

QFL 4325 –Química Orgânica Experimental

Técnicas Cromatográficas

Cromatografia

- IUPAC = Método físico de separação no qual os componentes a serem separados são distribuídos entre duas fases, uma das quais é estacionária (fase estacionária) enquanto a outra (fase móvel) se move numa direção definida.
- Não se limita a técnicas analíticas

Tipos de Cromatografia

Adsorption chromatography	
Liquid–solid column chromatography	
(LSC)	
Paper chromatography (PC)	
Thin–layer chromatography (TLC)	
Gas–solid chromatography (GSC)	
	Packed columns
	Open tubular columns (OTC)

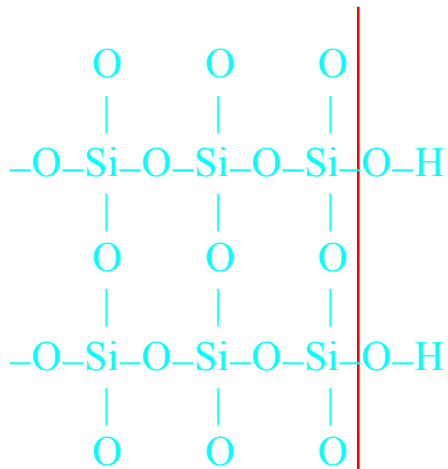
Partition chromatography	
Liquid–liquid column chromatography	
(LLC)	
Paper chromatography (PC)	
Thin–layer chromatography (TLC)	
Foam chromatography (FC)	
Emulsion chromatography (EC)	
Gas–liquid chromatography (GLC)	
	Packed columns
	Open tubular columns (OTC)

Ion exchange	
Liquid–solid chromatography (LSC)	
	Ion chromatography
Paper chromatography (PC)	
Thin–layer chromatography (TLC)	

Size exclusion chromatography (SEC)	
Gel filtration (GFC) or gel	
Permeation chromatography (GPC)	
Molecular sieves	

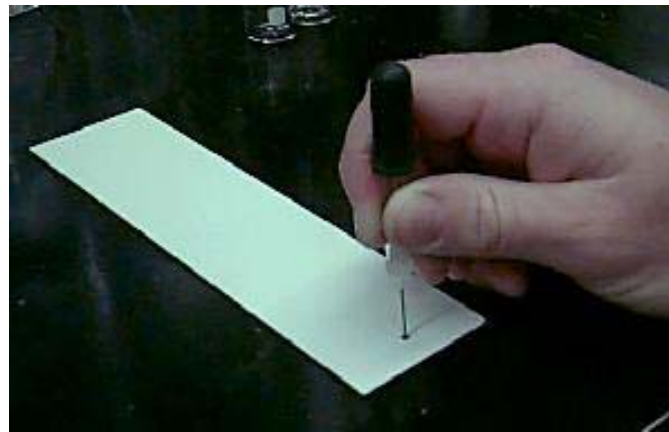
Cromatografia em Camada Delgada (CCD)

Fase Estacionária



$(\text{SiO}_2)_x$

superfície



aplicação



CCD

Fase estacionária: sílica (SiO_2), alumina (Al_2O_3), celulose, etc.

