

EXAM FOUR – TUESDAY
APRIL 20th

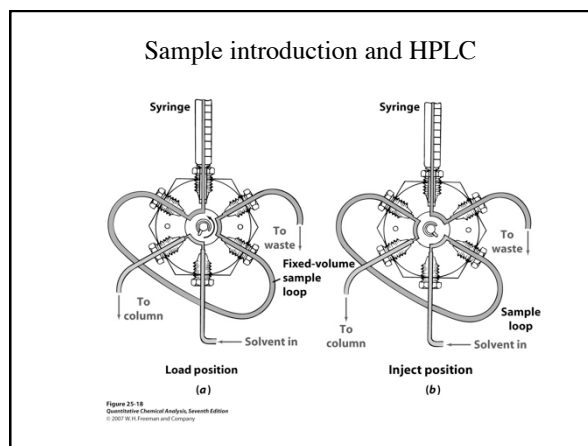
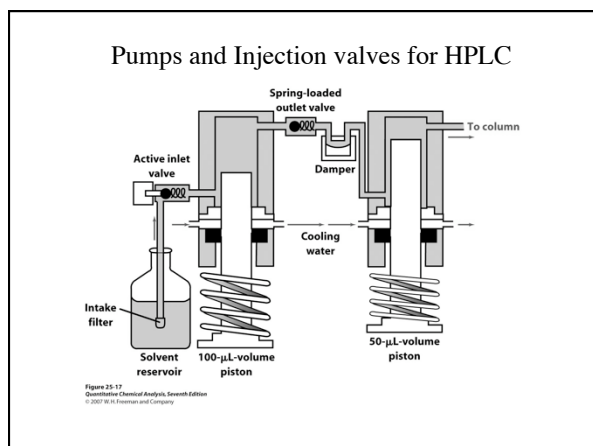
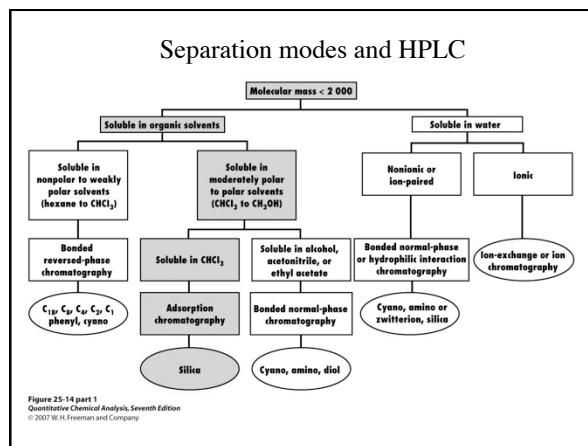
Daniel C. Harris

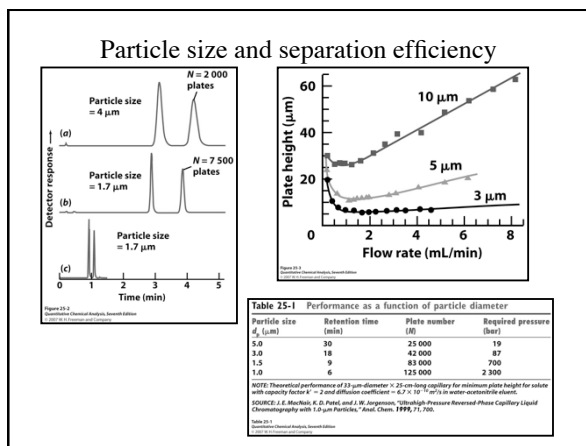
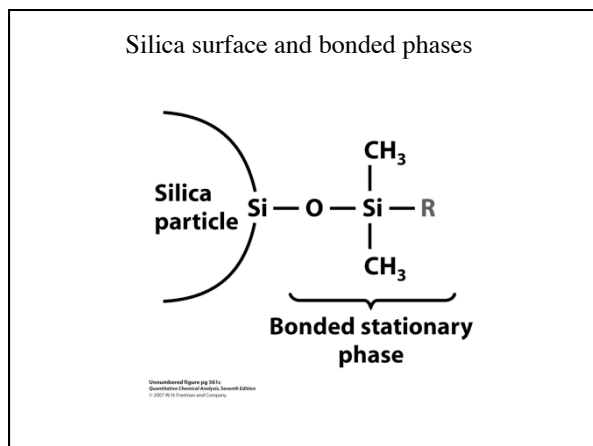
**Quantitative Chemical
Analysis**
Seventh Edition

