

341- 350

“ You have very intelligent chromatographers on hand to answer method development questions.

Timothy E. Mason
AkzoNobel

The opinions stated herein are solely those of the speaker and not necessarily those of any company or organization.

SFC Media

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Supercritical Fluid Chromatography (SFC)

SFC is recognized by scientists worldwide as a clean, green, and efficient tool for analysis and purification. With recent advancements and accessibility of instrumentation, improved column hardware, and the wide variety of surface chemistries available, SFC has enjoyed an ever-increasing range of applications in many industries:

- Pharmaceutical
- Nutraceutical
- Petrochemical
- Natural Products
- Food & Beverage
- Environmental
- Academic
- and more...



Expanding the Range of Selectivity for SFC

Selecting a column is one of the most critical parameters during SFC method development. Having a variety of complementary and orthogonal selectivities to choose from can mean the difference between partial or no separation and achieving an optimal fully resolved separation that can be validated and scaled-up in your lab or contract lab.

Phenomenex offers a large collection of packed SFC analytical and preparative columns that have earned their reputations for performance, reliability, high efficiency, reproducibility, and long lifetimes.

Complete SFC Product Offering

Phenomenex offers solutions for your SFC needs.

- Over 20 selectivities for use in SFC
- Chiral and achiral phases available
- Multiple particle sizes ranging from 1.7 µm to 20 µm*
- Scalable packed column dimensions (2.0 mm – 50.0 mm ID)

Chiral columns (pp. 343-347)

6 Coated Lux Polysaccharide Chiral Stationary Phases

- Lux Amylose-1
- Lux Amylose-2
- Lux Cellulose-1
- Lux Cellulose-2
- Lux Cellulose-3
- Lux Cellulose-4

2 Immobilized Lux Phases

- Lux i-Amylose-1
- Lux i-Amylose-3
- Lux i-Cellulose-5

Achiral columns (pp. 348-350)

- | | |
|------------------------|------------------------|
| • Kinetex Phenyl-Hexyl | • Luna NH ₂ |
| • Kinetex F5 | • Luna Si |
| • Kinetex Biphenyl | • Luna CN |
| • Kinetex HILIC | • Synergi Polar-RP |
| • Luna HILIC | |
| • Luna PFP(2) | |

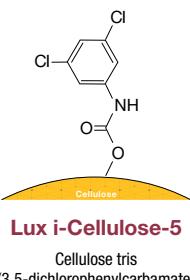
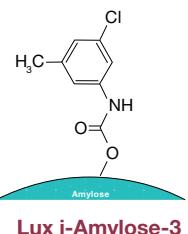
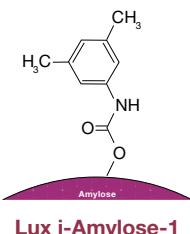


*Not all media available in a full range of particle sizes, please inquire.

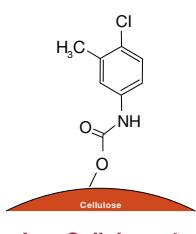
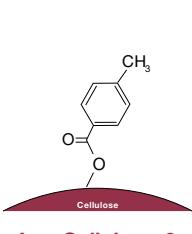
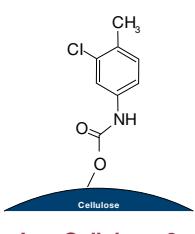
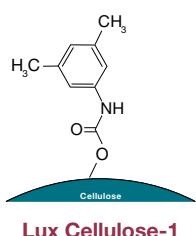
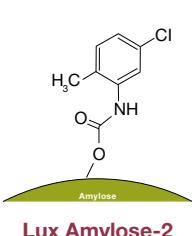
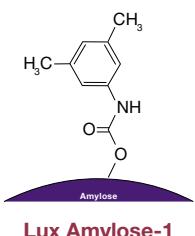
SFC Supercritical Fluid Chromatography

Chiral SFC Media

Three Robust Immobilized Chiral Columns



Combined with Six Coated Lux Polysaccharide LC/SFC Chiral Stationary Phases



Easily upgrade from your existing chiral columns to Lux LC/SFC columns!

If you are using one of the DAICEL® columns below:	Guaranteed alternative:	Phase description:
CHIRALPAK IA® and IA-3	Lux i-Amylose-1	Amylose tris(3,5-dimethylphenylcarbamate)
CHIRALPAK IG® and IG-3	Lux i-Amylose-3	Amylose tris(3-chloro-5-methylphenylcarbamate)
CHIRALPAK IC® and IC-3	Lux i-Cellulose-5	Cellulose tris(3,5-dichlorophenylcarbamate)
CHIRALPAK AD®, AD-H®, AD-3, AD-RH®, and AD-3R	Lux Amylose-1	Amylose tris(3,5-dimethylphenylcarbamate)
CHIRALPAK AY®, AY-H®, AY-3, AY-RH, and AY-3R	Lux Amylose-2	Amylose tris(5-chloro-2-methylphenylcarbamate)
CHIRALCEL® OD®, OD-H®, OD-3, OD-RH®, and OD-3R	Lux Cellulose-1	Cellulose tris(3,5-dimethylphenylcarbamate)
CHIRALCEL OZ, OZ-H®, OZ-3, OZ-RH, and OZ-3R	Lux Cellulose-2	Cellulose tris(3-chloro-4-methylphenylcarbamate)
CHIRALCEL OJ®, OJ-H®, OJ-3, OJ-RH®, and OJ-3R	Lux Cellulose-3	Cellulose tris(4-methylbenzoate)
CHIRALCEL OX-H, OX-3, OX-RH, and OX-3R	Lux Cellulose-4	Cellulose tris(4-chloro-3-methylphenylcarbamate)

