This presentation describes what Supelco ionic liquid GC columns are, and includes several column selection charts.

Agenda

- Overview
- GC Column Polarity Scale
- Temperature Effects on Selectivity
- Column Selection
- Summary / Related Products / Resources

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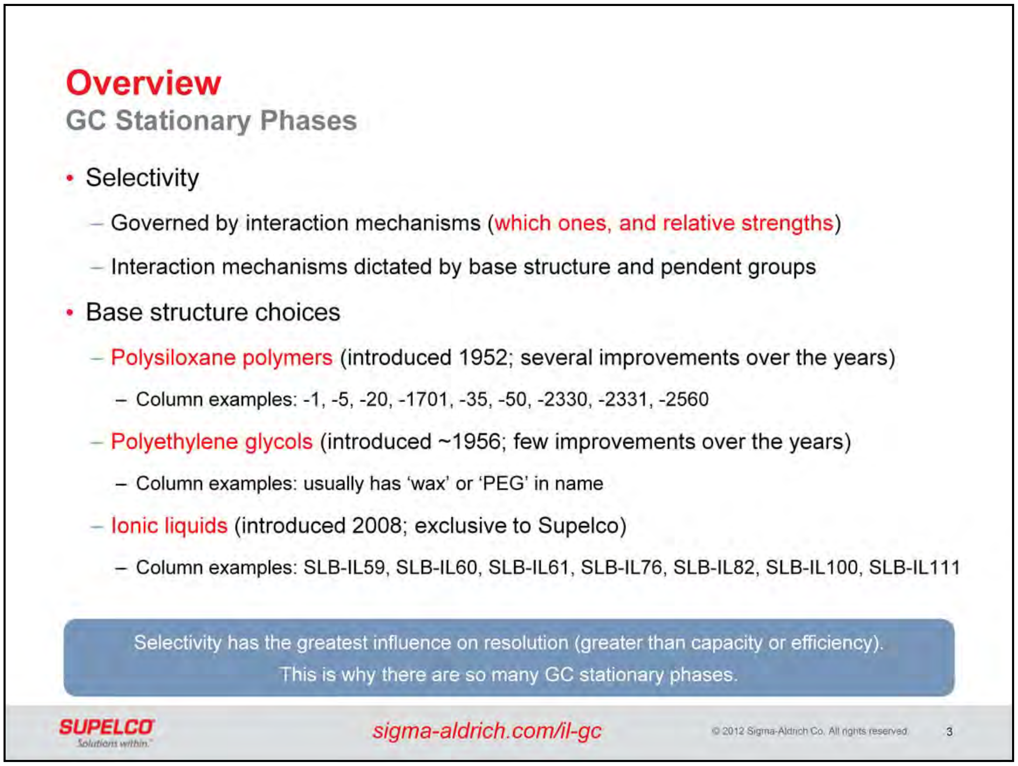
Overview



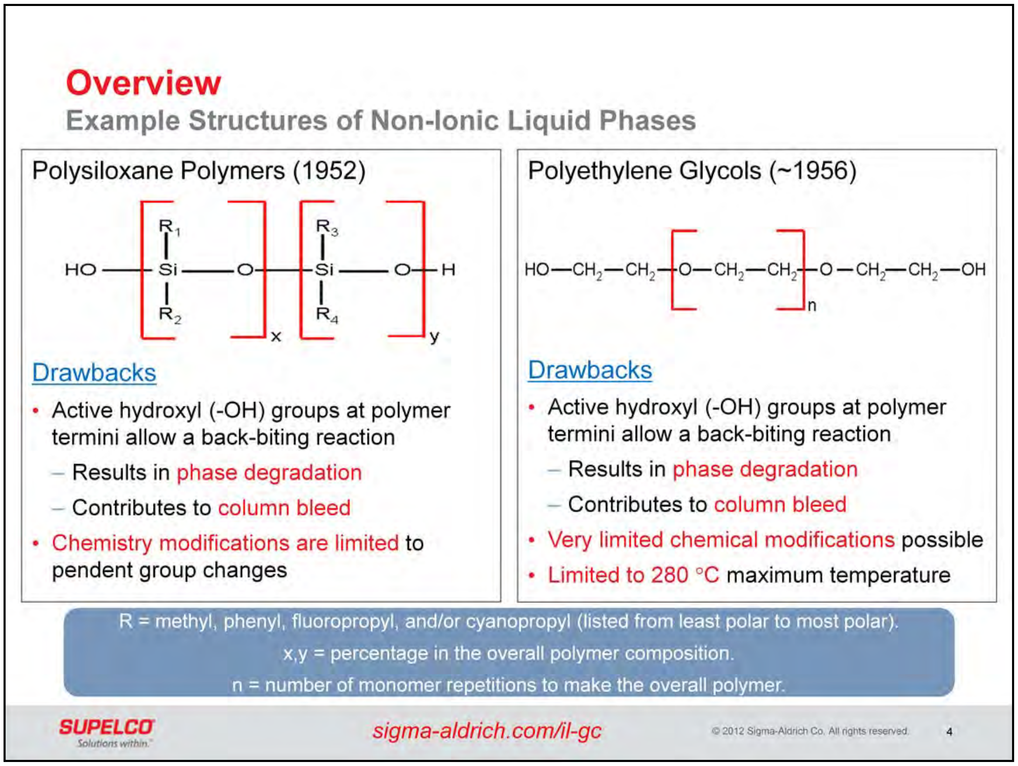
Selectivity is important as it has the greatest influence on resolution; greater than either capacity or efficiency. Selectivity is governed by two aspects of interaction mechanisms:

- Which mechanisms are available
- The relative strengths of these mechanisms to each other

Interaction mechanisms are dictated by the base structure of the stationary phase, as well as any pendent groups.

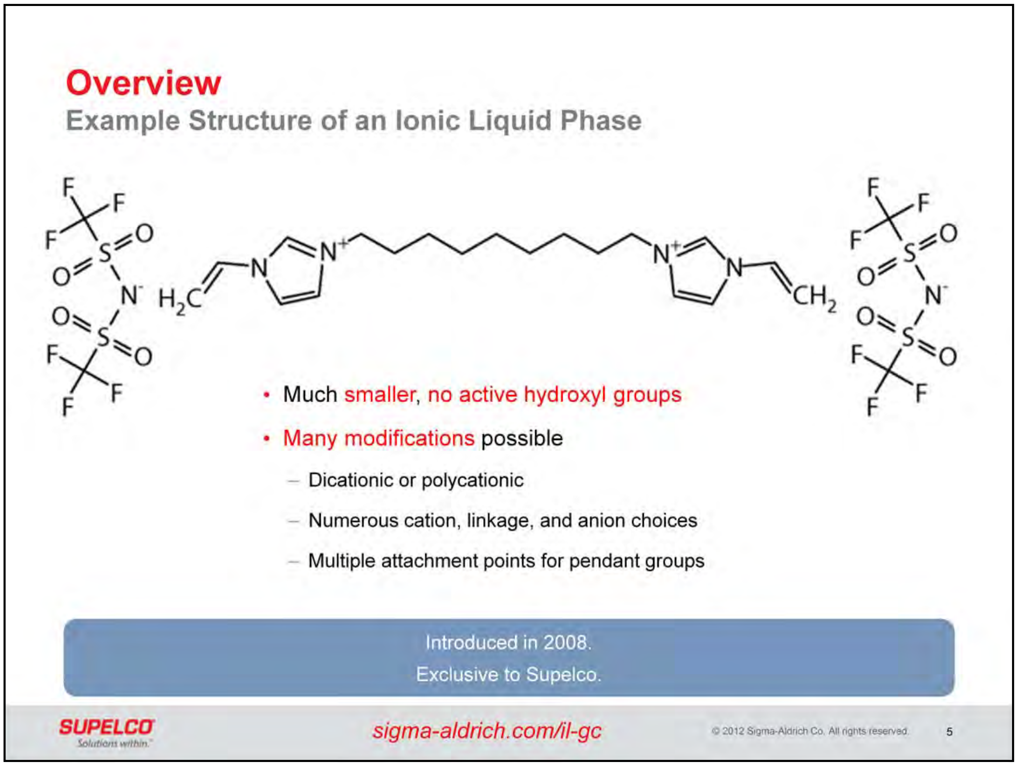
With a few exceptions (namely chiral and PLOT columns), just three base structures are typically used as GC stationary phases.