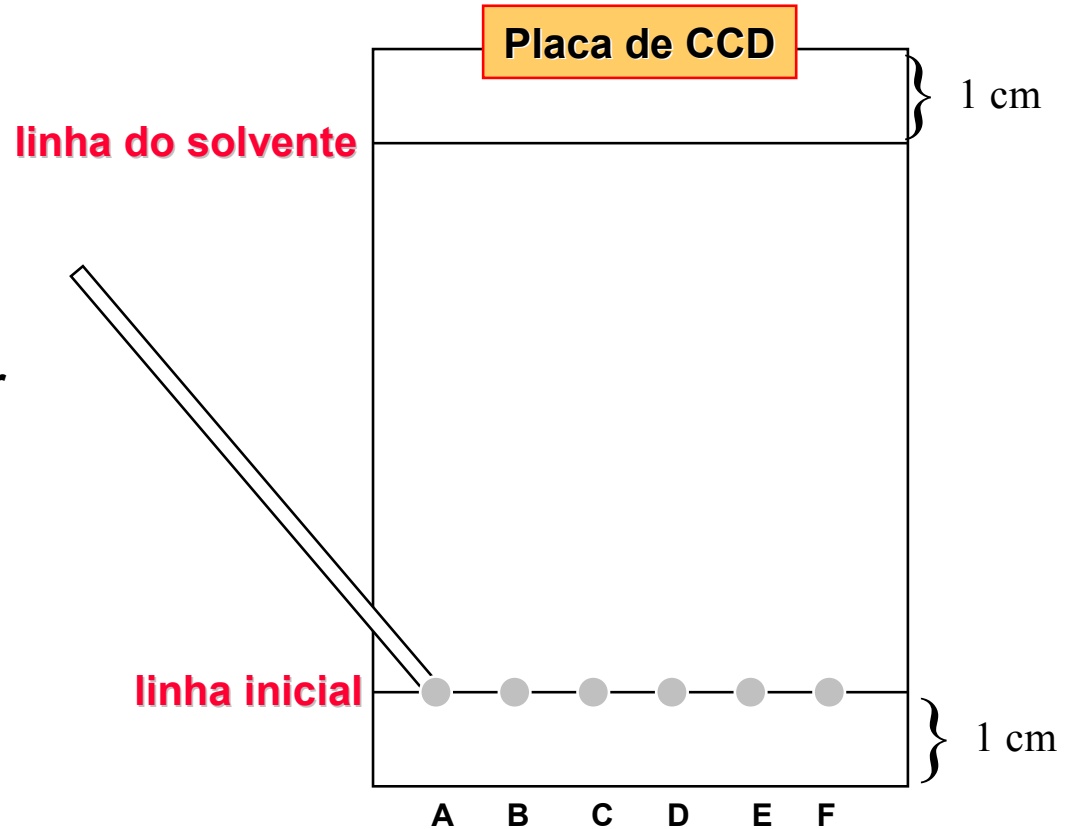
Fase móvel: solvente.

Força motriz: capilaridade.

Revelação: UV, câmara de I_2 , vanilina, anilina, etc.

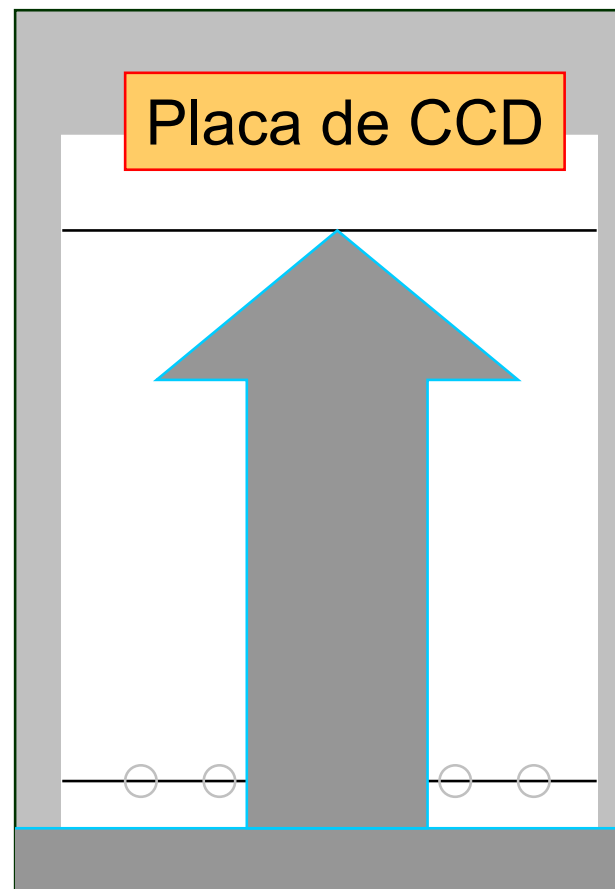
1. Aplicação da amostra

- A. Desenhe linhas-guia com um lápis
- B. Dissolva as amostras em um solvente adequado
- C. Use um capilar para aplicar as amostras e padrões na placa de CCD



2. Corrida

- A. Coloque a placa de CCD na cuba com o solvente
- B. O solvente sobe por capilaridade e arrasta as amostras e padrões
- C. Retire a placa quando o solvente atingir a linha superior