SFC Supercritical Fluid Chromatography

Chiral SFC Media (cont'd)

Exceptional Stability and Separating Power under SFC Conditions

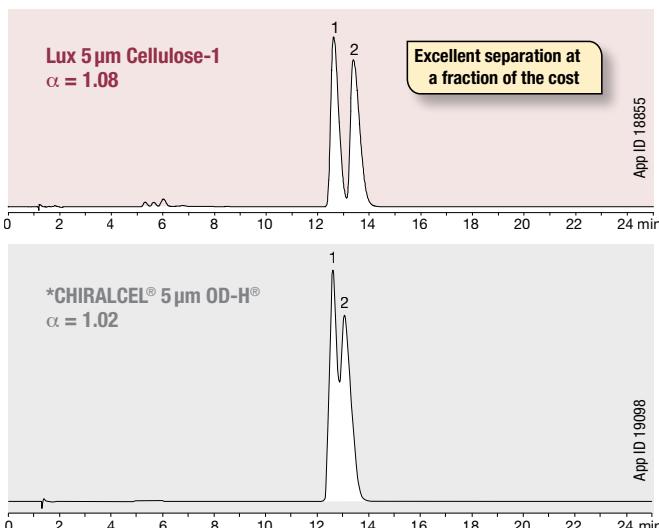
- Multiple complementary polysaccharide stationary phases
- High efficiency and loading capacity
- Pressure stability up to 300 bar
- 3 µm, 5 µm packed columns and 10 and 20 µm bulk media for scale-up

Extreme Stability and Separating Power under SFC Conditions.

Never fear crushed media or loss in efficiency again. With a pressure stability up to 300 bar (4350 psi), you can feel confident about running at high operating pressures (if necessary). Lux media is

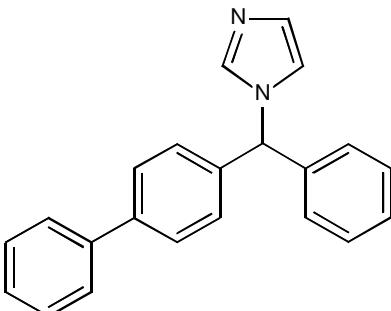
SFC approved and versatile enough to satisfy all of your chiral separation needs.

Bifonazole



Conditions for both columns:

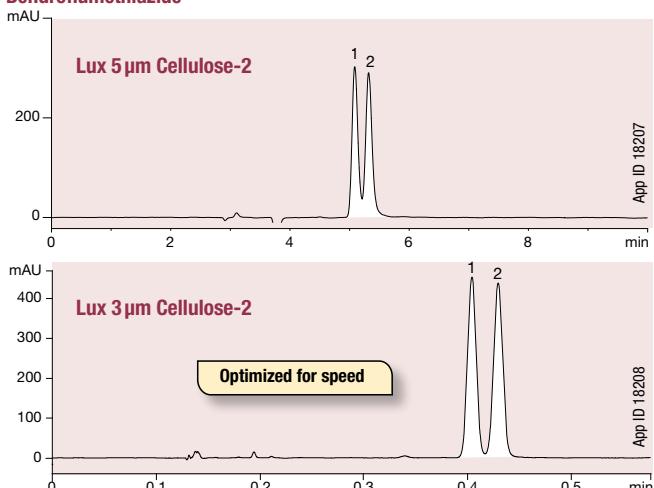
Dimensions: 250 x 4.6 mm
Mobile Phase: 0.1 % Diethylamine in Methanol / Carbon Dioxide (15:85)
Flow Rate: 2.5 mL/min
Temperature: 35 °C
Detection: Diode Array Detector



Smaller Particles for Higher Efficiency

Scaling down to a 3 µm particle gives you exceptional efficiencies and significantly reduced runtimes without compromising enantioselectivity.

Bendroflumethiazide



Column: Lux 5 µm Cellulose-2
Dimensions: 250 x 4.6 mm
Part No.: [00G-4457-E0](#)
Mobile Phase: 0.1 % Diethylamine with 0.1 % Trifluoroacetic acid in Methanol / Carbon Dioxide (30:70)
Flow Rate: 2 mL/min
Detection: UV @ 273 nm
Temperature: Ambient

Column: Lux 3 µm Cellulose-2
Dimensions: 50 x 4.6 mm
Part No.: [00B-4456-E0](#)
Mobile Phase: 0.1 % Diethylamine with 0.1 % Trifluoroacetic acid in Methanol / Carbon Dioxide (30:70)
Flow Rate: 4 mL/min
Detection: UV @ 273 nm
Temperature: Ambient

* Comparative separations may not be representative of all applications.
Columns used for comparison were manufactured by DAICEL Corporation.

