

Better HPLC Methods at Elevated Temperature with a New Standalone Preheater

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Faster and More Efficient Separations

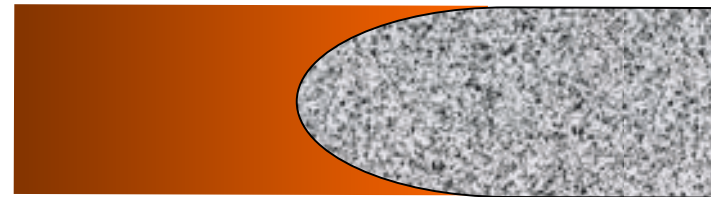
- **Speed**
 - Flatter van Deemter curves allow operation at flow rates many times optimal velocity
- **Higher efficiency - better resolution**
 - Increased diffusion rates provide lower plate heights at higher flow rates
 - Lower viscosity and back pressure permits higher flow rates with smaller particle size packings



Thermal Mismatch Effects

No Preheating

Parabolic flow caused by mobile phase heating up faster along column wall



Flow

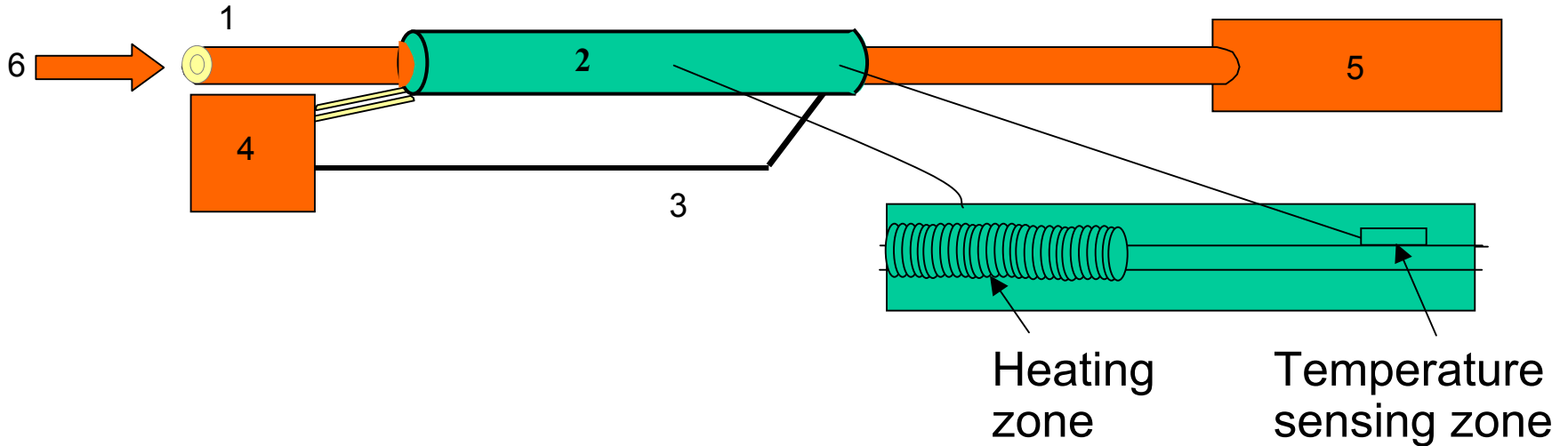


With Preheating

Mobile phase at column temperature eliminates parabolic flow



Solvent Preheater Design



(1) stainless steel tubing, (2) heater, (3) thermocouple sensor, (4) temperature controller, (5) column, (6) from pump

Patent pending Selerity Technologies, Inc.