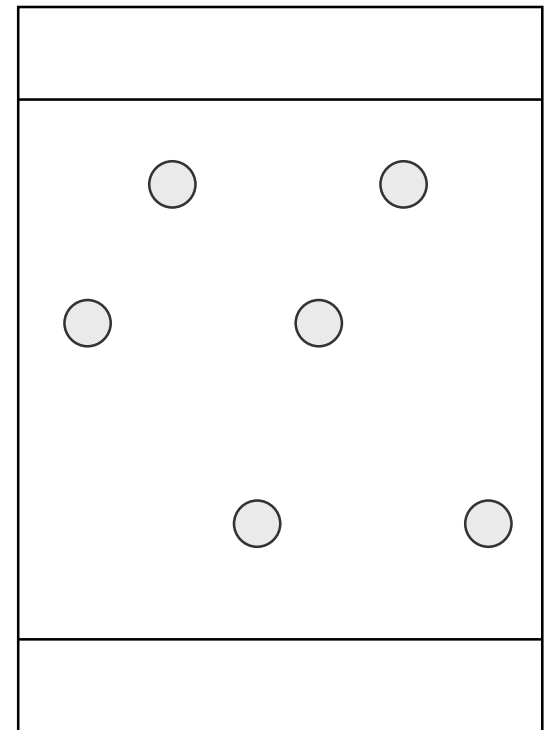
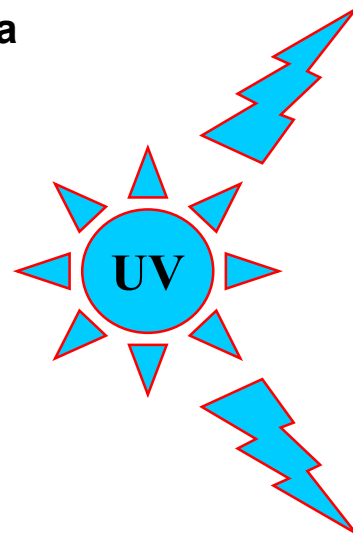
Câmara de CCD
uma cuba com solvente tampada

3. Revelação

A. Deixe o solvente evaporar da placa

B. Deixe a placa sob luz UV, em uma câmara de I₂ ou borrife um reagente de revelação específico

C. Marque as manchas com um lápis e guarde a placa



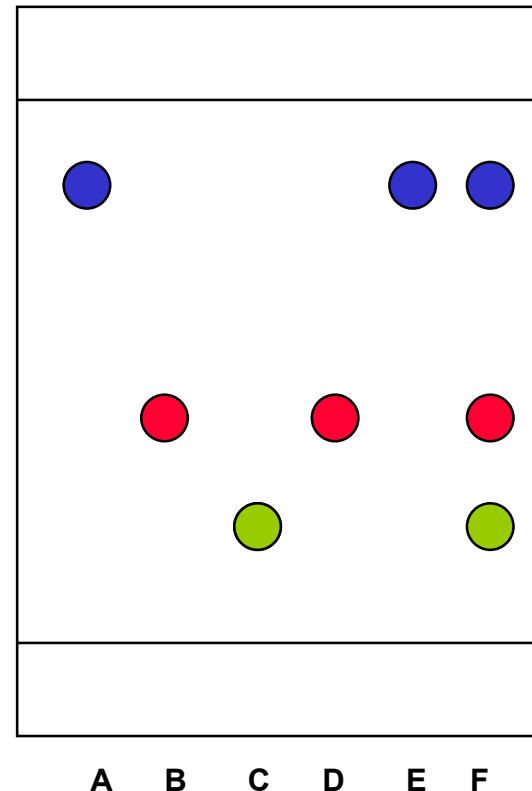
A B C D E F

4. Determinação do Fator de Retenção (R_F)

A. Determine os R_F para cada mancha

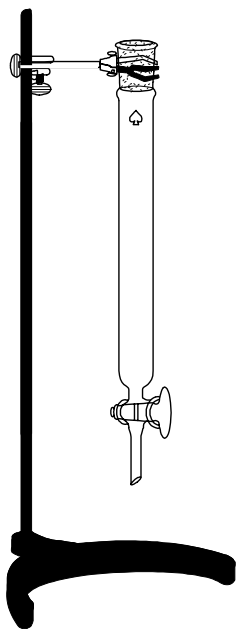
B. Use os valores de R_F para identificar os compostos de uma mistura

$$R_F = \frac{\text{distância do centro da mancha}}{\text{distância percorrida pelo solvente}}$$

