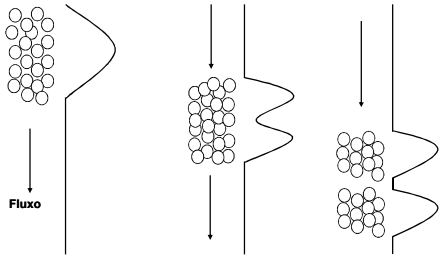
CROMATOGRAFIA GASOSA

Profª Drª Maria Eugênia Queiroz Nassur

Cromatografia Gasosa

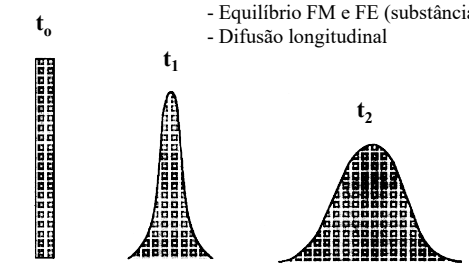
- Método físico-químico de separação dos diferentes componentes da amostra baseado na distribuição destes entre uma fase estacionária (sorção) e uma fase móvel (gasosa).

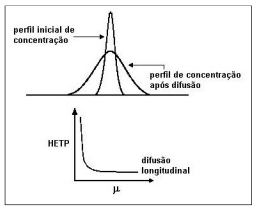
Separação cromatográfica

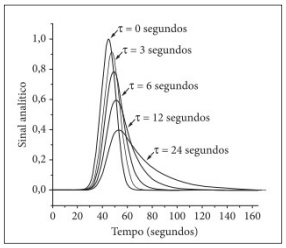


ALARGAMENTO DE UMA BANDA CROMATOGRÁFICA

- Equilíbrio FM e FE (substância)
- Difusão longitudinal





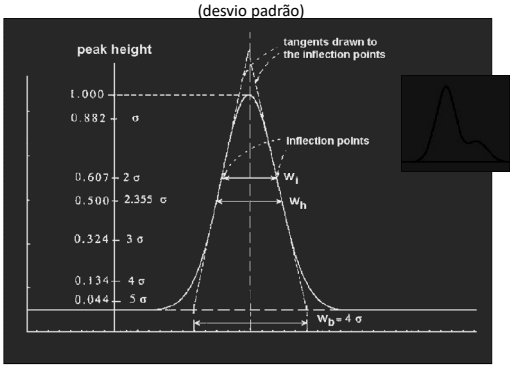


$\mu = L/t_M$

μ - velocidade linear média da fase móvel

Características de um Pico Gaussiano

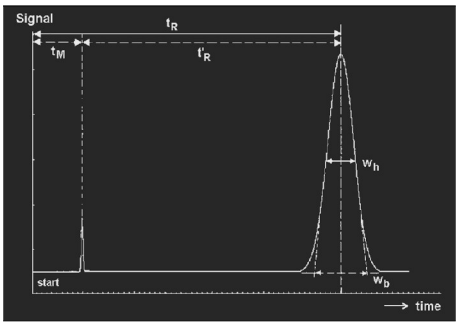
(desvio padrão)



Cromatograma Esquemático

Forma Gaussiana

t'_R ajustado



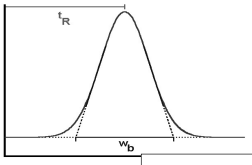
Detector	Recommended Dead Volume Compounds
FID	methane, propane, butane
TCD	air*, methane, butane
NPD	acetonitrile headspace vapors, air*
ECD	air*, methylene chloride headspace vapors
ELCD	dichlorodifluoromethane or methylene chloride headspace vapors
MS	air*, propane, butane, argon
PID	acetylene ethylene

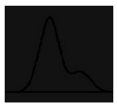
* should be used only at lower oven temperatures

1. EFICIÊNCIA DA COLUNA (picos mais estreitos)



Cada "estágio" equilíbrio de distribuição - **PRATO TEÓRICO**



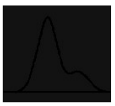


$$N=\left(\frac{t_R}{\sigma}\right)^2 \quad N=16\left(\frac{t_R}{w_b}\right)^2 \quad N=5{,}54\left(\frac{t_R}{w_h}\right)^2$$

Fatores que afetam N

- L (comprimento da coluna)
- Diâmetro interno
- Temperatura
- Volume da amostra

- Picos mal resolvidos
- N = 5,545 (tr/wh)^2



1. Eficiência da Coluna

Outra maneira de medir-se a eficiência é através da Altura Equivalente a um Prato Teórico, *H*:

$$H=\frac{L}{N} \quad \text{(cm)}$$

Comparação entre diferentes colunas

☞ *H* é o comprimento necessário de uma coluna para gerar um prato teórico. Quanto maior *N* menor será *H* e mais eficiente a coluna.

2. Resolução, R_S

$$R_S=\frac{t_{R2}-t_{R1}}{\frac{1}{2}(w_{b1}+w_{b2})}$$

R_S = Resolução entre dois picos; t_{R1} = Tempo de retenção do pico 1; t_{R2} = Tempo de retenção do pico 2; w_{b1} = Largura do pico 1 na base; w_{b2} = Largura do pico 2 na base.

