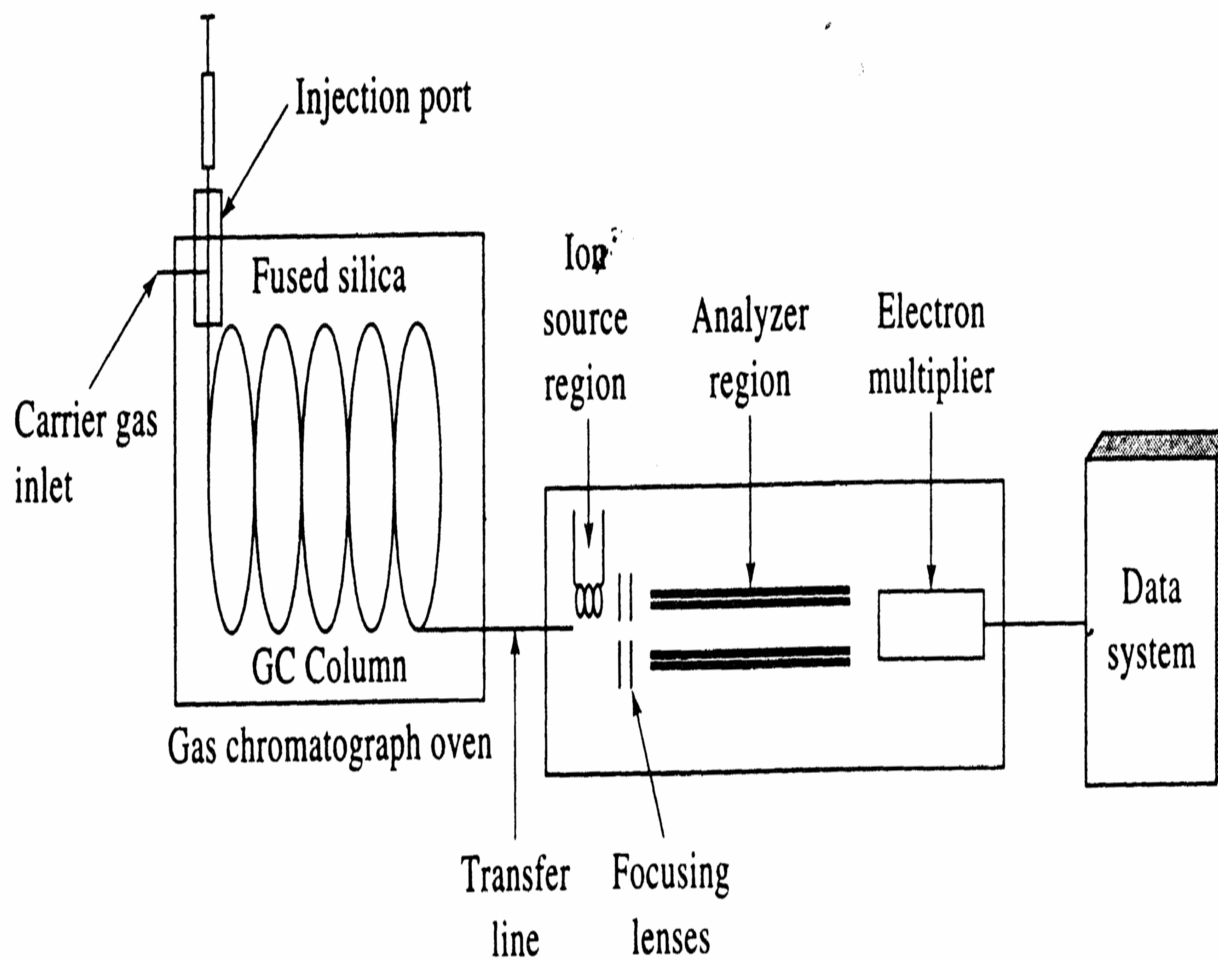
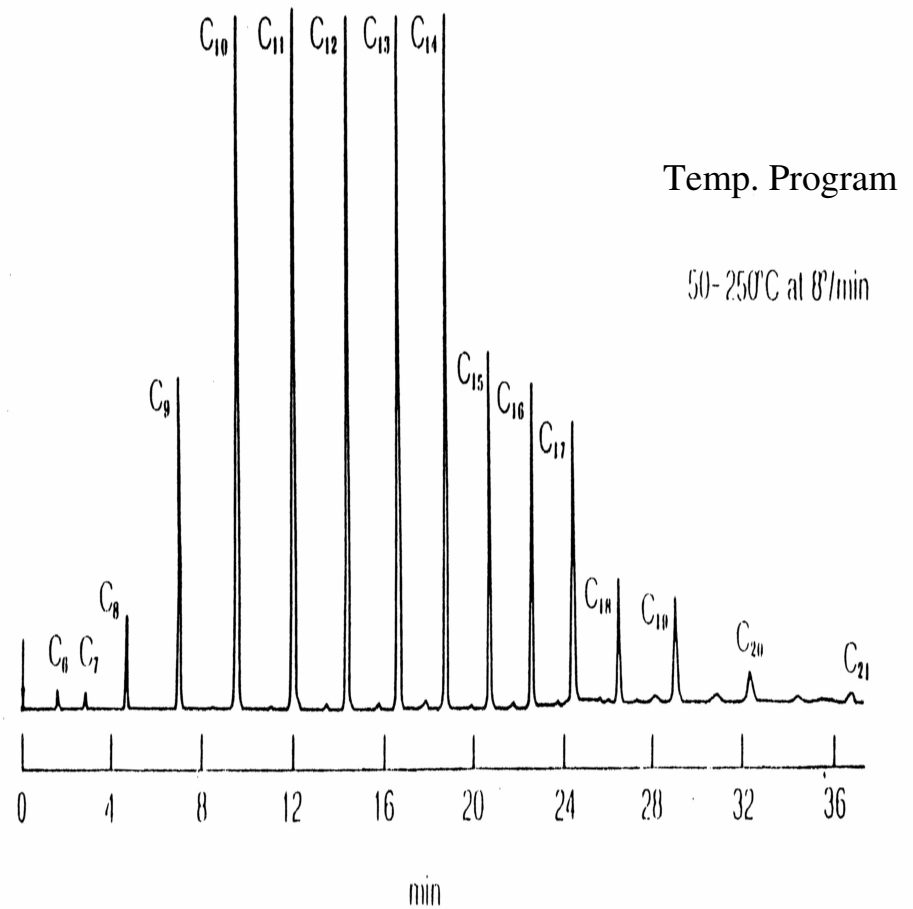
