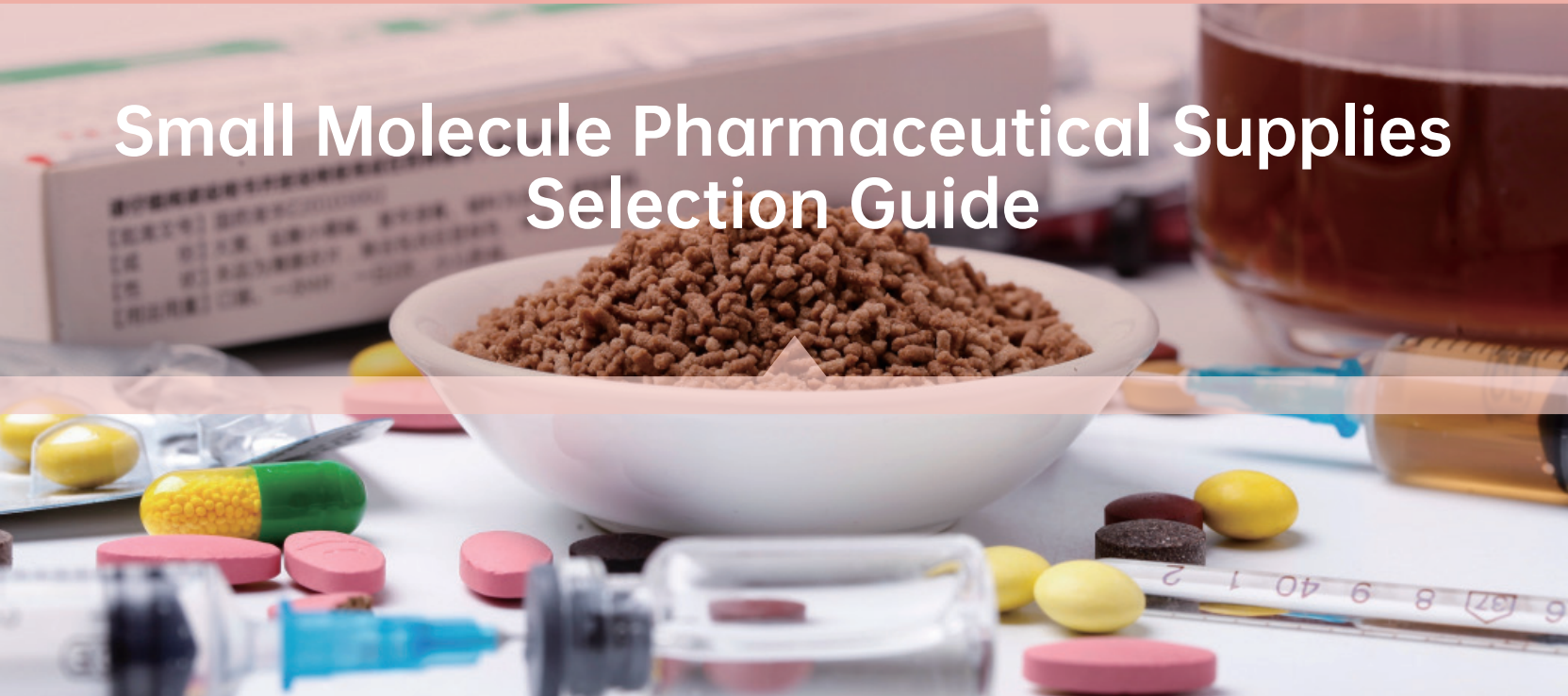
SHIMADZU
Excellence in Science

SGLC-POS-016

Small Molecule Pharmaceutical Supplies Selection Guide



SGLC offers "one-stop" consumables solutions

You are not alone with SGLC

Preface

SGLC is providing this Small Molecule Pharmaceutical Supplies Selection Guide based on the needs of Pharmaceutical Analysis.

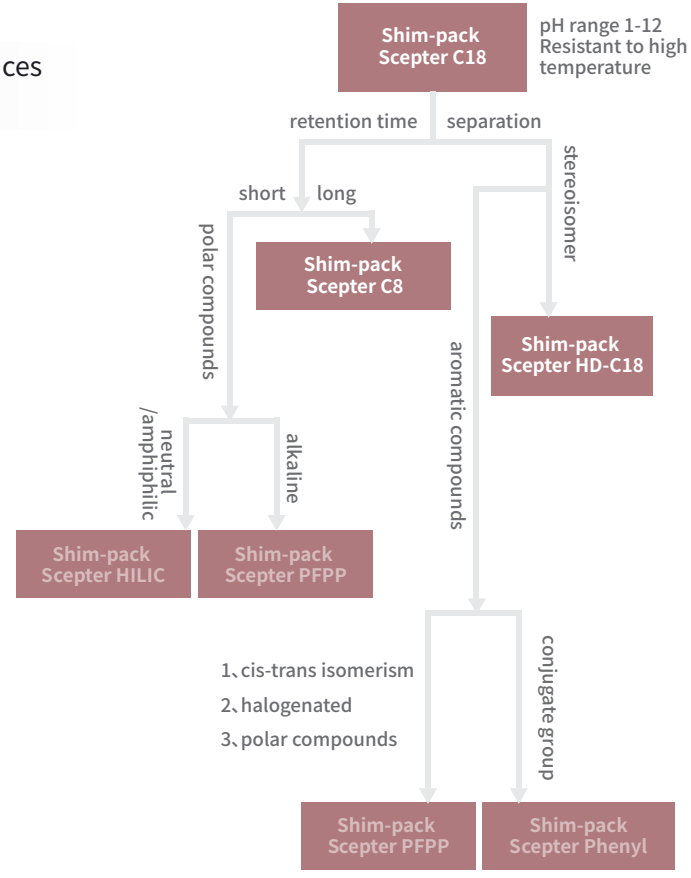
CONTENTS

Related Substances

Separation is the primary concern for related substances analysis.

