemical, high temp., caustic, etc.
- Progressing on Siltek II which will also allow highly efficient Semi-Volatile analyses
- Future - Siltek III to include amines (i.e., one surface for all analyses)