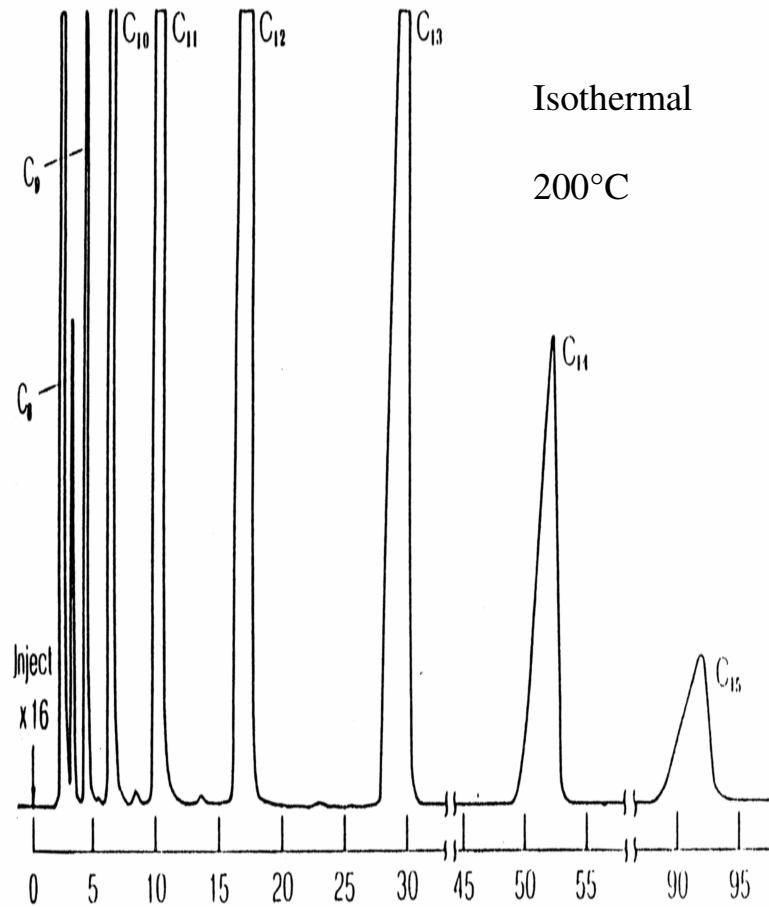


