Synthesis and Use of a New Covalently Bonded C18 Modified Carbon Clad Microporous Zirconia for Fast High Temperature Separations

by

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HPLC Columns

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OUTLINE

- Analytical Advantages of Column Stability
- Development of a New Type of RP Column: DiamondBond-C18
- Selectivity Comparison of DiamondBond-C18 with Luna C18 (2) Silica and Other Columns
- Applications
- Conclusions

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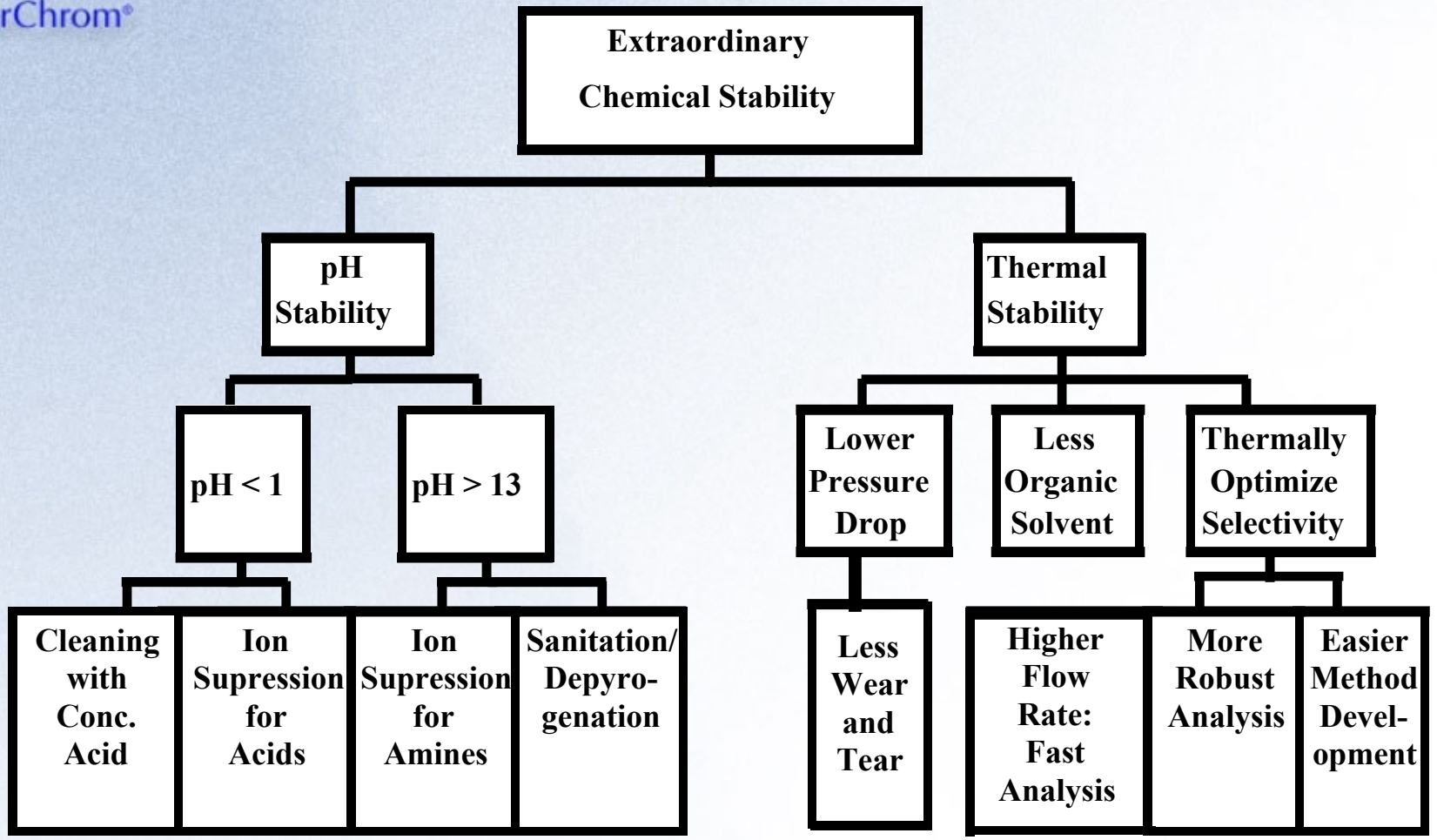
Analytical Advantages of Column Stability



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Patented Synthesis of Carbon Clad Zirconia

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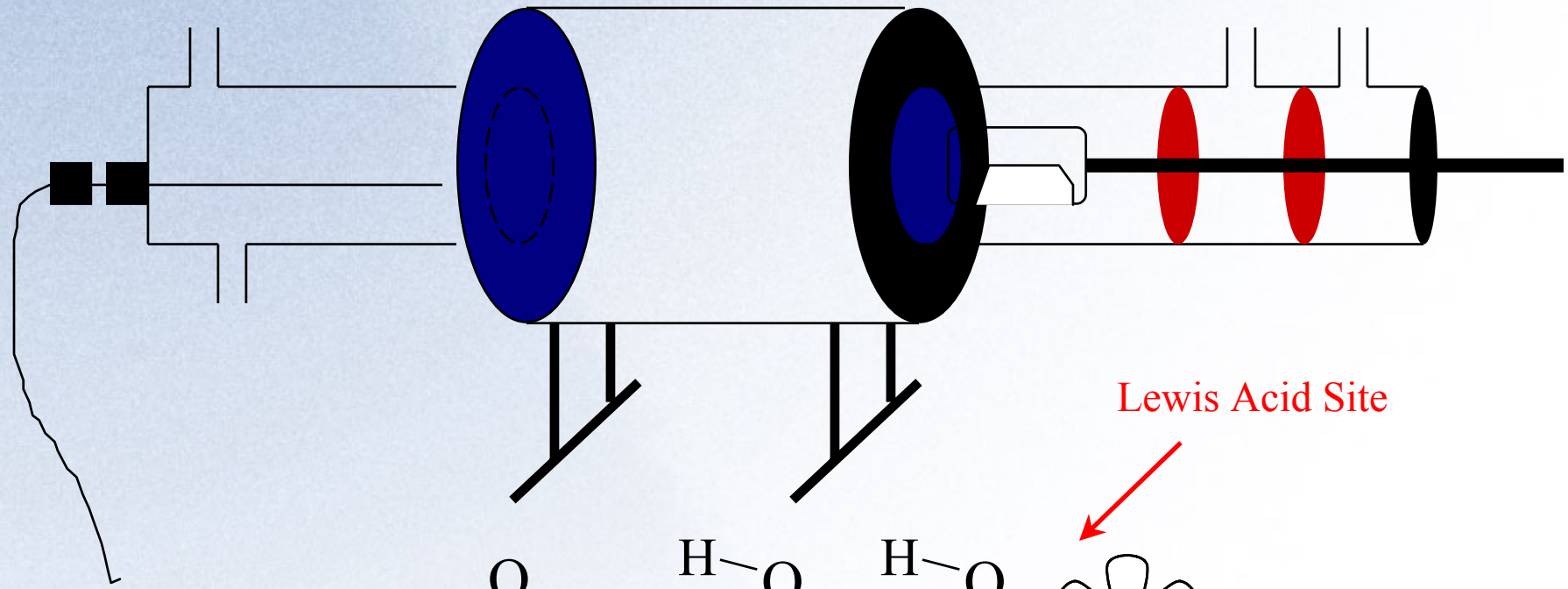
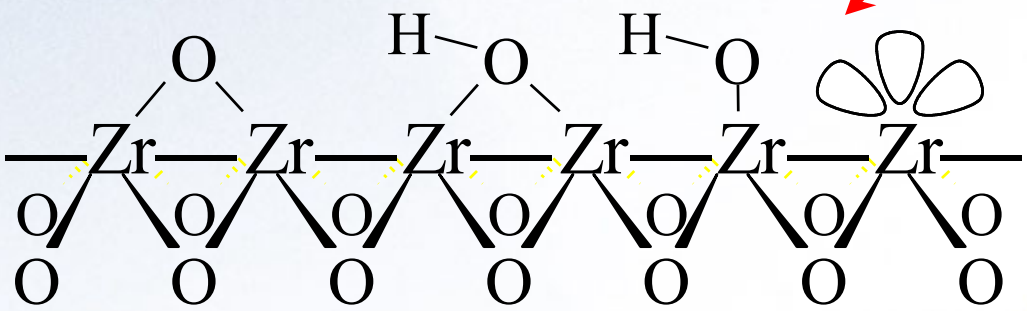