- Substituted polysiloxane polymers, the origin of which can be traced back to 1952 and the very birth of the GC technique
- Polyethylene glycols, introduced ~1956
- Ionic liquids, a new GC stationary phase platform introduced in 2008



The most widely used GC phase platforms are based on polysiloxane polymers or polyethylene glycols. While some improvements have occurred, these phase platforms have remained virtually unchanged nearly as long as GC has existed. Drawbacks include:

- Active hydroxyl (-OH) groups at the polymer termini make these phases susceptible to a back-biting reaction if exposed to moisture and oxygen, leading to phase degradation and contributing to column bleed
- The limited ability to modify the phase limits the ability to alter selectivity
- A major limitation of PEG phases is their thermal limit of around 280 ºC



Ionic liquids consist of two or more organic cations, joined by a linkage, and associated with anions, which can be either inorganic or organic. Ionic liquids differ physically and chemically from other phases.

- They are much smaller compared to big, bulky polysiloxane polymer and polyethylene glycol phases, plus there are no active hydroxyl groups. These features lead to greater stability, even in the presence of moisture and/or oxygen.
- Many modifications are possible to make columns with unique selectivity.
  - The base structure can be dicationic or polycationic
  - There are numerous cation, linkage, and anion choices
  - Pendant groups can be added to cations and/or linkages

GC Column Polarity Scale

GC Column Polarity Scale

Description of our Procedure

- Each column is characterized with a series of five probes plus several n-alkane markers to determine the retention index for each probe
  - Benzene
  - Butanol
  - 2-Pentanone
  - Nitropropane
  - Pyridine
- McReynolds Constants are then calculated using the retention index data of the column relative to the retention index data for the same five probes on squalane, the most non-polar GC stationary phase
- The five McReynolds Constants are summed to obtain Polarity (P) values, which are then normalized to SLB-IL100 (set at P=100) to obtain Polarity Number (P.N.) values

Our procedure was proposed by Prof. Luigi Mondello (University of Messina, Italy).

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Our GC column polarity scale is a convenient tool to classify ionic liquid GC columns against each other, and also against non-ionic liquid GC columns. The procedure we follow was proposed to us by Prof. Luigi Mondello (University of Messina, Italy). Each column is characterized with a series of five probes plus several n-alkane markers to determine the retention index for each probe. McReynolds Constants are then calculated using the retention index data of the column relative to the retention index data for the same five probes on squalane, the most non-polar GC stationary phase. The five McReynolds Constants are summed to obtain Polarity (P) values, which are then normalized to SLB-IL100 (set at P=100) to obtain Polarity Number (P.N.) values.

Our GC column polarity scale can be used for column selection because it allows multiple columns to be compared easily, because all P.N. values are relative to both squalane (0 on the scale) and SLB-IL100 (100 on the scale).

GC Column Polarity Scale

Experimentally Determined Polarity Numbers

Column	McReynolds Constants					P	P.N.
	Benzene	n-Butanol	2-Pentanone	Nitropropane	Pyridine		
SPB-Octyl	17	-20	6	19	6	28	1
Equity-1	11	10	33	60	16	130	3
SLB-5ms	33	30	55	91	43	252	6
SPB-20	76	79	104	167	109	535	12
Equity-1701	82	131	150	233	136	732	16
SPB-35	175	113	151	225	175	839	19
SPB-50	154	134	176	266	218	948	21
SPB-225	233	342	342	501	375	1793	40
PAG	276	459	320	508	428	1991	45
SUPELCOWAX 10	334	509	375	601	505	2324	52
SLB-IL59	338	505	549	649	583	2624	59
SLB-IL60	362	492	525	679	564	2622	59
SLB-IL61	371	551	516	624	648	2710	61
SP-2330	469	663	608	859	712	3311	75
SLB-IL76	456	690	643	845	745	3379	76
SP-2331	495	674	622	856	735	3382	76
SP-2560	510	724	652	913	773	3572	81
SLB-IL82	532	676	701	921	808	3638	82
TCEP	622	871	772	1072	957	4294	97
SLB-IL100	602	853	884	1017	1081	4437	100
SLB-IL111	766	930	957	1192	1093	4938	111

P (Polarity) = sum of the first 5 McReynolds Constants.
P.N. (Polarity Number) = Polarity (P) normalized to SLB-IL100 (set at P=100).

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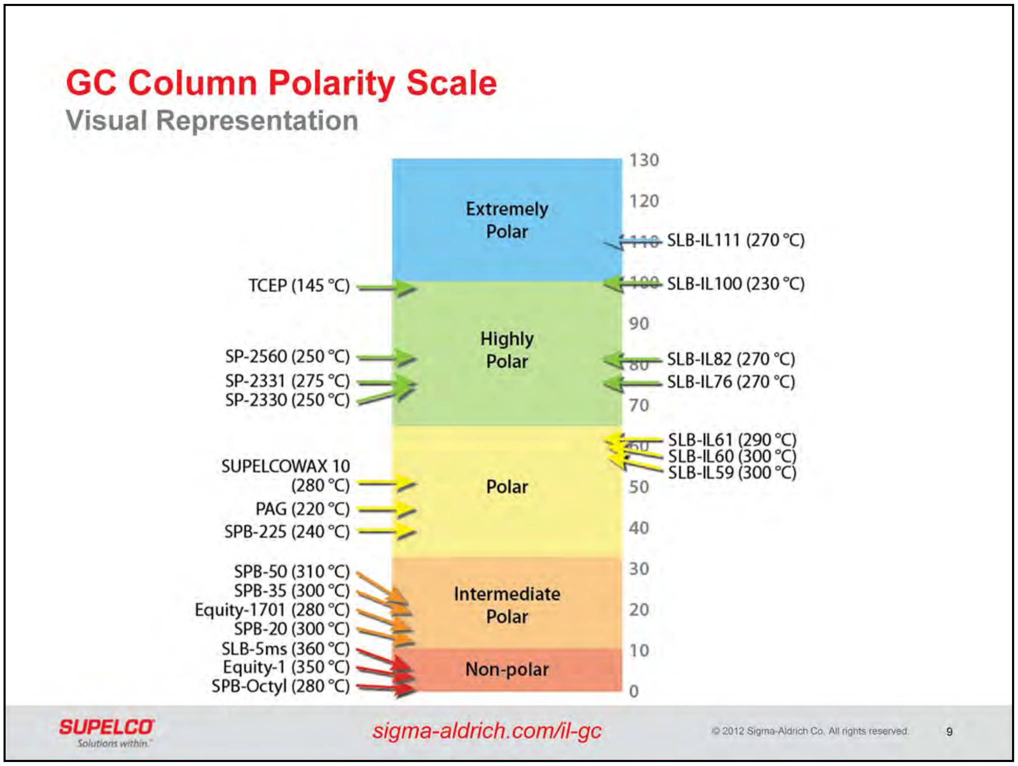
McReynolds Constants, Polarity (P) values, and Polarity Number (P.N.) values for several ionic liquid and non-ionic liquid columns are shown.