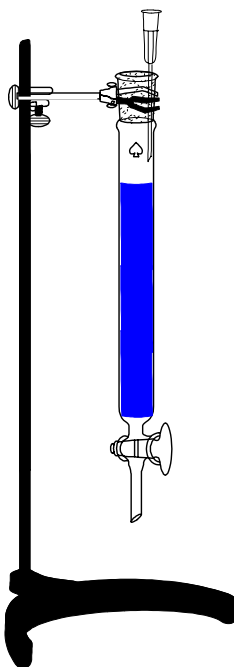


Cromatografia em Coluna

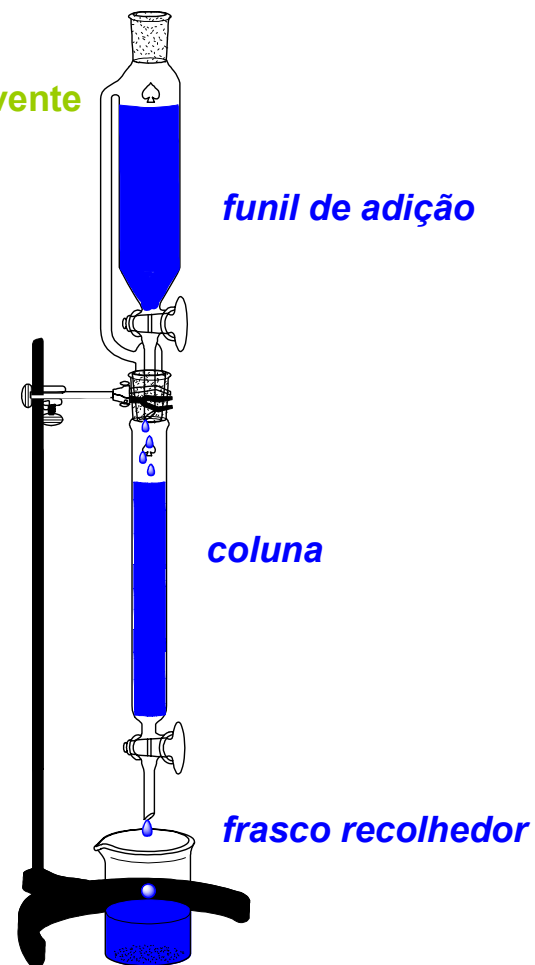
1. preencha a coluna com a fase estacionária