Lewis Acid Site



Bulk Zirconia

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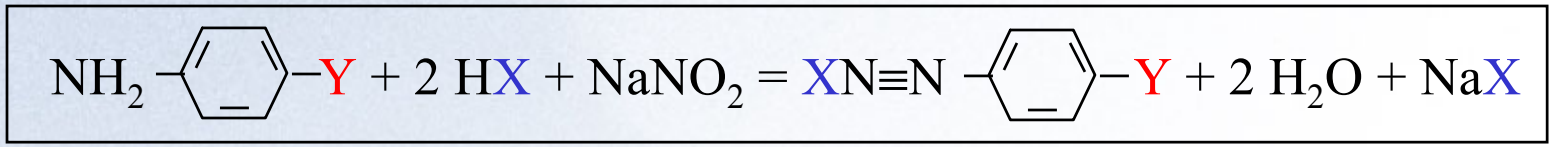
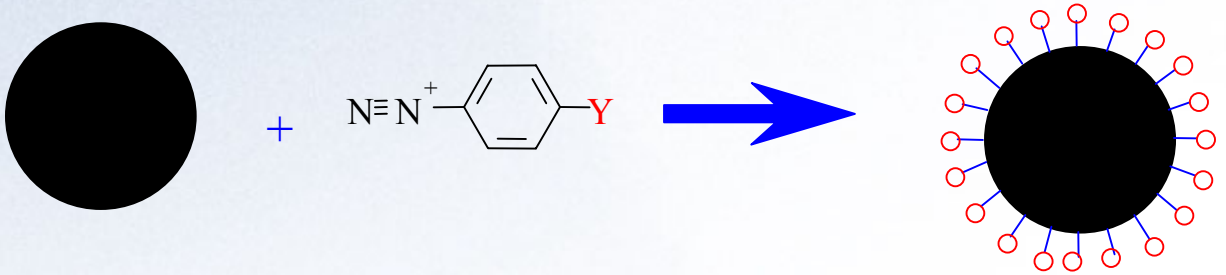
A New Class of Stationary Phase Media

- General approach - Cabot Corporation (Billerica, MA):
  - functionalizing agent X-R-Y
  - X reacts with surface
  - Y = functional group
- X is typically a diazonium salt





Carbon Clad Zirconia Diazonium Salt Modified Carbon Clad Zirconia

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Chemical Structure of DiamondBond-C18

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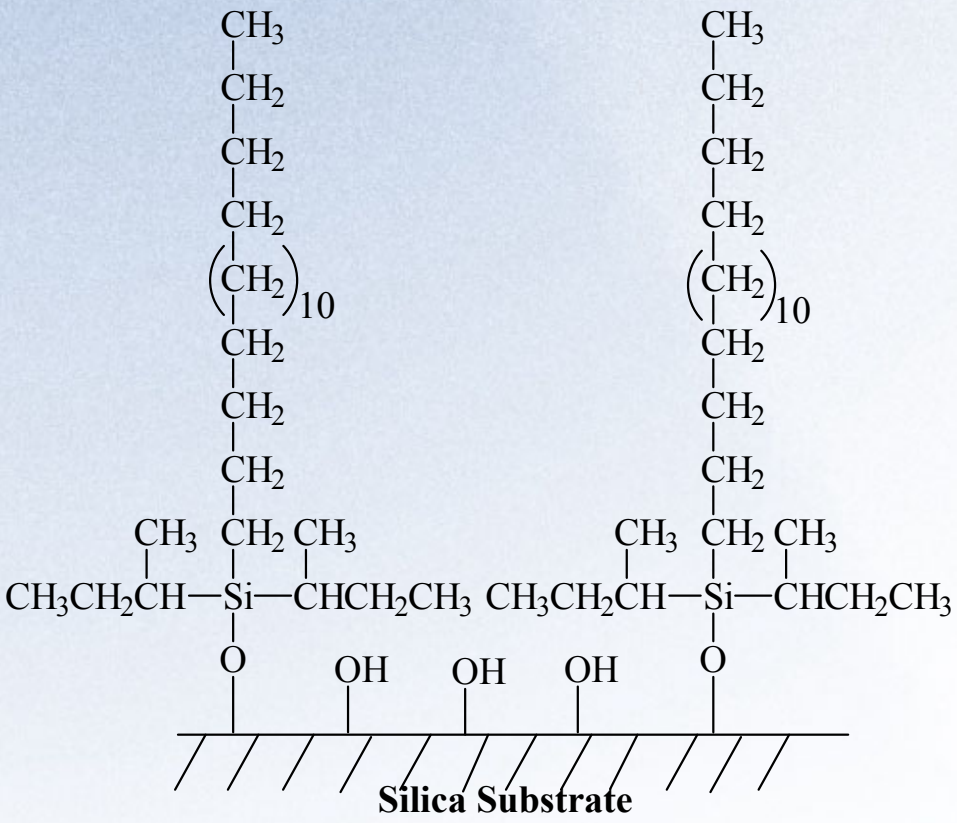
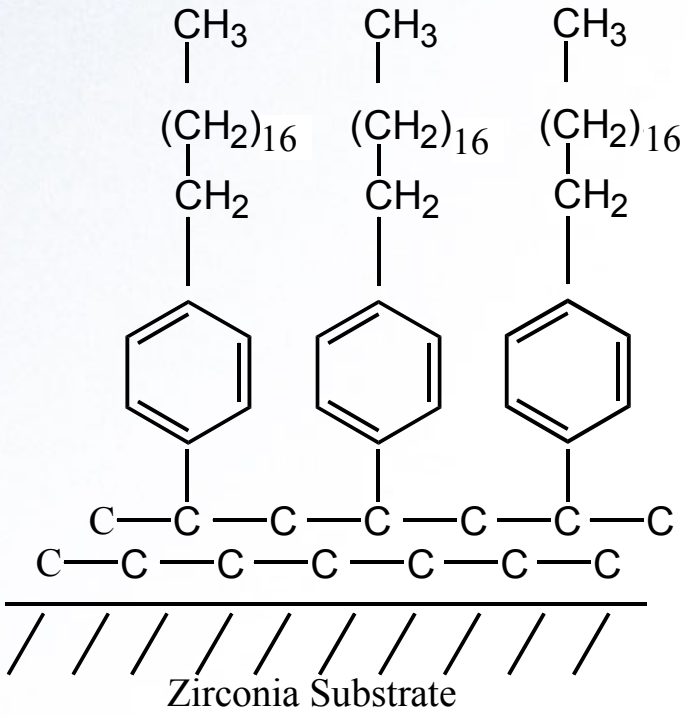
ODS