SFC Supercritical Fluid Chromatography

Chiral SFC Media (cont'd)

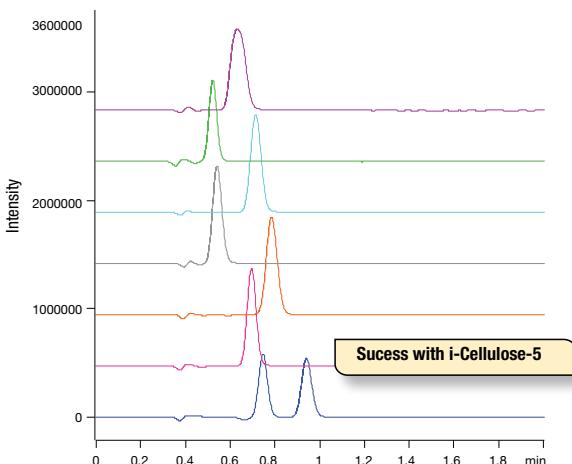
Eight distinct yet complementary Lux® CSPs allow for excellent success rate over reversed phase, polar organic, normal phase, and SFC conditions, with the i-Amylose-3, i-Cellulose-5, and i-Amylose-1, adding strong solvent capability to this versatile family of products.

For SFC, having this breadth of selectivities is incredibly useful for

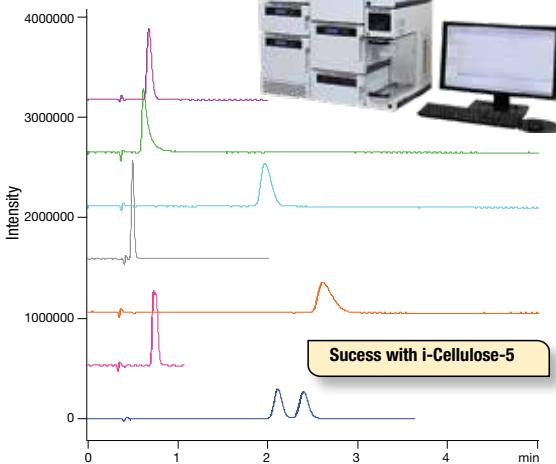
A variety of compounds were separated including:

- Beta-Blockers
- Anti-Anxiety
- Pain Relievers
- Anti-Allergenic agents
- Anti-Arrhythmia
- Anti-Asthmatic
- Anti-Coagulants
- Anti-Depressive
- Anti-Inflammatory
- Calcium Channel Blockers

Nimopidine



Acebutolol



Nimopidine and Acebutolol

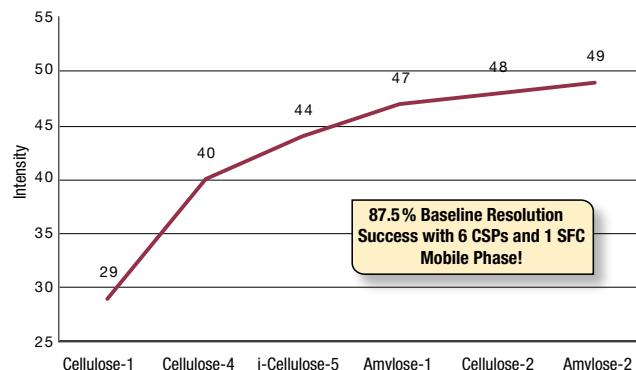
Columns: Lux 3 µm Amylose-2
Lux 3 µm Amylose-1
Lux 3 µm Cellulose-4
Lux 3 µm Cellulose-3
Lux 3 µm Cellulose-2
Lux 3 µm Cellulose-1
Lux 3 µm i-Cellulose-5

Dimensions: 150 x 3.0 mm



screening and discovery work. Below is a portion of a study where 56 racemic pharmaceutical compounds were run on a variety of Lux stationary phases under various mobile phase options to help develop useful screening protocols. Over the course of the study, it was determined that with one SFC mobile phase and the use of 6 different Lux CSPs, a lab could get 87.5 % success (baseline resolution).

Cumulative baseline separation with Lux phases



SFC Screen

Columns: Lux 5 µm Cellulose-1
Lux 5 µm Cellulose-4
Lux 5 µm i-Cellulose-5
Lux 5 µm Amylose-1
Lux 5 µm Cellulose-2
Lux 5 µm Amylose-2

Dimensions: 250 x 4.6 mm

Conditions for all columns:

Mobile Phase: 80 % CO₂ / 20 % Methanol + 0.1 % Isopropylamine and 0.1 % TFA

Flow Rate: 3 mL/min

Detection: UV @ 220 nm

Temperature: 30 °C

System: JASCO® 4000 Series Analytical SFC



Lux columns are interchangeable between normal phase and SFC modes with a simple solvent switch.

Request Technical Note, [TN-9004](#), for more details on chiral SFC screening strategies.