2. Aplique a amostra

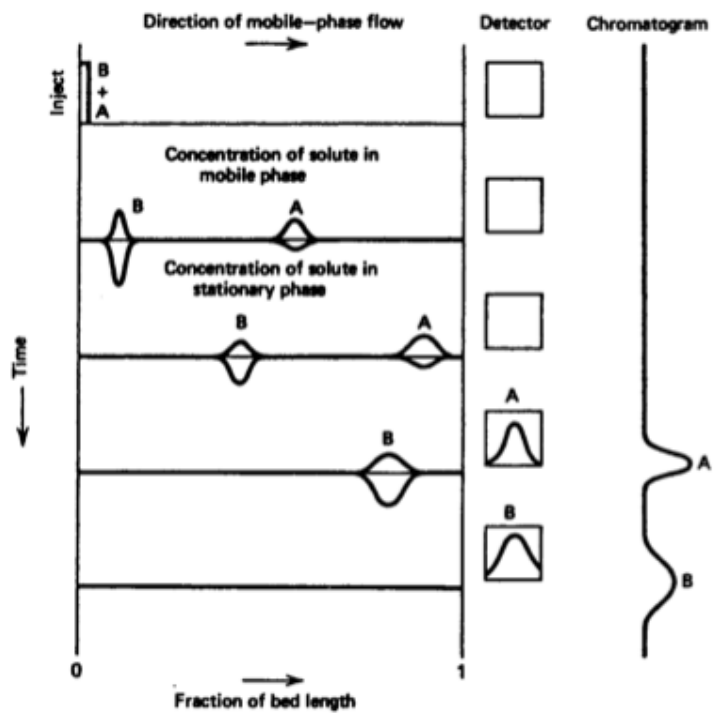


3. Corra o solvente

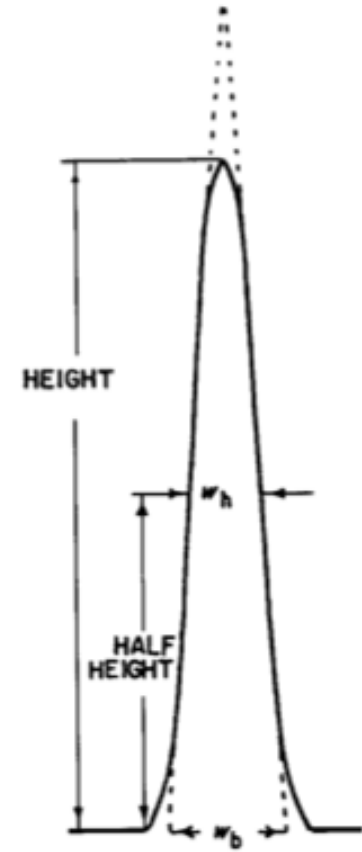
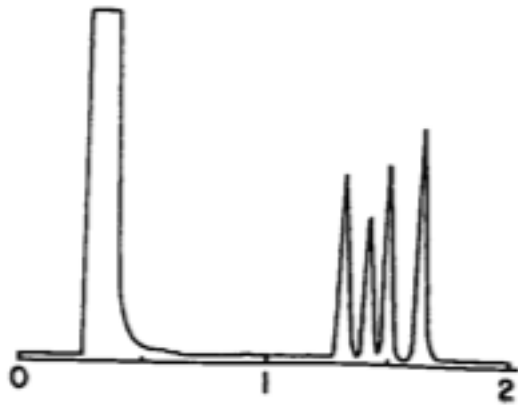


Fase estacionária: sílica (SiO_2), alumina (Al_2O_3), celulose, etc.
Fase móvel: solvente.
Força motriz: gravidade.