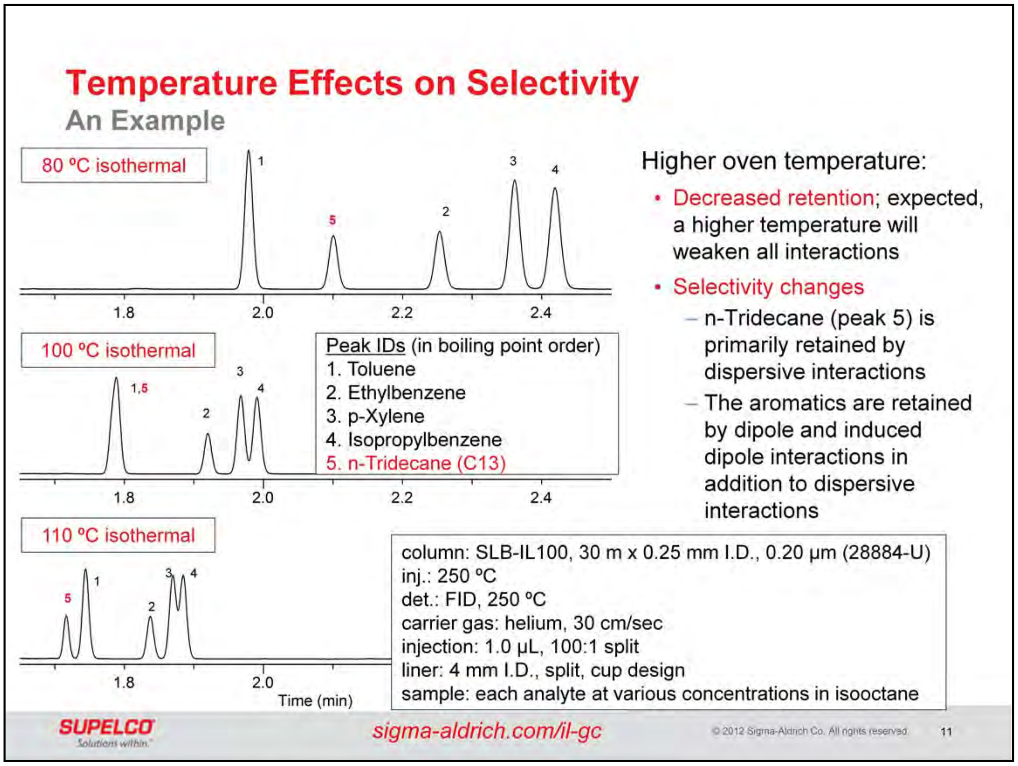


The GC Column Polarity Scale illustrates the relative polarities of various column phases to one another. To the left of the scale are the positions and maximum temperatures of several non-ionic liquid columns. To the right of the scale are the positions and maximum temperatures of current Supelco ionic liquid columns. We expect ionic liquid columns will be introduced into the intermediate polar region. The scale is divided into five regions:

- The first four regions are generally accepted and used by several GC column manufacturers
- The fifth region (extremely polar) was required with the introduction of the ionic liquid column SLB-IL111 in 2010, because no column existed within this polarity region before

Note that there are several gaps to the left of the scale. These are attributed to the limited modifications possible with polysiloxane polymer and polyethylene glycol phases.

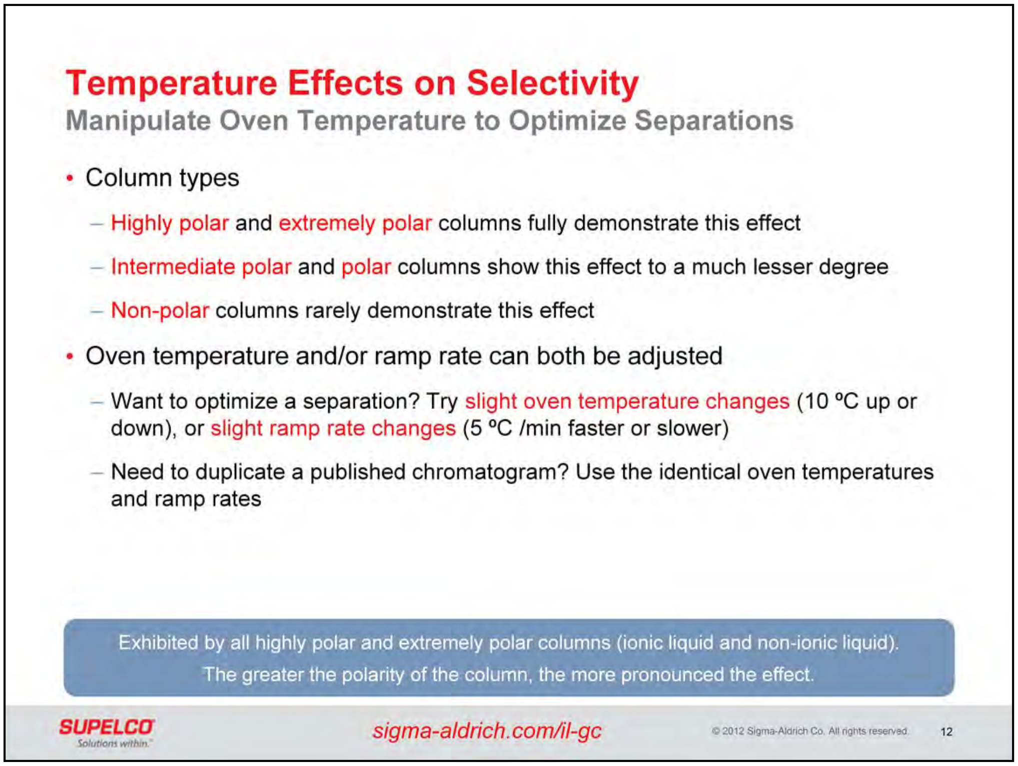
Temperature Effects on Selectivity



A visual example of this temperature effect phenomenon is shown using a simple analyte list comprised of one alkane (n-tridecane) and four aromatics (toluene, ethylbenzene, p-xylene, and isopropylbenzene) on the SLB-IL100. Three runs are shown, each using a different isothermal oven temperature. All other conditions remain constant.

A higher oven temperature results in the decreased retention of all analytes. This is expected because a higher oven temperature will weaken all stationary phase-analyte interactions.

But what about selectivity? A higher oven temperature results in a selectivity change. Why? Let's first identify the stationary phase-analyte interactions at work here. Alkanes (such as n-tridecane, peak 5) are primarily retained by dispersive interactions, whereas aromatics are retained by dipole and induced dipole interactions in addition to dispersive interactions. As mentioned above, a higher temperature will weaken all stationary phase-analyte interactions. However, all interactions do not weaken at the same rate. The alkane loses retention quicker than the aromatics because the dispersive interactions used for its retention weaken quicker with a higher oven temperature than the dipole and induced dipole interactions used for part of the retention of the aromatics.



Can we manipulate oven temperature to optimize a separation? We can try.