# General Instrument Schematic for a Gas Chromatograph/Mass Spectrometer



From Skoog, Holler and Nieman, "Principles of Instrumental Analysis, 5 th. ed., p. 719

# General Elution Problem in Gas Chromatography



From D.C. Harris, "Quantitative Chemical Analysis", 3 rd. ed., 1991, W.H. Freeman, p. 653.

Depiction of the most common modes of injection into GC capillary columns.

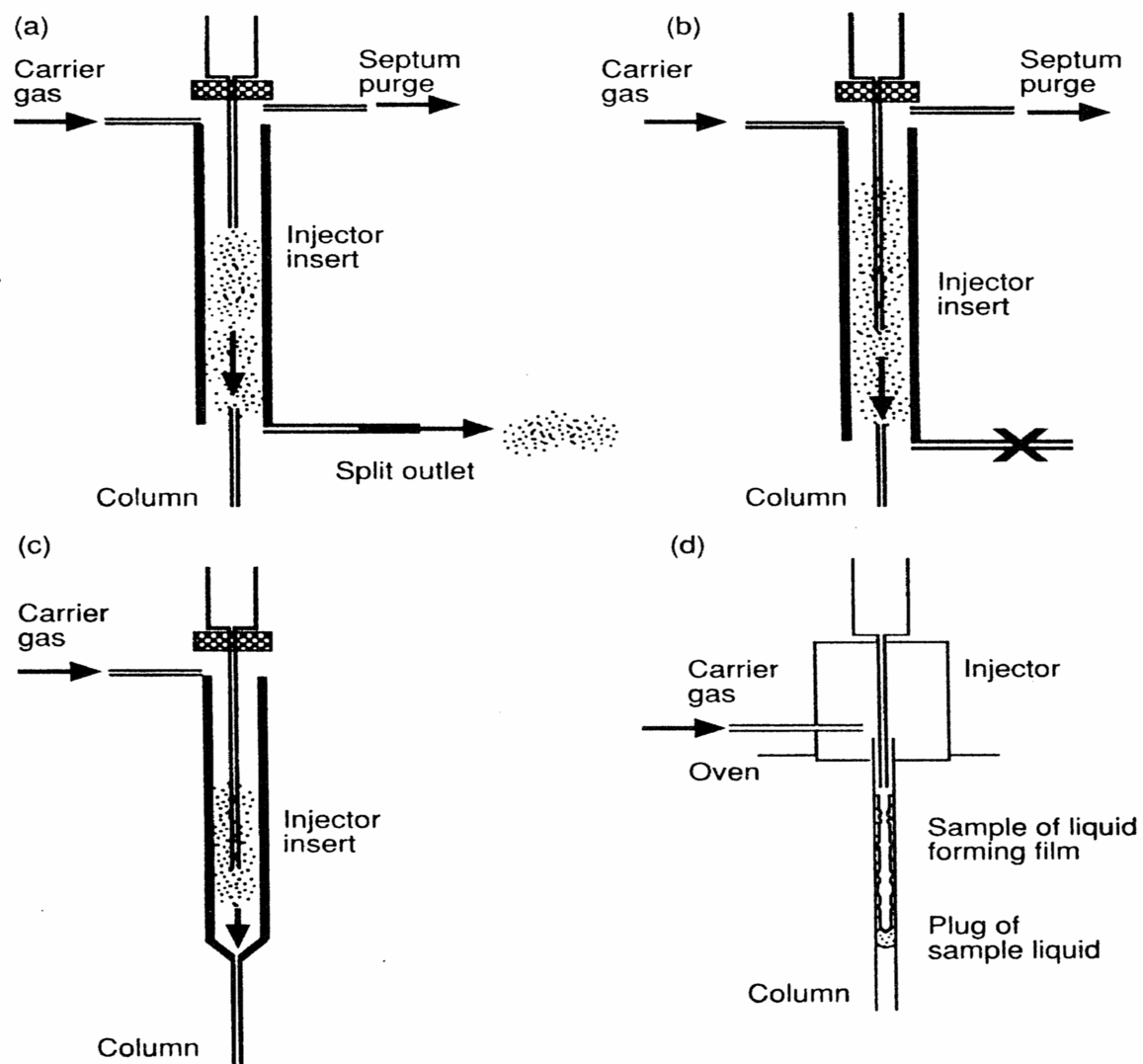


Fig. 1 of K. Grob, "Injection Techniques in Capillary GC", *Anal. Chem.*, 1994, **66** (22), 1010A.

