OPTIMIZING THE GC TEMPERATURE PROGRAM

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Resolution

$$R_s = \frac{\sqrt{N}}{4}\left(\frac{k}{k+1}\right)\left(\frac{\alpha-1}{\alpha}\right)$$

Efficiency	$N = f$ (gas, L, r_c)	L = Length
Retention	$k = f$ (T, d_f, r_c)	r_c = column radius
Selectivity	$\alpha = f$ (T, phase)	d_f = film thickness
		T = temperature

Temperature, the TRUMP card

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Column Temperature
Developing Temperature Programs

Most powerful variable

Changes Selectivity and Retention

Natural log (ln) relationship between retention and temperature

Most to difficult predict and development

Often involves trial and error

NOTE: A carrier gas flow change, in a temp. program run, will affect the temperature a compound sees in that run.

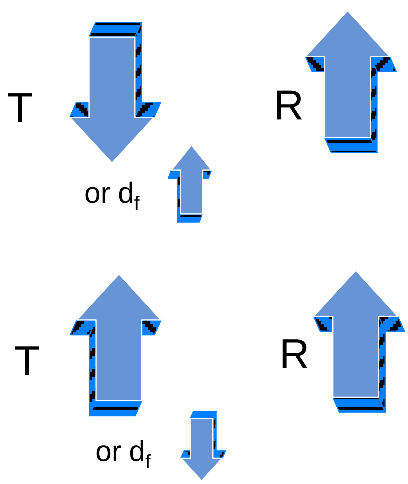
Temperature, Retention and Resolution

When solute k < 5

(early eluters)

When solute k > 5

(later eluters)



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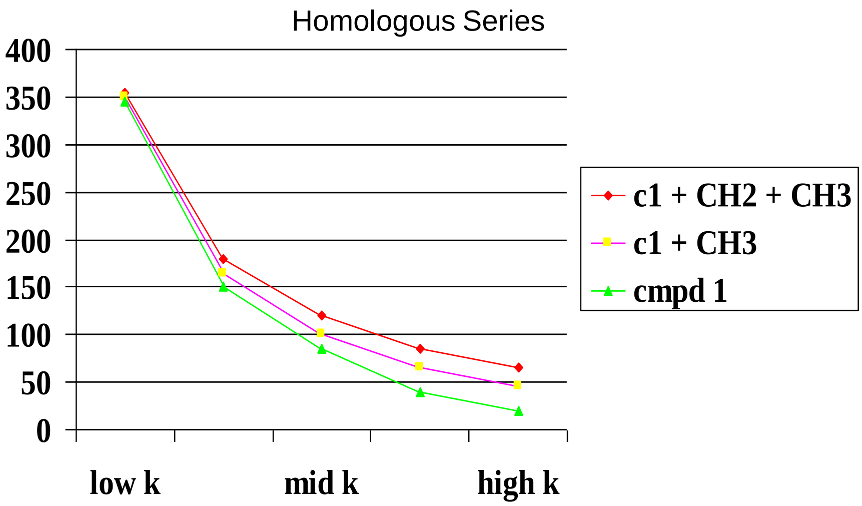
Temperature VS Retention



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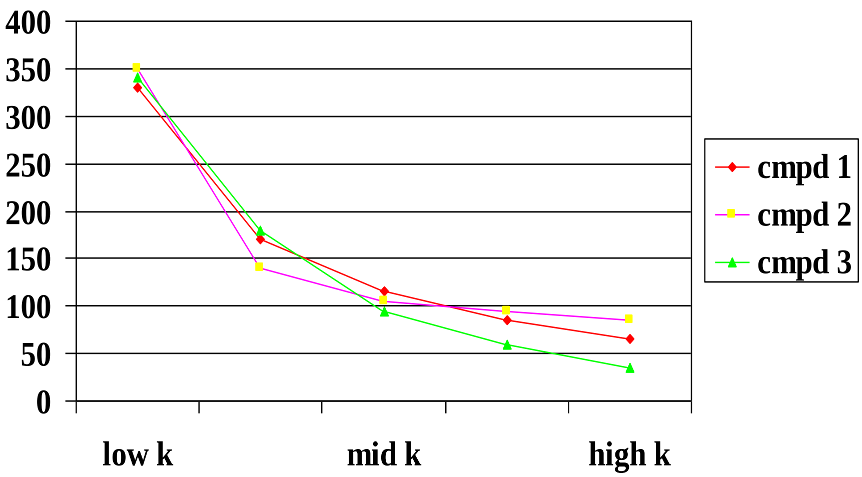
Temperature VS Retention