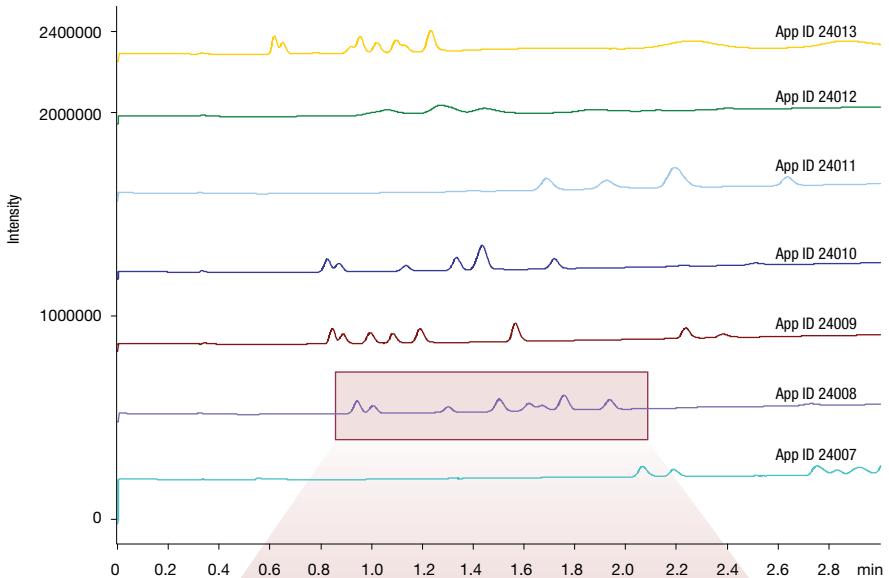
SFC Supercritical Fluid Chromatography

Chiral SFC Media (cont'd)

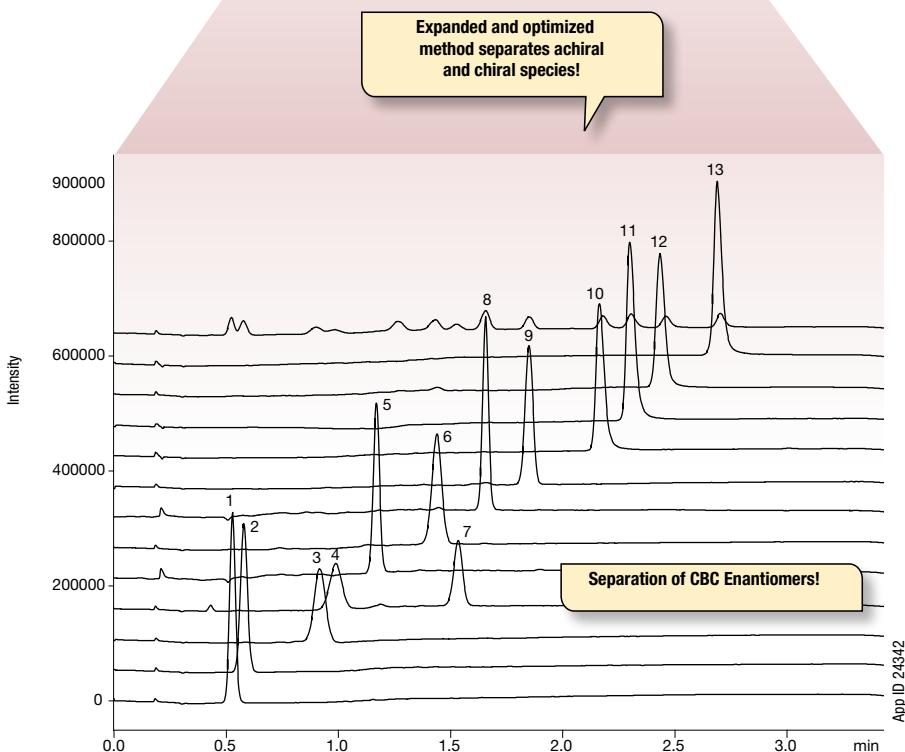
Achiral SFC Success with Chiral Columns!

While the incredible range of interaction mechanisms (polar, electrostatic, hydrophobic, van der Waals, and others) present in each Lux material are fundamental for ensuring baseline separation of chiral compounds, these same interaction mechanisms can also be used as an excellent screening tool for achiral work. Here we

present an achiral screening of natural cannabinoids using 7 Lux selectivities under one SFC mobile phase. The initial resolution and separation provided by the Lux Cellulose-2 was then further optimized to provide even greater resolution.



Conditions for all columns:
Columns: Lux 3 µm i-Cellulose-5
Lux 3 µm Amylose-2
Lux 3 µm Amylose-1
Lux 3 µm Cellulose-4
Lux 3 µm Cellulose-3
Lux 3 µm Cellulose-2
Lux 3 µm Cellulose-1
Dimensions: 150 x 3.0 mm
Mobile Phase: A: Carbon Dioxide
B: Methanol
Gradient: Time (min) % B
0 5
2.5 25
3 25
Flow Rate: 3 mL/min
Detection: UV @ 220 nm
Temperature: 40 °C
Sample: Cannabinoid mix of 8