DiamondBond-C18



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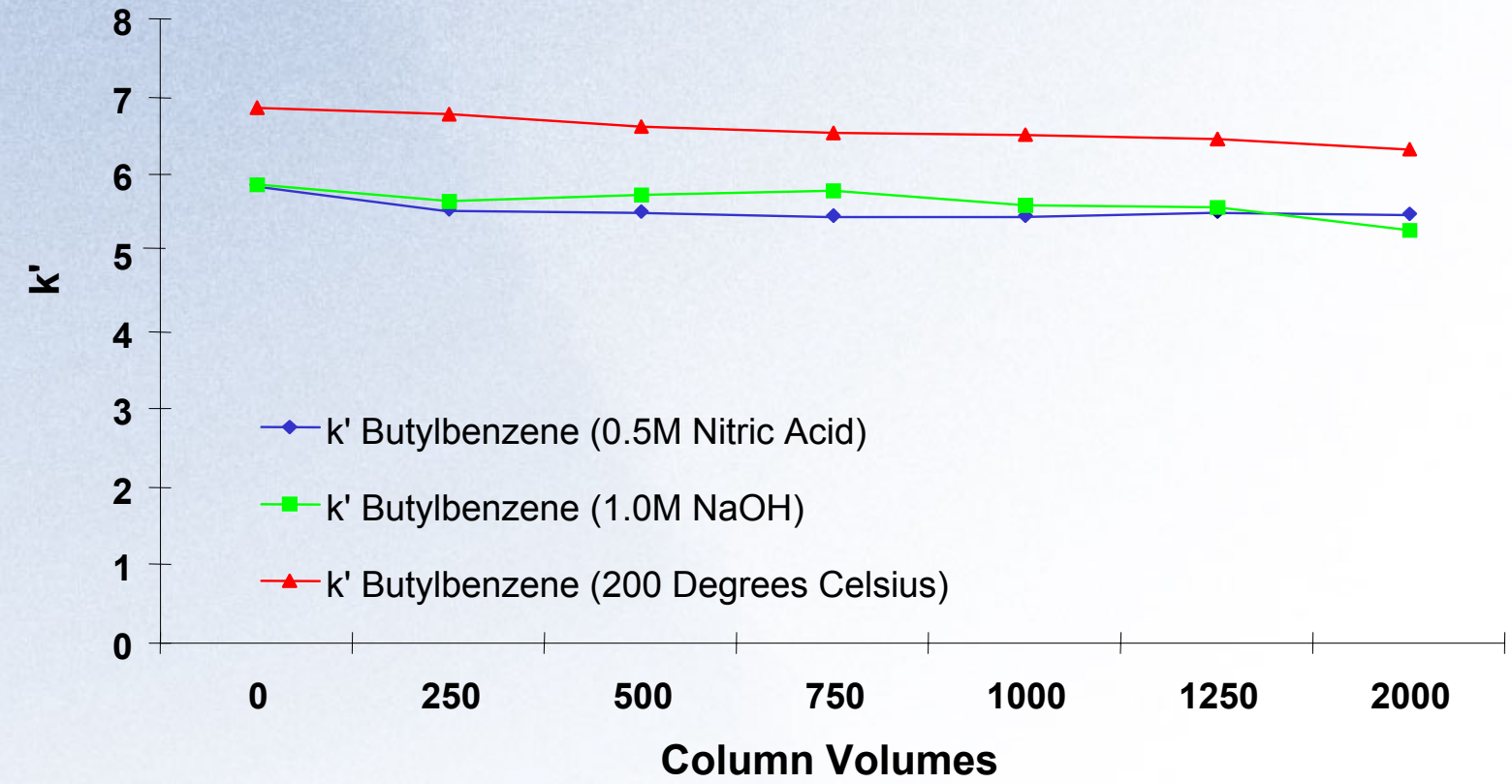
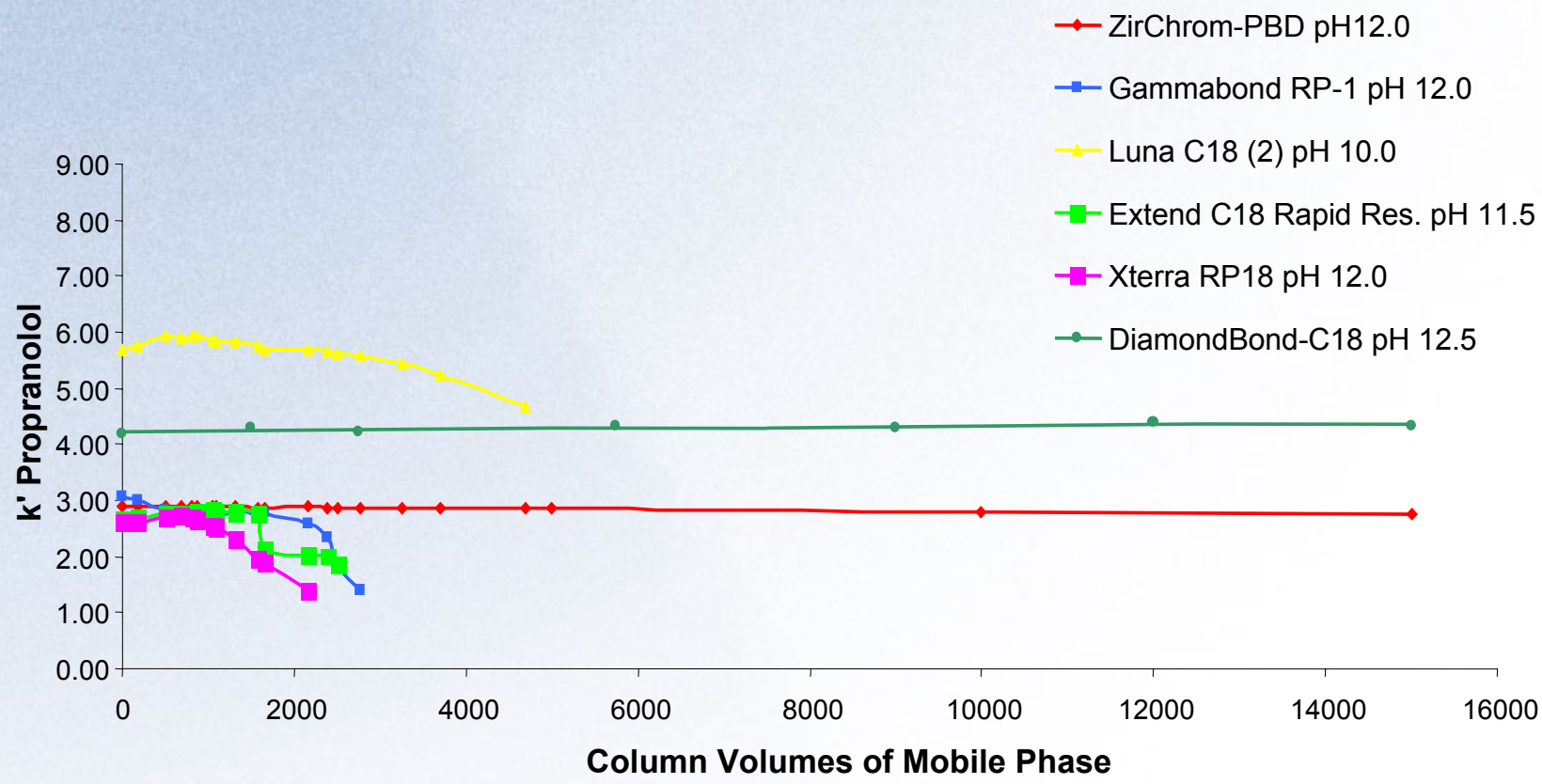
Stability Testing of DiamondBond