2. Resolução, R_S

Para 2 picos adjacentes $w_{b1} \approx w_{b2}$, podendo-se escrever:

$$R_S = \frac{t_{R2} - t_{R1}}{w_{b2}} = \frac{\Delta t_R}{w_{b2}}$$

Rs = 1,25 (fins quanti)

Rs > 1,5 (completa)

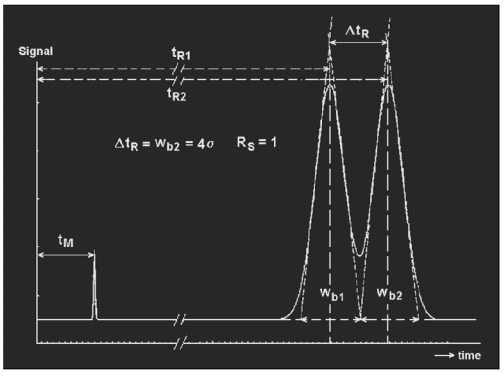
R_S valerá 1 quando $\Delta t_R = w_{b2}$

% Separação em função de R_S

R_S	0,50	0,90	1,00	1,10	1,25	1,50	2,00	2,50
%	68,28	92,82	95,44	97,22	98,76	99,73	99,99	100,00
Δt_R (em σ)	2,0	3,6	4,0	4,4	5,0	6,0	8,0	10,0

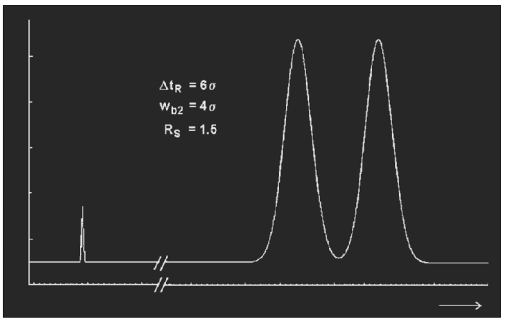
13

2. Resolução, R_S



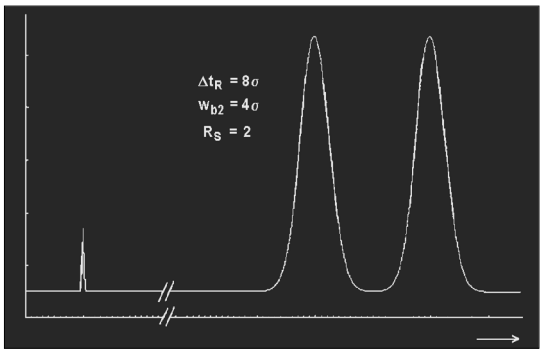
14

2. Resolução, R_S



15

2. Resolução, R_S



16

3. Fator de Retenção, *k*

$$k = \frac{t_R - t_M}{t_M} \qquad k = \frac{t'_R}{t_M}$$

t_M = tempo que uma molécula da fase móvel passa na coluna; denominado de "hold-up time", tempo morto ou tempo de retenção de um soluto não retido.

t'_R = tempo de retenção ajustado ou tempo que o soluto passa na fase estacionária.

FATOR DE RETENÇÃO ($1 \leq k \leq 10$)

Análise de múltiplos componentes ($0{,}5 \leq k \leq 20$)

17

4. Retenção Relativa, α seletividade da fase líquida

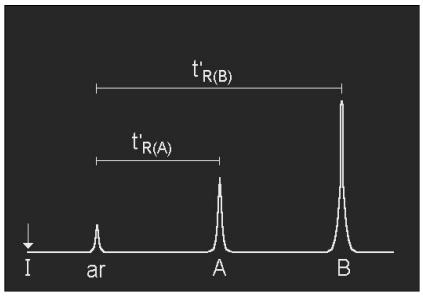
O fator de separação, α, é a relação existente entre o tempo que dois picos permanecem na fase estacionária.

$$\alpha = \frac{t'_{R2}}{t'_{R1}} = \frac{K_2}{K_1}$$

☞ Quanto maior o valor de α, mais seletiva será a fase estacionária para aquele par de compostos.

18

4. Retenção Relativa, α



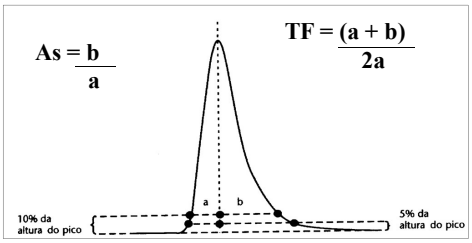
19

FATOR DE ASSIMETRIA

$$As = \frac{b}{a}$$

FATOR DE ALARGAMENTO

$$TF = \frac{(a + b)}{2a}$$



20

CROMATOGRAFIA GASOSA

GASES OU SUBSTÂNCIAS VOLATILIZÁVEIS

PONTOS DE EBULIÇÃO de até 300ºC

TERMICAMENTE ESTÁVEIS

DEMAIS SUBSTÂNCIAS

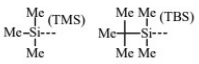
REAÇÕES DE DERIVATIZAÇÃO

(reações de alquilação, acetilação e sililação)

21

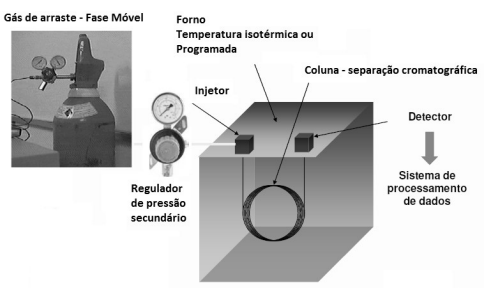
GC analysis, compounds containing functional groups with active hydrogens (e.g., -COOH, -OH, - NH and –SH)

intermolecular hydrogen bonds affects the inherent volatility of compounds containing them, their tendency to interact deleteriously with column packing materials and their thermal stability.