Column: Lux 3 µm Cellulose-2
Dimensions: 150 x 3.0 mm
Part No.: [00F-4456-Y0](#)
Mobile Phase: A: Carbon Dioxide
B: Methanol
Gradient: Time (min) % B
0 4
3 25
3.5 25
Flow Rate: 5 mL/min
Detection: UV @ 220 nm
Temperature: 40 °C
Sample: Cannabinoid mix of 12
1. CBDV 8. THCV
2. CBN 9. CBG
3. Delta-8-THC 10. CBDA
4. CBC (Enantiomer 1) 11. CBDVA
5. CBD 12. THCA
6. Delta-9-THC 13. CBGA
7. CBC (Enantiomer 2)

SFC Supercritical Fluid Chromatography

Chiral SFC Media (cont'd)

Chiral Material Characteristics

Packing Material Porous	Particle Size (μm)	Pressure Stability (bar)	pH Stability
Lux Cellulose	3, 5, 10, 20	300	2.0 - 9.0
Lux Amylose	3, 5, 20*	300	2.0 - 9.0

* Please inquire

3.0 mm ID Lux Screening Columns

Ordering Information

3 μm MidBore™ Columns (mm)*		SecurityGuard™ Cartridges (mm)
Phases	150 x 3.0	4 x 2.0*
		/10pk
i-Cellulose-5	00F-4755-Y0	AJ0-8631
Cellulose-1	00F-4458-Y0	AJ0-8402
Cellulose-2	00F-4456-Y0	AJ0-8398
Cellulose-3	00F-4492-Y0	AJ0-8621
Cellulose-4	00F-4490-Y0	AJ0-8626
Amylose-1	00F-4729-Y0	AJ0-9337
Amylose-2	00F-4471-Y0	AJ0-8471

for ID: 2.0–3.0 mm

* Additional dimensions available upon request.

* SecurityGuard Analytical Cartridges require holder, Part No.: [KJ0-4282](#)



Ordering Information

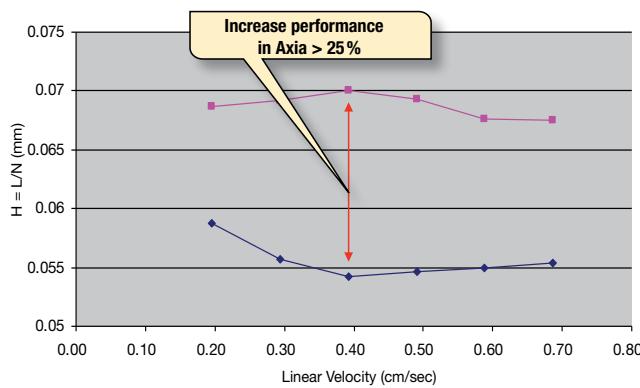
Supercritical Fluid Chromatography (SFC) Columns (mm)

Phase	150 x 4.6**	250 x 4.6**	250 x 10	SecurityGuard™ Cartridges (mm)	
Chiral Columns†				4 x 3.0*	10 x 10.0‡
Lux 5 μm i-Amylose-1	00F-4762-E0	00G-4762-E0	00G-4762-N0	AJ0-8641	AJ0-8642
Lux 5 μm i-Amylose-3	00F-4779-E0	00G-4779-E0	00G-4779-N0	AJ0-8650	AJ0-8652
Lux 5 μm i-Cellulose-5	00F-4756-E0	00G-4756-E0	00G-4756-N0	AJ0-8632	AJ0-8633
Lux 5 μm Cellulose-1	00F-4459-E0	00G-4459-E0	00G-4459-N0	AJ0-8403	AJ0-8404
Lux 5 μm Cellulose-2	00F-4457-E0	00G-4457-E0	00G-4457-N0	AJ0-8366	AJ0-8399
Lux 5 μm Cellulose-3	00F-4493-E0	00G-4493-E0	00G-4493-N0	AJ0-8622	AJ0-8623
Lux 5 μm Cellulose-4	00F-4491-E0	00G-4491-E0	00G-4491-N0	AJ0-8627	AJ0-8628
Lux 5 μm Amylose-1	00F-4732-E0	00G-4732-E0	00G-4732-N0	AJ0-9336	AJ0-9344
Lux 5 μm Amylose-2	00F-4472-E0	00G-4472-E0	00G-4472-N0	AJ0-8470	AJ0-8472

**Available in 3 μm . †Additional dimensions available upon request.

SFC Preparative Advantage Using Axia™ Packed Technology

Expect up to 25 % higher resolution when using the same material packing in Axia versus standard hardware.



for ID: 3.2–8.0 mm 9–16 mm

* SecurityGuard Analytical Cartridges require holder, Part No.: [KJ0-4282](#).



† SemiPrep SecurityGuard Cartridges require holder, Part No.: [AJ0-9281](#).



** SFC PREP 21.2 mm ID SecurityGuard Cartridges require holder, Part No.: [AJ0-8617](#).

* SFC PREP 30.0 mm ID SecurityGuard Cartridges require holder, Part No.: [AJ0-8618](#).