Cromatografia

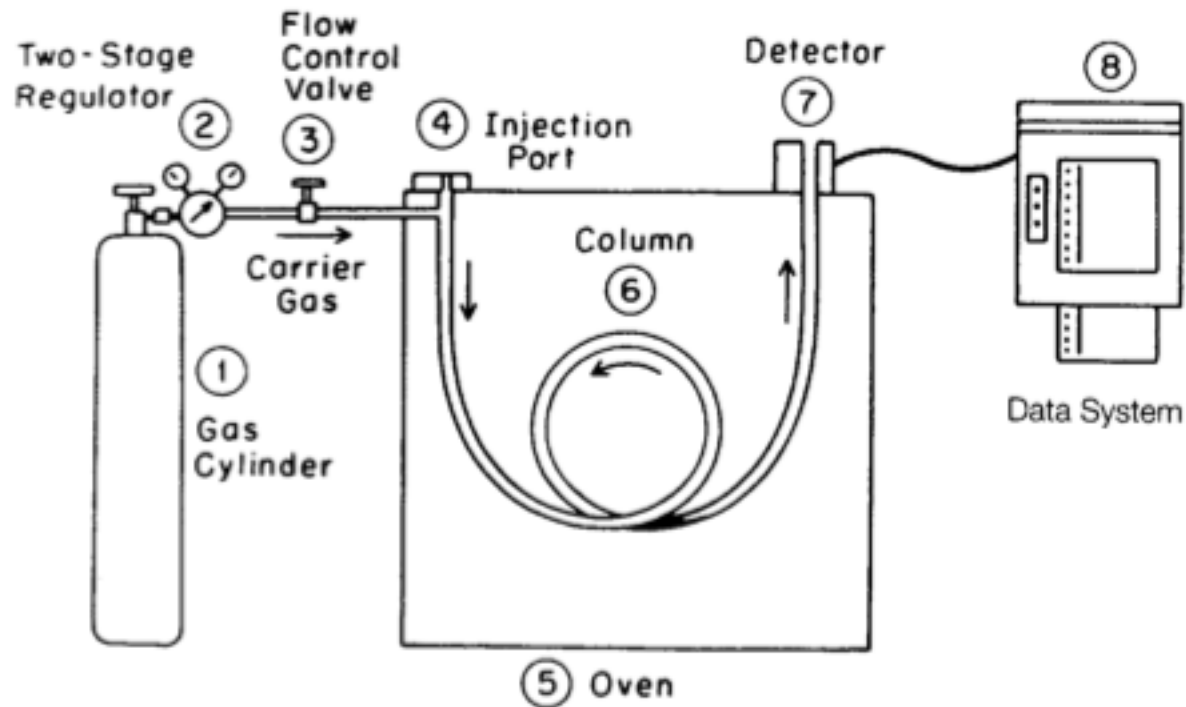


Cromatograma

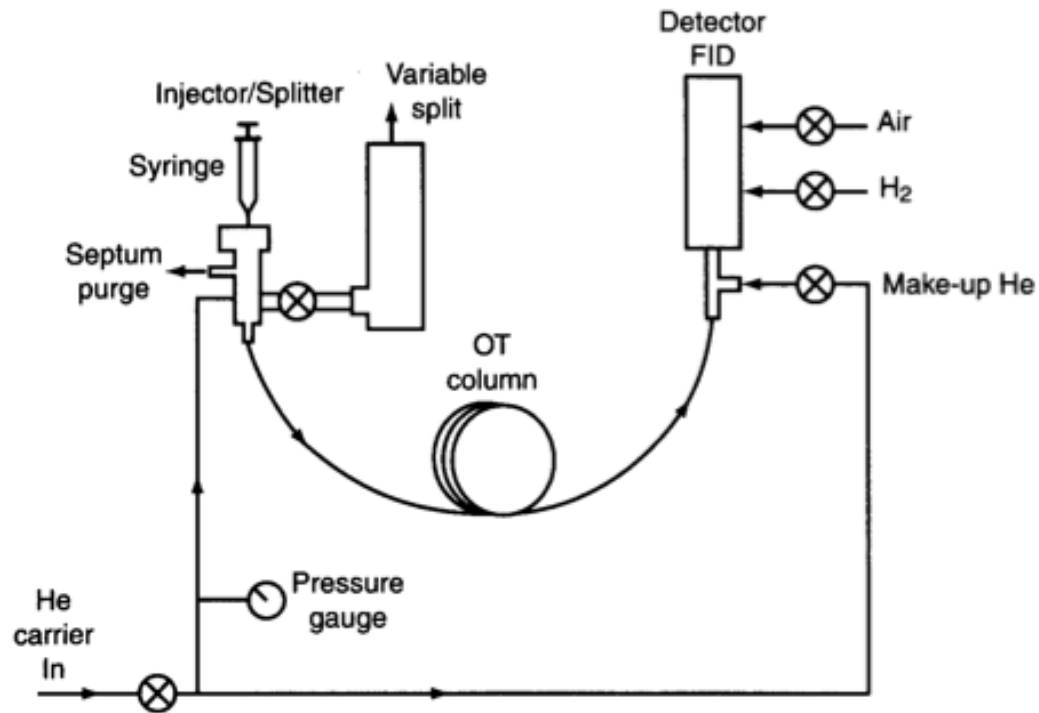


Cromatografia a Gás (ou Gás-Líquido)