Mobile Phase Preheater

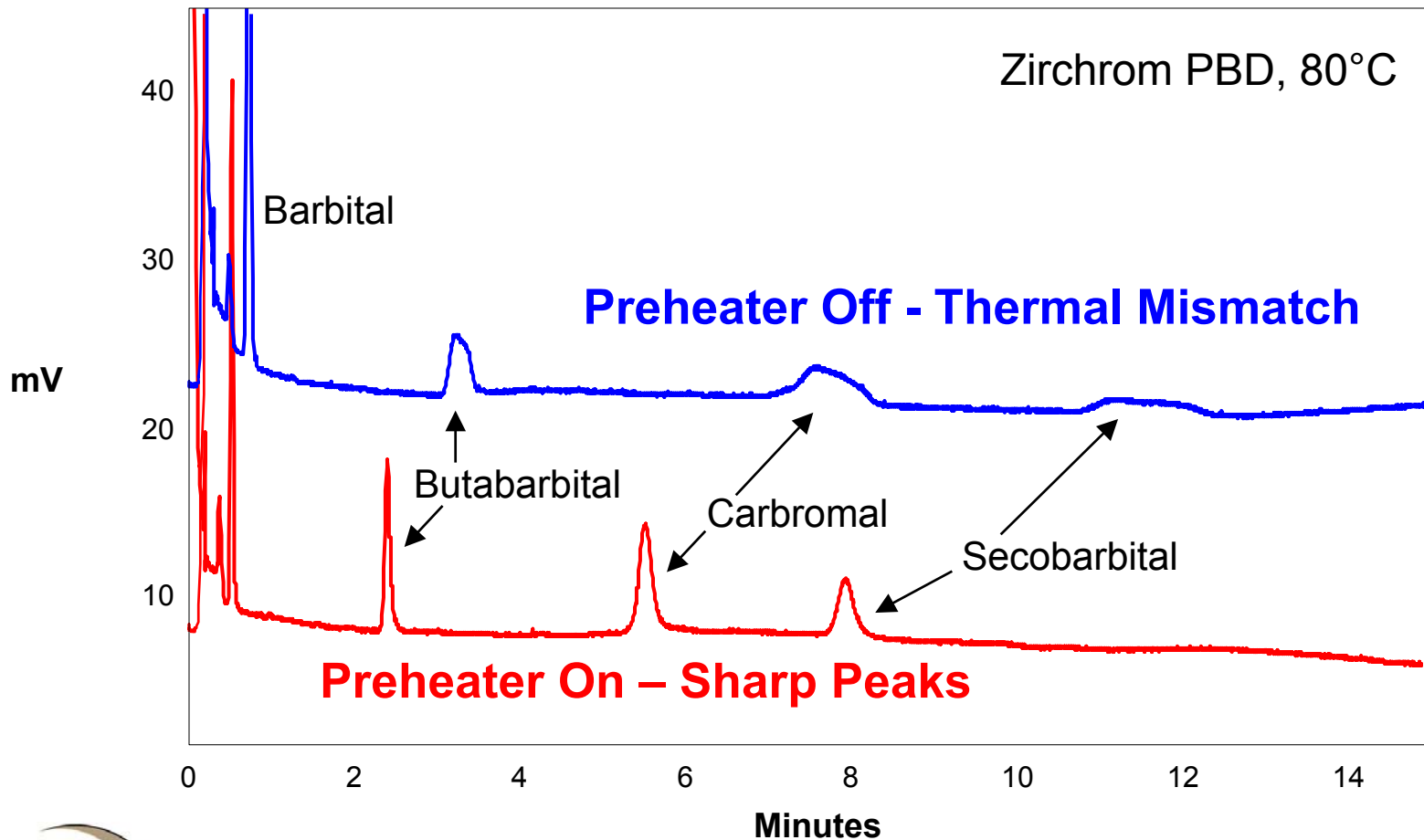


- Very responsive and non-invasive
- Low-mass and low-volume: <2 grams mass (including the tubing), <1 μL totally swept volume

