Review of Liner Selection Criteria for Gas Chromatographic Analysis

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Project Objective

The objective is to investigate the effects of intermediate polarity, Siltek™, and base deactivation, and liner geometry for the analysis of neutral, acidic, and basic compounds in EPA Method 8270.

Overview

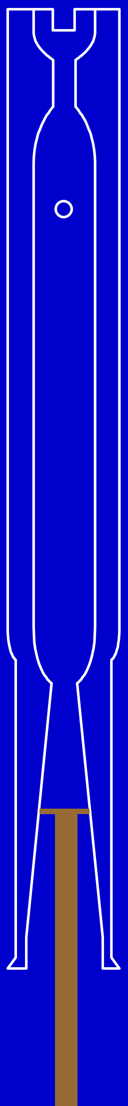
- Compare inlet liner deactivations
  - Intermediate Polarity, Siltek™, Base
- Effects of liner geometry
  - Single gooseneck, Drilled Uniliner®, double gooseneck, Cycloliner
    - Injection conditions
      - Constant flow
      - Pressure pulse

Inlet Sleeve Deactivation

- Standard Intermediate Polarity (IP)
  - Polymeric deactivation
- Siltek™ Deactivation
  - Chemical vapor deposition
- Base deactivation
  - Deactivation leaves a basic character to the glass surface

Experimental Conditions

- Rtx®-5Sil MS
  - 30m x 0.25mm ID, 0.25um film
- Drilled Uniliner®
  - Eliminate metal contact in injection port
- Standard concentration
  - 4, 10, 16, 24, 32, 80 ng/µl
  - ISTD at 8 ng/µl
- 1µl injections, 0.4 min. purge time
- Injection port temp. at 300°C
- HP 6890 w/5973 GC/MS
- 35°C (2 min.) 20°C/min. 260° (0 min.) 6°C/min. 330° (1 min.)



Compound List

- Standard mix: 104 compound mix of US EPA 8270 list including ISTD
- Compounds used for comparisons:
  - Neutral compounds
    - Benzo(b)fluoranthene
    - Benzo(ghi)perylene
  - Acidic compounds
    - 2,4-dinitrophenol
    - Pentachlorophenol
  - Basic compounds
    - N-nitrosodimethyl amine
    - N-nitroso-di-n-propyl amine
    - Benzidine