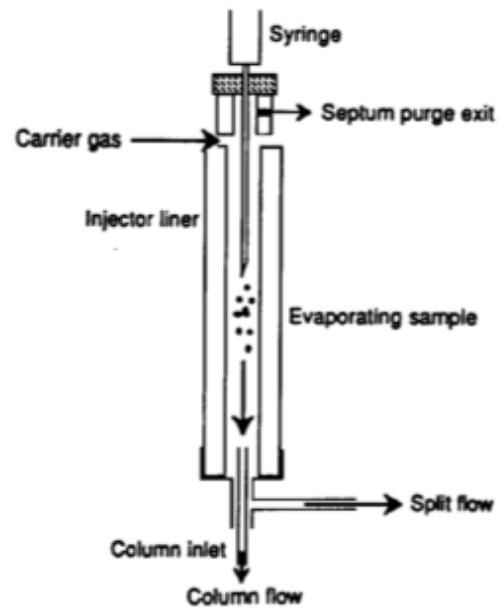
- Cromatógrafo a gás



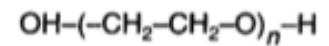
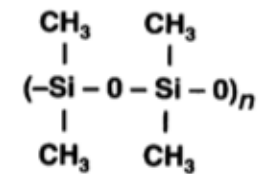
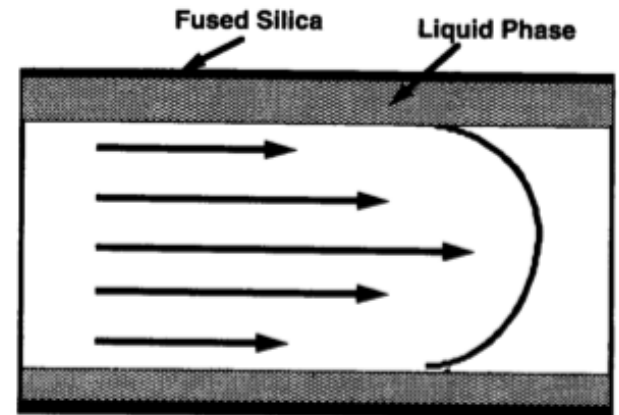
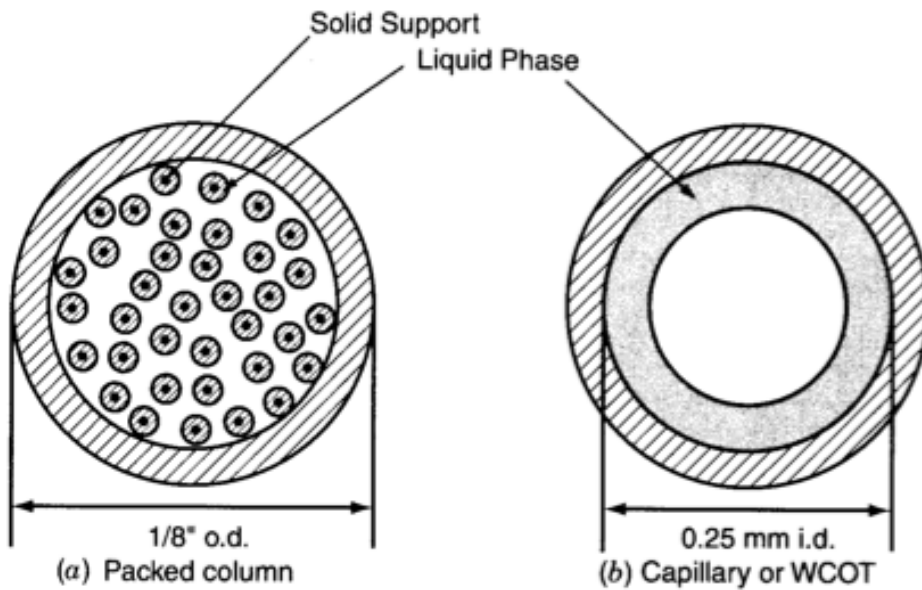
Cromatógrafo a Gás com Detetor FID