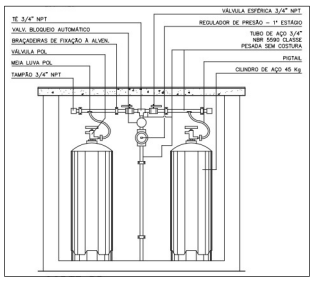


22

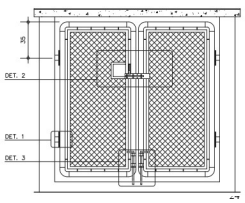
Esquema de um cromatógrafo a gás



23

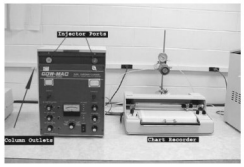


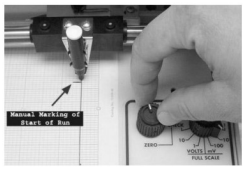




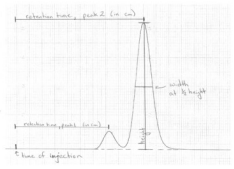
ABRIGO DOS GASES

Cromatógrafo a gás

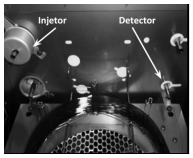


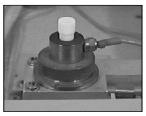


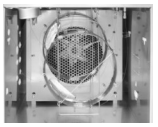


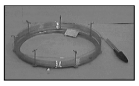


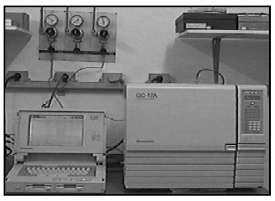
25

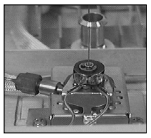




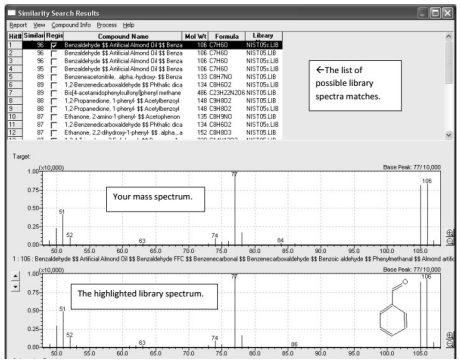








26



27



Gás de arraste – Fase Móvel

INERTE Não deve interagir com a amostra, fase estacionária ou superfícies do instrumento.

PURO Deve ser isento de impurezas que possam degradar a fase estacionária.

ADEQUADO AO DETECTOR

CUSTO E DISPONIBILIDADE

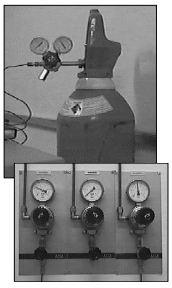
Impurezas típicas em gases e seus efeitos:

H_2O, O_2 → Oxidação FE / hidrólise e quebra de ligações algumas FE

hidrocarbonetos → incompatíveis com ECD → ruído no sinal de FID (estabilidade e resposta)

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Gás de arraste - detector

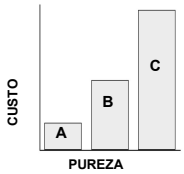


•Detector	Gás de arraste
•Condutividade térmica •(resposta universal)	Hélio, Hidrogênio
•Ionização de chama •(número de átomos C)	Hélio, N_2
•Captura de elétrons •(compostos halogenados)	Nitrogênio
•Espectrômetro de massas	He

29

GÁS DE ARRASTE

CUSTO Gases de altíssima pureza podem ser muito caros.



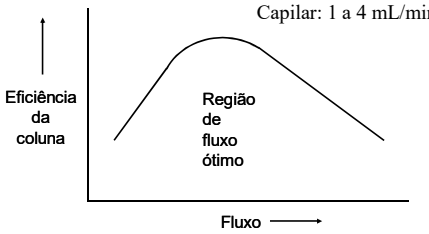
A = 99,995 % (4.5)
B = 99,999 % (5.0)
C = 99,9999 % (6.0)

30

Efeito do fluxo de gás na eficiência de uma coluna

Empacotadas: 30 a 80 mL/min

Capilar: 1 a 4 mL/min



31

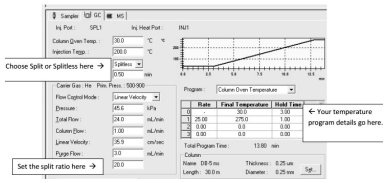
Coluna d.i. (mm)	Espessura do filme (µm)	Fluxo (mL/min)	Velocidade linear média (cm/s)
0,10	0,10	0,2 – 0,5	38
0,22	0,5	0,8 – 2,0	36
0,32	0,5	1,7 – 4,0	34
0,32	1,0	1,7 – 4,0	28
0,53	2,0	30-50	28

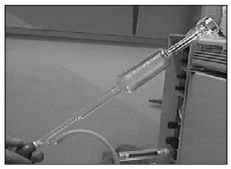
$$\mu = \frac{L\ (cm)}{t_M\ (s)}$$

32

Medida de fluxo do gás de arraste

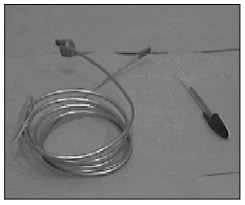




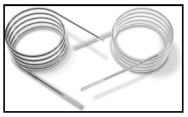


33

Colunas Empacotadas



- Comprimento: 1 a 3 m
- Diâmetro interno: 2 a 4 mm
- Aço inox ou vidro
- Fácil de fazer e usar
- Grande variedade de fases líquidas
- Modesto No. de pratos (máximo 8.000)
- Compostos: µg



$$N=\left(\frac{t_R}{\sigma}\right)^2 \quad N=16\left(\frac{t_R}{w_b}\right)^2 \quad N=5{,}54\left(\frac{t_R}{w_h}\right)^2$$