## Typical Gas Chromatographic Columns

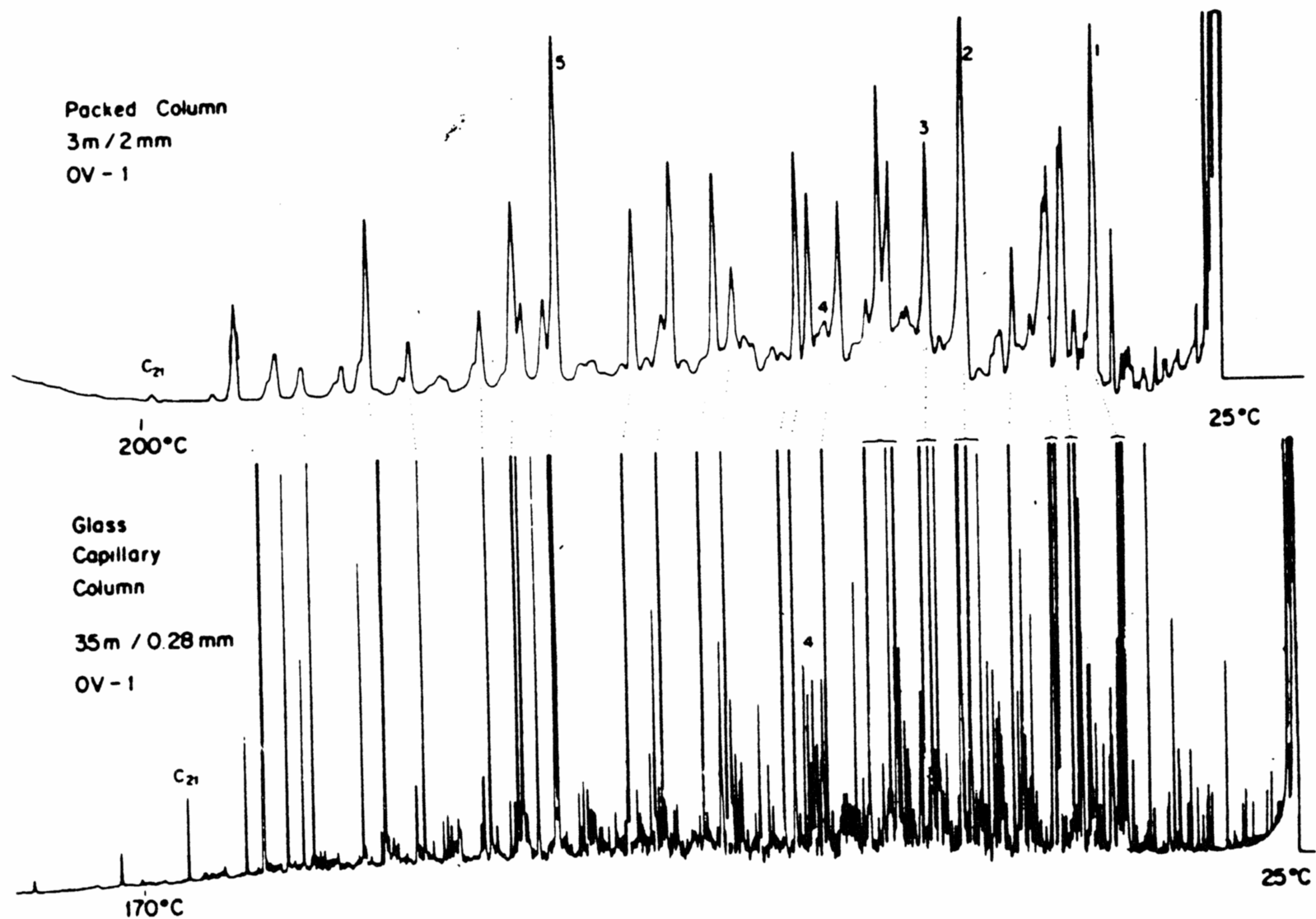
Column Name	Inner Diameter	Column Length	Plates per meter	Total Plates in column (30m)	Rel. Loading Capacity
<b>Packed</b>	<b>3.2-6.4 mm</b>	<b>1 - 6 m</b>	<b>2,000</b>	<b>6,000 (2m)</b>	<b>500</b>
<b>Capillary* - Fused Silica Open Tubular (FSOT)</b>					
<b>Megabore</b>	<b>0.53 mm</b>	<b>10-60 m</b>	<b>2,340</b>	<b>70,000 (30m)</b>	<b>50</b>
<b>Narrow-bore</b>	<b>0.32 mm</b>	<b>10-60 m</b>	<b>5,000</b>	<b>150,000</b>	<b>10</b>
<b>Narrowbore</b>	<b>0.25 mm</b>	<b>10-60 m</b>	<b>6,400</b>	<b>192,000</b>	<b>5</b>
<b>Minibore</b>	<b>0.20 mm</b>	<b>10-30 m</b>	<b>8,000</b>	<b>240,000</b>	<b>1</b>
<b>Minibore</b>	<b>0.10 mm</b>	<b>10-20 m</b>	<b>16,000</b>	<b>480,000</b>	<b>0.1</b>

•Types Capillary Columns:FSOT; Wall-Coated Open Tubular (WALCOT); Support-Coated OT (SCOT).

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Adapted from C.F. Poole, "The Essence of Chromatograph", Elsevier, 2003, p. 80-83 and Skoog, Holler and Nieman, "Principles of Instrumental Analysis", 5 th. ed. 1998, Harcourt Brace & Co., p. 713.

# Comparison of the same water extract by packed and capillary column GC



From C.F. Poole and S.K. Poole, "Chromatography Today", Elsevier Scientific, p. 133