Shim-pack Scepter Series

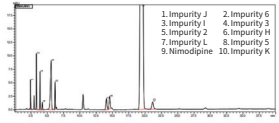
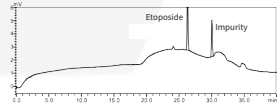
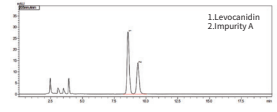
- The next generation organic silica hybrid based columns!
- A wide range of LC conditions!
- Best choice of Related substances analysis

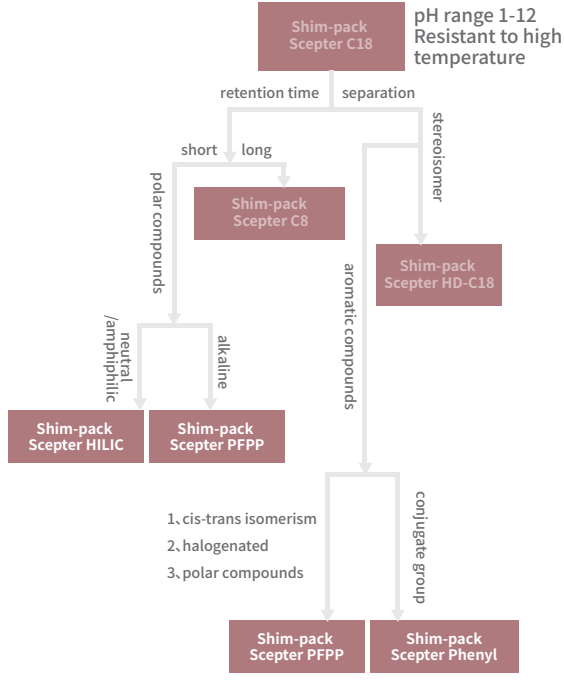


Reversed phase

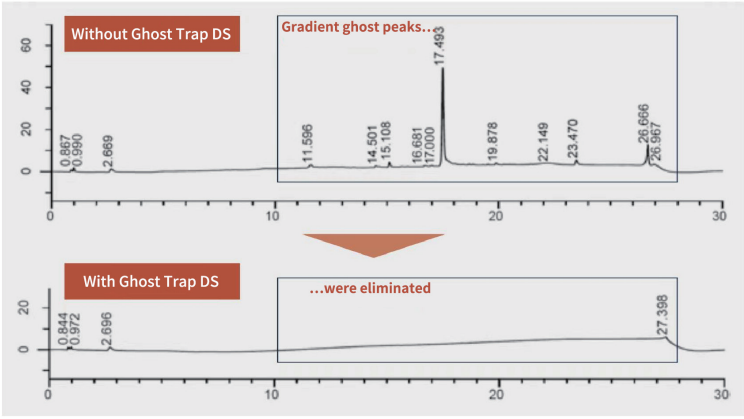
Classification	Drug	LC Column	Feature	Classification	Drug	LC Column	Feature
Antibiotics	Azithromycin, Penicillin Sodium, Cefaclor, Cefazolin Sodium, Cefoperazone Sodium	Shim-pack Scepter C18 (4.6mm×250mm, 5μm, PN: 227-31020-06)	Good separation and reproducibility.		Aceclofenac	Shim-pack Scepter C18 (4.6mm×250mm, 5μm, PN: 227-31020-06)	Perfect separation and peak symmetry.
	Sulbactam Sodium, Flucloxacillin Sodium	Shim-pack Scepter C18 (4.6mm×150mm, 3μm, PN:227-31016-05)	Good separation of impurities and API.	Anti-inflammatory	Dexibuprofen	Shim-pack Scepter C18 (4.6mm×150mm, 3μm, PN:227-31016-05)	Baseline separation of isomeric impurities.
Hormone	Rednisone Acetate, Hydrocortisone, Cortisone Acetate, Hydrocortisone Acetate, Ethinyl Estradiol, Triamcinolone Acetate	Shim-pack Scepter C18 (2.1mm×100mm, 1.9μm, PN:227-31012-05)	UHPLC method with good separations& fast.		Chlortetracycline	Shim-pack Scepter C18 (4.6×100 mm, 3 μm, PN：380-31016-04)	Symmetrical peaks & good separation.
	Dexamethasone Acetate	Shim-pack Scepter C18 (4.6mm×250mm, 5μm, PN: 227-31020-06)	Baseline separation & good peak shapes.	Artemisinin-based	Dihydroartemisinin, Artesunate	Shim-pack Scepter C18 (4.6×100 mm, 3 μm, PN：380-31016-04)	Good separation & perfect peak shapes.
Antihyper-tensive	Rbesartan, Captopril	Shim-pack Scepter C18 (4.6mm×150mm, 3μm, PN:227-31016-05)	Good separation& perfect peak shapes.	Antivirals	Adefovir	Shim-pack Scepter C18 (4.6mm×150mm, 5μm PN:227-31020-05)	Baseline separation.
	Nifedipine	Shim-pack Scepter C18 (2.1mm×100mm, 1.9μm, PN:227-31012-05)	Separation Rs>1.5.		Valaciclovir Hydrochloride	Shim-pack Scepter C18 (2.1mm×100mm, 1.9μm, PN:227-31012-05)	UHPLC method & baseline separation.
	Nimodipine	Shim-pack Scepter HD-C18 (4.6mm×250mm, 5 μm, PN: 227-31024-06)	Baseline separation & perfect peak shapes.	Antihyper-glycemic	Gliquinone	Shim-pack Scepter C18 (2.1mm×100mm, 1.9μm, PN:227-31012-05)	UHPLC method.
	Perindopril Indapamide	Shim-pack Scepter C8 (4.6mm × 250mm, 5μm, PN：227-31041-06)	Good separation & symmetric peaks.	Hyperuri-cemia/gout	Febuxostat	Shim-pack Scepter HD-C18 (4.6mm×250mm, 5 μm, PN: 227-31024-06)	The baseline separation; good peak shapes and reproducibility.
Anti-tumor	Flutamide	Shim-pack Scepter C18 (4.6mm×150mm, 3μm, PN:227-31016-05)	Perfect peak shape & good separation.	Muscle relaxant	Baclofen	Shim-pack Scepter C18 (4.6 mm×250 mm, 5μm, PN: 227-31020-06) Shim-pack Scepter C18 (2.1 mm×100 mm, 1.9μm, PN:227-31012-05)	Baseline separation, good retention of the main peak.
	Letrozole	Shim-pack Scepter C18 (4.6mm×150mm, 3μm, PN:227-31016-05)	UHPLC method achieves good baseline separation of the 4.4min main peak from isomeric.	Antimicro-bial	Terbinafine Hydrochloride	Shim-pack Scepter C18 (2.1mm×100mm, 1.9μm, PN:227-31012-05)	Good separation & perfect peak shapes.