34

Colunas Empacotadas

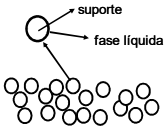
CGS

-ADSORÇÃO

- VOLATILIDADE DIFERENTES

- PEQUENA FAIXA LINEAR

- FE MAIS ESTÁVEL a maiores temperaturas



CGL

- SIMILAR SOLUBILIZA SIMILAR – 95%

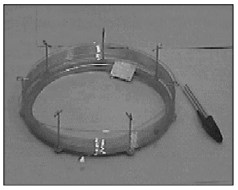
-LIMITE DE TEMPERATURA DAS FASES LÍQUIDAS

Suporte - Terras diatomáceas

Esqueletos silicosos – 900°C – sílica microamorfa

35

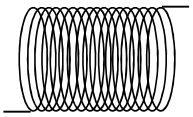
Colunas Capilares



- Comprimento: 5-100 metros
- Diâmetro interno: 100-530 µm
- Parede de sílica fundida
- Grande variedade de fases líquidas
- Separações muito eficientes (100.000 pratos teóricos)
- Compostos: ng, pg

36

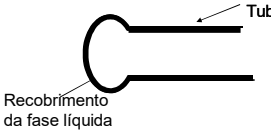
Sílica Fundida



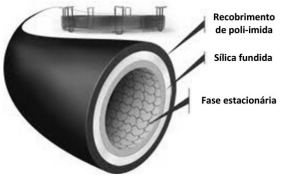
- Flexível
- Cobertura de poliimida
- Bastante inerte

37

WCOT - Wall Coated Open Tubular

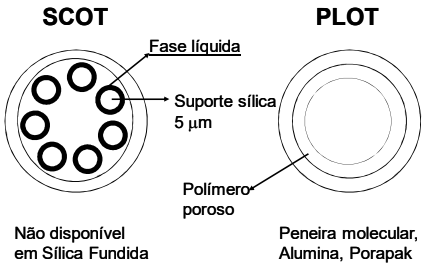


95% das aplicações



38

Outros Tipos de Colunas Capilares



39

Capilar x Empacotada

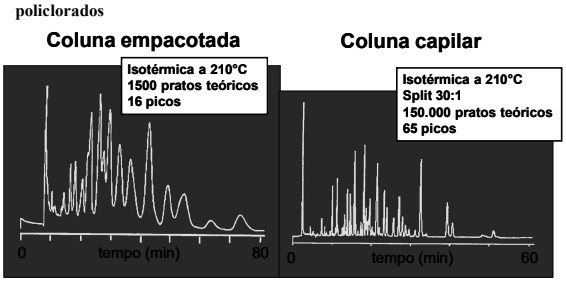


•	Capilar	Empacotada
•Comprimento (L)	60 metros	2 metros
•Pratos teóricos (N/m)	3.000-5.000	2.000
•Pratos totais (L x N/m)	180.000-300.000	4.000

40

COLUNA EMPACOTADA *"VERSUS"* COLUNA CAPILAR

Bifenil policlorados



41

CROMATOGRAFIA GASOSA

GASES OU SUBSTÂNCIAS VOLATILIZÁVEIS

PONTOS DE EBULIÇÃO de até 300°C

TERMICAMENTE ESTÁVEIS

DEMAIS SUBSTÂNCIAS

REAÇÕES DE DERIVATIZAÇÃO

(reações de alquilação, acetilação e sililação)

42

Parâmetros de Injeção

TEMPERATURA DO INJETOR Deve ser suficientemente elevada para que a amostra vaporize-se imediatamente, mas sem decomposição

Regra Geral: T_{inj} **= 40ºC** acima da temperatura de ebulição do componente menos volátil

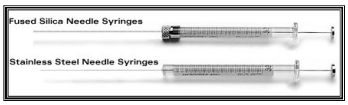
VOLUME INJETADO Depende do tipo de coluna e do estado físico da amostra

COLUNA	*Amostras Líquidas*	*Amostras Gasosas*
empacotada $\varnothing$ *= 3,2 mm (1/4")*	1 μL ... 10 μL	0,1 mL ... 10 mL
capilar $\varnothing$ *= 0,25 mm*	0,001 μL ... 0,5 μL	0,1 μ L ... 10 μ L

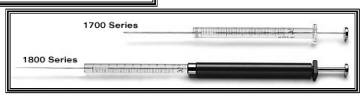
Sólidos: convencionalmente se dissolve em um solvente adequado e injeta-se a solução

43

Microsseringas









Gases: "gas tight"