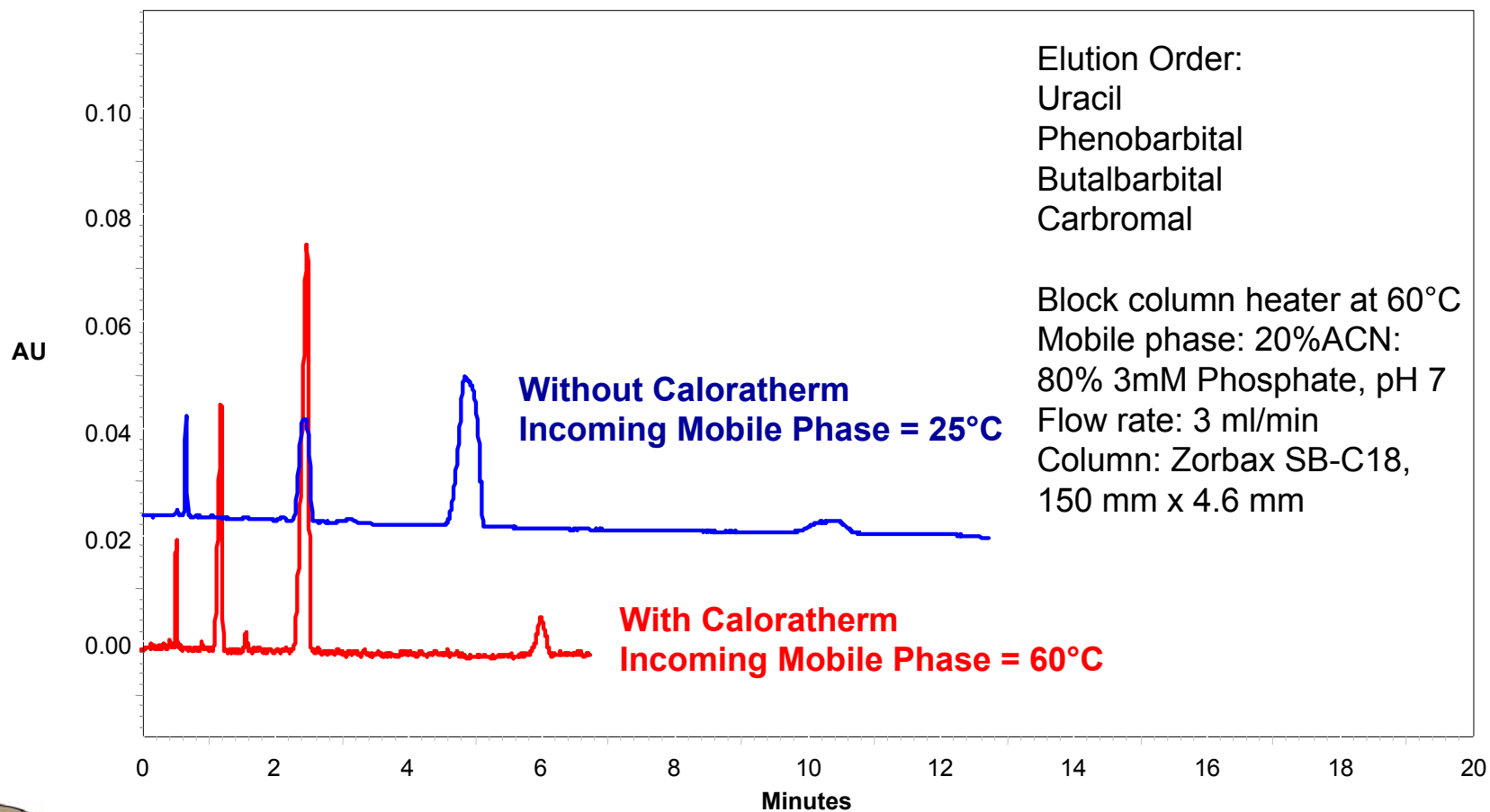
Selerity Caloratherm™ Mobile Phase Temperature Controller