Related Substances

Drug	LC column	Feature	Chromatogram
Nimodipine	Shim-pack Scepter Phenyl-120 (4.6 mm × 250 mm, 5 µm, PN:227-31071-06)	Good peak shapes; separation meets the requirements.	
Etoposide	Shim-pack Scepter Phenyl-120 (4.6 mm × 250 mm, 5 µm, PN:227-31071-06)	Separation and retention time were in accordance with the requirements of pharmacopoeia.	
Levocanidin	Shim-pack Scepter Phenyl-120 (4.6 mm × 250 mm, 5 µm, PN:227-31071-06)	Symmetric peaks & good separation.	



"Magic tool" for Related Substances Analysis-Ghost Trap DS



Column : ODS column
Mobile Phase : A) 25 mmol/L Phosphate (Potassium) buffer solution (pH 4.0) /Acetonitrile = 9/1
B) Water/Acetonitrile = 1/9
Flow Rate : 0.65 mL/min
Col. Temp : 45 °C
Detection : UV 210 nm

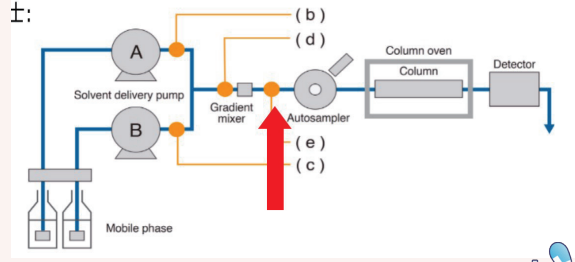
Ghost Trap DS* was co-developed with Daiichi Sankyo Co., Ltd.
Higher trapping efficiency and longer life!

* DS: Abbreviation of Daiichi Sankyo (D) and Shimadzu (S)

Ghost Trap DS

Economic type

TIPS.



- Installation position: (e). Between the injector and mixer. Only capture the ghost peaks from the mobile phase, pipeline, system; no effect on the sample.
- Not apply to ion-pair reagent.

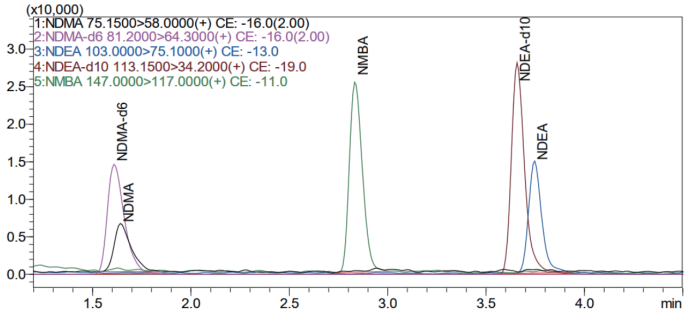
Genotoxic Impurities

Better separation, higher sensitivity!