Supercritical Fluid Chromatography (SFC) Columns (mm) (cont'd)

Phase	250 x 21.2	250 x 30	250 x 50	SecurityGuard™ Cartridges (mm)	
Chiral Columns†				15 x 21.2	15 x 30.0*
Lux 5 μm i-Amylose-1	00G-4762-P0-AX	00G-4762-U0-AX	00G-4762-V0-AX	AJ0-8643	AJ0-8644
Lux 5 μm i-Amylose-3	00G-4779-P0-AX	00G-4779-U0-AX	00G-4779-V0-AX	AJ0-8653	AJ0-8654
Lux 5 μm i-Cellulose-5	00G-4756-P0-AX	00G-4756-U0-AX	00G-4756-V0-AX	AJ0-8634	AJ0-8635
Lux 5 μm Cellulose-1	00G-4459-P0-AX	00G-4459-U0-AX	00G-4459-V0-AX	AJ0-8405	AJ0-8406
Lux 5 μm Cellulose-2	00G-4457-P0-AX	00G-4457-U0-AX	00G-4457-V0-AX	AJ0-8400	AJ0-8401
Lux 5 μm Cellulose-3	00G-4493-P0-AX	00G-4493-U0-AX	00G-4493-V0-AX	AJ0-8624	AJ0-8625
Lux 5 μm Cellulose-4	00G-4491-P0-AX	00G-4491-U0-AX	00G-4491-V0-AX	AJ0-8629	AJ0-8630
Lux 5 μm Amylose-1	00G-4732-P0-AX	00G-4732-U0-AX	00G-4732-V0-AX	AJ0-9338	AJ0-9339

for ID: 18-29 mm 30-49 mm

†Additional dimensions available upon request.



Bulk SFC media is available. Please contact your Phenomenex representative for more information.

SFC Supercritical Fluid Chromatography

Achiral SFC Media

- Core-shell and fully porous media
- High surface area for increased loading
- Easy scale-up from lab to pilot plant
- Polar and non-polar selectivities for screening
- Columns interchangeable between SFC and HPLC modes

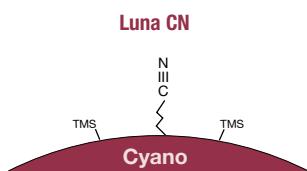
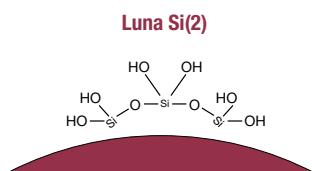
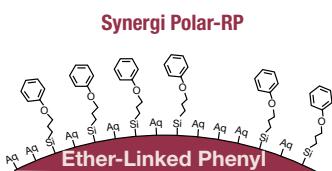
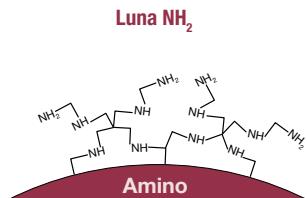
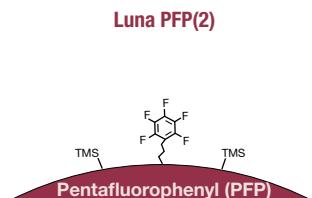
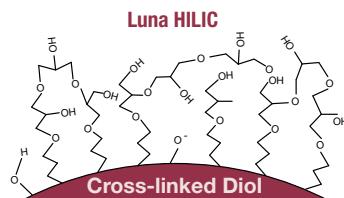
Media Selectivity is Critical for Success

Utilizing differences in surface chemistries will ensure that you achieve a successful separation for any given project, as in the example below. Once the ideal column phase is identified, you have the ability to optimize for additional improvements in performance:

- Changing retention
- Increasing efficiency
- Altering selectivity
- Reversing elution orders

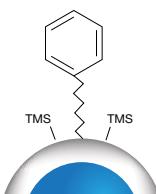
These optimization steps can easily be achieved by adjusting a few simple parameters. For instance, you can try different modifiers and/or additives, change the percent concentration of your modifier, or you can simply change your pressure, temperature, and/or flow rate.