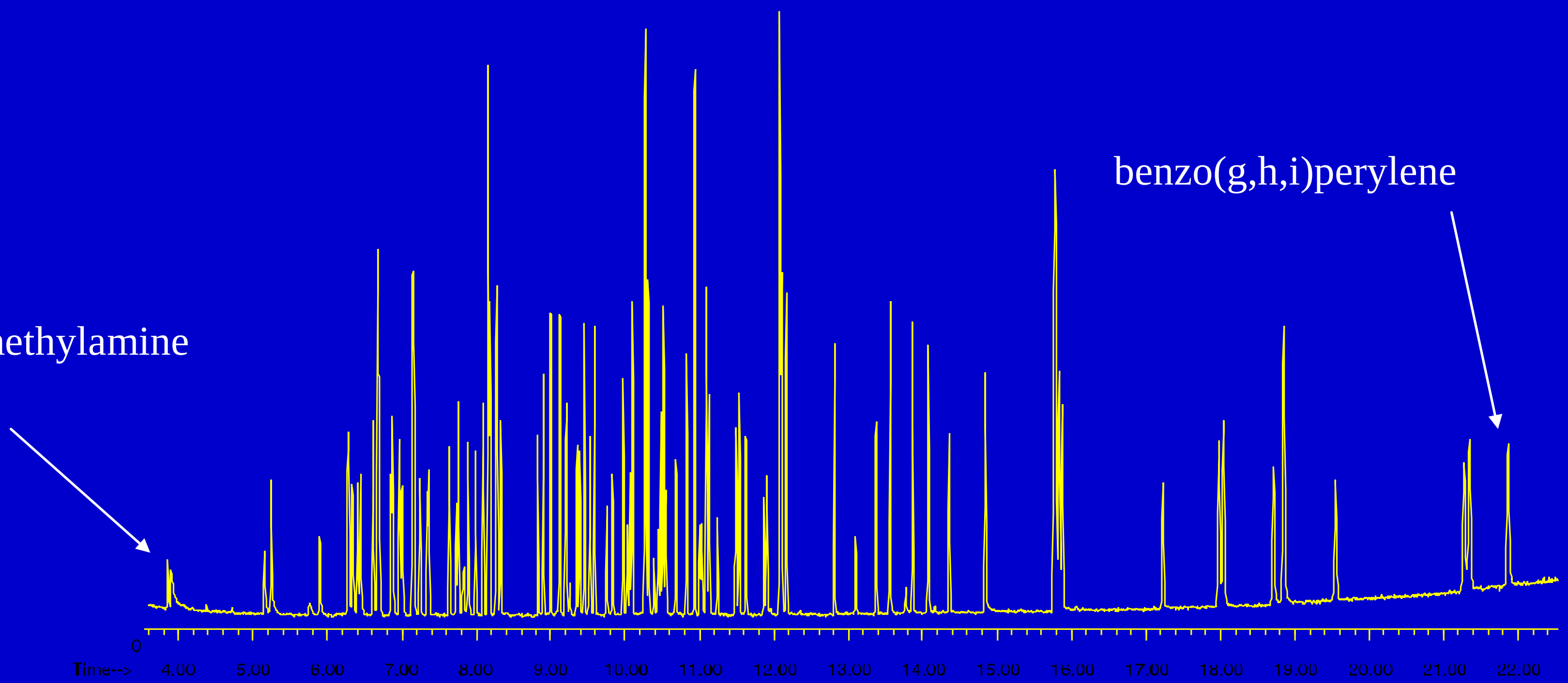
4ppm 8270 Calibration Standard

- Excellent signal-to-noise for 4ng on-column injection
- Low column bleed

benzo(g,h,i)perylene

N-nitrosodimethylamine

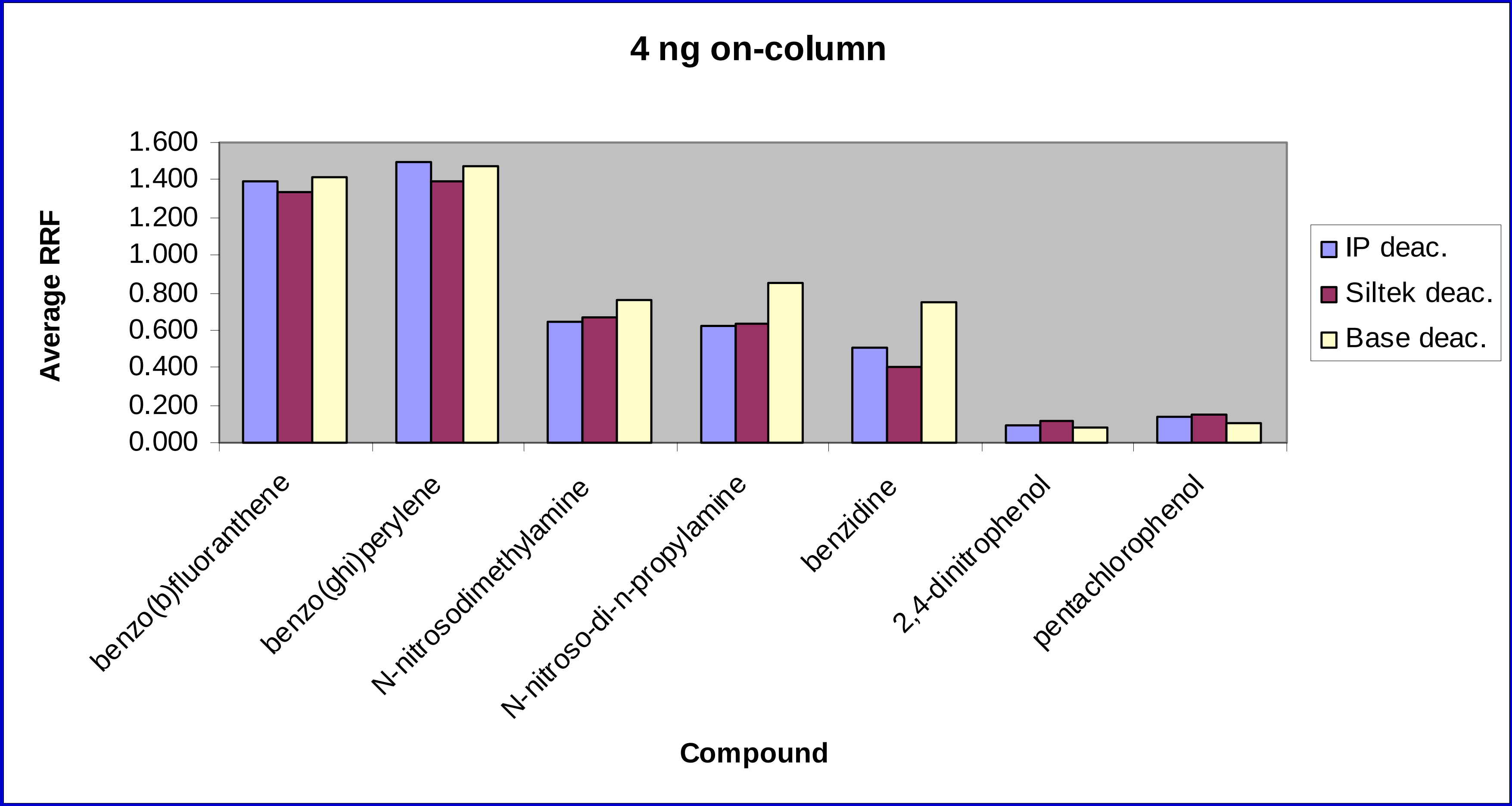
& pyridine



Comparison of Deactivations

- Deactivated Drilled Uniliner®
  - IP, Siltek™, and base procedure
- Run sequence
  - 7 reps at 4ppm
    - Show largest difference in RRF due to active sites
  - Calibration curve
    - 4, 10, 16, 24, 32, and 80 ppm
    - ISTD at 8ppm

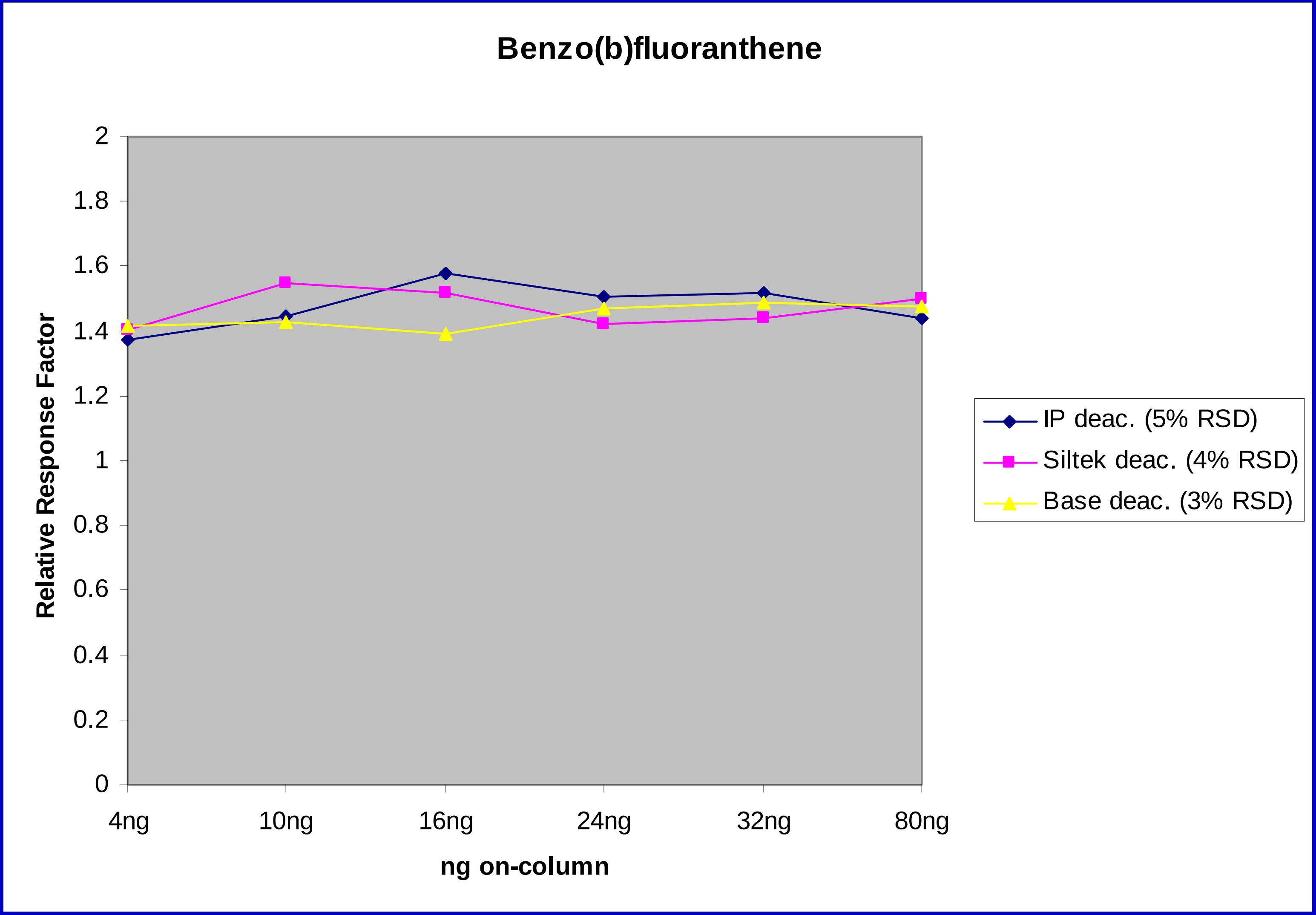
Liner Deactivation
Average RRF from 4ppm Standards



Effects of Deactivation on Linearity

- Response factors of 4ppm standard gave a good indication of the activity of the liner surfaces.
- What are the effects of deactivation on linearity?