USP <1469> specifies detection methods for nitrosamine-based toxic impurities that include HPLC-MS/MS and GC-MS/MS.

Application 1 Quantification of Nitrosamines (LCMS/MS)

Shim-pack Velox C18 -- High sensitivity & fast

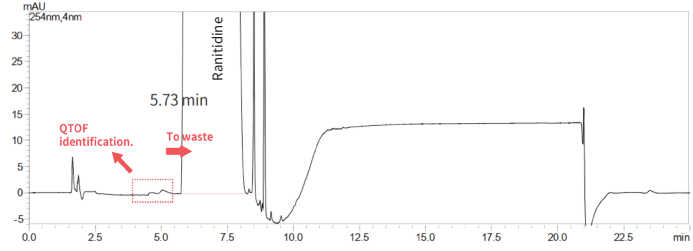


LC conditions :

Column: Shim-pack Velox C18 (2.1mm×100 mm, 1.8μm, PN: R227-32007-03)
Eluent: A-0.1% formic acid; B-methanol
Needle wash mode: before and after injection
Flow rate: 0.3 mL/min
Temperature: 40 °C
Injection volume: 20 μL
Elution mode: gradient elution

Application 2 Structural characterization of Nitrosamines (LCMS-QTOF)

Shim-pack Scepter C18 -- Excellent separation of impurities near the main peak



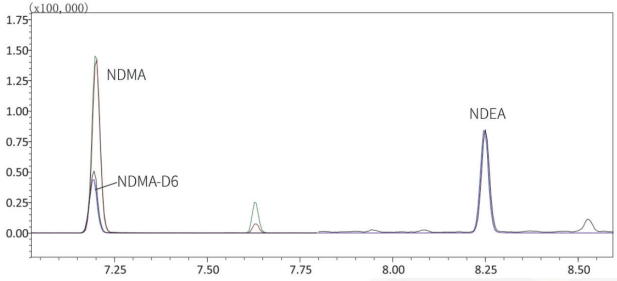
LC conditions :

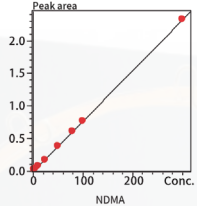
Column: Shim-pack Scepter C18 (4.6mm×150mm, 5.0μm, PN: 227-30017-07)
Eluent: A-0.1% formic acid; B-methanol
Flow rate: 1.0 mL/min
Injection volume: 5 μL
Temperature: 40 °C
Elution mode: gradient elution

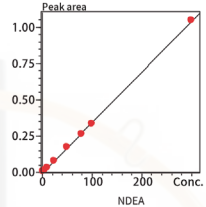
*EMA/409815/2020 Rev.17 ;
FDA/Control of Nitrosamine Impurities in Human Drugs Guidance for Industry)

Application 3 Quantification of Nitrosamines (HS-GC-MS)

SH-I-624Sil MS -- Excellent linearity and signal-to-noise ratio!







ID	NAME	R	S/N (2.5 μg/L)
1	NDMA	0.9999	23.39
2	NDEA	0.9998	35.81

GC conditions :

Column: SH-I-624Sil MS (60 m×0.32 mm×1.8 μm PN:221-75963-60)

Temperature program: 40°C(1 min)_40°C/min_180°C_20°C/min_300°C(2 min)

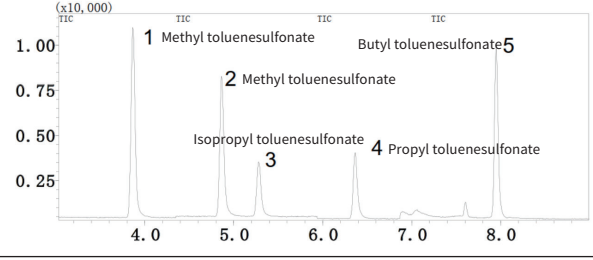
Injection volume: 2 μL

Carrier gas control mode: constant linear velocity (36.1 cm/sec) Inlet mode: non-split injection Ion source temperature: 230°

Interface temperature: 300°C Detector voltage: tuned voltage +0.6kV Acquisition method: MRM

Application 4 Quantification of Methanesulfonates (HS-GC-MS)

SH-PolarWax -- Bonded Crosslink™ polyethylene glycol stationary phase, low loss ensures column lifetime



☐ Headspace conditions

Equilibration temperature: 60°C; Equilibration time: 30 min; Sample pathway temperature: 100°C.

☐ GCMS conditions

Column: SH-PolarWax Column (30m×0.25mm×0.25 μm, PN:227-36305-02)

Temperature program: 40°C(1 min)_10°C/min_ 130°C_40°C/min_ 230°C(8 min)

Carrier Gas: He, constant flow mode.

Split ratio: 20:1

Ionization mode: EI

Ion source temperature: 220°C

Interface temperature：220°C

Flow rate: 0.8 mL/min to 2 mL/min after 10 min.

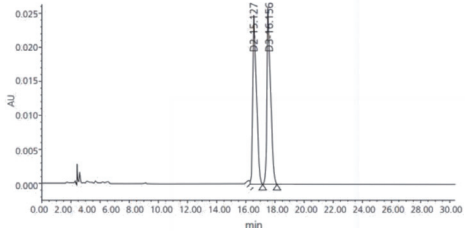
ASSAY

ShimNex UP Series

- Silica-based, multi-specifications!
- High column-to-column performance reproducibility!
- Best choice for ASSAY!