Fully Porous Particles

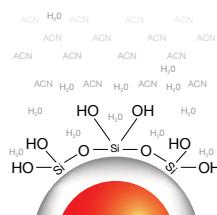


Core-Shell Particles

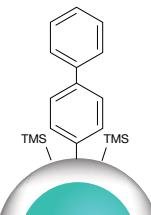
Kinetex Phenyl-Hexyl



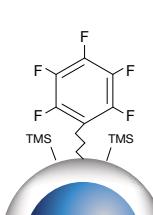
Kinetex HILIC



Kinetex Biphenyl



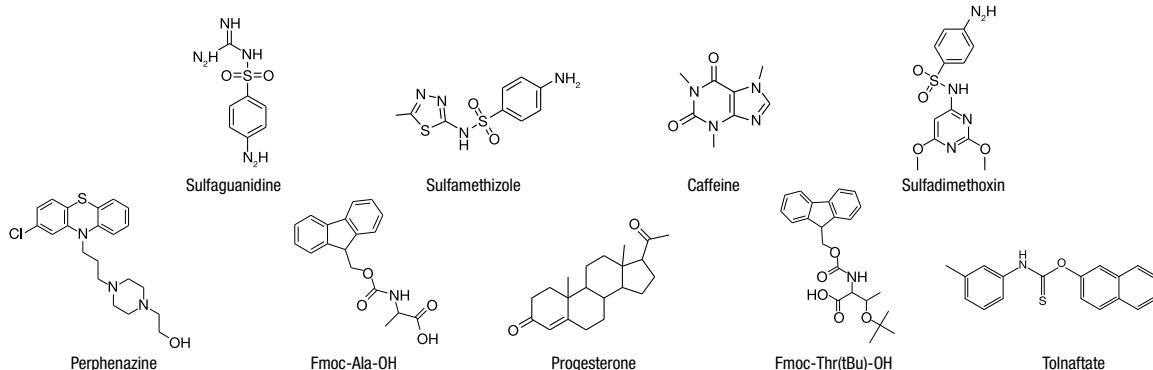
Kinetex F5



SFC Supercritical Fluid Chromatography

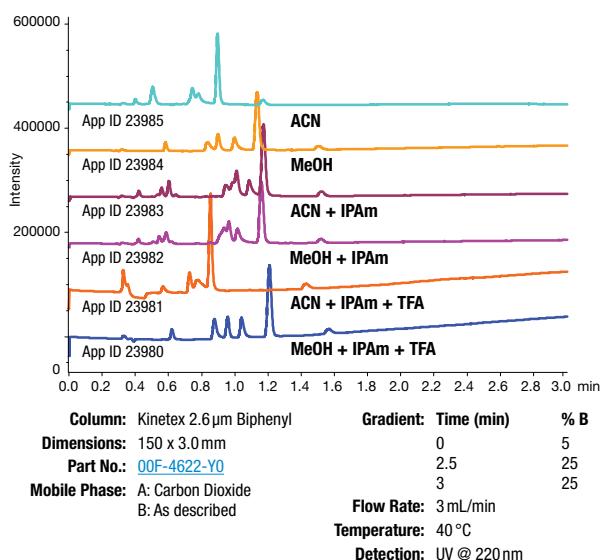
Achiral SFC Media (cont'd)

Screening Strategy Applied to the Separation of Pharmaceutically Related Compounds



Step 1. Screen Co-Solvents

- Use an appropriate sample that has a representative chromatographic profile
- Use a single column; this work used a Kinetex core-shell Biphenyl LC column
- Evaluate additives; this work used methanol to evaluate acidic, basic, acid/base mixed, and without any additives
- Use a fast gradient, an example would be 5 % to 25 % over 2 min with a 30 second hold
- Interpret results by comparing peak shape, retention and how many peaks were observed
- Evaluate other solvents such as acetonitrile, isopropanol, or mixtures if necessary
- Select the most promising conditions and move on to Step 2



Step 3. Method Optimization

Expand the gradient around the observed peaks

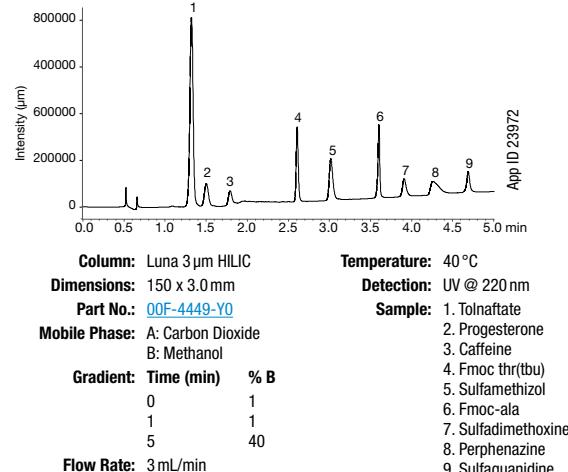
- If all of the peaks are early, lower the final gradient % co-solvent
- If all of the peaks are late, raise the initial gradient % co-solvent
- If the peaks are very close, extend the gradient over a longer period of time

Determine if a gradient is needed

- Evaluate if the chromatographic selectivity is dependent on the eluent density by screening with backpressure set higher and lower than typical; 20 – 30 bar difference is suitable

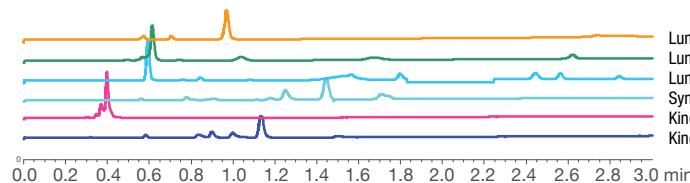
Finalize the gradient slope (if necessary)

- If the peaks are well resolved, shorten the time for the gradient
- If the peaks need more resolution, lengthen the time for the gradient



Step 2. Column Screening

- Use the best co-solvent additive combination found in Step 1
- Evaluate columns that have been previously successful with achiral SFC
- Use a gradient similar to the one used in Step 1
- Interpret results by comparing peak shape, retention and how many peaks were observed
- If nothing is promising, select other column chemistries and repeat
- If promising conditions are found, move on to Step 3



Column: As described
Dimensions: 150 x 3.0 mm
Mobile Phase: A: Carbon Dioxide
B: Methanol
Gradient: Time (min) % B
0 5
2.5 25
3 25
Flow Rate: 3 mL/min
Temperature: 40 °C
Detection: UV @ 220 nm

SFC Supercritical Fluid Chromatography

Achiral SFC Media (cont'd)

