Purification of Binol compounds using PF-30SIHP-F0120

Application Note #032

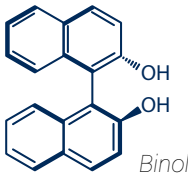
1. TLC method development

Mobile phase:
Hexane / MTBE

Compound of interest:
Compound 2

Compound	Rf	CV
1	0.5	2
ΔCV_{2-1} = 1.33		
2	0.3	3.33
ΔCV_{3-2} = 6.67		
3	0.1	10

According to ΔCV calculation, compounds 1 and 2 are difficult to separate.



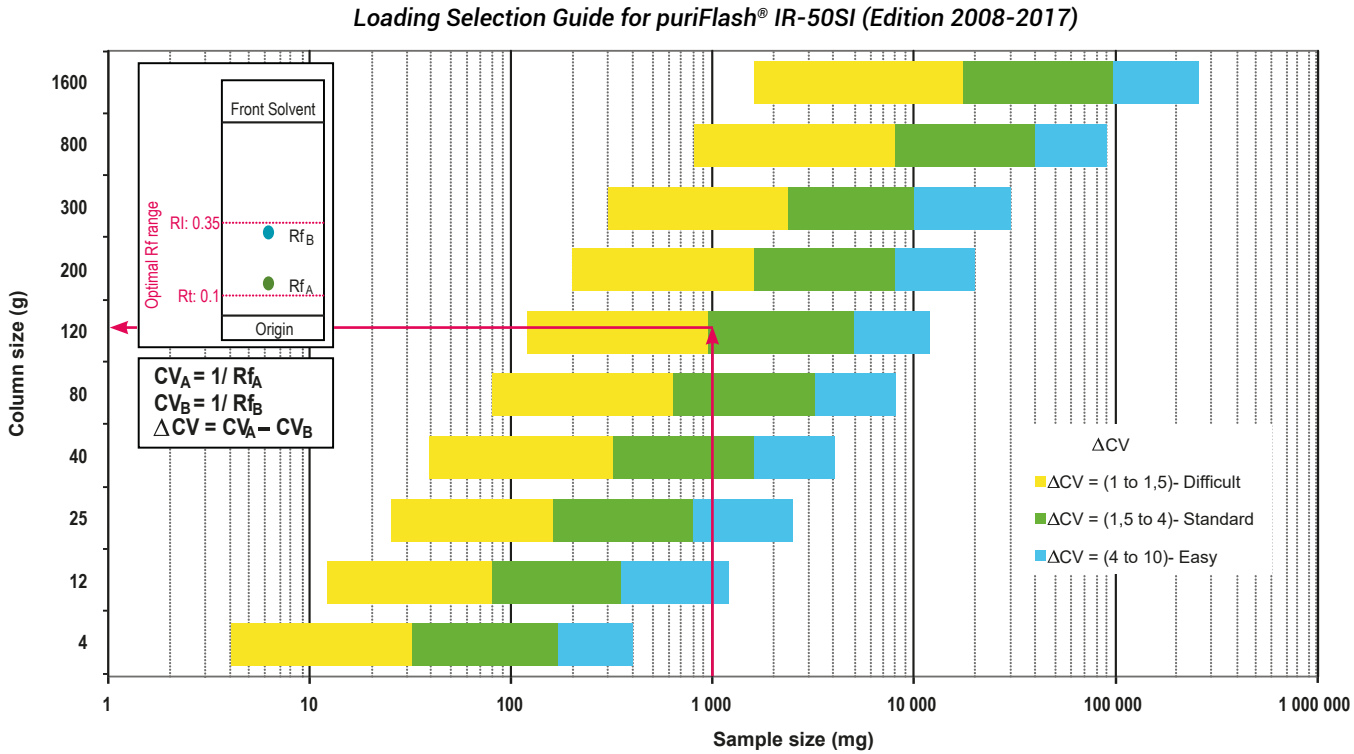
Binol (compound 2)