ShimNex UP Series								
Ligand Type	Particle Size	Pore Size	LD.:mm					
			2.1	3.0	4.0	4.6	10.0	21.2
C18	1.8μm	100 Å	●	●	○	○	○	○
	2.5μm		●	●	○	●	○	○
	3.5μm		●	●	●	●	○	○
	5μm		●	●	●	●	●	●
	10μm		○	○	●	●	●	●
C8	1.8μm	100 Å	●	●	○	○	○	○
	2.5μm		●	●	○	●	○	○
	3.5μm		●	●	●	●	○	○
	5μm		●	●	●	●	●	●
	10μm		○	○	●	●	●	●
C4	1.8μm	100 Å	●	●	○	○	○	○
	2.5μm		●	●	○	●	○	○
	3.5μm		●	●	●	●	○	○
	5μm		●	●	●	●	●	●
	10μm		○	○	●	●	●	●
Length : 50mm, 100mm, 150mm, 250mm								
注: ● :available; ○ :unavailable								

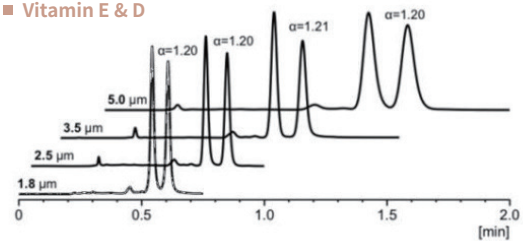
Vitamin D2 & D3



LC conditions:

Column: ShimNex UP C18, 4.6x250mm, 5μm (PN:380-01231-49)
Eluent: methanol:water=98:2
Flow rate: 1 .0 mL/min
Temperature: 40°C
Detection: UV 265nm
Injection volume: 10 μL

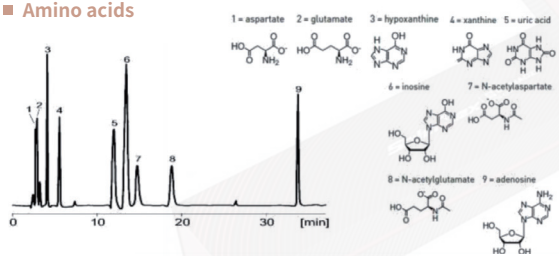
Vitamin E & D



LC conditions:

Column: ShimNex UP C4, 2.1x50mm, 1.8μm or 4.6x50mm, ⩾2.5μm
Eluent: acetonitrile
Flow rate: 5.0 μm: 1.0 mL/min; 3.5 μm: 1.5 mL/min:2.5 μm: 2.0 mL/min; 1.8 μm: 0.6mL/min
Temperature: 20°C
Detection: UV 215nm

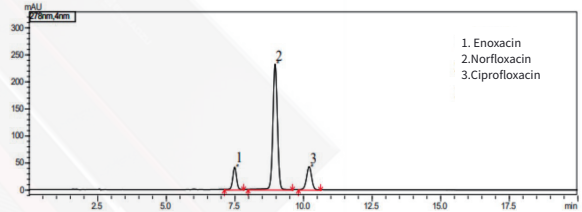
Amino acids



LC conditions:

Column: ShimNex UP C18, 4.6x250mm, 5μm (PN:380-01231-49)
Eluent: 28 mM tetrabutylammonium hydroxide, 25 mM potassium dihydrogen phosphate, 1.25% methanol (pH 7)
Flow rate: 1 .0 mL/min
Temperature: 23°C
Detection: UV 210nm

Norfloxacin



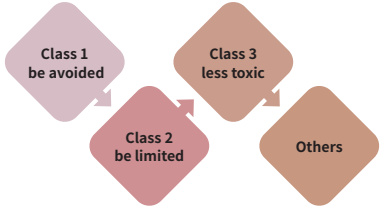
LC conditions:

Column: ShimNex UP C18, 4.6×250 mm, 5 μm (PN:380-01231-49)
Eluent: 0.025 mol/L Phosphoric acid (adjusted to pH 3.0 with triethyl-amine)-acetonitrile=87:13
Flow rate: 1.5 mL/min
Temperature: 40°C
Detection: UV 278nm
Injection volume: 20 μL

Analysis of Residual Solvents

SH-I-624Sil MS

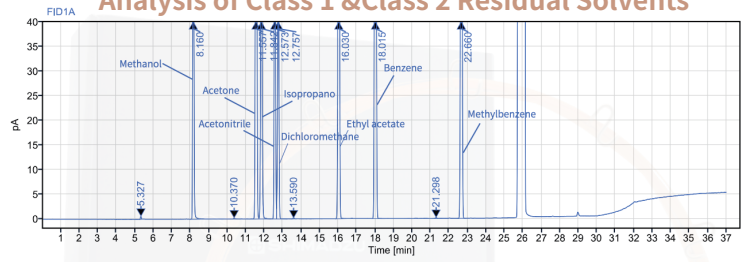
- Mid-polarity Crossbond™ silarylene phase.
- Ultra-inert & high-thermal stability!
- Best choice for residual solvent analysis!