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DEVELOPING TEMPERATURE PROGRAMS

First Step - Linear Program

Initial temperature: 40-50°C

Ramp rate: 10°C/min

Final temperature: Column's upper limit*

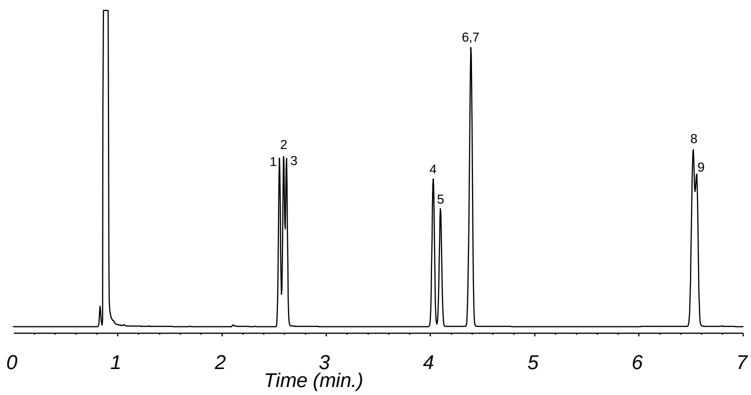
Final hold: Until the last peak elute

*Or until the last peak elutes from the column

DEVELOPING TEMPERATURE PROGRAMS

Linear Program

50-130°C at 10°/min



DB-1, 15 m x 0.25 mm I.D., 0.25 µm

DEVELOPING TEMPERATURE PROGRAMS
Second Step

Change initial hold time

or

Change initial temperature

Early Eluters

When solute k < 5 $d_f \uparrow$ $R \uparrow$

Need an increase in retention

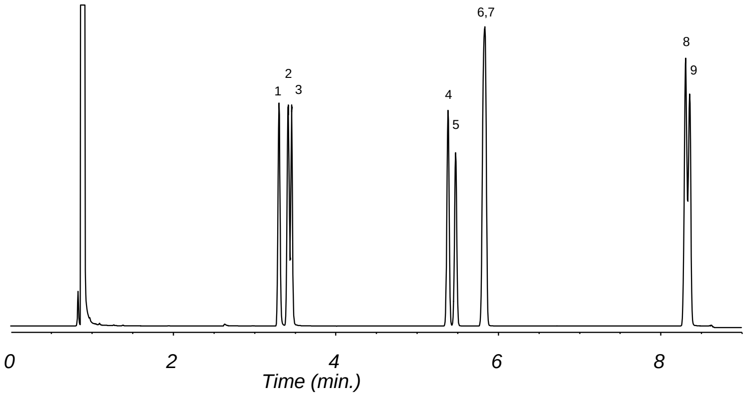
Therefore, if early eluter (k < 5)