Chapter 25
High-Performance Liquid
Chromatography

Suggested problems: Chapter 25: 1a, 1s
Chapter 26: 25, 28

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Selecting the right solvent for HPLC

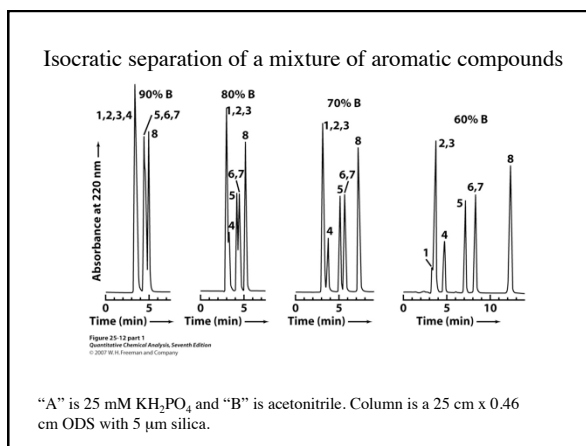
Table 25-2 Eluotropic series and ultraviolet cutoff wavelengths of solvents for adsorption chromatography on silica

Solvent	Eluent strength (ϵ')	Ultraviolet cutoff (nm)
Pentane	0.00	190
Hexane	0.01	195
Heptane	0.01	200
Trichlorofluoroethane	0.02	231
Toluene	0.22	284
Chloroform	0.26	245
Dichloromethane	0.30	233
Diethyl ether	0.43	215
Ethyl acetate	0.48	256
Methyl <i>t</i> -butyl ether	0.48	210
Dioxane	0.51	215
Acetonitrile	0.52	190
Acetone	0.53	330
Tetrahydrofuran	0.53	212
2-Propanol	0.60	205
Methanol	0.70	205

NOTE: The ultraviolet cutoff for water is 190 nm.

SOURCES: L. R. Snyder, in *High-Performance Liquid Chromatography* (C. Horváth, ed.), Vol. 3 (New York: Academic Press, 1983); Burdick & Jackson Solvent Guide, 3rd ed. (Muskegon, MI: Burdick & Jackson Laboratories, 1990).

Table 25-2
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Isocratic separation of a mixture of aromatic compounds cont'd

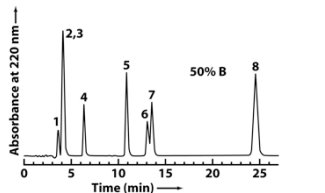


Figure 25-12 (part 2)

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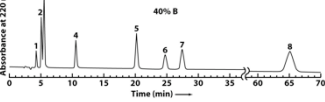


Figure 25-12 (part 3)

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Isocratic separation of a mixture of aromatic compounds cont'd

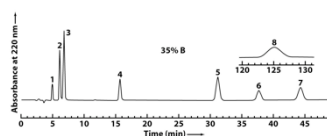


Figure 25-12 (part 5)

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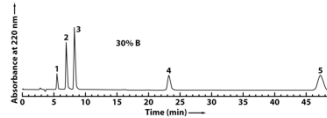


Figure 25-12 (part 4)

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Gradient elution of the same mixture!