List of GC Columns for Residual Solvent Analysis

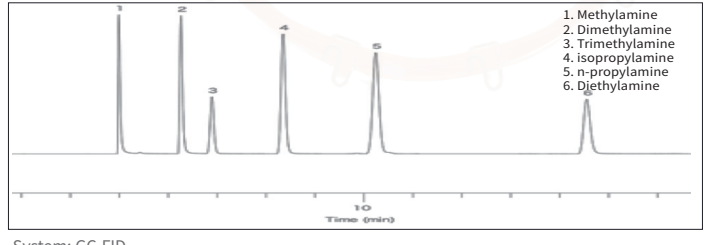
Category	Columns	S/N No.
Non-polar (-1)	SH-I-1MS Cap. Column, 30mx0.32mmx1μm	R227-36010-01
Medium polarity (-624)	SH-I-624Sil MS Cap. Column, 30mx0.32mmx1.8μm	R227-36077-01
Polar (WAX)	SH-PolarWax Column, 30mx0.25mmx0.25μm SH-PolarWax-MS, 30mx0.25mmx0.25μm	R227-36305-02 R227-36322-01
Columns for amine analysis	SH-Volatil Amin Cap. Column, 30mx0.32mmx5μm	R227-36326-01

Analysis of Class 1 &Class 2 Residual Solvents



Column: SH-I-624Sil MS, 30 m × 0.53 mm × 3.0 μm (PN: R227-36078-01)
Injection method: Headspace injection
Split ratio: 5: 1
Equilibrium temperature: 80 °C
Inlet temperature: 250 °C
Quantification loop temperature: 100 °C
GC cycle time: 45 min
GC cycle time: 45 min
Temperature program: 35 °C (hold 5 min) to 160 °C at 5 °C/min (hold 5 min)) to 240 °C at 40 °C/min (hold 5 min)

Analysis of Amines



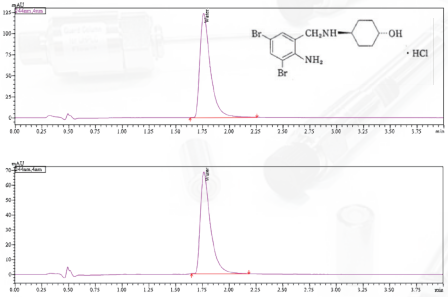
System: GC-FID
Column: SH-Volatil Amin (60 m x 0.32 mm x 5.00 μm)
Temperature: 60°C
Carrier gas: He 150kPa
Split ratio: 1:50

Dissolution Test

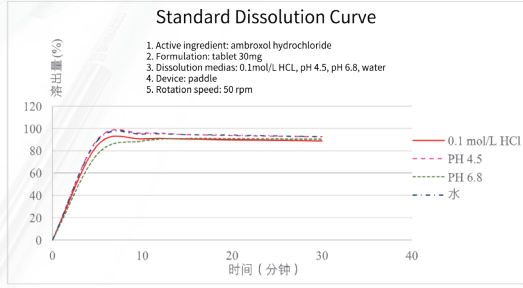
Shim-pack Scepter Series

- Ultra-high tolerance, fast, easy to handle complex sample matrices!
- The best choice for analyzing dissolved samples!

In Vitro Dissolution Study of Ambroxol Hydrochloride Tablets in Four Dissolution Medias



Chromatogram of Ambroxol Hydrochloride



Chromatogram of Ambroxol Hydrochloride

LC conditions:

Column: Shim-pack Scepter C18（4.6mm×50mm, 5μm, PN: 227-31020-02)
Eluent: A- 0.01molL Ammonium dihydrogen phosphate B: acetonitrile, A/B = 1:1(v/v)
Flow rate: 0.4 mL/min
Temperature: 40°C
Detection: 244nm
Injection volume: 5μL
The conditions of dissolution determination were operated according to the draft of generic drug consistency evaluation of Amoxicillin Hydrochloride Tablets drafted by National Medical Products Administration.

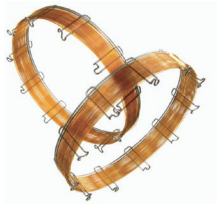
TIPS.

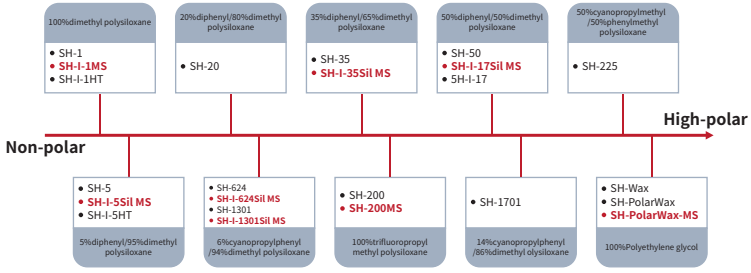
Reasons for unstable retention time: column contamination, insufficient column equilibration time, selective volatilization due to different nature of mobile phase, unstable column temperature

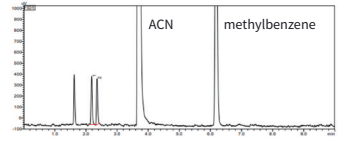
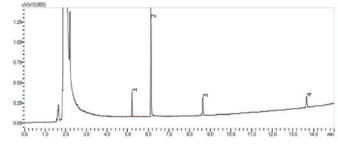
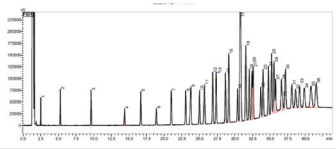
Pharmaceutical Excipients & Packing Materials

SH Series GC Columns

- Excellent chromatographic performance, reproducibility & lifetime!