Common packed column applications of porous polymers

Polymer	Application
Chromosorb 101 Porapak P and PS	Esters, ethers, ketones, alcohols, hydrocarbons, fatty acids, aldehydes and glycols. Not recommended for amines and anilines.
Chromosorb 102 Porapak Q	Light and permanent gases, volatile carboxylic acids, alcohols, glycols, ketones, hydrocarbons, esters, nitriles and nitroalkanes. Not recommended for amines, anilines. Nitrated by nitrogen oxide gases.
Chromosorb 103 Porapak S	Amines, amides, alcohols, aldehydes, hydrazines and ketones. Not recommended for acids, amines, glycols, and nitriles. Reacts with nitroalkanes.
Chromosorb 104	Nitriles, nitro compounds, sulfur gases, ammonia, carbon dioxide, vinyl chloride, moisture in solvents and xylenols. Not recommended for amines and glycols.
Chromosorb 105 Porapak N	Aqueous mixtures of formaldehyde, acetylene from lower hydrocarbons and most gases. Not recommended for glycols, acids and amines.
Chromosorb 106 Porapak QS	Alcohols, C <sub>2</sub> -C <sub>5</sub> carboxylic acids, alcohols and sulfur gases. Not recommended for glycols and amines.
Chromosorb 107 Porapak T	Formaldehyde from water and acetylene from lower hydrocarbons. Sulfur compounds. Not recommended for glycols and amines.
Chromosorb 108	Gases, water, alcohols, aldehydes and glycols.
Porapak R	Esters, ethers, nitriles and nitro compounds. Not recommended for glycols and amines.
Tenax-GC	High boiling polar compounds, diols, phenols, methyl esters of dicarboxylic acids, amines, diamines, ethanolamines, amides, aldehydes and ketones.

From C.K. Poole, "The Essence of Chromatography", Elsevier Scientific, p. 117.

## Common Gas Chromatographic Detectors

Detector Name	Mode of Operation	Approx. Min. Detection Level	Linear Dynamic Range
Flame Ionization (FID)	Change in the conductivity of H <sub>2</sub> /air flame	2 pg/sec (carbon)	10 <sup>7</sup>
Thermal Conductivity (TCD)	Change in the resistance of a heated wire	400 pg/mL (propane)	10 <sup>5</sup>
Electron Capture (ECD)	Reduction of the ionization of a cascading radioactive emission.	5-500 fg/mL (lindane)	10 <sup>4</sup> -10 <sup>6</sup>
Photoionization (PID)	Ionization caused by a intense UV source	0.2 pg/sec (benzene)	10 <sup>7</sup>
Atomic Emission (AED)	Elemental emission caused by a plasma source	0.1-20 pg/sec (element)	10 <sup>4</sup>
Flame Photometric (FPD)	Specific emission of an excited state in a flame	20 pg S/sec, 1 pg P/sec	10 <sup>3</sup> -10 <sup>4</sup>
Thermionic Emission (TED)	Enhanced ionization due to an alkali bead	0.1 pg N/sec, .01 pg P/sec	10 <sup>4</sup>
Mass Spectrometric (MSD)	Monitoring mass/charge as a result of electron impact	10pg/sec-10ng.sec	10 <sup>6</sup>

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Adapted from various sources and D.C. Harris, "Quantitative Chemical Analysis", 5 th. ed. 1999, W.H. Freeman and Co., p. 691-693.

PRINCIPAL INTERMOLECULAR FORCES CHARACTERIZING SOLUTE/STATIONARY PHASE INTERACTIONS

$\alpha_A$  and  $\alpha_S$  = polarizabilities of solute and solvent molecules;  $I_A$  and  $I_S$  = ionization energies of solute and solvent molecules;  $r$  = distance between dipoles;  $\mu_A$  and  $\mu_S$  = dipole or induced dipole moments in solute and solvent molecules;  $K$  = Boltzmann constant;  $T$  = temperature (K).