Separation of Sedatives at 80°C



Separation of Sedatives at 60°C

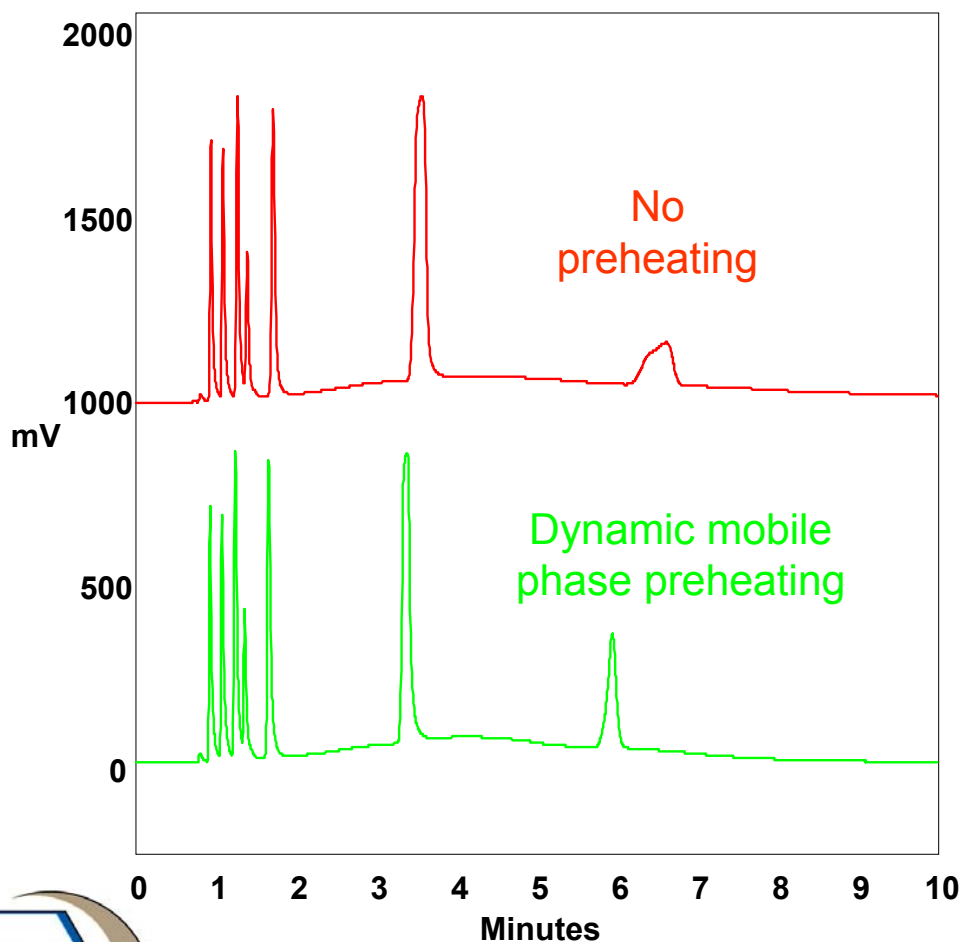


Actual tubing wall temperatures measured at the inlet and outlet of the column

Column Dimensions	Oven Temperature	Flow Rate ml/min	Preheater On		Preheater Off	
			Inlet Temp.	Outlet Temp.	Inlet Temp.	Outlet Temp.
50 x 7.8 mm, 3 μ m	60.0	1.0	60.0	61.0	32.5	50.6
	60.0	3.0	60.0	60.1	26.9	42.2
	60.0	5.0	60.0	61.0	26.9	40.0
	70.0	1.0	70.0	69.7	35.8	56.5
	70.0	3.0	70.0	70.7	30.3	46.6
	70.0	5.0	70.0	72.0	28.1	41.3
150 x 4.6 mm, 3 μ m	60.0	1.0	60.0	57.2	36.4	55.0
	60.0	2.0	60.0	64.5	32.6	53.1
	70.0	1.0	70.0	73.3	35.8	61.8
	70.0	2.0	70.0	73.8	31.7	58.3



Separation of Analgesics With and Without Mobile Phase Preheating



Column: Selerity Blaze C₈, 3 μm
100 x 4.6 mm

Mobile Phase: 40:60
acetonitrile:water with 0.1%TFA

Flow Rate: 1.5 mL/min

Detection: UV 220 nm

Temperature Program: hold at 50°C
for one minute, ramp to 100°C at
30°C/min, hold six min.