Cases	GC Column	Features	Chromatograms
Determination of ethylene oxide (EO) in pharmaceutical packaging materials (pre-filled syringes)	**SH-624** 30 m×0.25 mm × 1.4 μm PN:R221-75863-30; SH-I-624Sil MS 30 m×0.32 mm × 1.8 μm PN:R227-36077-01	Ultra-high inertness and low loss, temperature resistant to 300/320°C, unique selectivity for volatile organic compounds with boiling points below 200°C, such as ethylene oxide, propylene oxide, epichlorohydrin, etc. EO peak is baseline separated from the adjacent impurity acetaldehyde peak in the spectrum.	
Glycol, diethylene glycol, triethylene glycol in Polysorbate 80	**SH-PolarWax** 30 m×0.25 mm × 0.25 μm PN:227-36305-02	Less interference between ethylene glycol and propylene glycol; better peak shapes!	
Fatty acid composition of lipid substances for pharmaceutical and excipients	**SH-Wax** 30 m×0.53 mm × 1 μm PN:221-75899-30	Determination of 36 fatty acids, meets the system suitability requirements of the fatty acid composition determination of the regulation.	

Preparative Scale LC Column

Shim-pack Scepter PREP series

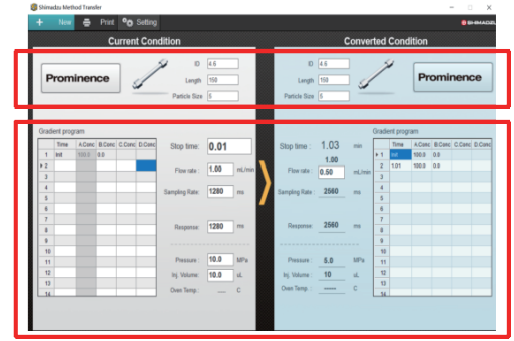
- A wide range of LC conditions & multiple phases!
- High column-to-column performance reproducibility
- Best choice of Preparative columns

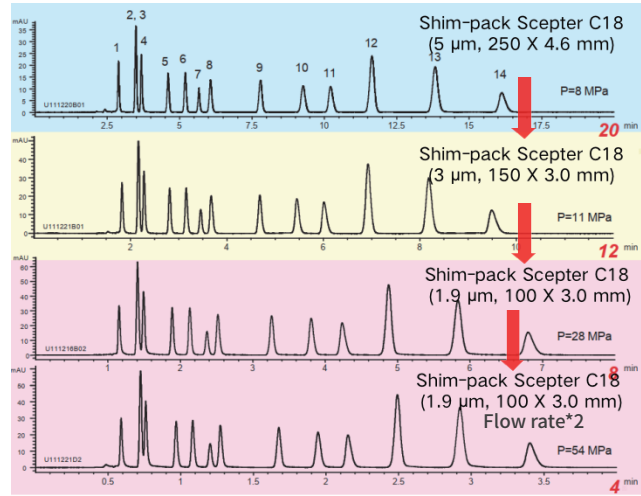
Flow rate equation (Same with Loading) :

$$\text{Prep flow rate} = \text{Analytical flow rate} \times \frac{(\text{Prep I.D.})^2}{(\text{Analytical I.D.})^2}$$

HPLC >>> PREP HPLC

1. LC column specifications
2. Mobile phase/injection/-flow rate/column temperature, etc.
3. Mixing mode/mixer volume





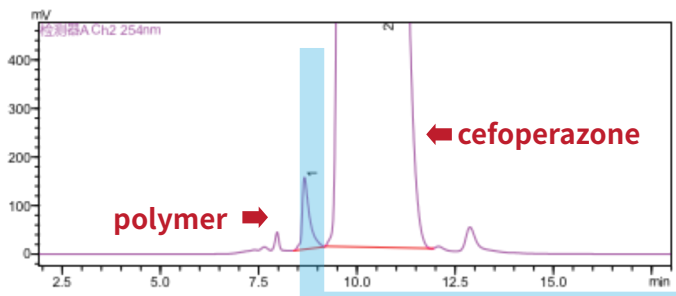
Lab Scale→Production Scale

	Loading (mg)	Lab Scale	10-50		50-100	100-1000	1g-10g	10g-100g	⩾100g
	I.D.(mm) / Particle Size (μm)	4.6	10	20	30	50	100	200	⩾300
High ↑ Efficiency, Cost, Perssure ↓ Low	5	●	●	●	●	●	●	●	
	10	●	●	●	●	●	●	●	●
	10~20	●	●	●	●	●	●	●	●
	15~30		●	●	●	●	●	●	●
	50					●	●	●	●

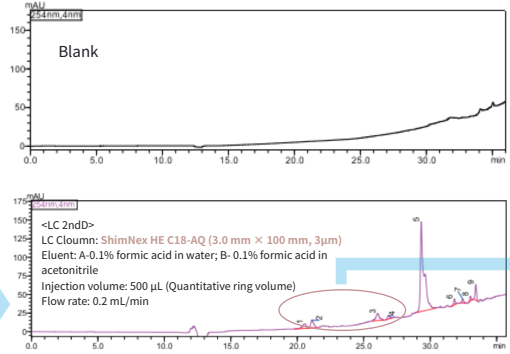
● Most appropriate ● Appropriate ● Depending on purpose

Prep LC Column

Featured Solution– desalted by 2D chromatography & identification of cephalosporin polymers



Chromatogram of cefoperazone in 1st dimensional LC system



QTOF LCMS-9030

Four polymer structures were identified.