LC Conditions: Base Stability—DiamondBond™ Phase A, 30 x 4.6 mm id; Mobile phase, 50/50 ACN/Water; Flow rate, 1.0 ml/min.; Temperature, 30 °C; Injection volume, 5ul; Detection at 254nm. Acid Stability—DiamondBond™ Phase A, 50 x 4.6 mm id; Mobile phase, 50/50 ACN/Water; Flow rate, 1.0 ml/min.; Temperature, 30 °C; Injection volume, 5ul; Detection at 254nm. Temperature Stability—DiamondBond™ Phase B, 50 x 4.6 mm id; Mobile phase, 50/50 ACN/Water; Flow rate, 1.0 ml/min.; Temperature, 30 °C; Injection volume, 5ul; Detection at 254nm.

Phase Stability Comparison at High pH*



* Column names are the trademarks of their respective manufacturers.

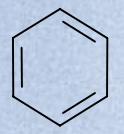
Chromatographic Selectivity Comparison of DiamondBond-C18 to Other Stable RP Phases

- **LC Conditions**: Mobile phase, 40/60 Acetonitrile/50mM phosphate at pH 3.2, Flow rate, 1.0 ml/min., Temperature = 30 °C, Detection at 254nm, 5µl Injection volume.
  (LC-GC, Vol 13, No. 9, September 1995, 720-726.)
- **Columns Tested***: DiamondBond-C18, Luna C18 (2), ZirChrom-PBD, Gammabond RP-1, Xterra RP18, Polymer Labs PLRP-S

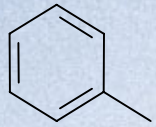
* Column names are the trademarks of their respective manufacturers.