$T \downarrow$ $R \uparrow$

DEVELOPING TEMPERATURE PROGRAMS

Increase Initial Hold Time

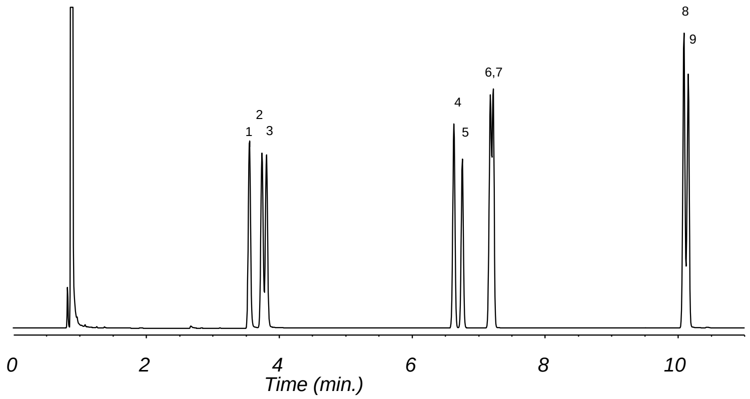
50°C for **2 min**, 50-120°C at 10°/min



DEVELOPING TEMPERATURE PROGRAMS

Increase Initial Hold Time

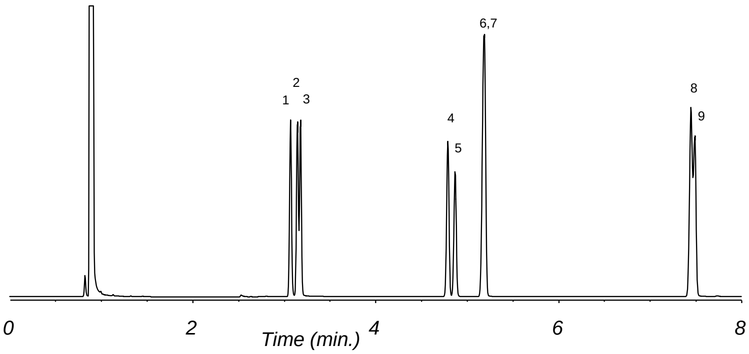
50°C for **4 min**, 50-120°C at 10°/min



DEVELOPING TEMPERATURE PROGRAMS

Decrease Initial Temperature

40-120°C at 10°/min



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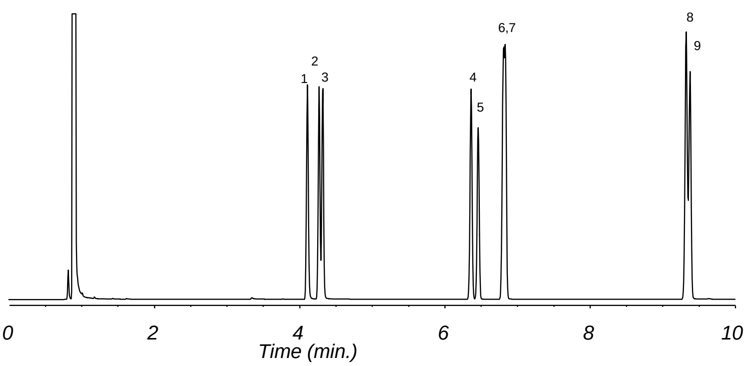
DEVELOPING TEMPERATURE PROGRAMS

Increase Initial Hold Time

40°C for 2 min, 40-120°C at 10°/min



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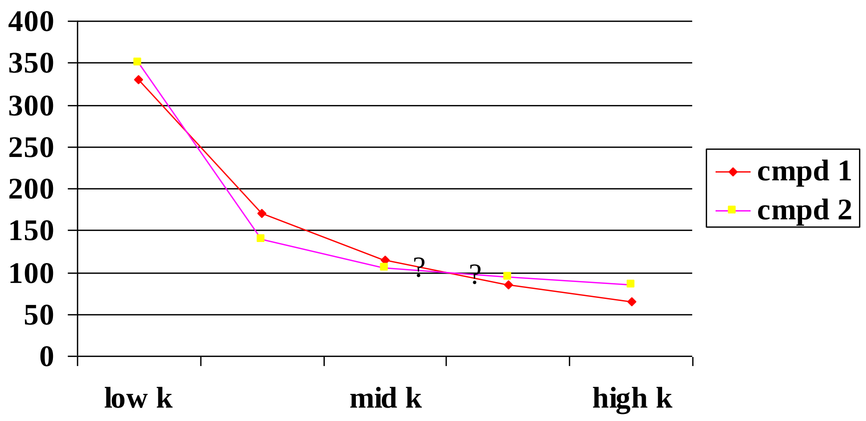
Temperature VS Retention