Effects of Deactivation on Linearity



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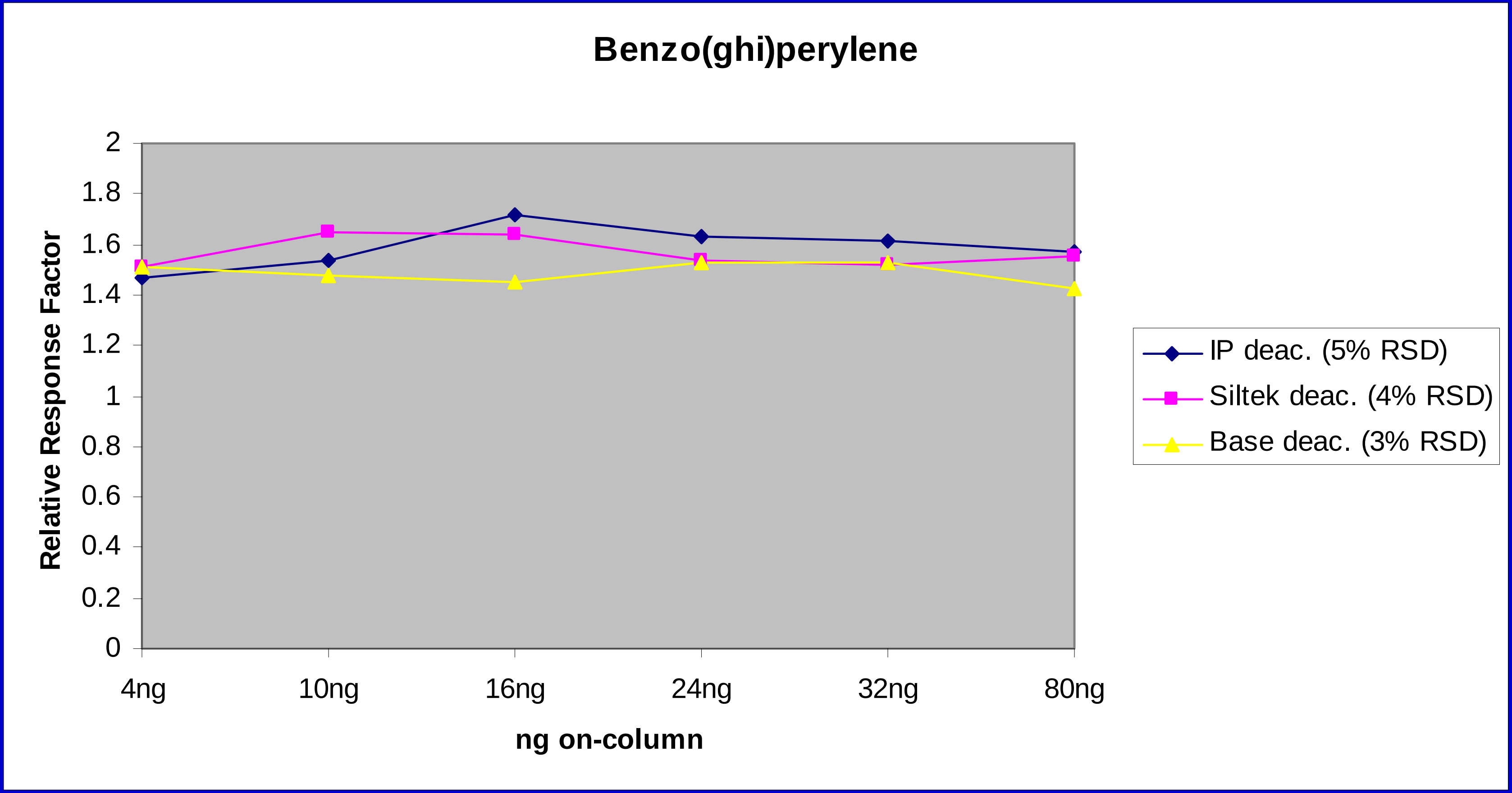

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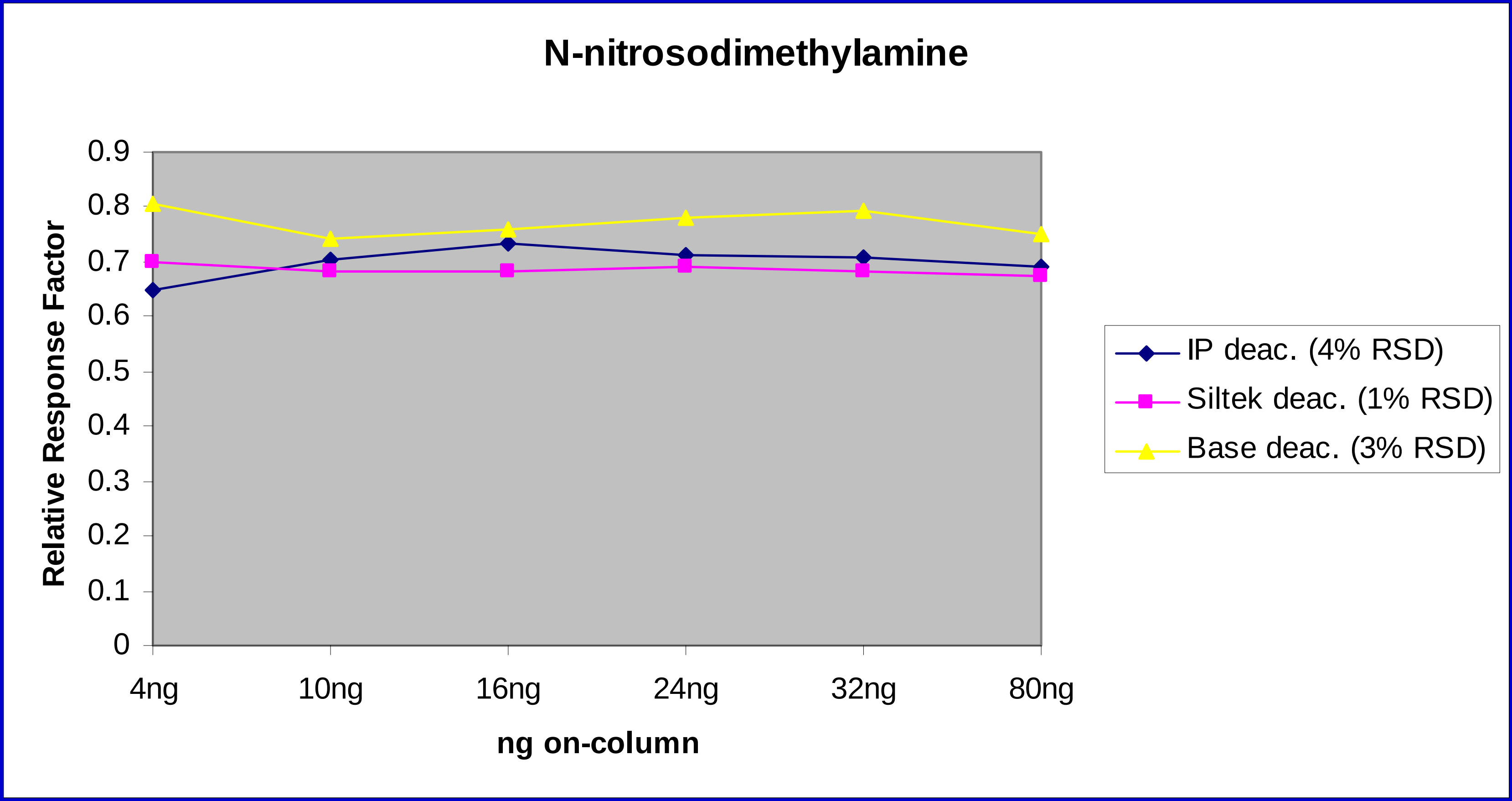