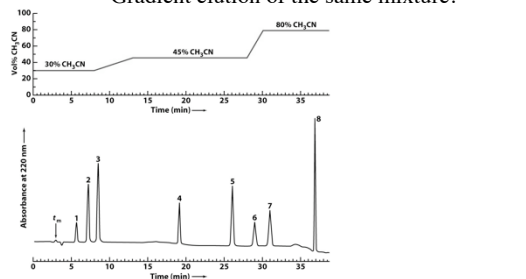


Figure 25-13

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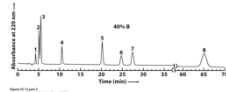


Figure 25-12 (part 3)

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Detectors for HPLC

Table 25-3 Comparison of commercial HPLC detectors

Detector	Approximate limit of detection ^a (ng)	Useful with gradient?
Ultraviolet	0.1–1	Yes
Refractive index	100–1 000	No
Evaporative light-scattering	0.1–1	Yes
Electrochemical	0.01–1	No
Fluorescence	0.001–0.01	Yes
Nitrogen ($\text{N} \xrightarrow{\text{combustion}} \text{NO} \xrightarrow{\text{O}_1} \text{NO}_2 \xrightarrow{h\nu}$)	0.3	Yes
Conductivity	0.5–1	No
Mass spectrometry	0.1–1	Yes
Fourier transform infrared	1 000	Yes

^a Most detection limits from E. W. Yeung and R. E. Synovec, "Detectors for Liquid Chromatography," *Anal. Chem.* **1986**, *58*, 1237A.

Table 25-3

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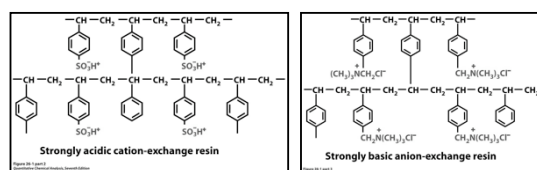
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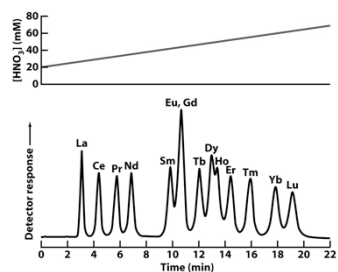
Chapter 26
 Chromatographic Methods and
 Capillary Electrophoresis

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Ion exchange resins and ion chromatography