Elution Order:
Acetaminophen

Caffeine

Salicylamide

Aspirin

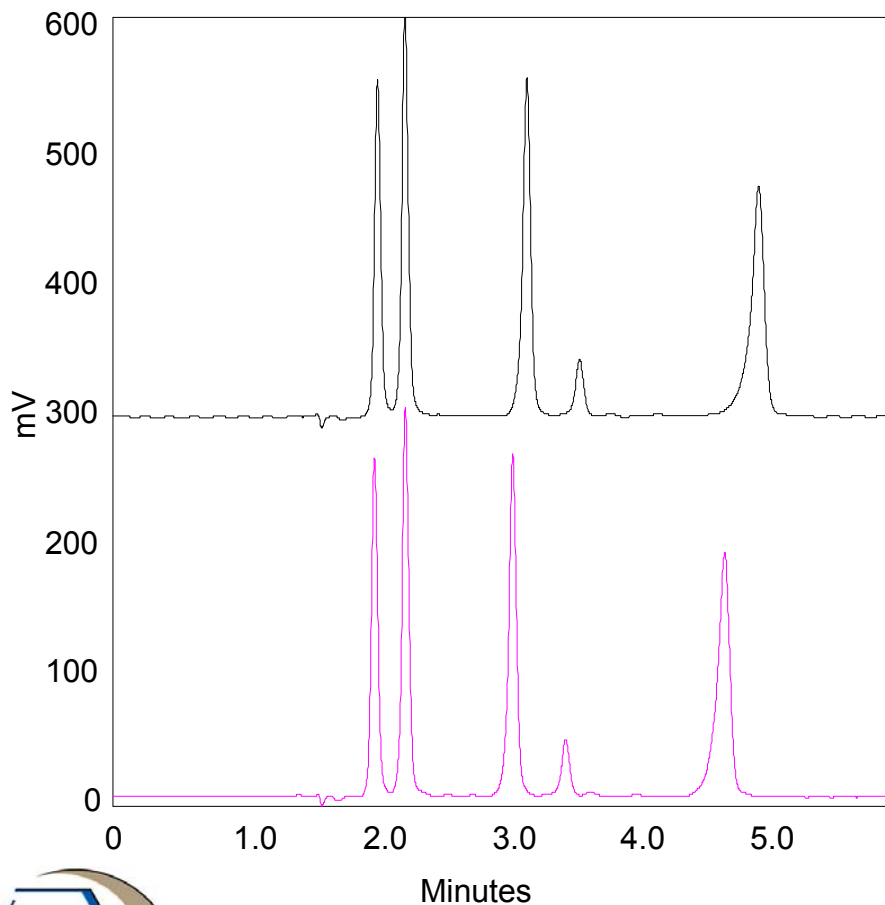
Salicylic acid

Ibuprofen

Naproxen



Separation of Analgesics With and Without Mobile Phase Preheating at 35°C



Column: Selerity Blaze C₁₈, 3 μm
100 x 4.6 mm

Mobile Phase: 30:70
acetonitrile:water with 0.1%TFA

Flow Rate: 1.0 mL/min

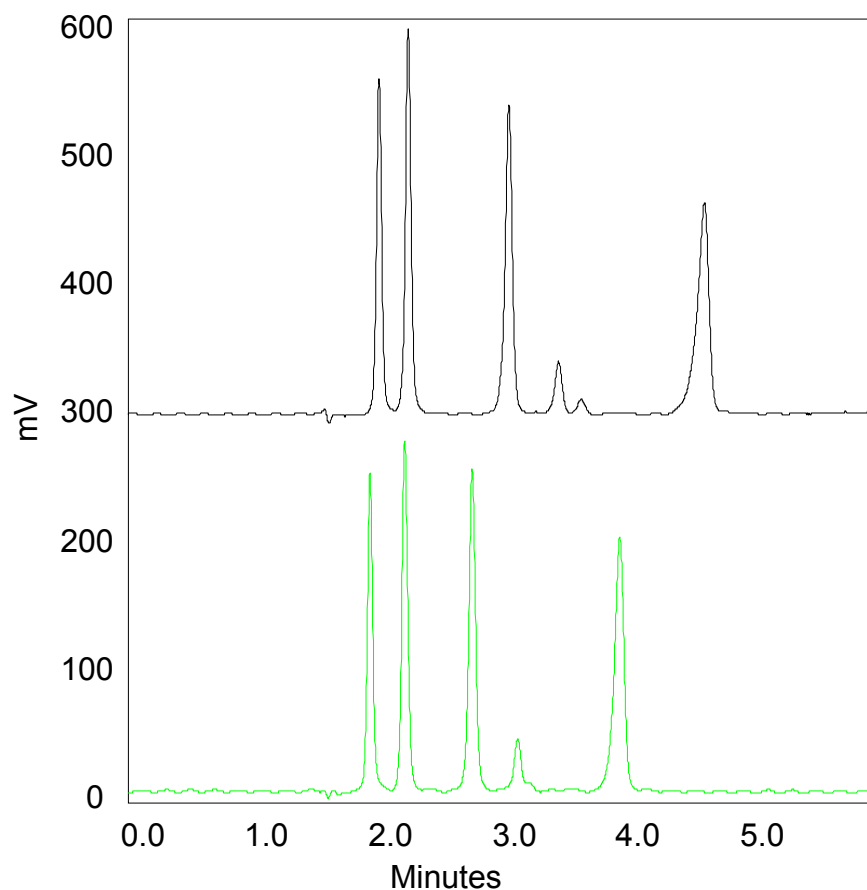