This temperature effect is exhibited by all columns (ionic liquid and non-ionic liquid). The greater the polarity of the column, the more pronounced the effect. Highly polar and extremely polar columns fully demonstrate this effect. Intermediate polar and polar columns show this effect to a much lesser degree. Non-polar columns rarely demonstrate this effect.

Oven temperature and/or ramp rate can both be adjusted. Want to optimize a separation? Try slight oven temperature changes (10 ºC up or down), or slight ramp rate changes (5 ºC/min faster or slower). Need to duplicate a published chromatogram? Use the identical oven temperatures and ramp rates.

Column Selection

Column Selection

- Ionic liquid columns are relatively new (2008) compared to non-ionic liquid columns (1950's)
- Not all suitable applications are yet discovered
- The following charts serve as a starting point

Testing Type	Application	SLB-IL59	SLB-IL60	SLB-IL61	SLB-IL76	SLB-IL82	SLB-IL100	SLB-IL111
Environmental	PCBs by GC-HRMS	X				X		X
	PAHs by GC or GC-MS	X	X					
Petroleum	Aromatics in Fuel							X
	Sulfur Compounds in Fuel	X	X					
Biofuel	Biodiesel: FAME Profile		X					X

Chromatograms for some applications can be found in the "Supelco Ionic Liquid GC Columns – Applications" presentation.

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Ionic liquid GC columns are relatively new. They were introduced in 2008. Compare this to the 1950's when polysiloxane polymers and polyethylene glycols were introduced. Not all suitable applications that ionic liquid columns can perform are yet discovered. The following charts serve as a starting point.

Chromatograms for some applications can be found in the "Supelco Ionic Liquid GC Columns – Applications" presentation.

Column Selection

Testing Type	Application	SLB-IL59	SLB-IL60	SLB-IL61	SLB-IL76	SLB-IL82	SLB-IL100	SLB-IL111
Chemical	Solvents		X					
	Aromatics	X	X					X
	Impurities in Toluene						X	
	Xylene Isomers	X	X					
	Chlorinated Solvents		X					
	Alcohols		X					
	Glycol Ethers (Cellosolves), Diols, Glycols		X					
	Ketones		X					
	Carboxylic Acids as Methyl Esters							X
	Dicarboxylic Acids as Dimethyl Esters							X
	Tall Oil Fatty Acids as Methyl Esters							X
	Esters and Ethers		X					
	Terpenes	X	X					X
	Amines	X	X					
	Aromatic Amines (Anilines)	X	X					
	Sulfur Compounds	X	X					

Chromatograms for some applications can be found in the "Supelco Ionic Liquid GC Columns – Applications" presentation.

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Chromatograms for some applications can be found in the "Supelco Ionic Liquid GC Columns – Applications" presentation.

Column Selection

Testing Type	Application	SLB-IL59	SLB-IL60	SLB-IL61	SLB-IL76	SLB-IL82	SLB-IL100	SLB-IL111
Agriculture	Edible Oils	X	X					X
	FAMEs by Degree of Unsaturation	X	X					
	Omega 3 and Omega 6 FAMEs	X	X					
	cis/trans FAME Isomers							X
	Flavors & Fragrances, Aroma		X					
Food and Beverage	FAMEs by Degree of Unsaturation	X	X					
	Omega 3 and Omega 6 FAMEs	X	X					
	cis/trans FAME Isomers							X
	Fatty Acid Ethyl Esters (FAEEs)	X	X					X
	Organic Acids		X					
	Flavors & Fragrances, Aroma		X					
	Benzene	X	X					X
	Nitrosamines	X	X					
	PAHs	X	X					
	Beverage Analysis		X					
	Sulfur Compounds in Beverages	X	X					

Chromatograms for some applications can be found in the
"Supelco Ionic Liquid GC Columns – Applications" presentation.

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Chromatograms for some applications can be found in the "Supelco Ionic Liquid GC Columns – Applications" presentation.

Column Selection

Testing Type	Application	SLB-IL59	SLB-IL60	SLB-IL61	SLB-IL76	SLB-IL82	SLB-IL100	SLB-IL111
Flavor and Fragrance	Volatiles		X					
	Essential Oils		X					
Cosmetic and Personal Care/Cleaning Product	Fragrance Compounds		X					
	Glycols		X					
Clinical	Antihistamines	X	X					
	Bacterial Acid Methyl Esters (BAMEs)	X	X					X
	Basic Drug Screen	X	X					
	Benzodiazepines as TBDMS Derivatives	X	X					
	Carboxylic Acids as Methyl Esters							X
	Cold and Sinus Medications	X	X					
	FAMEs by Degree of Unsaturation	X	X					
	Omega 3 and Omega 6 FAMEs	X	X					
	cis/trans FAME Isomers							X
	Sympathomimetic Amines	X						
Forensic	Basic Drug Screen	X						
GCxGC	Polar Secondary (2°) Column	X		X	X	X		X
	Polar Primary (1°) Column	X	X	X	X	X		X

Chromatograms for some applications can be found in the
"Supelco Ionic Liquid GC Columns – Applications" presentation.