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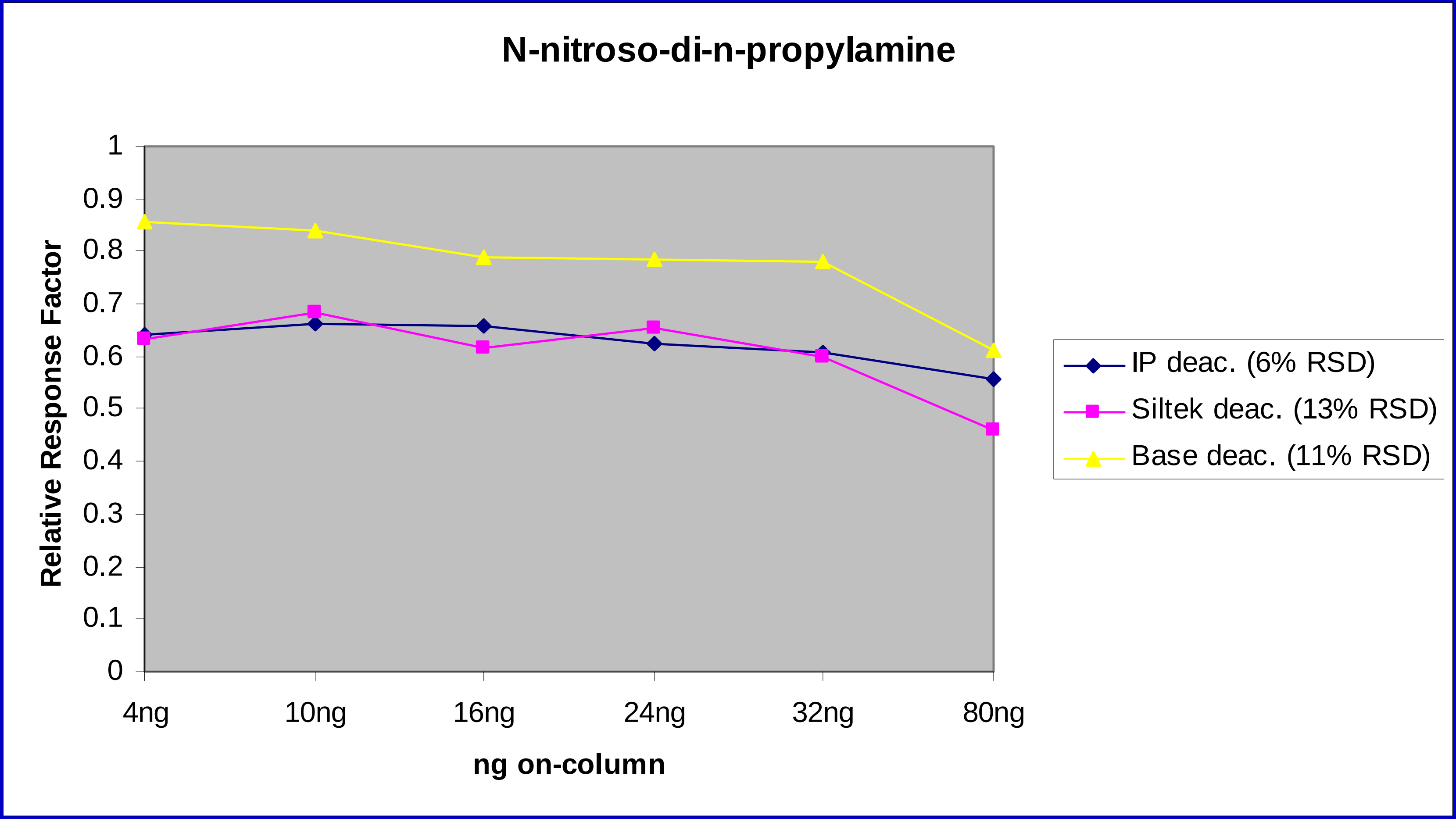

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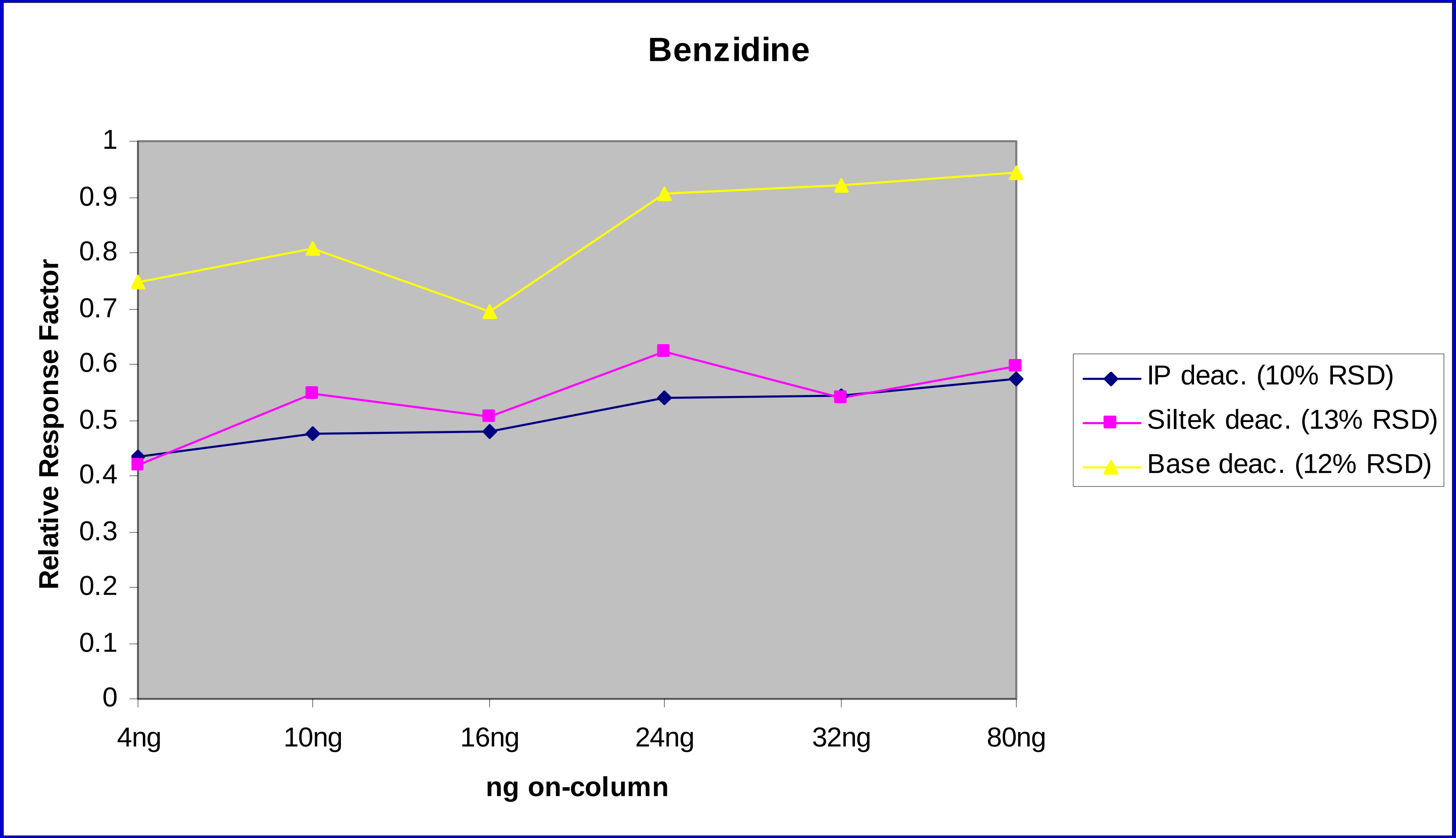