Vedação especial

44

INJETOR AUTOMÁTICO



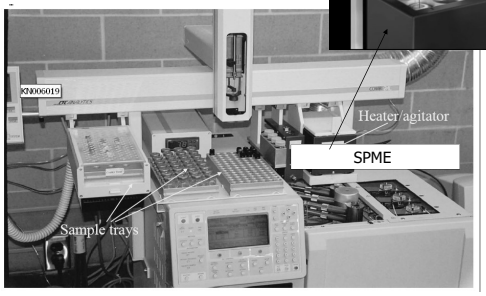




45

SPME



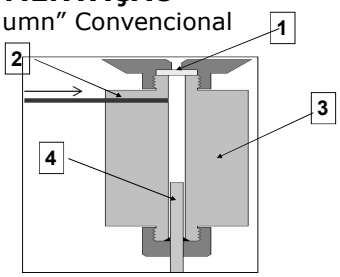


Combi Pal

46

INSTRUMENTAÇÃO

Injetor "on-column" Convencional

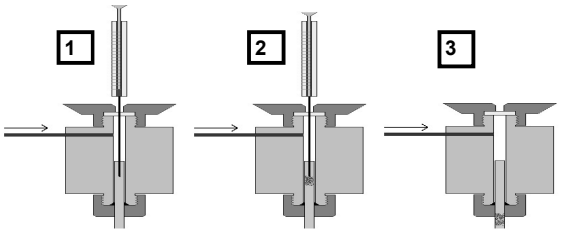


1 - Septo (silicone, PTFE - politetrafluoretileno) – vazamento e sangria – trocas periódicas
2 - Alimentação de gás de arraste
3 - Bloco metálico aquecido
4 - Coluna cromatográfica

47

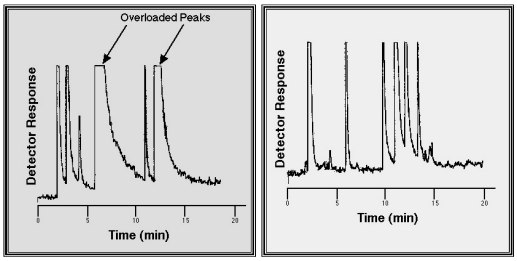
INSTRUMENTAÇÃO

Injeção "on-column" de líquidos



1 - Ponta da agulha da microsseringa é introduzida no início da coluna.
2 - Amostra injetada e vaporizada instantaneamente no início da coluna.
3 - "Plug" de vapor de amostra forçado pelo gás de arraste a fluir pela coluna.

48

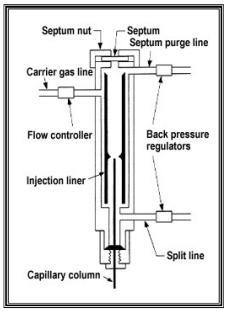


LIMITE DA CAPACIDADE DA COLUNA - volume FE

- QUANTIDADE INJETADA < 1 μL
- COLUNA CAPILAR 25 m – 10 mg FL
- COLUNA EMPACOTADA 10 m – 2 a 3 g FL

49

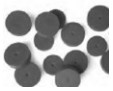
Injetor split/splitless







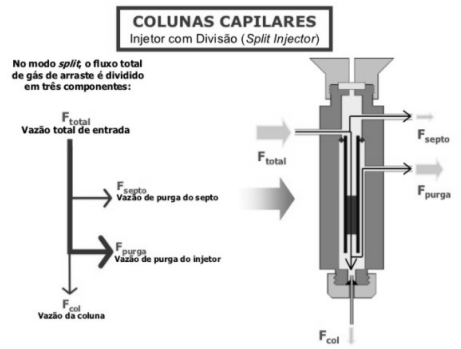
Ambiente inerte no injetor



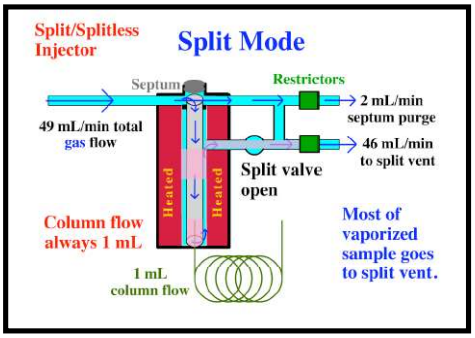
Silicone (trocas periódicas)

PTFE (politetrafluoretileno)

50

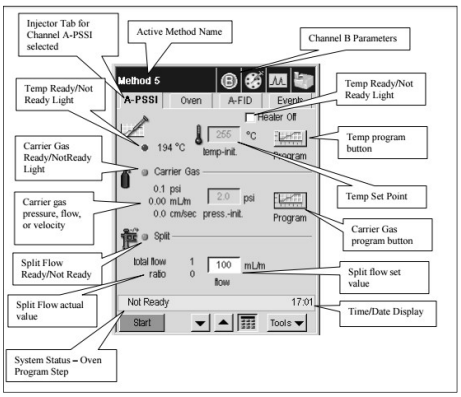


51



Analitos presentes em altas concentrações

52



53

INJETOR SPLIT

- TÉCNICA SIMPLES
- CONTROLAR A RAZÃO SPLIT
- SEPARAÇÕES DE ALTA RESOLUÇÃO
- AMOSTRAS LIMPAS E SUJAS
- LIMITAÇÃO – ANÁLISE DE TRAÇOS

54

Fluxo do Split

A quantidade de amostra introduzida na coluna depende do volume injetado e da razão de divisão (fração da amostra não descartada):

$$SR = 1 : \left[\frac{(F_{purga} + F_{col})}{F_{col}}\right]$$

SR Razão de Divisão
F_{purga} Vazão da Purga do Injetor (split)
F_{col} Vazão na Coluna

EXEMPLO F_{col} = 1.1 mL min^{-1}, F_{purga} = 145 mL min^{-1} ➡ $SR = 1 : \left[\frac{(145 + 1.1)}{1.1}\right]$ ➡ SR = 1 : 133

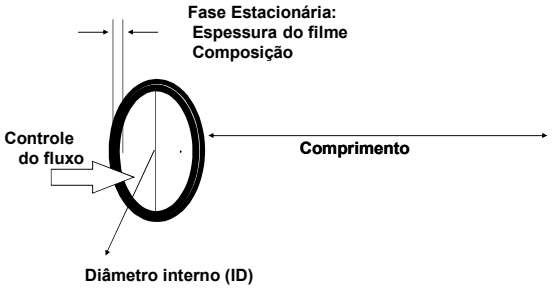
Se V_{inj} = 0.5 µL, o volume de amostra introduzido na coluna é (0.5/133) = 0.0038 µL = 38 nL !!!