Achiral Material Characteristics

Packing Material Porous	Particle Size (μm)	Pore Size (\AA)	Surface Area (m^2/g)	Carbon Load %	End Capping	pH Stability
Luna Silica(2)	3, 5, 10, 15	100	400	0	No	2.0 - 7.5
Luna HILIC	3, 5	200	200	5.7	No	1.5 - 8.0
Luna PFP(2)	3, 5	100	400	11.5	Yes	1.5 - 9.0
Luna CN	3, 5, 10	100	400	7.0	Yes	1.5 - 7.0
Luna NH ₂	3, 5, 10	100	400	9.5	No	1.5 - 11.0
Synergi Polar-RP	2.5, 4, 10	80/100*	475/400*	11	proprietary	1.5 - 7.0
Packing Material Core-Shell						
Kinetex HILIC	1.7, 2.6, 5	100	200	0	No	2.0 - 7.5
Kinetex Biphenyl	1.7, 2.6, 5	100	200	11	Yes	1.5 - 8.5**
Kinetex Phenyl-Hexyl	1.7, 2.6, 5	100	200	11	Yes	1.5 - 8.5**
Kinetex F5	1.7, 2.6, 5	100	200	9	Yes	1.5 - 8.5

**Columns are pH stable from 1.5 - 10 under isocratic conditions. Columns are pH stable under 1.5 - 8.5 under gradient conditions.

*Specs. for 2.5 μm Synergi Polar-RP

Ordering Information

Supercritical Fluid Chromatography (SFC) Columns (mm)					Axia™ Packed Preparative Columns		SecurityGuard™ Cartridges (mm)			
Phase	150 x 4.6	250 x 4.6	250 x 10	250 x 21.2	250 x 30		4 x 3.0*	10 x 10*	15 x 21.2**	15 x 30°
Achiral Columns[†]							/10pk	/3pk	/ea	/ea
Luna 5 μm Silica(2)	00F-4274-E0	00G-4274-E0	00G-4274-N0	00G-4274-PO-AX	00G-4274-U0-AX		AJ0-4348	AJ0-7223	AJ0-7229	AJ0-8312
Luna 5 μm HILIC	00F-4450-E0	00G-4450-E0	00G-4450-N0	00G-4450-PO-AX	00G-4450-U0-AX		AJ0-8329	AJ0-8902	—	—
Luna 5 μm PFP(2)	00F-4448-E0	00G-4448-E0	00G-4448-N0	00G-4448-PO-AX	—		AJ0-8327	AJ0-8376	AJ0-8377	AJ0-8378
Luna 5 μm CN	00F-4255-E0	00G-4255-E0	00G-4255-N0	00G-4255-PO-AX	00G-4255-U0-AX		AJ0-4305	AJ0-7313	AJ0-8220	AJ0-8311
Luna 5 μm NH ₂	00F-4378-E0	00G-4378-E0	00G-4378-N0	00G-4378-PO-AX	—		AJ0-4302	AJ0-7364	AJ0-8162	AJ0-8309
Synergi 4 μm Polar-RP	00F-4336-E0	00G-4336-E0	00G-4336-N0	00G-4336-PO-AX	00G-4336-U0-AX		AJ0-6076	AJ0-7276	AJ0-7845	AJ0-8307
Phase	150 x 4.6	250 x 4.6	250 x 10	250 x 21.2	250 x 30	4.6*	10 x 10	15 x 21.2	15 x 30	
Core-Shell Kinetex Technology						/3pk	/3pk	/ea	/ea	
Kinetex 2.6 μm HILIC	00F-4461-E0	00G-4461-E0	—	—	—		AJ0-8772	—	—	—
Kinetex 5 μm Biphenyl	00F-4627-E0	00G-4627-E0	00G-4627-N0	00G-4627-PO-AX	—		AJ0-9207	AJ0-9280	AJ0-9272	AJ0-9273
Kinetex 5 μm F5	00F-4724-E0	00G-4724-E0	00G-4724-N0	00G-4724-PO-AX	00G-4724-U0-AX		AJ0-9320	AJ0-9323	AJ0-9324	AJ0-9325
Kinetex 5 μm Phenyl-Hexyl	00F-4603-E0	00G-4603-E0	—	00G-4603-PO-AX	00G-4603-U0-AX		AJ0-8774	—	AJ0-9147	AJ0-9216

[†]Additional phases and dimensions available upon request.

for ID: 3.2-8.0 mm 9-16 mm 18-29 mm 30-49 mm

^{*} SecurityGuard ULTRA Cartridges require holder Part No.: [AJ0-9000](#)

[†] SecurityGuard Analytical Cartridges require holder Part No.: [KJ0-4282](#)

[‡] SemiPrep SecurityGuard Cartridges require holder Part No.: [AJ0-9281](#)

^{**} SFC PREP SecurityGuard Cartridges require holder Part No.: [AJ0-8617](#)

[°] SFC PREP SecurityGuard Cartridges require holder Part No.: [AJ0-8618](#)

Additional Non-Polar Phases Available

- C18/C8/C4
- Phenyl-Hexyl
- TWIN™ Technology C18
- TWIN Technology C6-Phenyl
- Fusion-RP
- Hydro-RP and more...



For more information on core-shell Kinetex media,
please see p. 227



Bulk SFC media is available. Please contact your Phenomenex
representative for more information.