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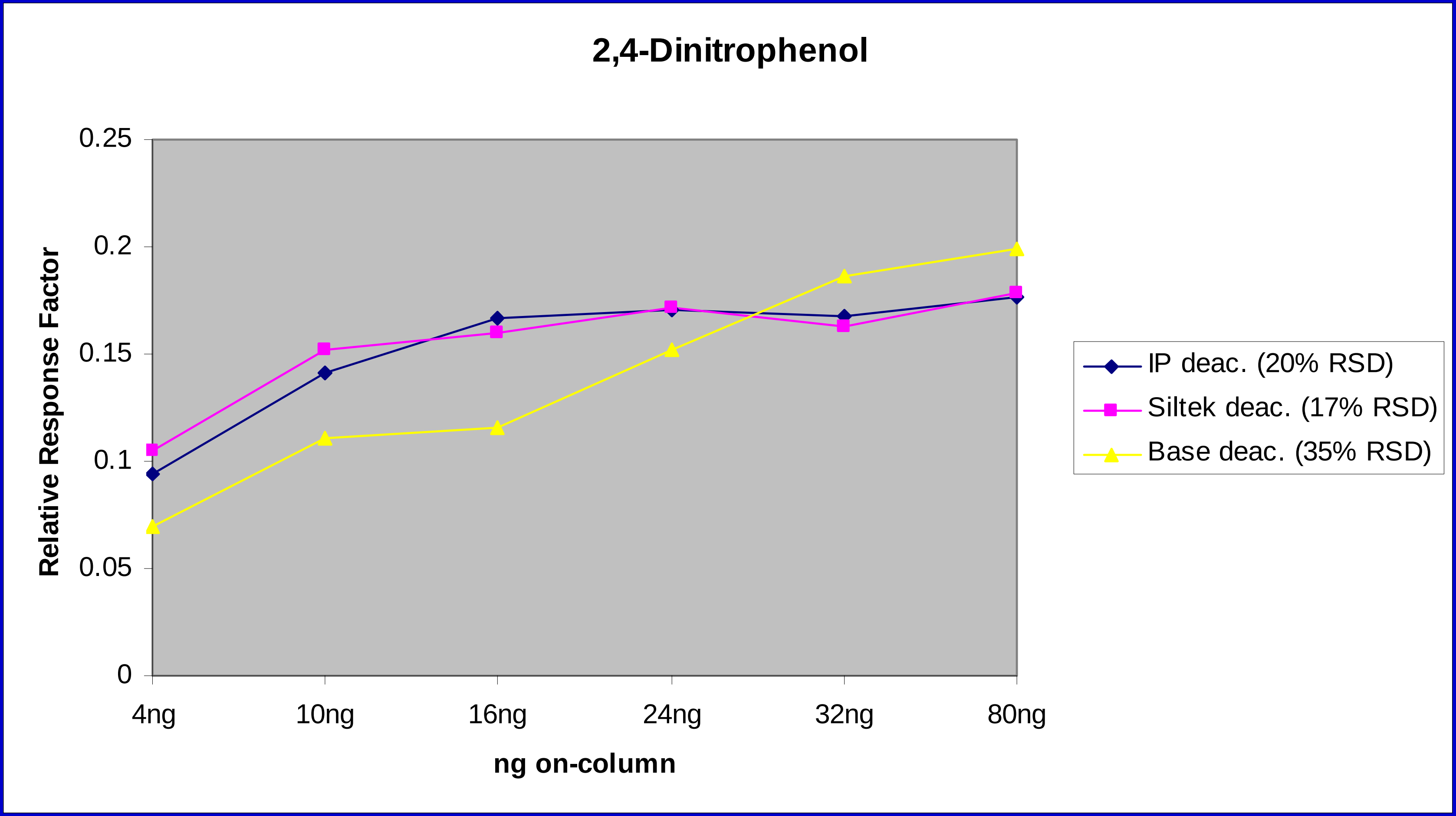