VALORES ADMISSÍVEIS TÍPICOS PARA RAZÕES DE DIVISÃO

- Colunas convencionais (d_c = 0.25 - 0.32 mm): 1:50 ... 1:500
- Colunas de diâmetro alto (d_c = 0.53 mm) ou filmes espessos de FE: 1:5 ... 1:50

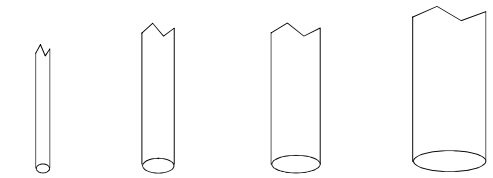
55

Parâmetros Importantes



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Diâmetro da Coluna



0,1 mm (100 µm) | 0,25 mm (250 µm) | 0,32 mm (320 µm) | 0,53 mm (530 µm)

A diminuição do ID aumenta a eficiência da coluna
Picos mais estreitos

57

Eficiência (N) vs Diâmetro Interno (ID)

•I.D. (mm)	H (mm)	Pratos/metro
• 0,53	0,47	2.100
• 0,32	0,29	3.400
• 0,25	0,22	4.500
• 0,10	0,09	11.000

$$H = \frac{L}{N}$$

Diminuindo-se o diâmetro, diminui-se H e aumenta-se a eficiência (N)

58

Diâmetro do Tubo vs. Quantidade de amostra injetada

(25 m; d_f = 0,25 µm)

• I.D. (mm)	ng/pico
• 0,53	2.000
• 0,32	500
• 0,25	100
• 0,10	1

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Diâmetro da Coluna vs

Tempo de Análise

• d_c (µm)	H_{min} (µm)	N_{max} (m)	L (m) para 10^5 pratos	t_R (min)
• 530	450	2200	60	100
• 320	270	3700	33	37
• 250	210	4700	26	22
• 150	130	8000	16	8

Maior transferência de massas

$$N = \left(\frac{t_R}{\sigma}\right)^2 \qquad N = 16\left(\frac{t_{R2}}{w_{b2}}\right)^2 \qquad N = 5{,}54\left(\frac{t_{R2}}{w_h}\right)^2$$

60

Comprimento da Coluna

- $N \alpha L$
- $R \alpha \sqrt{L}$
- $t_R \alpha L$

•Aumentando-se o comprimento da coluna (L) aumenta-se a Resolução (*R*), mas aumenta-se também o tempo de análise.

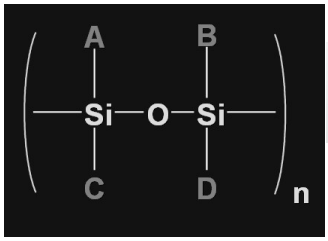
Diâmetro do Tubo (ID) vs Comprimento (L) vs Custo

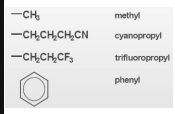
CONCLUSÃO

- 1. ***R*** é proporcional a $\sqrt{L}$
  (2 x ***L*** dará 1,4 x ***R*** => duplicação do tempo → diminui sinal analítico e aumento de sangria => custa mais caro)
- 2. ***R*** é proporcional a
- (I.D./2 dará 1,3 x ***R*** => mais barato)

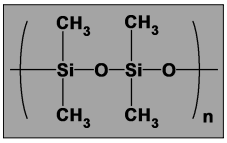
Estrutura Básica dos Silicones

Seletividade passou a ser menos importante



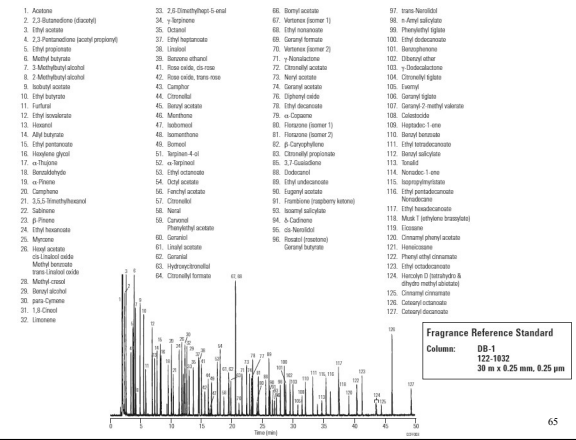


OV-1 (SE-30; SP-2100)*



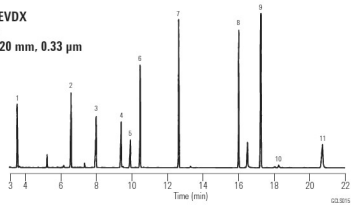
Dimetil Polisiloxano
Não polar (5%)
Fase líquida mais usada em Cromatografia Gasosa
Separação – diferenças nas volatilidades dos compostos

100 - 350°C

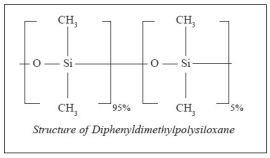


Sedative Hypnotics

Column: **DB-5ms EVDX**
128-8522
25 m x 0.20 mm, 0.33 µm

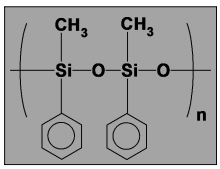


1. Ethchlorvynol
2. Ethinamate
3. Pyrithyldione
4. Talbutal
5. Meprobamate
6. Glutethimide
7. Methaqualone
8. Propiomazine
9. Haloperidol
10. Sulpiride
11. Droperidol