Solutions

SHIMSEN Ankylo-SEC

Fast & high column efficiency; excellent batch reproducibility.

ShimNex HE C18-AQ

Homogeneous particle size, rapid elution under the premise of ensuring the separation.

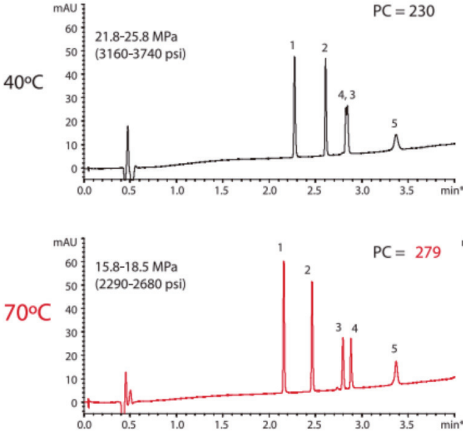
TIPS.

▶ **Non-volatile mobile phase additives are not allowed to enter the mass spectrometry detector.**

Featured Solution- column temperature adjustment

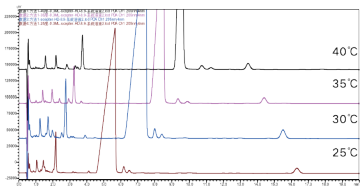
Improvement of separation and peak shapes by increasing column temperature

Peptides/Proteins

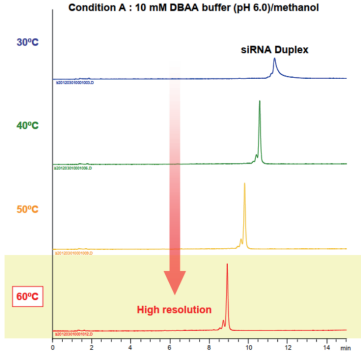


Analytes	MW	Peak width0.5h (min)	
		40°C	70°C
Condition A			
1. Oxytocin	1,007	0.017	0.014
2. Leu-Enkephalin	556	0.015	0.015
3. β-Endorphin	3,465	-	0.016
4. Insulin	5,733	-	0.015
5. β-Lactoglobulin A	18,400	0.043	0.030

Macrolide Antibiotic

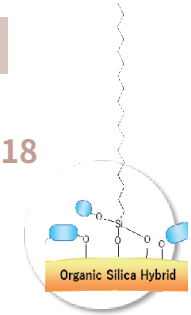


Ribonucleic acid

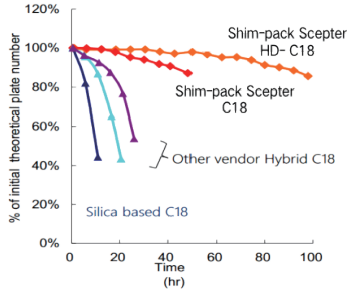


Shim-pack Scepter C18

Organic silica hybrid based column
pH range 1-12
High temperature resistance



Stability at high pH
(pH 11.5, 40C)

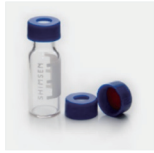


TIPS.

Be sure to use the column within the specified conditions.

化药相关消耗品推荐

Vials



SHIMSEN Vial
Laboratory General Vials

SHIMSEN ARC Vial
Economic choice

SHIMSEN Low adsorption
Low adsorption PP material

Labtotal Vial
LC/MS and GC/MS certification report
Cover gasket pre-installation

Needle Filter

HPTFE
For aquatic and organic

PTFE
For organic

PES
For Aquatic

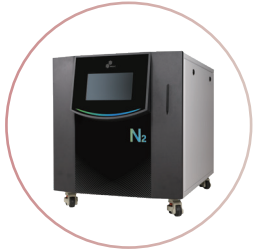
Other Consumables

Safety caps

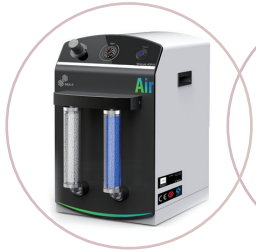
Solvent bottle

Pipette

Equipment



Nitrogen generator for LC-MS



Air compressor for GC

Hydrogen generator for GC

Air compressor for AA

Cooling circulating water unit for AA

Book Online

岛津消耗品全套方案 一站式解决!

行业解决方案

产品选型卡

Shimadzu (Shanghai) Global Laboratory Consumables Co.,Ltd.
www.sglc.shimadzu.com.cn
www.shimadzumall.com
Contact: contact@sglc.shimadzu.com.cn