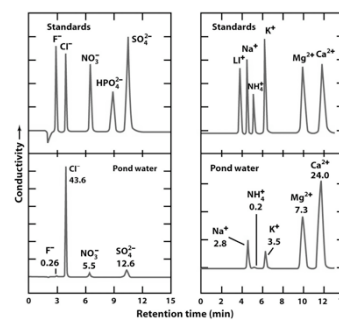


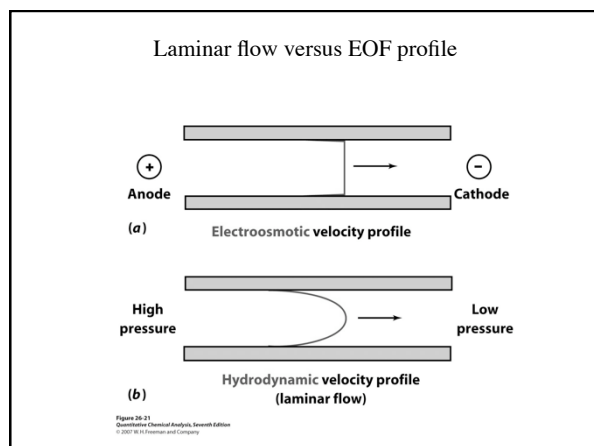
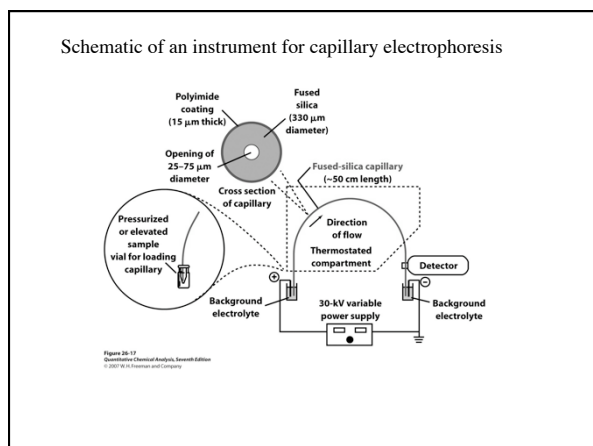
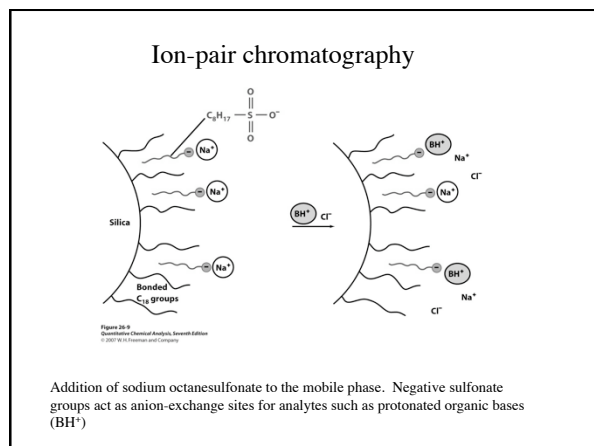
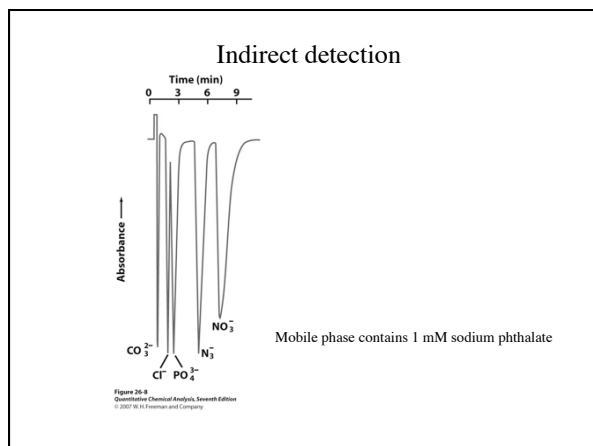
Gradient elution and ion exchange chromatography



Higher the atomic # of the lanthanide, the smaller its ionic radius and the more strongly it binds to the resin

Ion exchange chromatography - analysis of pond water





Separation efficiency in HPLC versus CE

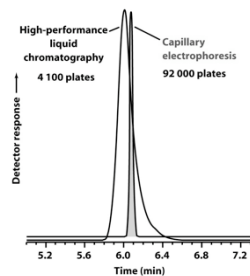


Figure 26-19
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Indirect detection of isotopes separated by CE

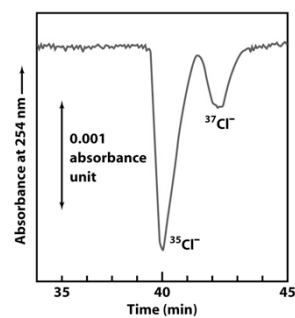


Figure 26-31
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Detection challenges in CE

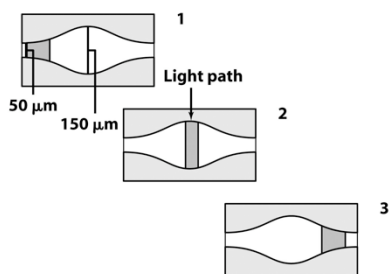


Figure 26-28a
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Electrochemical Detection in CE

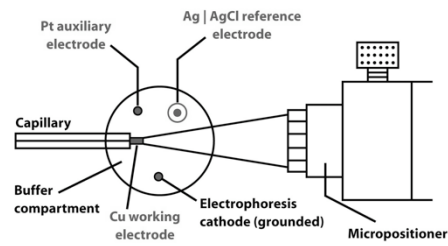


Figure 26-29a
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