Detection: UV 220 nm

Temperature: 35°C

Elution Order:
Acetaminophen
Caffeine
Salicylamide
Aspirin
Salicylic acid



Separation of Analgesics With and Without Mobile Phase Preheating at 50°C

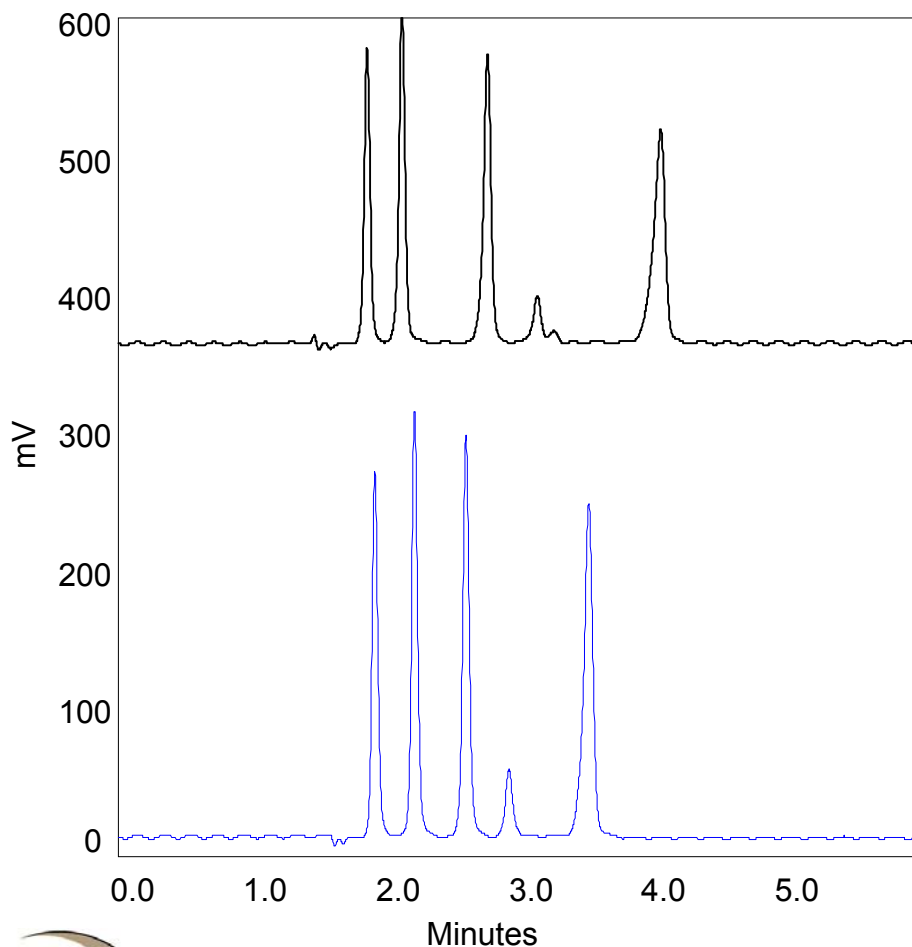


Column: Selerity Blaze C₁₈, 3 μm
100 x 4.6 mm
Mobile Phase: 30:70
acetonitrile:water with 0.1%TFA
Flow Rate: 1.0 mL/min
Detection: UV 220 nm
Temperature: 50°C