Injetor



Columnas

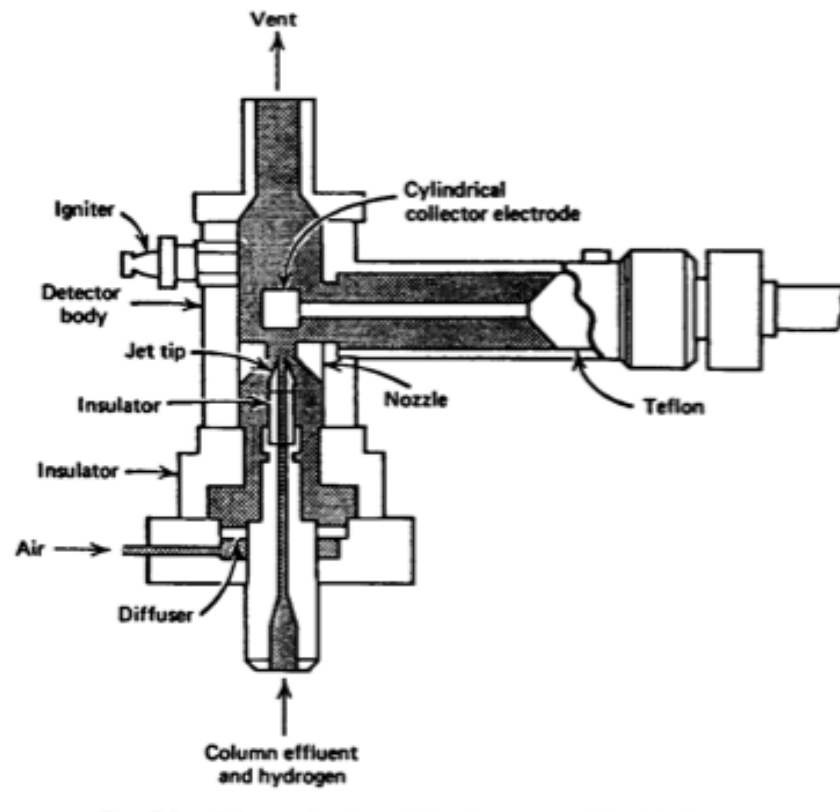


Detetores

TABLE 7.1 Common Commercially Available Detectors

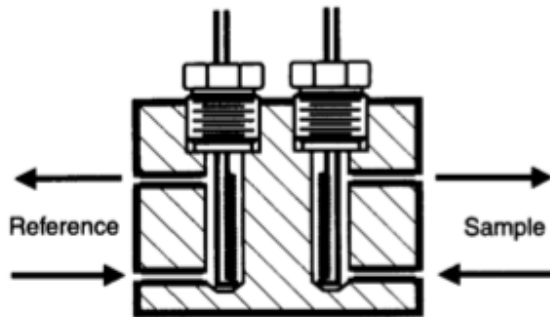
Name	Selective*	References
1. Flame Ionization Detector (FID)	No	1, 2, 3, 5, 12, 13
2. Thermal Conductivity Detector (TCD) (Katharometer)	No	1, 3, 5
3. Electron Capture Detector (ECD)	X	1, 2, 3, 5
Other ionization type detectors		
4. Nitrogen/Phosphorous Detector (NPD); Alkali Flame Ionization Detector (AFID); Thermionic Ionization Detector (TID)	N, P, X	1, 2, 3, 5
5. Photoionization Detector (PID); Discharge Ionization Detector (DID)	Aromatics	2, 5
6. Helium Ionization Detector (HID)	No	1, 2, 3, 5
Emission type detectors		
7. Flame Photometric Detector (FPD)	S, P	1, 2, 3, 5
8. Plasma Atomic Emission (AED)	Metals, X, C, O	2, 4, 5
Electrochemical detectors		
9. Hall Electrolytic Conductivity (HECD)	S, N, X	1, 2, 5
Other types of detector		
10. Chemiluminescent	S	2, 4
11. Gas Density Detector (GADE)	No	1, 3, 5
12. Radioactivity Detector	^3H , ^{14}C	3
13. Mass Spectrometer (MS or MSD)	Yes	2, 4
14. Fourier Transform Infrared (FTIR)	Yes	2, 5

* X = Halogen

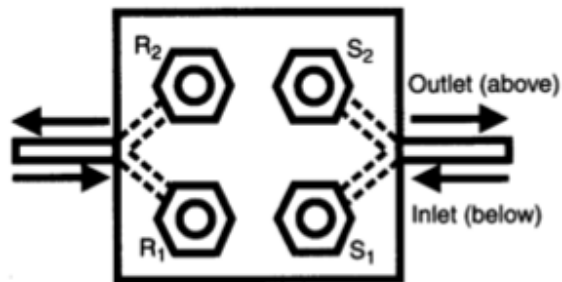


Detetor de Ionização por chama (FID)

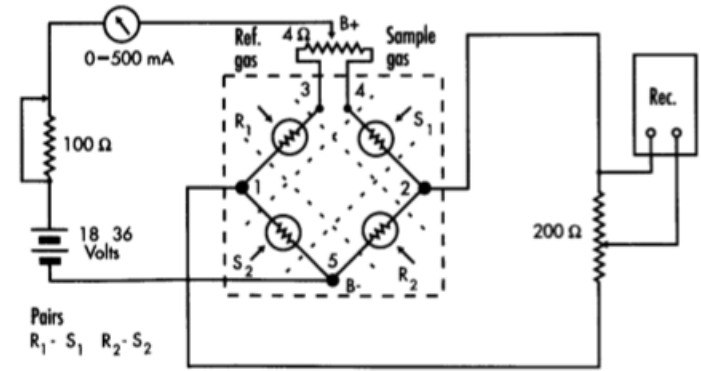
Detetor – Condutividade Térmica (TCD)



(a) Side View



(b) Top View



Circuitry for 4-filament cells