DEVELOPING TEMPERATURE PROGRAMS

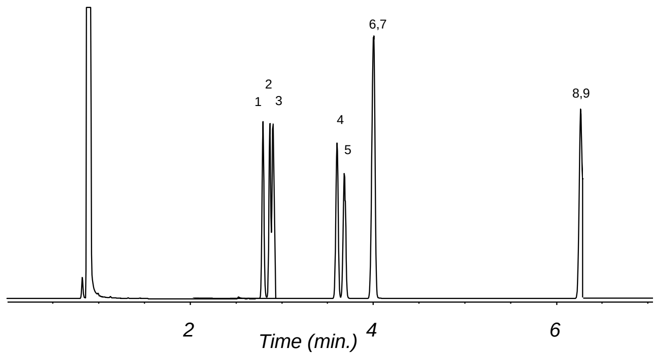
Third Step

Change the ramp rate

±5°C/min per change

DEVELOPING TEMPERATURE PROGRAMS

40°C for 2 min, 40-110°C at 15°/min



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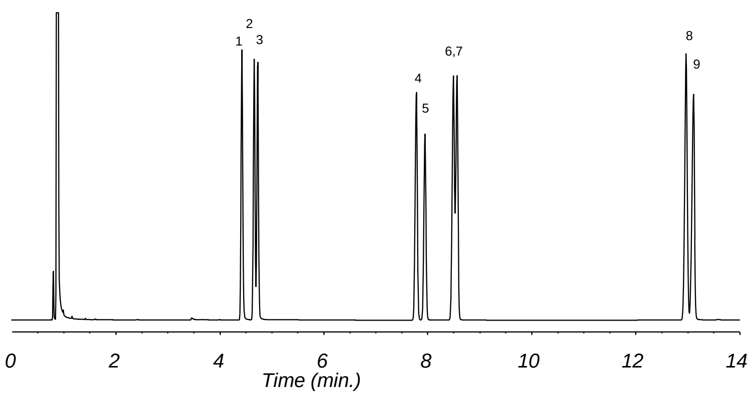
DEVELOPING TEMPERATURE PROGRAMS

40°C for 2 min, 40-110°C at 5°/min



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DEVELOPING TEMPERATURE PROGRAMS

Mid Ramp Holds

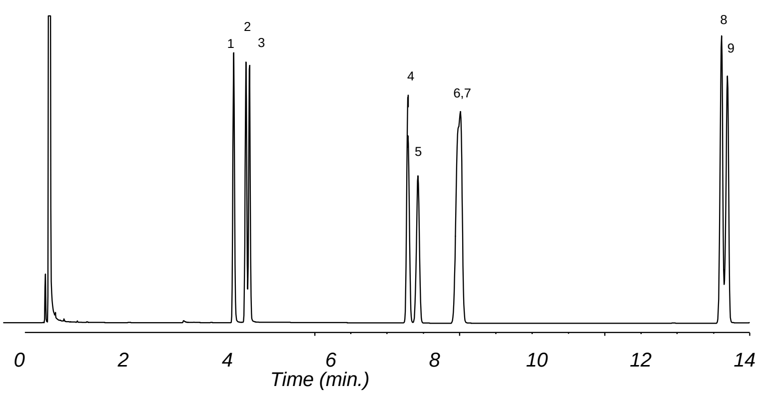
Isothermal portion during the temperature program