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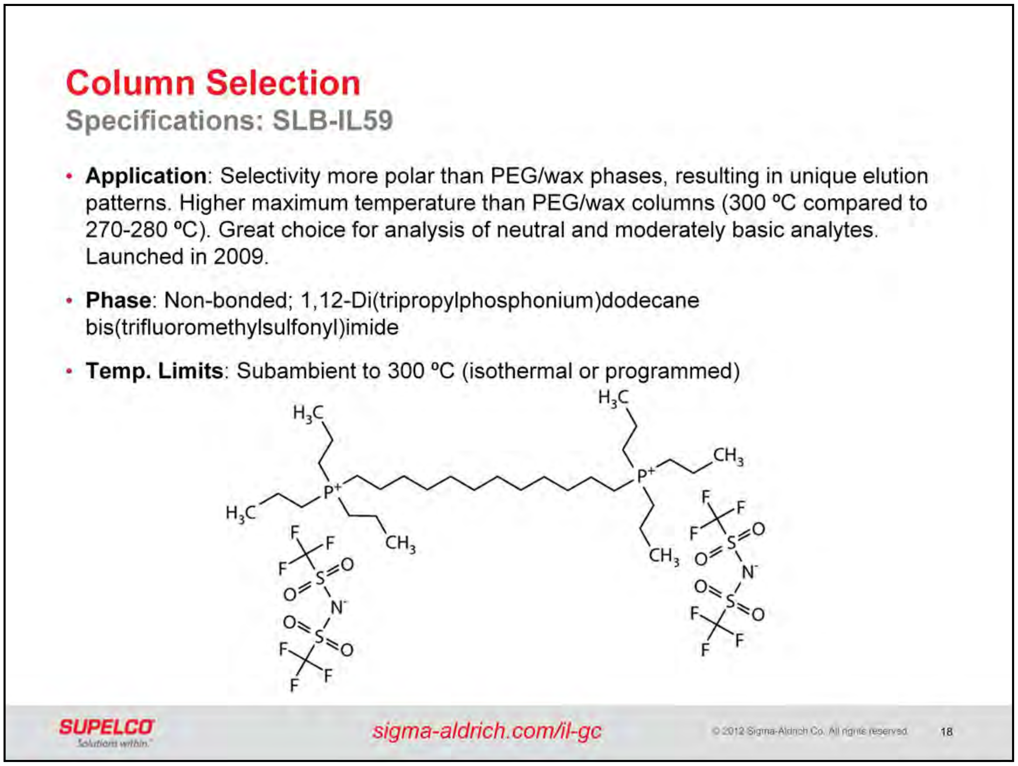
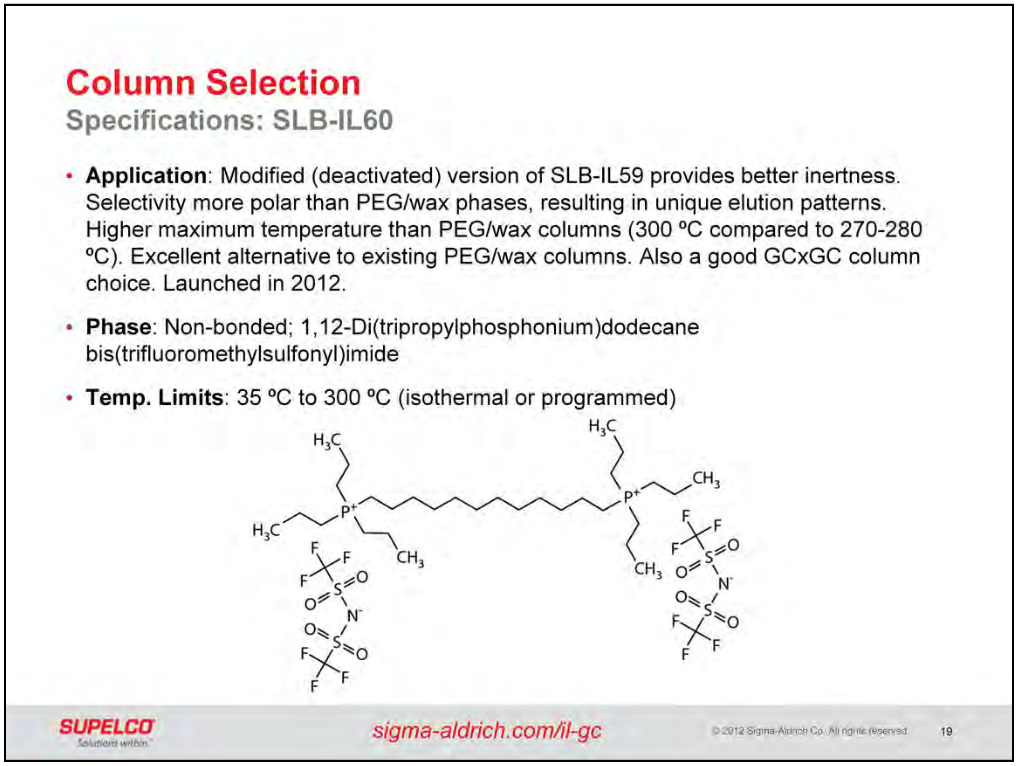
Chromatograms for some applications can be found in the "Supelco Ionic Liquid GC Columns – Applications" presentation.



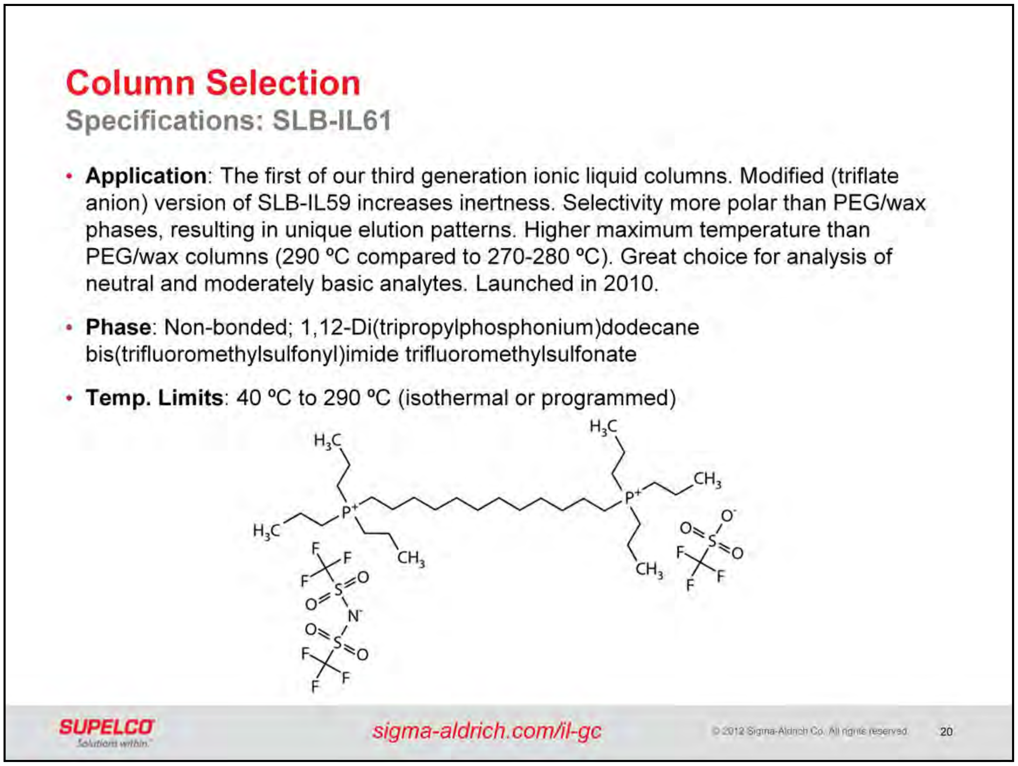
SLB-IL59

- **Application**: Selectivity more polar than PEG/wax phases, resulting in unique elution patterns. Higher maximum temperature than PEG/wax columns (300 ºC compared to 270-280 ºC). Great choice for analysis of neutral and moderately basic analytes. Launched in 2009.
- **Phase**: Non-bonded; 1,12-Di(tripropylphosphonium)dodecane bis(trifluoromethylsulfonyl)imide
- **Temp. Limits**: Subambient to 300 ºC (isothermal or programmed)