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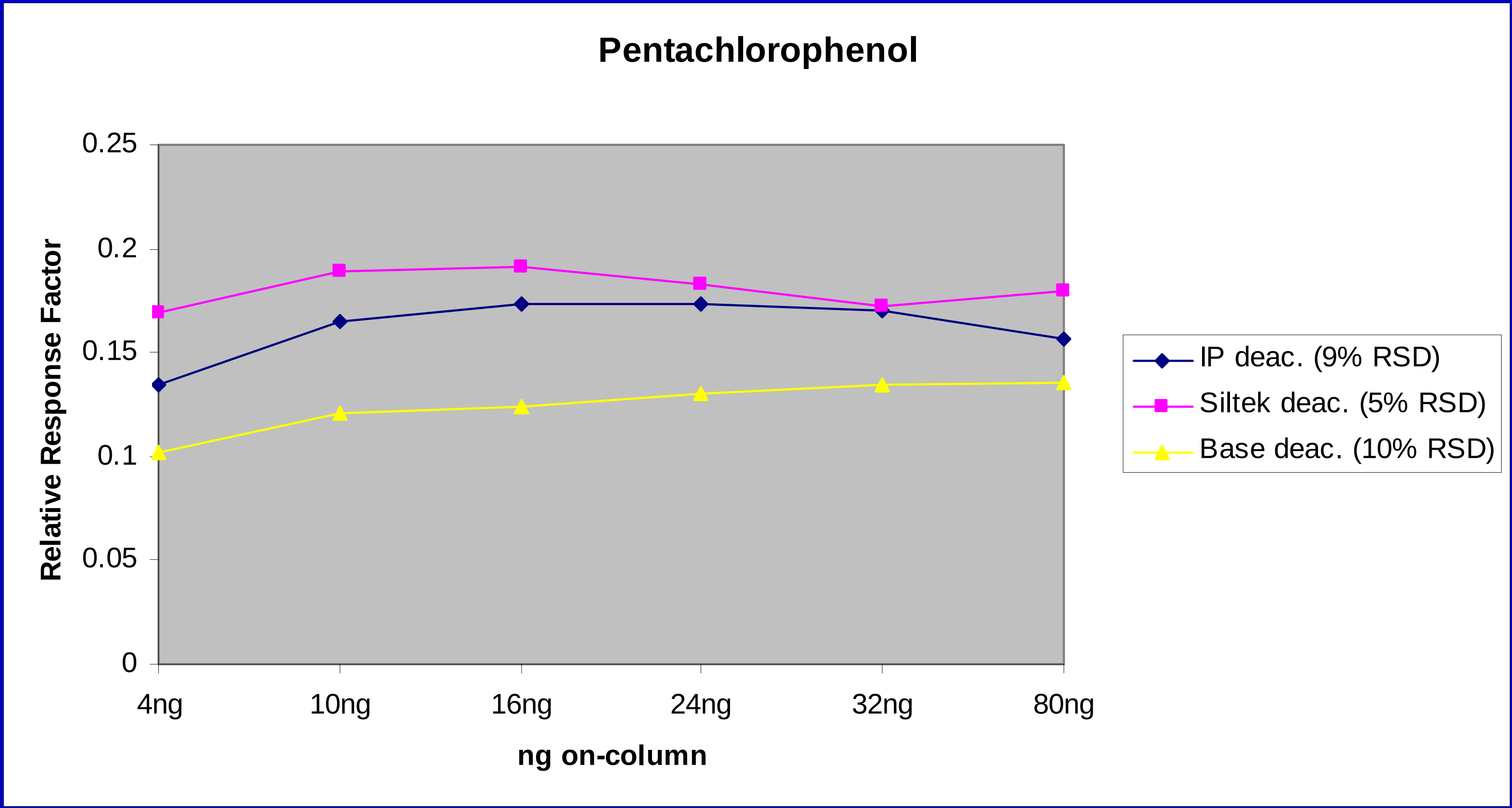


Summary of Deactivation

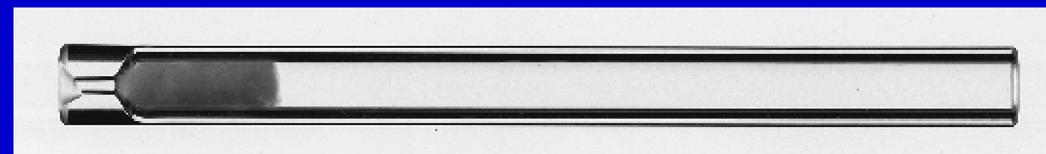
- The combination of response factors and linearity give a good picture of the effects of liner activity.
- Base deactivation results in low phenol compound response and variable linearity.
- IP and Siltek™ both exhibited acceptable response factors and linearity.

Liner Geometry

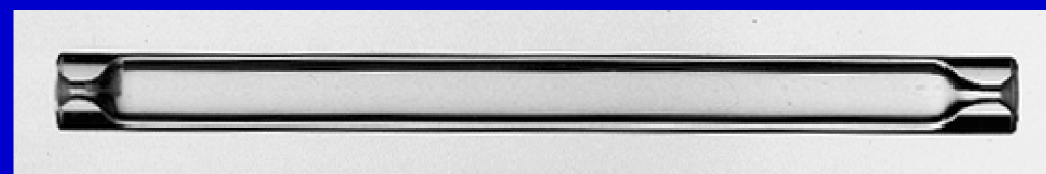
- Purpose
  - Vaporize sample prior to column
  - Shield sample from active metal parts of the injection port
- Problems
    - Need surface area and time to help vaporize sample
    - Opening at both ends of liner allows vapor cloud to expand out of glass liner, exposing sample to active sites

Liner Geometry

Single Gooseneck

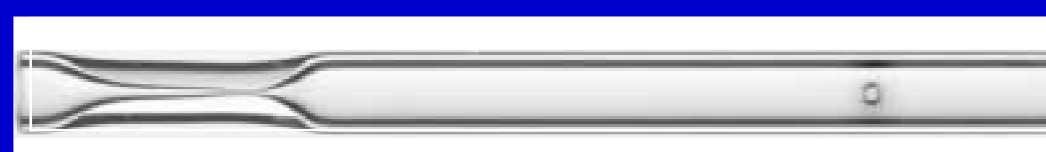


Double-Gooseneck



Cyclo Double
Gooseneck

Drilled Uniliner



Experimental Conditions for Liner Geometry

- Same conditions as deactivation study
  - Did not optimize conditions for each liner.
- 2 injection conditions
  - 1mL/min. constant flow (CF)
  - Pressure pulse (PP)
    - 30psig for 0.5 min., then constant flow at 1mL/min.
- Run sequence
  - 4, 10, 16, 24, 32, and 80ppm

Results of Liner Geometry

- Visual chromatographic differences
- Compare relative response factors (RRF) for different liner geometries
  - Pressure pulse versus constant flow
  - Average over 6 point curve
- Compare differences in linearity (%RSD)
  - Pressure pulse versus constant flow
  - Average over 6 point curve