2-5 minute hold

10-30°C below elution temperature of peaks

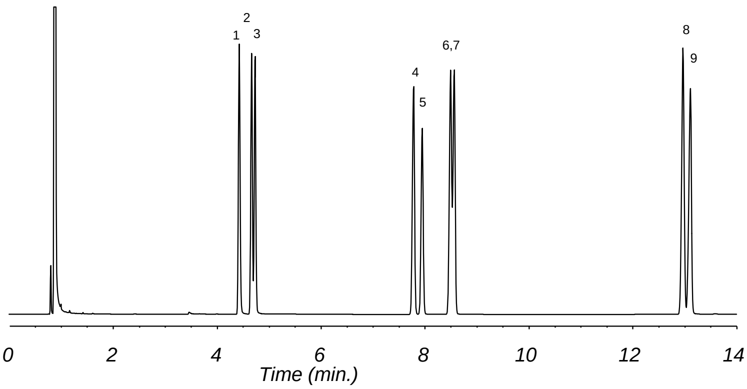
DEVELOPING TEMPERATURE PROGRAMS

40 for 2min, 40-70°C at 5°/min, 70°C for 2 min, (Mid Ramp Hold) 70-120°C at 10°/min



DEVELOPING TEMPERATURE PROGRAMS

40°C for 2 min, 40-110°C at 5°/min



Late Eluters

When solute k > 5 T ↑ R ↑

Need a decrease in retention

Sharper peaks even though they may be closer

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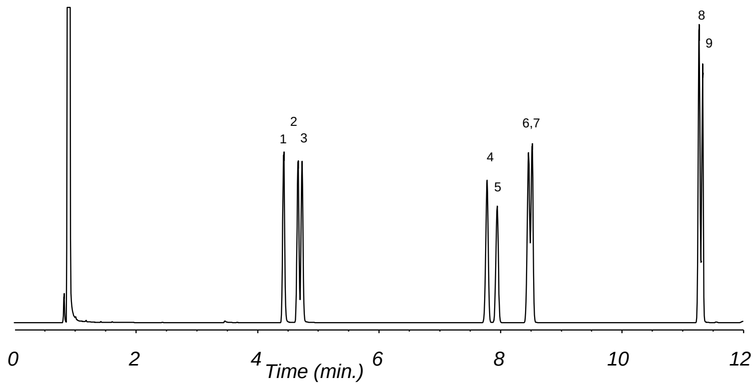
DEVELOPING TEMPERATURE PROGRAMS

40°C for 2 min, 40-70°C at 5°/min, 70-130°C at 15°/min



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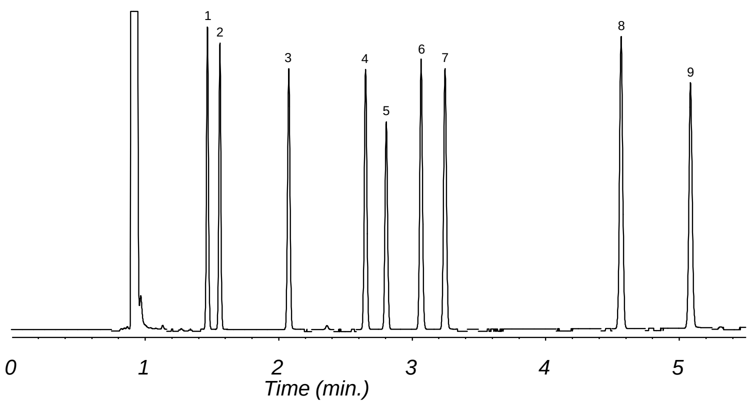
DEVELOPING TEMPERATURE PROGRAMS

80-190°C at 20°/min



DB-WAX, 15 m x 0.32 mm I.D., 0.25 µm

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DEVELOPING TEMPERATURE PROGRAMS
Lowering the Initial Temperature

Improves resolution of earlier peaks

Increasing Initial Temperature Hold Time

Similar, but smaller effect as lowering the initial temperature

Combine lower initial temperature and increased initial hold time*

*A higher initial temperature becomes feasible

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DEVELOPING TEMPERATURE PROGRAMS
Mid Ramp Hold

Sometimes improves resolution of co-eluting peaks in the middle of the chromatogram

Always causes peak broadening

Creates more complex programs

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DEVELOPING TEMPERATURE PROGRAMS
Combining Parameters

Offset retention increases by adjusting another parameter

Example:

Increase ramp rate when lowering initial temperature

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