DIAMONDBOND™ HPLC Columns

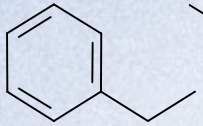
22 Non-electrolyte Solutes



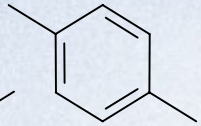
Benzene



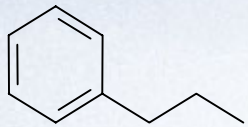
Toluene



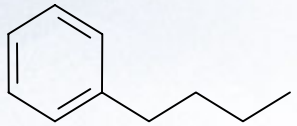
Ethylbenzene



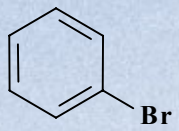
p-xylene



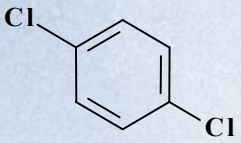
Propylbenzene



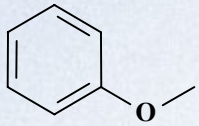
Butylbenzene



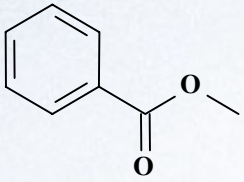
Bromobenzene



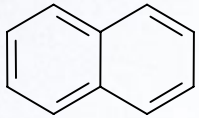
p-Dichlorobenzene



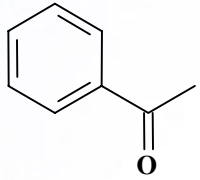
Anisole



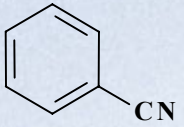
Methylbenzoate



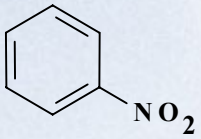
Napthalene



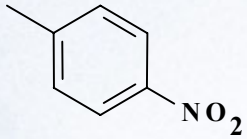
Acetophenone



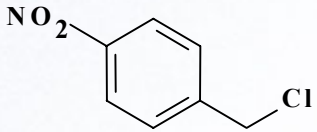
Benzonitrile



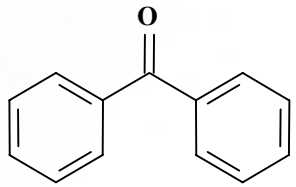
Nitrobenzene



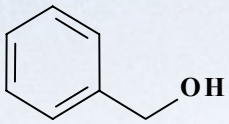
p-Nitrotoluene



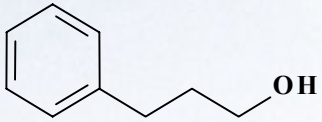
p-Nitrobenzyl Chloride



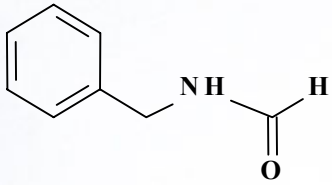
Benzophenone



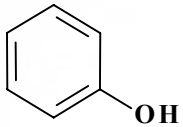
Benzylalcohol



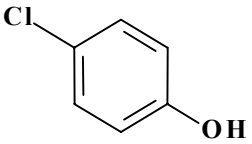
3-Phenyl Propanol



N-Benzyl Formamide

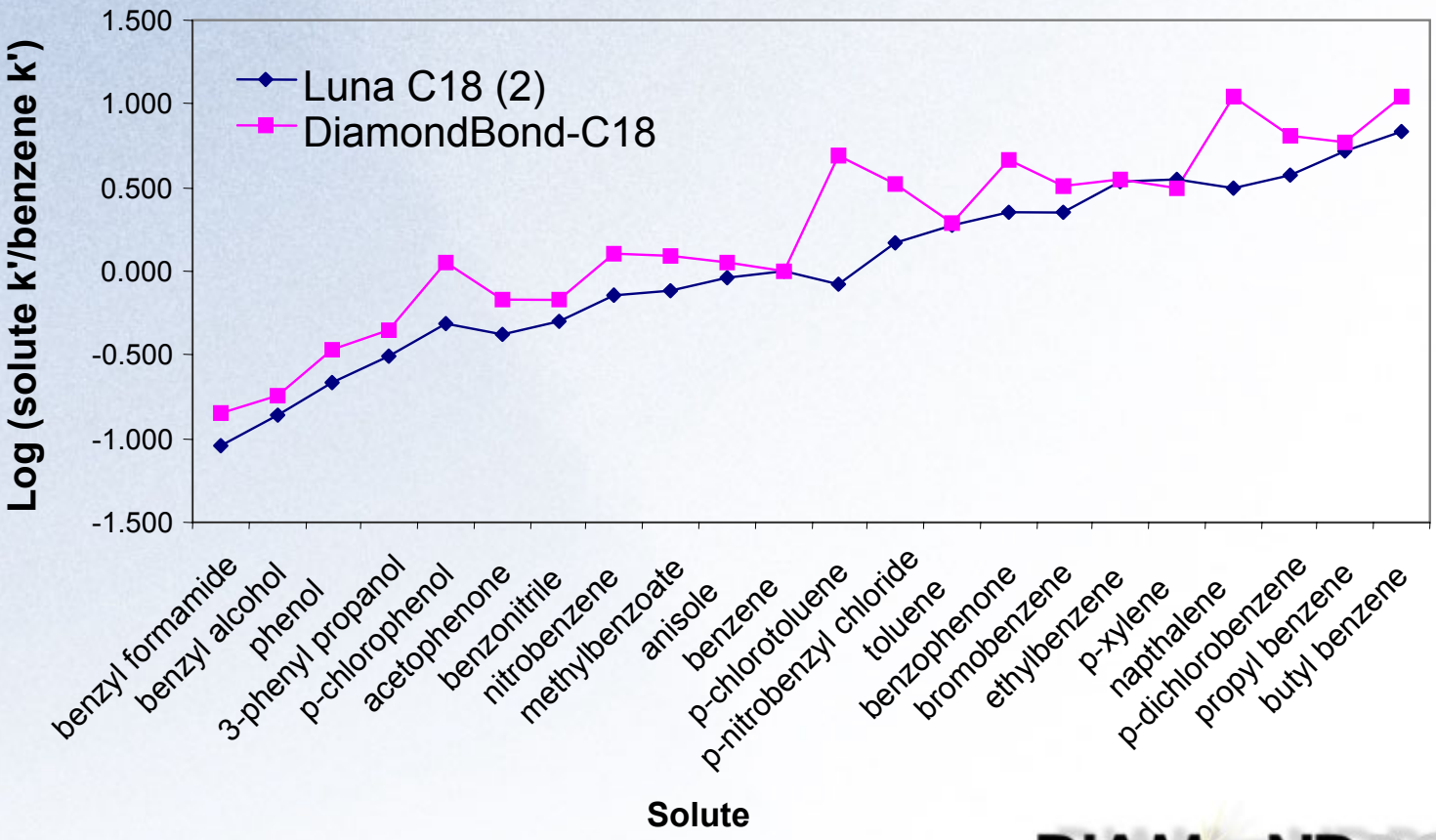


Phenol



p-Chlorophenol

Normalized Selectivity Comparison*: DiamondBond-C18 and Luna C18 (2)



* Column names are the trademarks of their respective manufacturers.

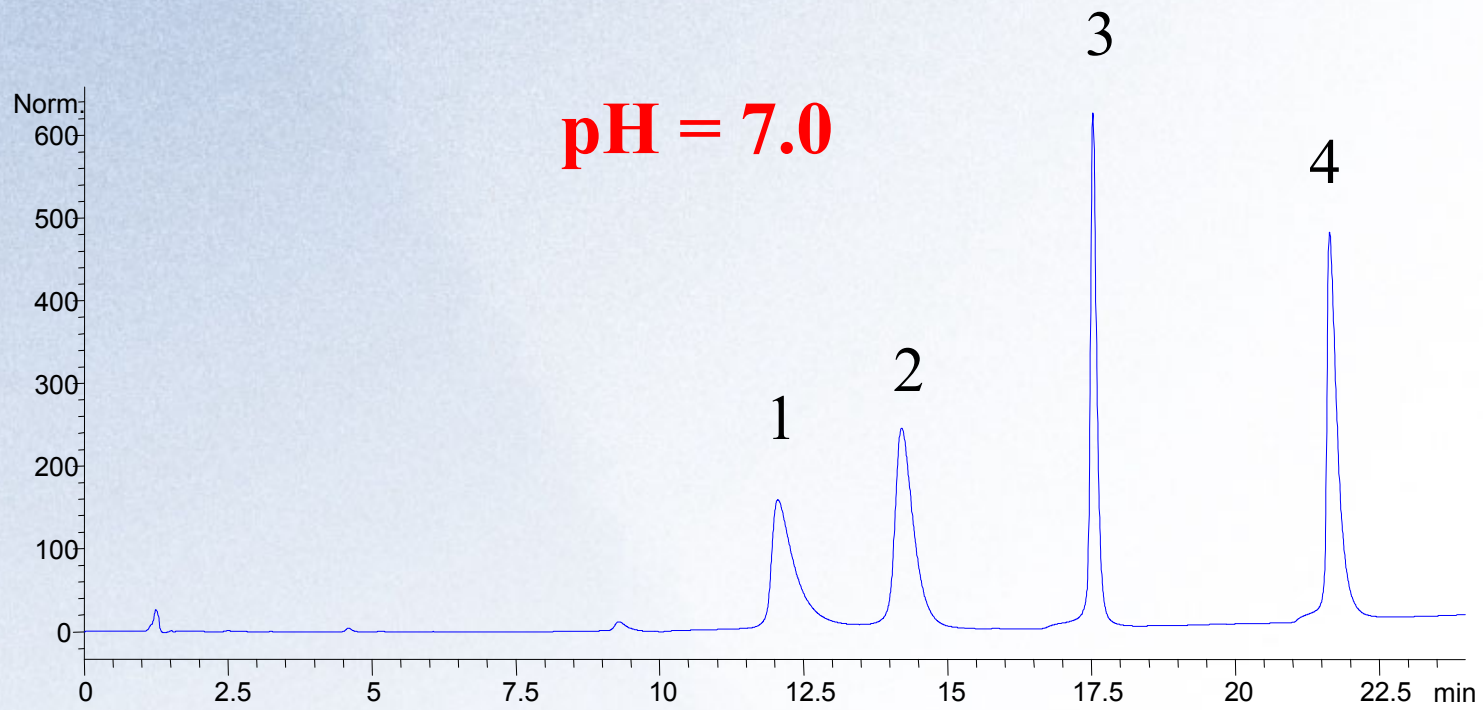
Regression Data from log k' vs. log k' Plots for All Columns versus Luna C18 (2)*