2. Choice of the column according to the ΔCV & crude sample mass

Crude sample: 1g

Column: PF-30SIHP-F0120

Loading capacity: 0.83%

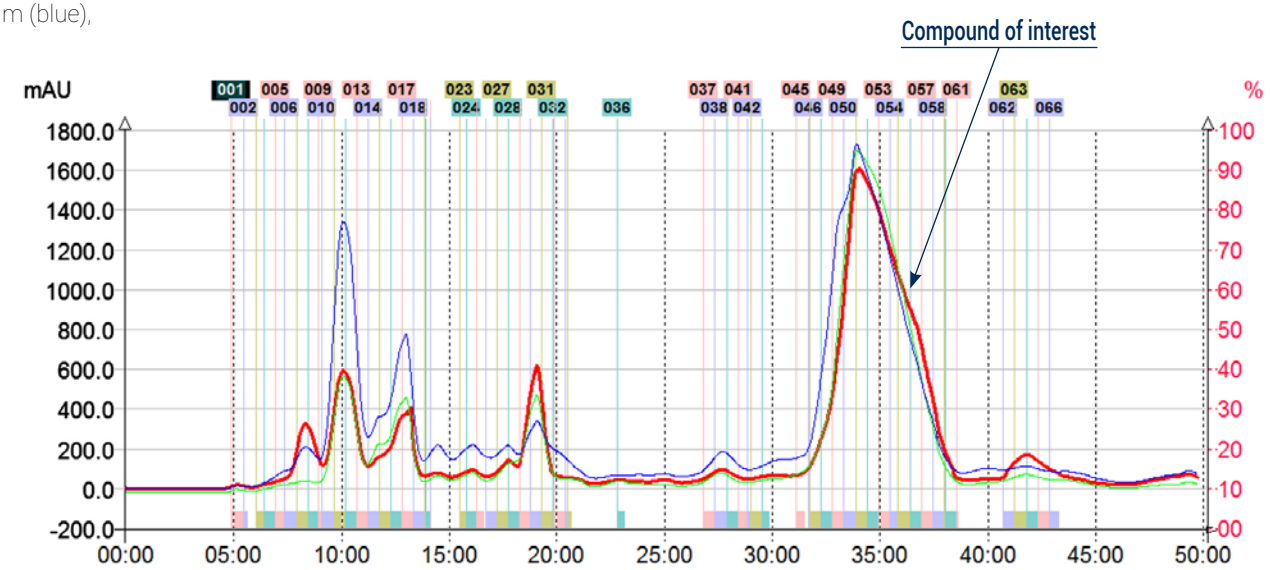


Customer has chosen to use a PF-30SIHP-F0120 column to obtain a better separation (efficiency & purity) than with a IR-50SI-F0120 column.

3. Flash conditions

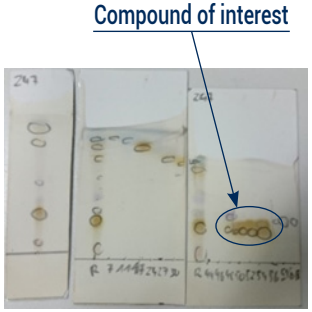
Device: puriFlash® 450-iELSD (or now puriFlash® 5.050 pack iELSD)
Solvents: A: Hexane
B: MTBE
Column: PF-30SIHP-F0120
Flow rate: 46mL/min
Injection mode: Solid deposit with celite (Dry-load F0012)
Crude sample: 1g
Detection: UV 280nm (green) & UV 300nm (blue),
UV Scan 250-600nm (red)
Elution conditions:

CV	A (%)	B (%)
0	88	12
1	88	12
11	25	75
13.5	25	75
14.5	10	90
16	10	90



4. TLC confirmation

Spoted collection tubes show that the compound of interest was pure in tubes 50 to 56.



The Interchim® separation revealed a lot of informations that the customer was not aware of.
The customer was quite impressed.

To achieve this purification:

You will need

- puriFlash® 5.050
  Discover it Add to card
- Integrated ELSD
  Discover it Add to card
- puriFlash® column PF-30SIHP-F0120
  Discover it Add to card
- puriFlash® Dry-load PF-DLE-F0012
  Discover it Add to card

We highly recommend

- 16x150mm Rack
  AYHE40 Add to card
- Tubes 16x150mm
  BX5400 Add to card
- Manometer ELSD
  FJ6720 Add to card

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