Elution Order:
Acetaminophen
Caffeine
Salicylamide
Aspirin
Salicylic acid



Separation of Analgesics With and Without Mobile Phase Preheating at 70°C



Column: Selerity Blaze C₁₈, 3 μm
100 x 4.6 mm

Mobile Phase: 30:70
acetonitrile:water with 0.1%TFA

Flow Rate: 1.0 mL/min

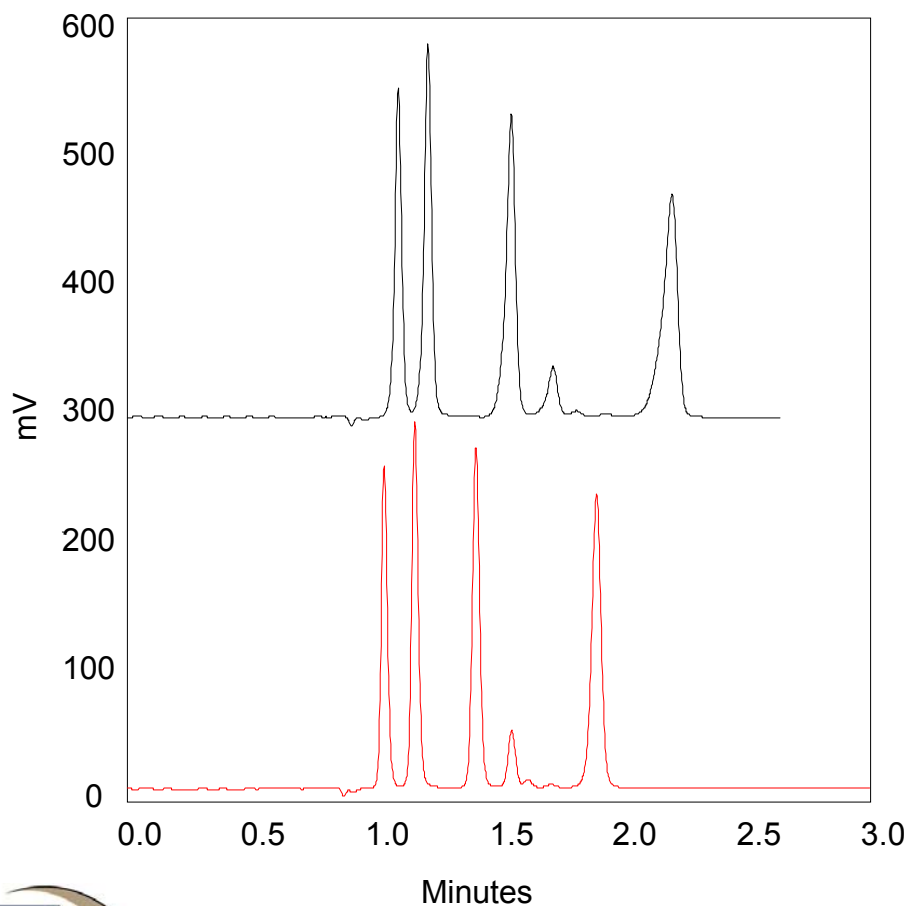
Detection: UV 220 nm

Temperature: 70°C

Elution Order:
Acetaminophen
Caffeine
Salicylamide
Aspirin
Salicylic acid



Separation of Analgesics With and Without Mobile Phase Preheating at 70°C



Column: Selerity Blaze C₁₈, 3 μm
100 x 4.6 mm
Mobile Phase: 30:70
acetonitrile:water with 0.1%TFA
Flow Rate: 2.0 mL/min
Detection: UV 220 nm
Temperature: 70°C

Elution Order:
Acetaminophen
Caffeine
Salicylamide
Aspirin
Salicylic acid



Analgesic Analysis with and without Preheating

Salicylamide					
		Preheater On		Preheater Off	
Temp	Flow rate	Retention Time	Plates	Retention Time	Plates
35.0	1.0	3.10	13,000	3.21	13,200
50.0	1.0	2.78	13,500	3.08	12,900
70.0	1.0	2.62	13,500	2.89	12,700
70.0	2.0	1.41	10,500	1.55	8,100
Salicylic Acid					
		Preheater On		Preheater Off	
Temp		Retention Time	Plates	Retention Time	Plates
35.0	1.0	4.74	12,000	5.01	12,100
50.0	1.0	3.97	12,600	4.66	11,600
70.0	1.0	3.54	12,300	4.19	11,000
70.0	2.0	1.90	11,500	2.20	6,400



Conclusions

- Thermal mismatch can occur at temperatures as low as 50 °C
- Mobile phase preheating is critical to good chromatography at elevated temperatures

Some aspects of this work are covered under
US and International Patents Pending