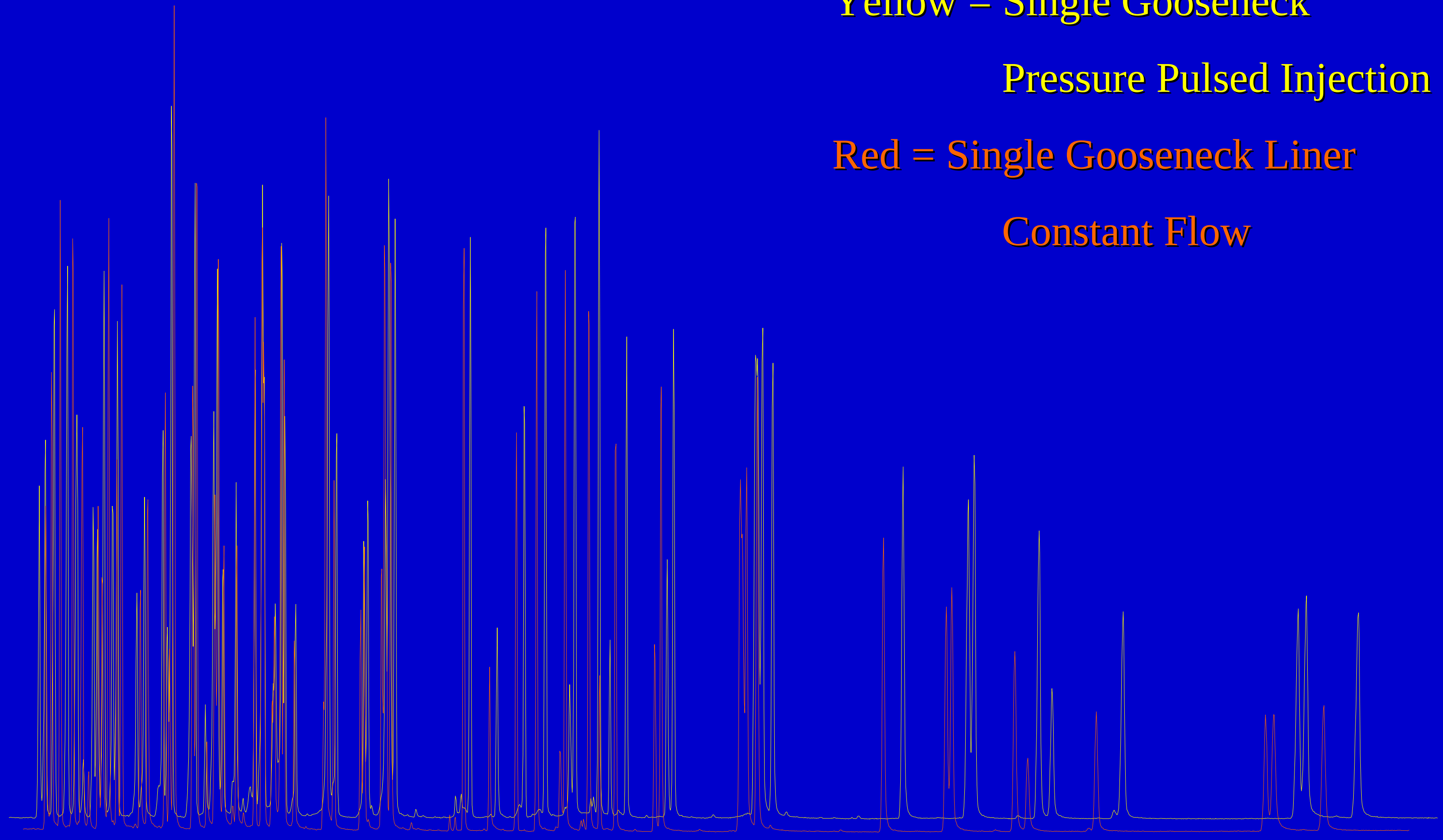
Single Gooseneck Liner
(Constant Flow vs Pressure Pulsed Injection)