Structure of Diphenyldimethylpolysiloxane

OV-17 (SP-2250)



50% metil, 50% fenil polisiloxano
Semi-polar (21%)
Principais aplicações: drogas, esteróides, carbohidratos

0 - 350°C

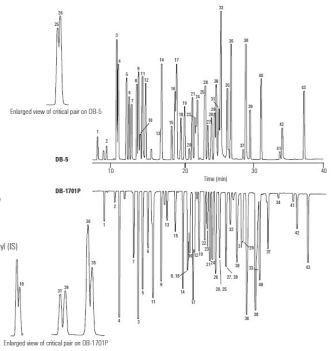
67

Organochlorine Pesticides, DB-5/DB-1701P

Column: DB-5
123-5032
30 m x 0.32 mm, 0.25 µm

Column: DB-1701P
123-7732
30 m x 0.32 mm, 0.25 µm

1. 2,4,5,6-Tetrachloro-m-xylene (IS)
2. α-BHC
3. β-BHC
4. γ-BHC
5. δ-BHC
6. Heptachlor
7. Aldrin
8. Heptachlor epoxide
9. γ-Chlordane
10. Endosulfan I
11. α-Chlordane
12. Dieldrin
13. p,p'-DDE
14. Endrin
15. Endosulfan II
16. p,p'-DDD
17. Endrin aldehyde
18. Endosulfan sulfate
19. p,p'-DDT
20. Endrin ketone
21. Methoxychlor
22. Decachlorobiphenyl (IS)



68

Narcotics and Adulterants

Column: DB-5
123-5032
30 m x 0.32 mm, 0.25 µm

Carrier: Helium at 40 cm/sec, measured at 140 °C

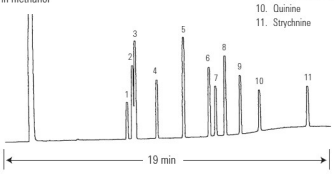
Oven: 140-320 °C at 12 °C/min
320 °C for 4 min

Injection: Split, 250 °C
Split ratio 1:75

Detector: FID, 300 °C
Nitrogen makeup gas at 30 mL/min

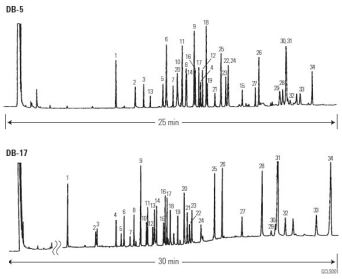
Sample: 1 µL of 0.5 µg/µL each in methanol

1. Caffeine
2. Ketamine
3. Lidocaine
4. Procaine
5. Cocaine
6. Codeine
7. Morphine
8. 6-Acetylcodeine
9. Diacetylmorphone (Heroin)
10. Quinine
11. Strychnine



69

1. Nicotine
2. Phenmetrazine
3. Ibuprofen
4. Procaine
5. Allobarbital
6. Aprobarbital
7. Butabarbital
8. Secobarbital
9. Pentobarbital
10. Phenacetin
11. Amobarbital
12. Benzphetamine
13. Acetaminophen
14. Hydroxyphenamate
15. Dimenhydrinate
16. Meprobamate
17. Benactyzine
18. Hexobarbital
19. Doxylamine
20. Caffeine
21. Chlorpheniramine
22. Methapyrilene
23. Thenyldiamine
24. Phenobarbital
25. Bromopheniramine
26. Chlorcyclizine
27. Cocaine
28. Pyrrobutamine
29. Codeine
30. Diazepam
31. Morphine
32. Hydrocodone
33. Oxymorphone
34. Heroin



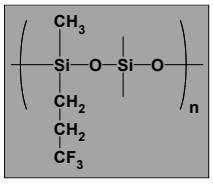
Common Drug Screen

Column: DB-5
122-5032
30 m x 0.25 mm, 0.25 µm

Column: DB-17
122-1732
30 m x 0.25 mm, 0.25 µm

70

OV-210 (QF-1; SP-2401)

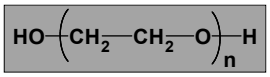


Trifluoropropil polisiloxano
Semi-polar (36%)
Seletivo para cetonas e pesticidas

0 - 350°C

71

Carbowax 20M



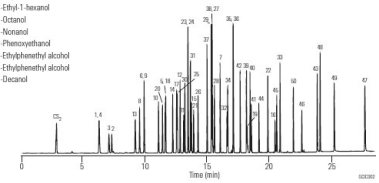
Glicóis poliméricos do polietileno
Polar (55%)
Muito usados para compostos polares