Type	Description	Representation	Comments
Dispersive (or London) Forces	Arise from the electric field produced by the very rapidly varying dipoles between nuclei and electrons in molecules with zero-point motion. Induced dipoles are formed in phase with the instantaneous dipoles producing them.	$E_D = \frac{-3\alpha_A\alpha_S I_A I_S}{2r^6(I_A + I_S)}$	Present in all solute/solvent systems. Only source of attraction between nonpolar molecules. Independent of temperature.
Induction (or Debye) Forces	Arise from the interaction of a permanent dipole with a polarizable molecule.	$E_i = 1/r^6(\alpha_S\mu_A^2 + \alpha_A\mu_S^2)$	Generally weak and decrease with increasing temperature. Dipole-induced dipole interactions are not the same in all directions and depend on relative molecular orientation.
Orientation (Keesom) Forces	Arise from the net attraction between molecules or portions of molecules possessing a permanent dipole moment.	$E_o = \frac{-2\mu_A^2\mu_S^2}{3r^6KT}$	Decrease with increasing temperature and approach zero at very high temperatures when all orientations are equally probable.
Donor- Acceptor Complexes	Special chemical bonding interactions which arise from the partial transfer of electrons from a filled orbital on the donor to a vacant orbital on the acceptor molecule.		Examples include coordination forces between metal ions and olefins, charge transfer forces, and hydrogen bonding interactions.

From S.K. Poole and S.A. Schuette, "Contemporary Practice of Chromatography" 1984, Elsevier Science, p. 32.



INTERACTIONS CHARACTERIZED BY MCREYNOLDS PROBES (ROHRSCHEIDER  
 PROBES IN PARENTHESES)

Symbol	Test Solute	Boiling Point (°C)	Dipole Moment	Interaction Measured
X'	Benzene	80.1	0.03-0.1	Primarily dispersion with weak proton acceptor properties. Polarizable in induction interactions.
Y'	Butanol (Ethanol)	117.5	1.75	Orientation properties with both proton donor and proton acceptor capabilities.
Z'	2-Pentanone (2-Butanone)	102	2.82	Orientation with some weak proton acceptor but not proton donor capabilities.
U'	Nitropropane (Nitromethane)	103.5	3.59	Orientation with some weak proton acceptor capability.
S'	Pyridine	115.5	2.37	Strong proton acceptor with moderate orientation properties. Proton donor properties are absent.
H'	2-Methyl-2 pentanol	121.5	---	See butanol.
J'	Iodobutane	130	1.81	Moderate orientation interactions.
K'	2-Octyne	125	---	See benzene.
L'	Dioxane	101	0.45	Proton acceptor with weak orientation properties. Proton donor properties are absent.
M'	cis-Hydrindane	159(*)	---	Dispersive properties.

(\*)boiling point is for trans isomer .

From C.K. Poole and S.K. Poole, "Chromatography Today", Elsevier Scientific, p. 189.

## Comparing McReynolds' (Rohrneider's) solutes to Poole's solvation parameter model.

Prototypical solutes used by McReynolds (Rorschneider solutes in parentheses) to characterize stationary phase properties

$R_2$  = excess refraction,  $\pi_2^H$  = dipolarity/polarizability,  $\Sigma\alpha_2^H$  hydrogen-bond acidity,  $\Sigma\beta_2^H$  = hydrogen-bond basicity and  $\log L^{16}$  partition coefficient on hexadecane at 25°C.

Symbol	Solute	Solute descriptors				
		$R_2$	$\pi_2^H$	$\Sigma\alpha_2^H$	$\Sigma\beta_2^H$	$\log L^{16}$
X'	Benzene	0.610	0.52	0	0.14	2.786
Y'	1-Butanol (Ethanol)	0.224	0.42	0.37	0.48	2.601
Z'	2-Pentanone (2-Butanone)	0.143	0.68	0	0.51	2.143
U'	1-Nitropropane (Nitromethane)	0.242	0.95	0	0.27	2.894
S'	Pyridine	0.631	0.84	0	0.52	3.022
H'	2-Methyl-2-pentanol	0.180	0.30	0.31	0.60	1.963
J'	Iodobutane	0.628	0.40	0	0.15	3.628
K'	2-Octyne	0.225	0.30	0	0.10	3.850
L'	Dioxane	0.329	0.75	0	0.64	2.892
M'	cis-Hydrindane	0.439	0.25	0	0	4.635

Copied from C.F. Poole, "The Essence of Chromatography", Elsevier Publishers, 2003, p. 139.