Yellow = Single Gooseneck

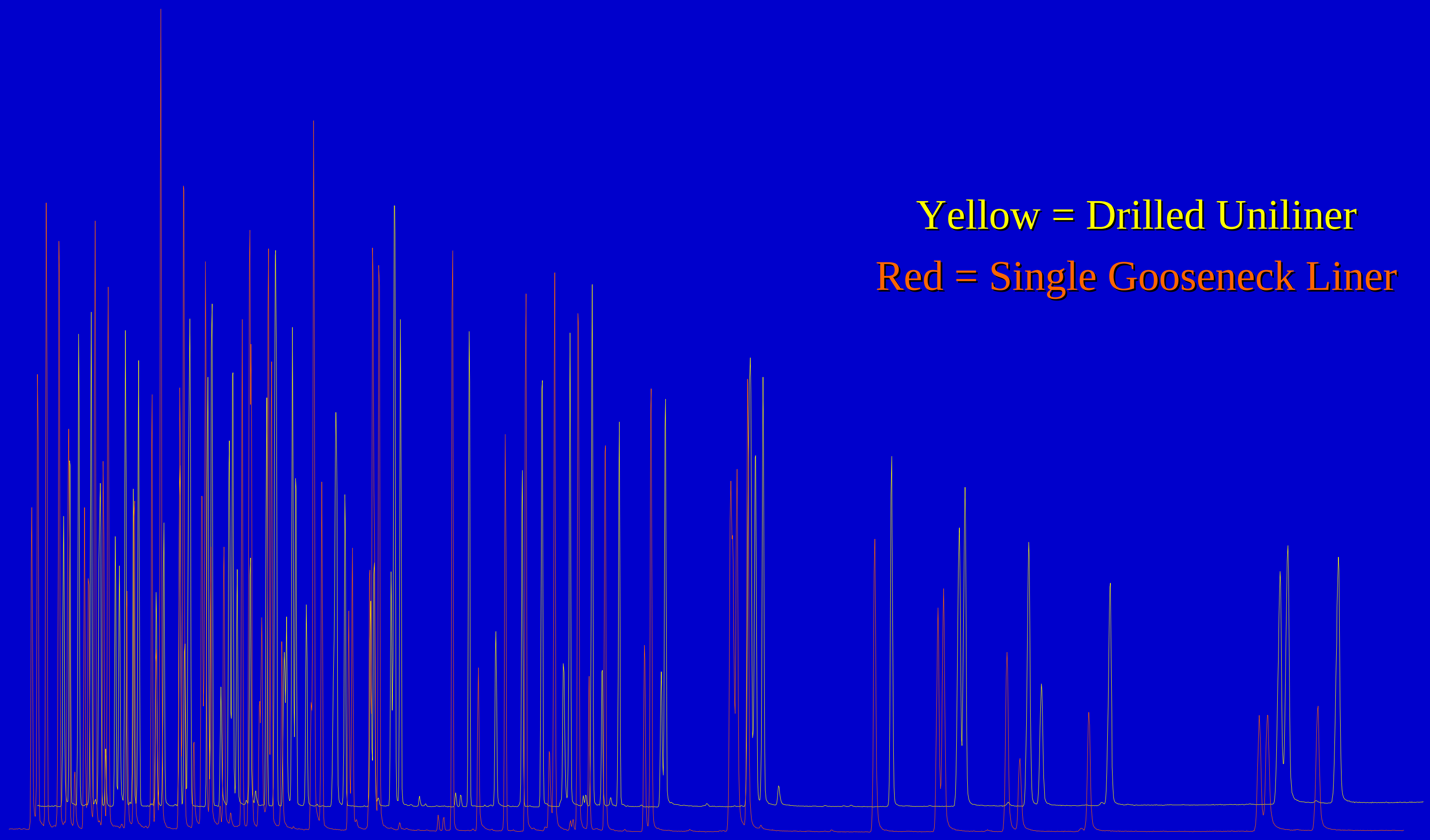
Pressure Pulsed Injection

Red = Single Gooseneck Liner

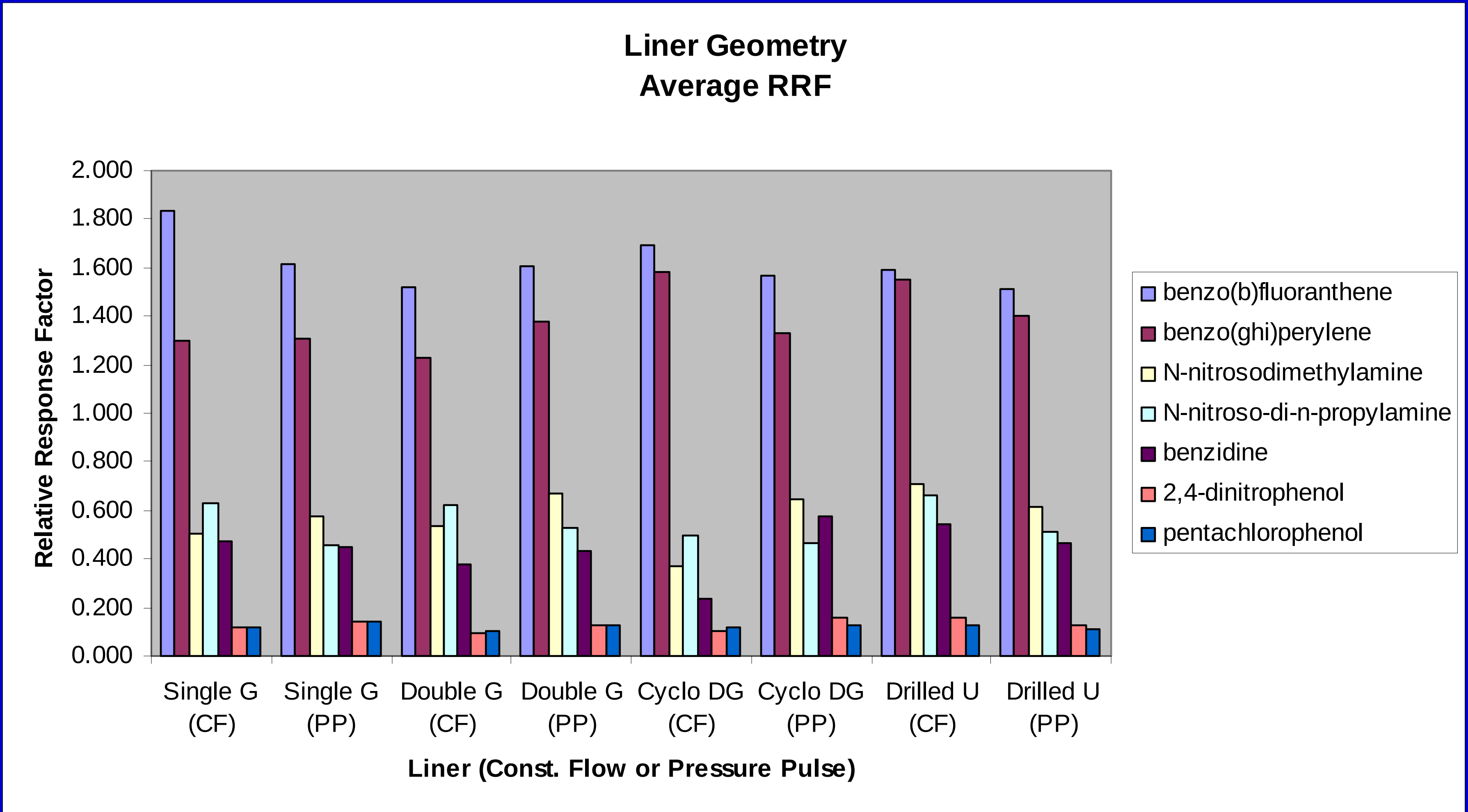
Constant Flow



Single Gooseneck vs Drilled Uniliner® Sleeve (Constant Flow)

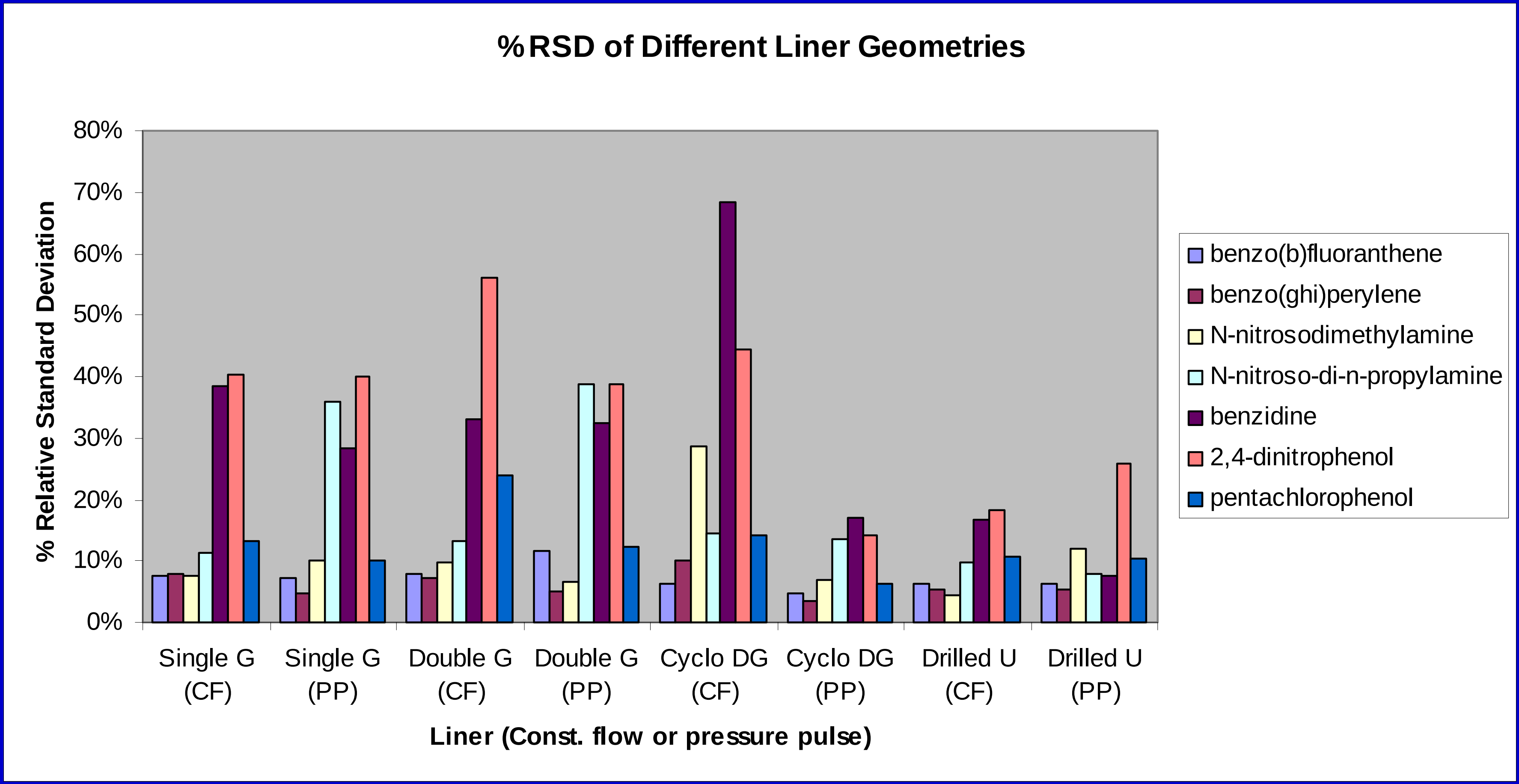


Average RRF of 6 Point Curves



Note: Higher is better

Linearity Results from 6 Point Curves



Note: Lower is better

Summary

- IP and Siltek™ deactivation are comparable for running method 8270.
- Pressure pulsing does improve the response of active compounds.
- Drilled Uniliner® appears to give the best overall results under constant flow conditions.

Future Work

- Continue comparison of experimental deactivations being designed
- Continue work with liner geometry