Column vs. Luna C18 (2)	R^2
ZirChrom-PBD	0.985
Gammabond RP-1	0.981
Xterra RP18	0.972
Polymer Labs PLRP-S	0.963
DiamondBond-C18	**0.889**

* Column names are the trademarks of their respective manufacturers.

Applications

Benzodiazepams - Anti-anxiety Drugs



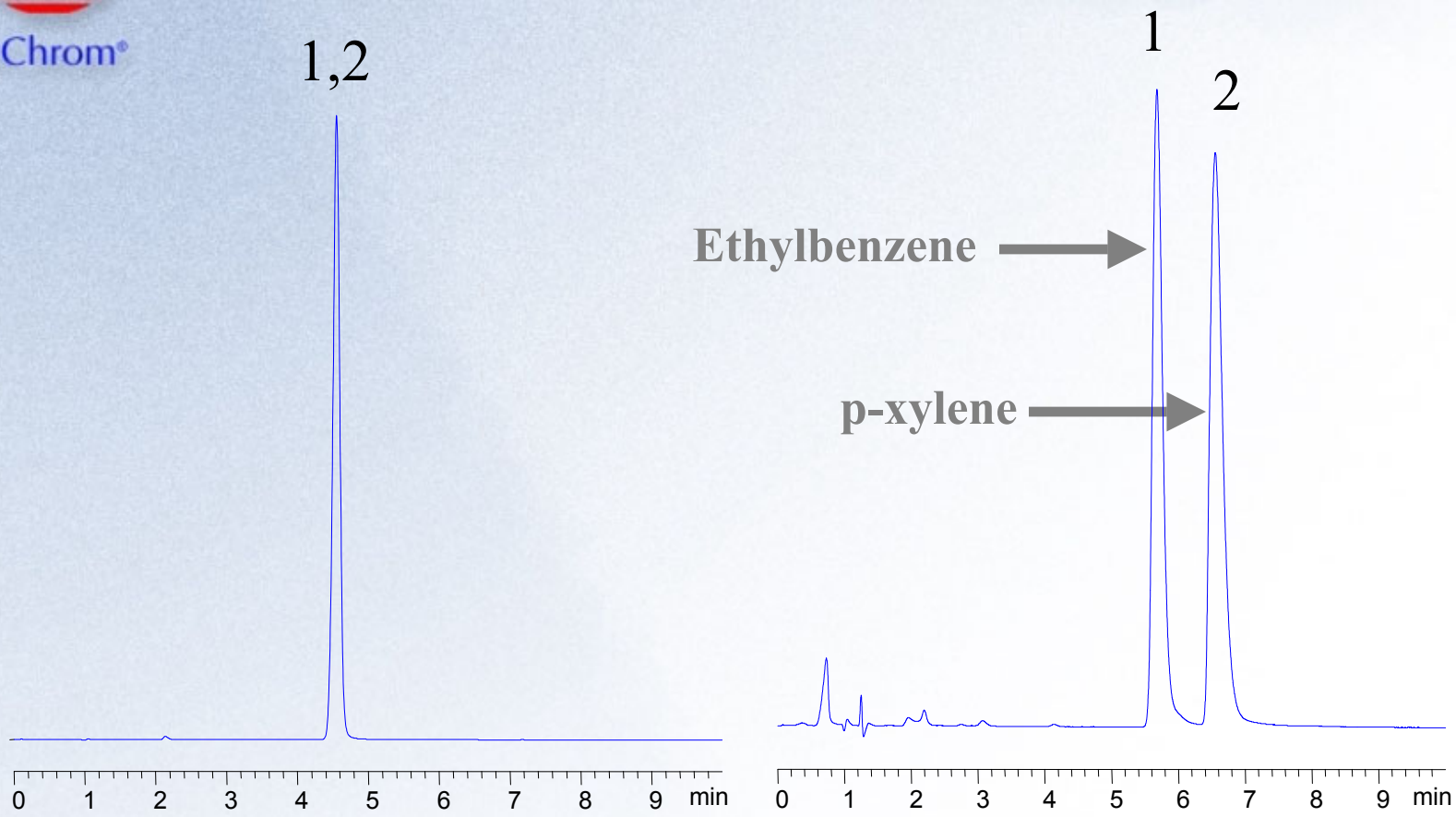
LC Conditions: Column, 50 x 4.6 DiamondBond™-C18; Mobile phase, 20 to 40% THF, 20 min ramp in 50mM potassium phosphate pH 7.0; Flow rate, 1.0 ml/min; Temperature, 30°C; Injection volume, 2 ul; Detection at 254 nm; Solute concentration 0.25 mg/ml; Solutes, 1=Oxazepam, 2=Temazepam, 3=Diazepam, 4=Medazepam.

DiamondBond-C18 Selectivity*

ZirChrom®

a) 150 x 4.6 mm ODS

b) 100 x 4.6 mm DiamondBond-C18



LC Conditions: a) Column, 150 x 4.6 Zorbax Eclipse XDB-C8 S/N: USRK010769; Mobile phase, 65/35 ACN/Water; Temperature, 30 °C; Flow rate, 1.0 ml/min.; Injection volume, 5 ul; Detection at 254 nm; Solutes: 1=Ethylbenzene, 2=p-xylene. b) Column, 100 x 4.6 DiamondBond-C18, OD082401A; Mobile phase, 37.5/5/57.5 ACN/THF/Water; Temperature, 60 °C; all other conditions the same as a).