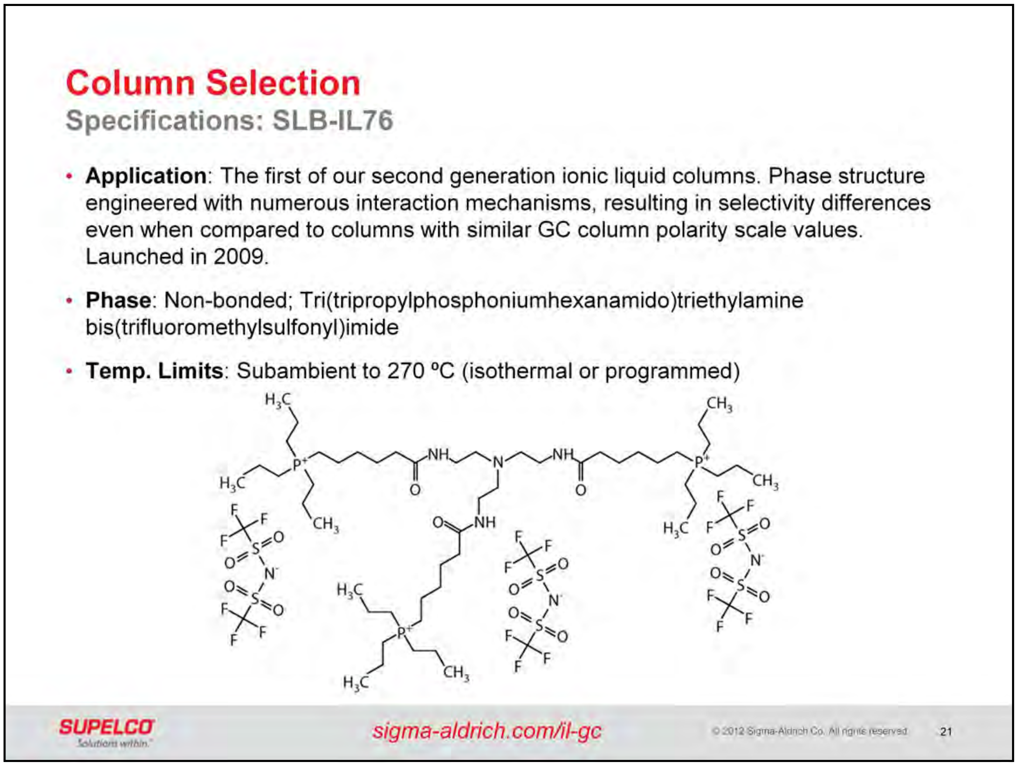
SLB-IL60

- **Application**: Modified (deactivated) version of SLB-IL59 provides better inertness. Selectivity more polar than PEG/wax phases, resulting in unique elution patterns. Higher maximum temperature than PEG/wax columns (300 ºC compared to 270-280 ºC). Excellent alternative to existing PEG/wax columns. Also a good GCxGC column choice. Launched in 2012.
- **Phase**: Non-bonded; 1,12-Di(tripropylphosphonium)dodecane bis(trifluoromethylsulfonyl)imide
- **Temp. Limits**: 35 ºC to 300 ºC (isothermal or programmed)



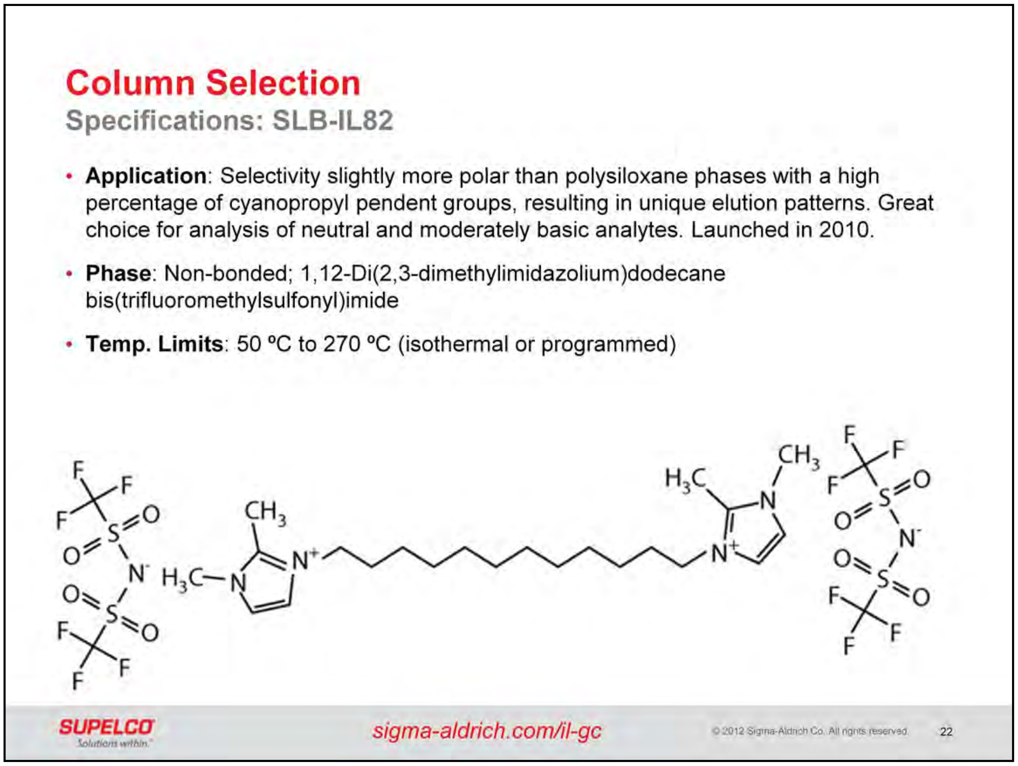
SLB-IL61

- **Application**: The first of our third generation ionic liquid columns. Modified (triflate anion) version of SLB-IL59 increases inertness. Selectivity more polar than PEG/wax phases, resulting in unique elution patterns. Higher maximum temperature than PEG/wax columns (290 ºC compared to 270-280 ºC). Great choice for analysis of neutral and moderately basic analytes. Launched in 2010.
- **Phase**: Non-bonded; 1,12-Di(tripropylphosphonium)dodecane bis(trifluoromethylsulfonyl)imide trifluoromethylsulfonate
- **Temp. Limits**: 40 ºC to 290 ºC (isothermal or programmed)



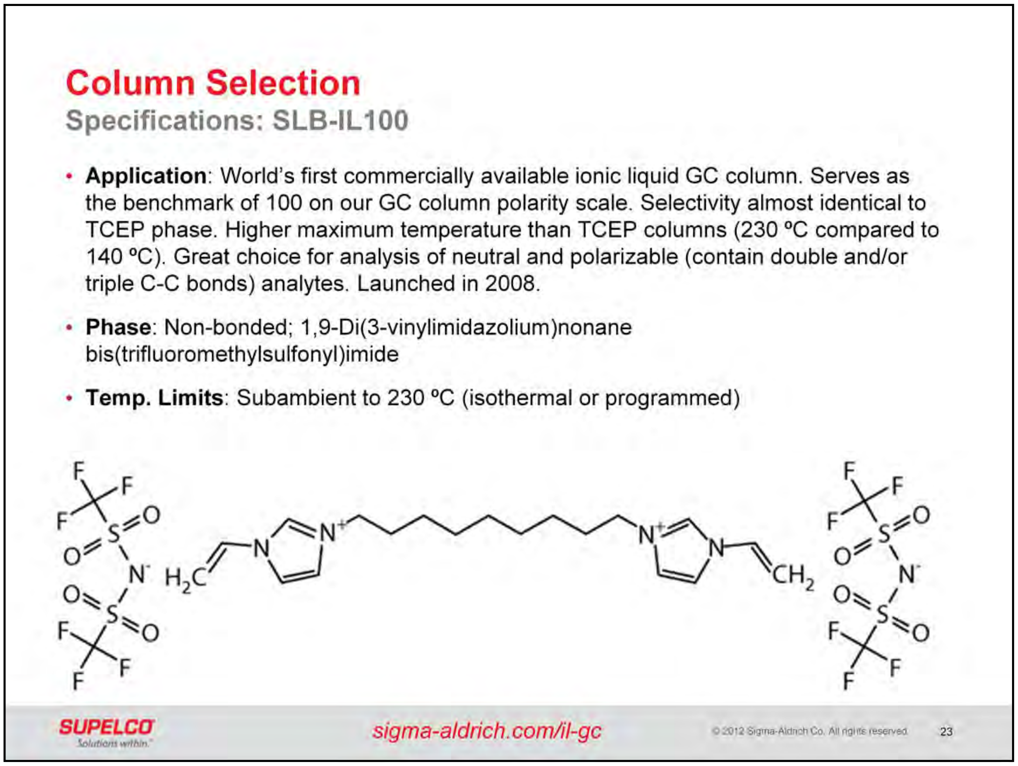
SLB-IL76

- **Application**: The first of our second generation ionic liquid columns. Phase structure engineered with numerous interaction mechanisms, resulting in selectivity differences even when compared to columns with similar GC column polarity scale values. Launched in 2009.
- **Phase**: Non-bonded; Tri(tripropylphosphoniumhexanamido)triethylamine bis(trifluoromethylsulfonyl)imide
- **Temp. Limits**: Subambient to 270 ºC (isothermal or programmed)