DIAMONDBOND™ HPLC Columns

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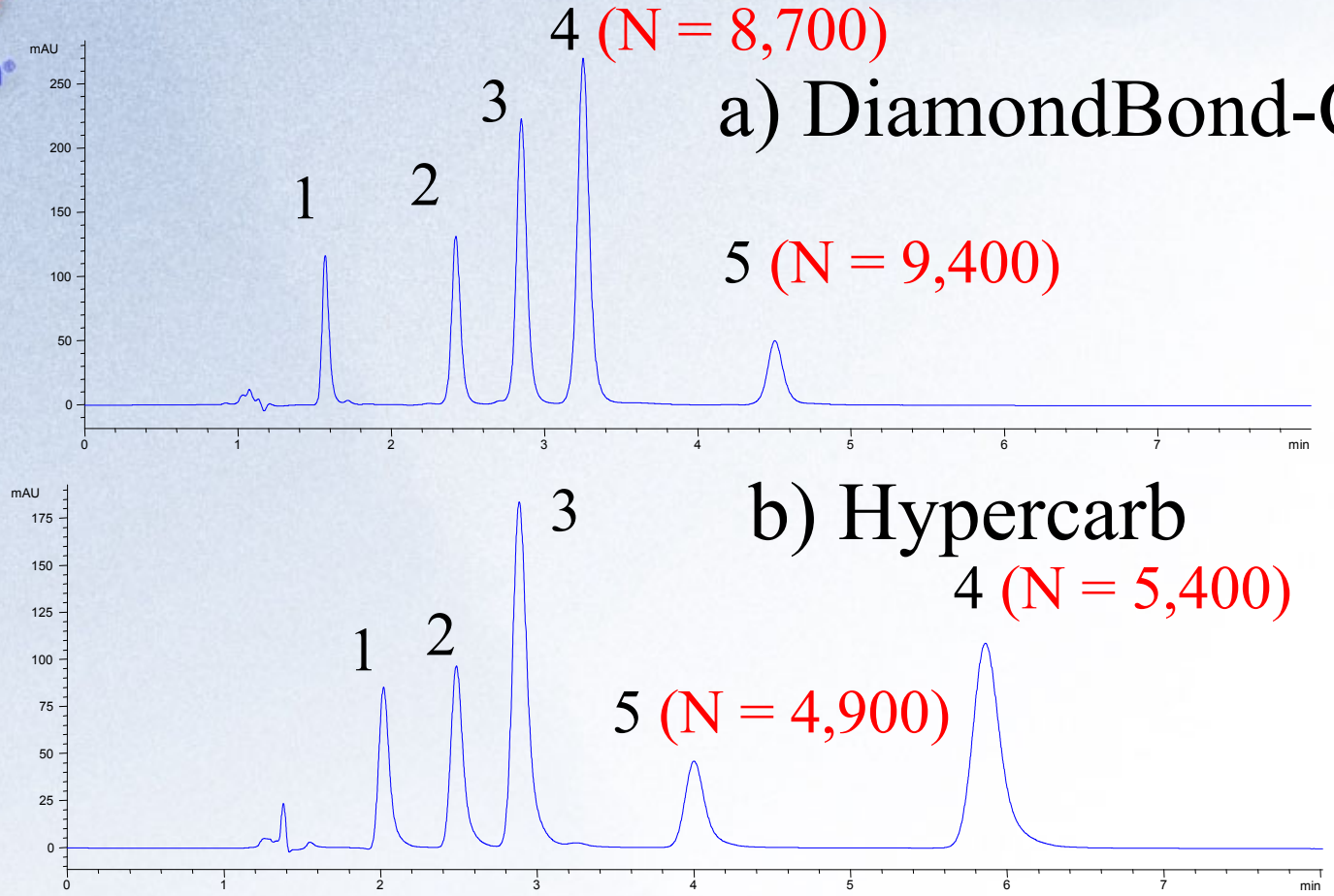
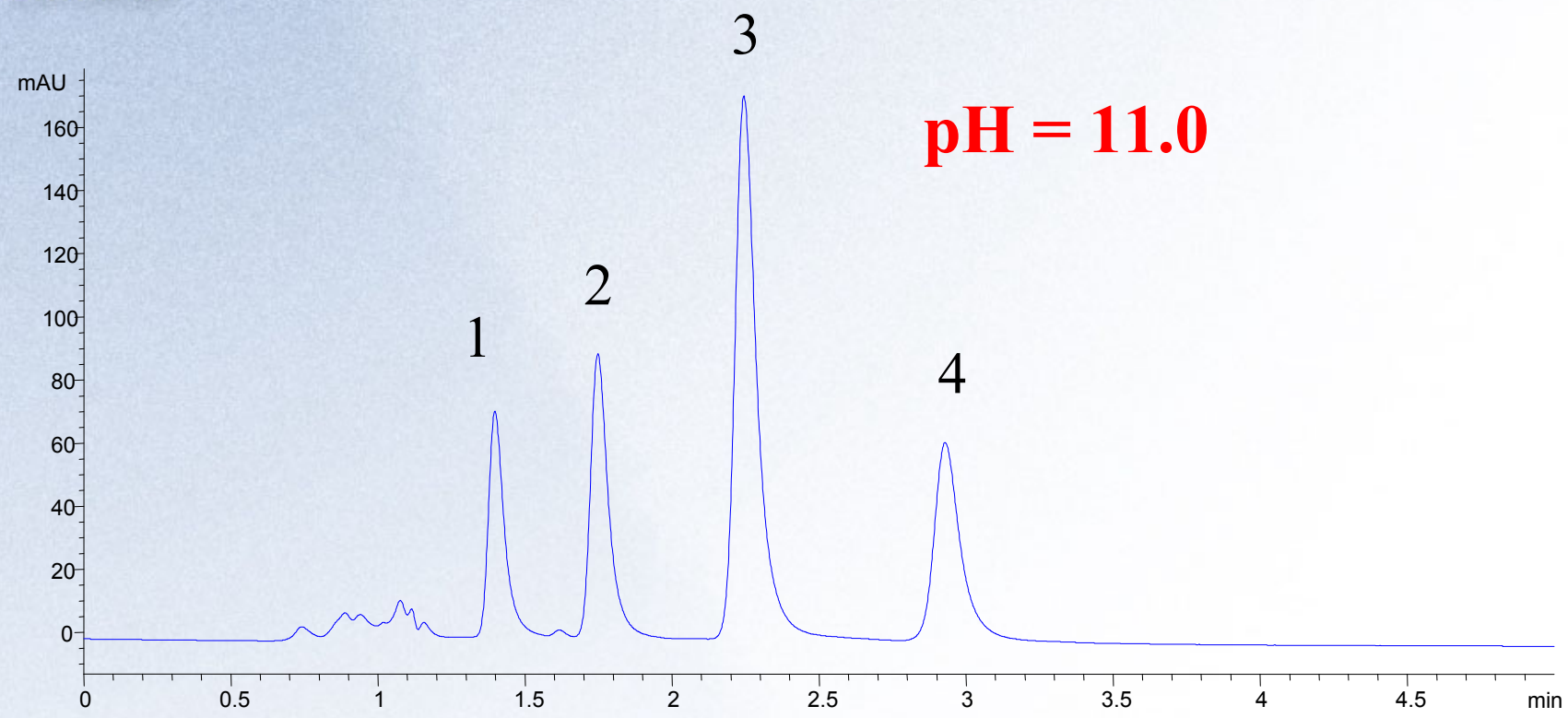
Separation of Anticonvulsants on DiamondBond™-C18 and Hypercarb*