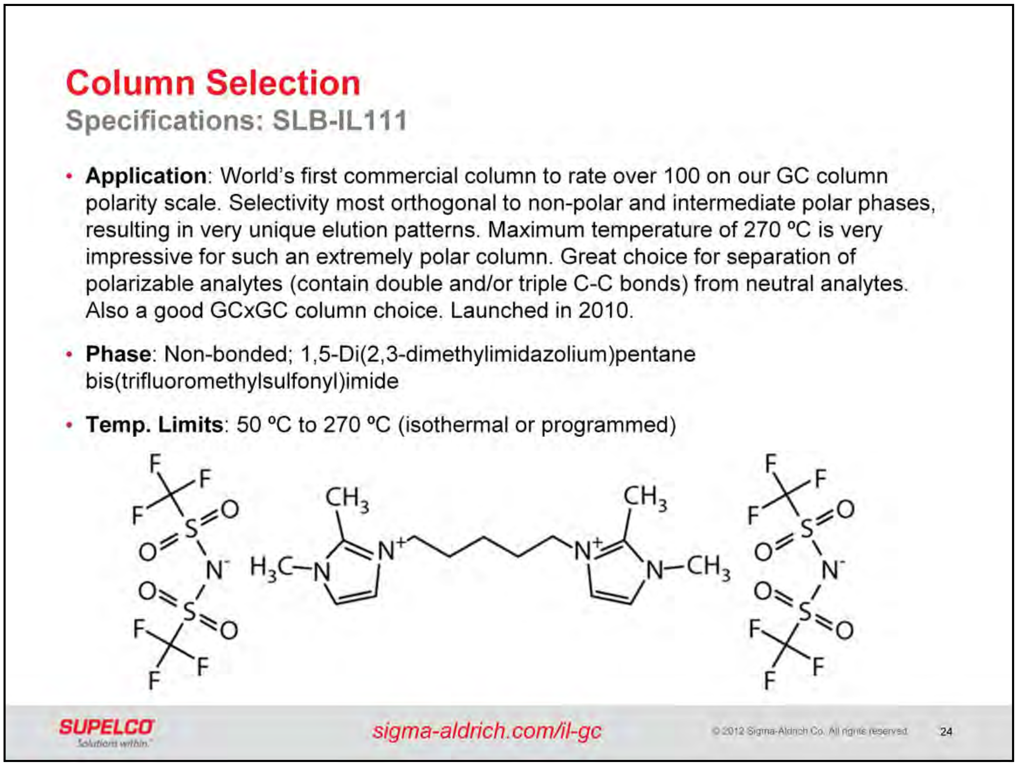
SLB-IL82

- **Application**: Selectivity slightly more polar than polysiloxane phases with a high percentage of cyanopropyl pendent groups, resulting in unique elution patterns. Great choice for analysis of neutral and moderately basic analytes. Launched in 2010.
- **Phase**: Non-bonded; 1,12-Di(2,3-dimethylimidazolium)dodecane bis(trifluoromethylsulfonyl)imide
- **Temp. Limits**: 50 ºC to 270 ºC (isothermal or programmed)



SLB-IL100

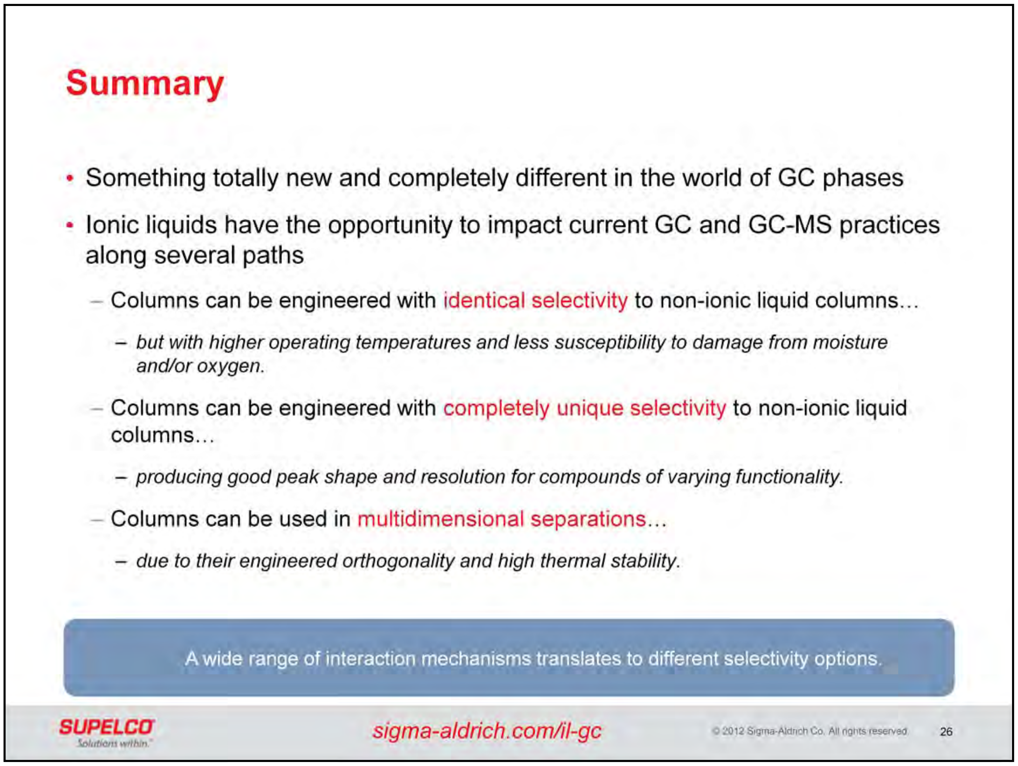
- **Application**: World's first commercially available ionic liquid GC column. Serves as the benchmark of 100 on our GC column polarity scale. Selectivity almost identical to TCEP phase. Higher maximum temperature than TCEP columns (230 ºC compared to 140 ºC). Great choice for analysis of neutral and polarizable (contain double and/or triple C-C bonds) analytes. Launched in 2008.
- **Phase**: Non-bonded; 1,9-Di(3-vinylimidazolium)nonane bis(trifluoromethylsulfonyl)imide
- **Temp. Limits**: Subambient to 230 ºC (isothermal or programmed)



SLB-IL111

- **Application**: World's first commercial column to rate over 100 on our GC column polarity scale. Selectivity most orthogonal to non-polar and intermediate polar phases, resulting in very unique elution patterns. Maximum temperature of 270 ºC is very impressive for such an extremely polar column. Great choice for separation of polarizable analytes (contain double and/or triple C-C bonds) from neutral analytes. Also a good GCxGC column choice. Launched in 2010.
- **Phase**: Non-bonded; 1,5-Di(2,3-dimethylimidazolium)pentane bis(trifluoromethylsulfonyl)imide
- **Temp. Limits**: 50 ºC to 270 ºC (isothermal or programmed)