LC Conditions: a) DiamondBond-C18 100 x 4.6; Mobile phase 25/75 THF/50mM Ammonium phosphate, pH 7.0; Flow rate, 1.0 ml/min.; Temperature, 50 °C; Injection volume, 0.5 ul; Detection at 220nm; Solutes: 1=Primidone, 2=Metharbital, 3=Mephenytoin, 4=Phenobarbital, 5=Phenytoin. b) Hypercarb 100 x 4.6; Mobile phase, 31/69 THF/50mM Ammonium phosphate, pH 7.0; all other conditions the same as in a).

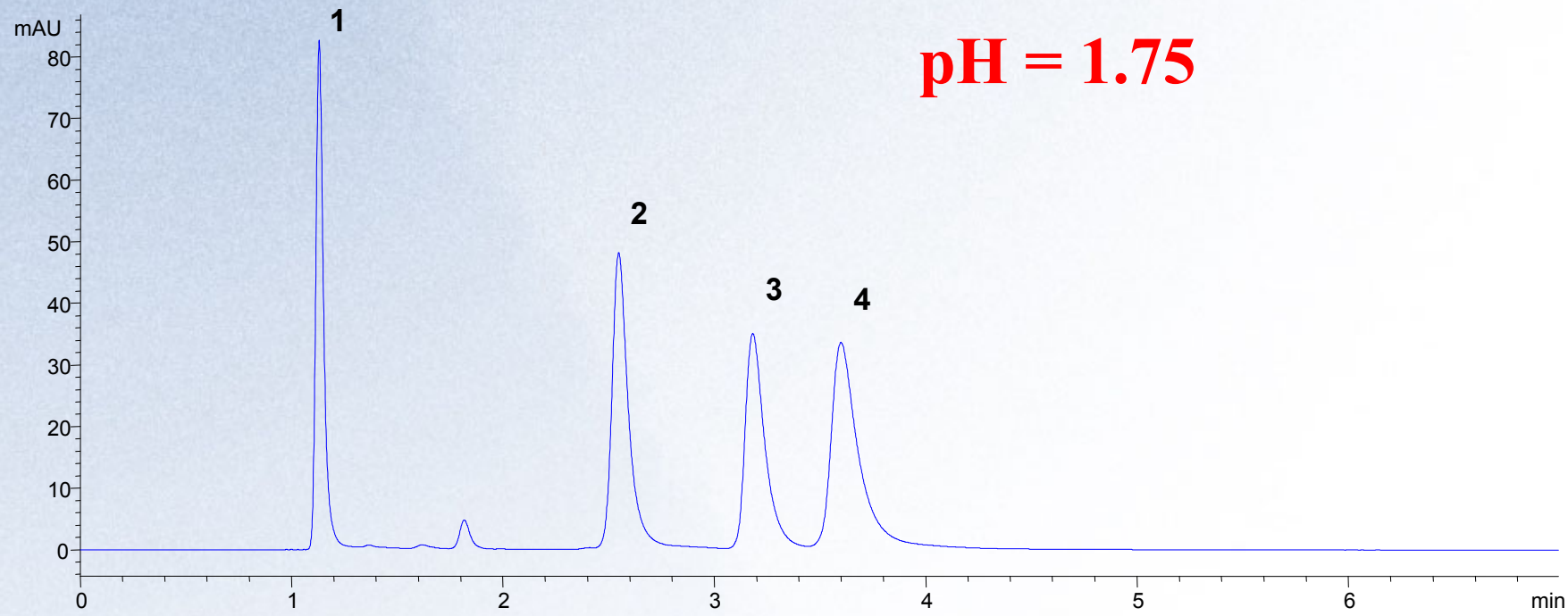
* Column names are the trademarks of their respective manufacturers.

Opioids



LC Conditions: Column, 100 x 4.6 DiamondBond™-C18; Mobile phase, 26.5/73.5 THF/20mM Ammonium phosphate, pH 11.0; Flow rate, 1.0 ml/min.; Temperature, 40 °C; Injection volume, 1.0 ul; Detection at 220nm; Solutes: 1=Naloxone, 2=Codeine, 3=Ethylmorphine, 4=Oxycodone

Fast Separations Non-Steroidal Anti-Inflammatories



pH = 1.75

LC Conditions: Column, 100 x 4.6 DiamondBond™-C18; LC Conditions: Mobile phase, 50/50 ACN/50mM Phosphoric acid, pH 1.75; Flow rate, 1.0 ml/min.; Temperature, 65 °C; Injection volume, 1.0 ul; Detection at 254nm; Solutes: 1=Acetominaphen, 2=Ketoprofen, 3=Ibuprofen, 4=Naproxen

ZirChrom®

Future Directions

- Ion-Exchangers
- Polar Bonded Phases (NPLC)
- Chiral Stationary Phases (CSPs)
- Reversed-Phases for Biomolecules
- Nonporous
- Preparative

DIAMOND BOND™
HPLC Columns

Conclusions

- **DiamondBond™-C18** is an ***ultra-durable*** and ***efficient*** carbon-based HPLC stationary phase.
- **DiamondBond™-C18** is stable at the ***extremes of pH*** and at column temperatures as high as ***200°C***.
- **DiamondBond™-C18** had the ***most different selectivity*** relative to conventional ODS phases for the 22 selected non-ionizable compounds.
- **DiamondBond™-C18's** durability allows for ***high and low pH*** separations for improved peak shape.
- **DiamondBond™'s** chemistry is ***as flexible as silane chemistry*** and in the future will be used to produce a ***broad family*** of novel ultra-durable phases.

DIAMONDBOND™ HPLC Columns