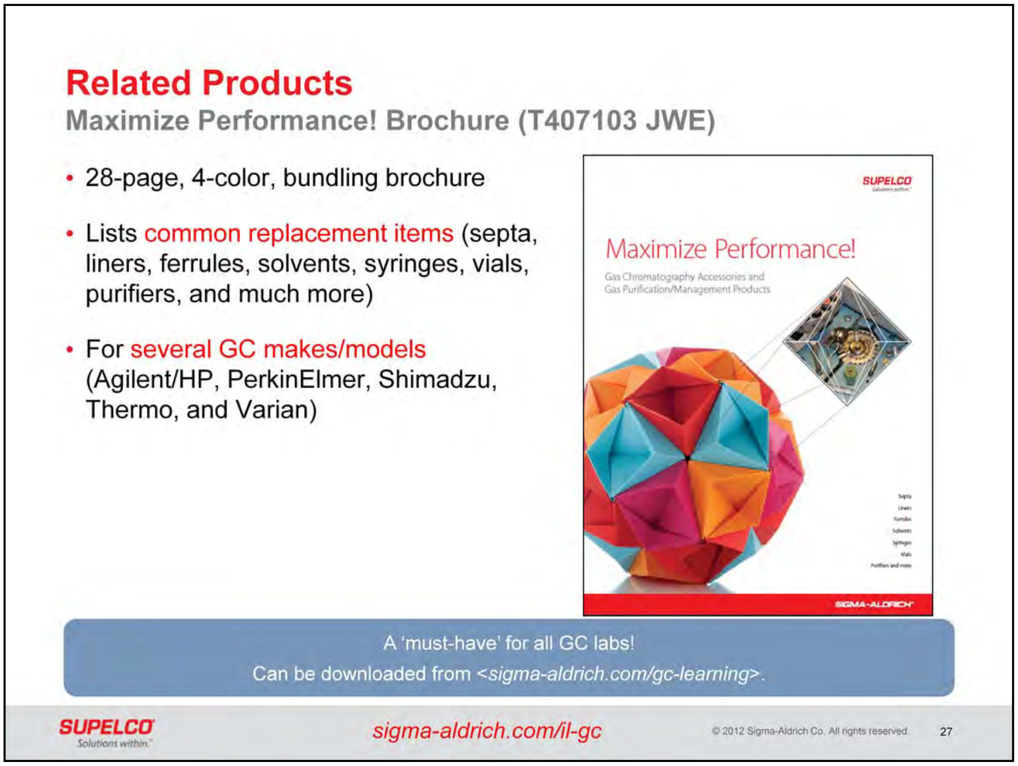
Summary / Related Products / Resources



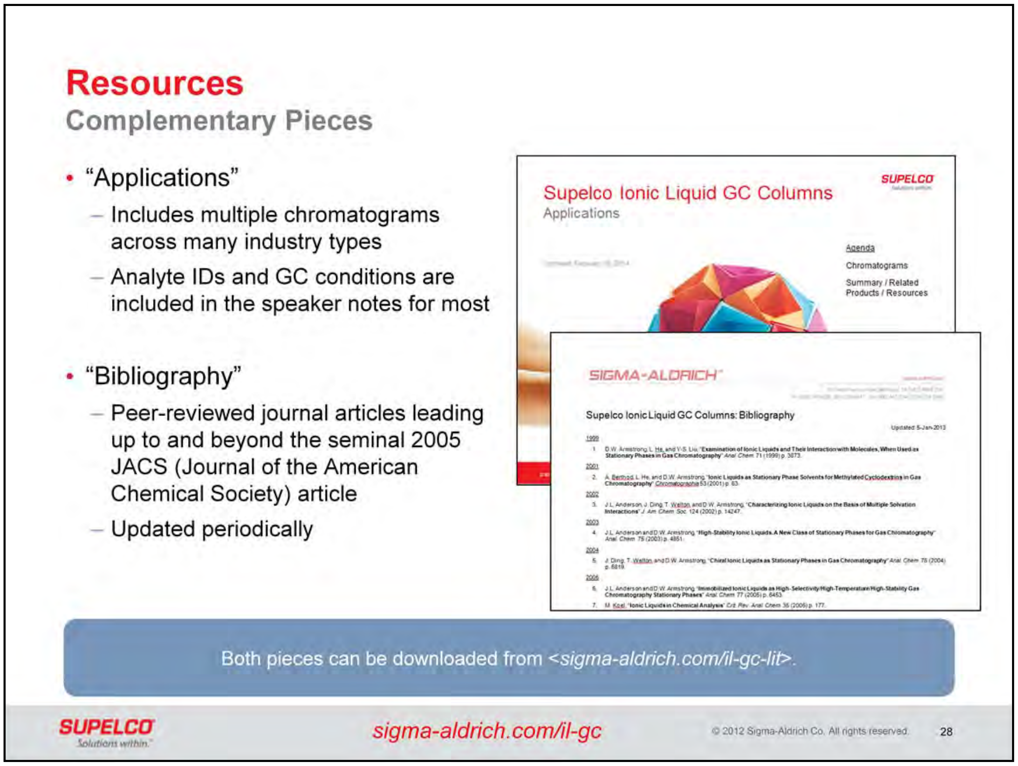
Ionic liquids are something totally new and completely different in the world of GC phases. They have the opportunity to impact current GC and GC-MS practices along several paths:

- Columns can be engineered with identical selectivity to non-ionic liquid columns… but with higher operating temperatures and less susceptibility to damage from moisture and/or oxygen
- Columns can be engineered with completely unique selectivity to non-ionic liquid columns… producing good peak shape and resolution for compounds of varying functionality
- Columns can be used in multidimensional separations… due to their engineered orthogonality and high thermal stability

A wide range of interaction mechanisms translates to different selectivity options.



There are multiple related products. Most of them are touched on in the 28-page, 4-color Maximize Performance! brochure. This piece lists all the common replacement items, including septa, liners, ferrules, solvents, syringes, vials, purifiers, and much more for several GC makes/models, including Agilent/HP, PerkinElmer, Shimadzu, Thermo, and Varian. This 'must-have' brochure can be downloaded from <sigma-aldrich.com/gc-learning>.



There are two complementary pieces. The first is the "Applications" presentation. It includes multiple chromatograms across many industry types. Analyte IDs and GC conditions are included in the speaker notes for most.

The second is a "Bibliography" of peer-reviewed journal articles leading up to and beyond the seminal 2005 JACS (Journal of the American Chemical Society) article. It is updated periodically.

Both pieces can be downloaded from <sigma-aldrich.com/il-gc-lit>.

Resources

People, Top-Level Literature, and Web Pages

- Contacts
  - Marketing (GC Columns): Mike Buchanan <*mike.buchanan@sial.com*>
  - Marketing (GC Accessories/Gas Purification): Jaime Martain <*jaime.martain@sial.com*>
  - Technical: Len Sidisky <*len.sidisky@sial.com*>
- Brochures
  - Ionic Liquid GC Columns brochure (planned)
  - Maximize Performance! brochure (T407103 JWE)
- Web pages (*sigma-aldrich.com/il-gc*)
  - Product information
  - Technical literature and application notes
  - Bibliography
  - Request an evaluation

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Contacts are as follows:

- Marketing for GC Columns is Mike Buchanan
- Marketing for GC Accessories and Gas Purification is Jaime Martain
- Technical is Len Sidisky

An “Ionic Liquid GC Columns” brochure is planned. Use the “Maximize Performance!” brochure for related products.

Visit our ionic liquid landing page to find:

- Detailed product information
- In-depth technical literature and applications notes
- A bibliography of journal articles featuring ionic liquid columns
- A form to request an evaluation column

Thank You