60 - 225°C

72

1. Methanol
2. Ethanol
3. Isopropanol
4. tert-Butanol
5. 2-Propen-1-ol (allyl alcohol)
6. 1-Propanol
7. 2-Propyn-1-ol (propargyl alcohol)
8. sec-Butanol
9. 2-Methyl-3-buten-2-ol
10. Isobutanol
11. 2-Methoxyethanol (methyl Cellosolve)
12. 3-Buten-1-ol
13. 2-Methyl-2-butanol (tert-amyl alcohol)
14. 1-Butanol
15. 2-Buten-1-ol (crotyl alcohol)
16. Ethylene glycol
17. 1-Penten-3-ol
18. 2-Pentanol
19. Glycidol
20. 3-Pentanol
21. 2-Ethoxyethanol (Cellosolve)
22. Propylene glycol
23. 3-Methyl-1-butanol (isoamyl alcohol)
24. 2-Methyl-1-butanol (active amyl alcohol)
25. 4-Methyl-2-pentanol
26. 1-Pentanol
27. 2-Penten-1-ol
28. 3-Methyl-2-buten-1-ol
29. Cyclopentanol
30. 3-Hexanol
31. 2-Hexanol
32. 4-Hydroxy-4-methyl-2-pentanone
33. Furfuryl alcohol
34. cis-3-Hexen-1-ol
35. cis-2-Hexen-1-ol
36. Cyclohexanol
37. 3-Heptanol
38. 2-Heptanol
39. 2-Butoxyethanol (butyl Cellosolve)
40. cis-4-Hepten-1-ol
41. trans-2-Hepten-1-ol
42. 1-Heptanol
43. Benzyl alcohol
44. 2-Ethyl-1-hexanol
45. 1-Octanol
46. 1-Nonanol
47. 2-Phenoxyethanol
48. a-Ethylphenethyl alcohol
49. b-Ethylphenethyl alcohol
50. 1-Decanol

Alcohols II
Column: **DB-WAXetr**
123-7354
50 m x 0.32 mm, 1.00 µm



73

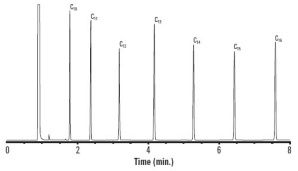
Figure 1: Boiling Point Elution Order for Homologous Series

Column: **DB-1, 15 m x 0.25 mm id, 0.25 µm**

Carrier: Helium at 30 cm/sec

Oven: 60 °C for 1 min, 60-180 °C at 20 °C/min

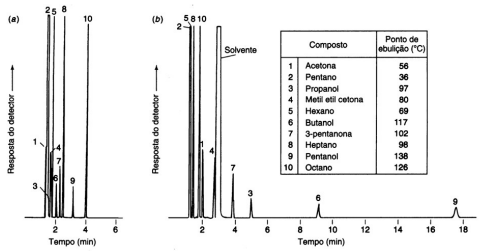
	Boiling Point (°C)
1. n-Decane (C_{10})	174
2. n-Undecane (C_{11})	196
3. n-Dodecane (C_{12})	216
4. n-Tridecane (C_{13})	234
5. n-Tetradecane (C_{14})	253
6. n-Pentadecane (C_{15})	268
7. n-Hexadecane (C_{16})	287



Homologous series of hydrocarbons. The solutes elute in order of their increasing boiling points; however, the peaks are not spaced in proportion to their respective boiling points.

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a) Polidimetilsiloxano b) polietilenoglicol
30 m x 0,32 mm x 0,1 µm



	Composto	Ponto de ebulição (°C)
1	Acetona	56
2	Pentano	36
3	Propanol	97
4	Metil etil cetona	80
5	Hexano	69
6	Butanol	117
7	3-pentanona	102
8	Heptano	98
9	Pentanol	138
10	Octano	126

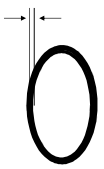
75

Espessura do Filme

- "Espesso" ≥ 0,3 µm
- "Padrão 0,1 µm - 0,3 µm
- "Fino" ≤ 0,1 µm

76

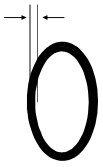
Filme "Fino" (≤ 0,1 µm)



- **Vantagens**
  - Maior eficiência.
  - Análises em temperaturas menores - análise rápida.
  - Menor sangria (eluição da fase estacionária)
- **Limitações**
  - Menor capacidade.
  - Ruim para análise de traços.

77

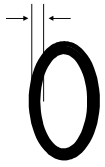
Filme Padrão (> 0,1 µm < 0,3 µm)



- **0,25 µm: bom começo.**
- **Baixa sangria.**
- **Boa resolução, boa capacidade**.

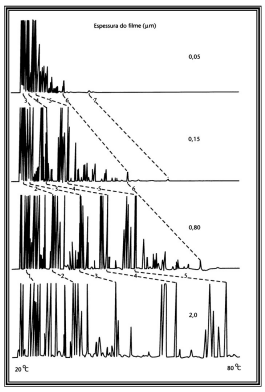
78

Filme Espesso (≥ 0,3 μm)



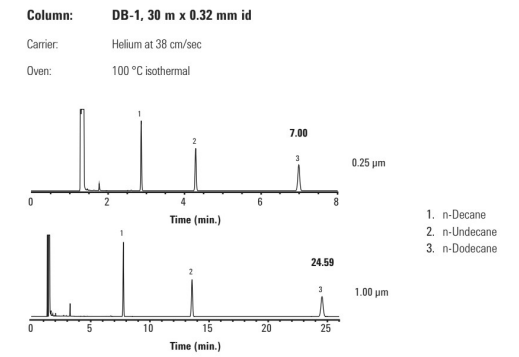
- **Vantagens**
  - **Aumenta retenção para voláteis.**
  - **Aumenta capacidade**
- **Limitações**
  - **Menos eficiente.**
  - **Requer maior temperatura.**
  - **Maior sangria.**

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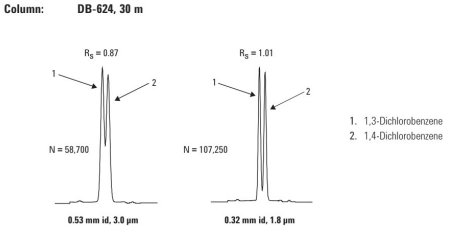


Componentes da gasolina

80

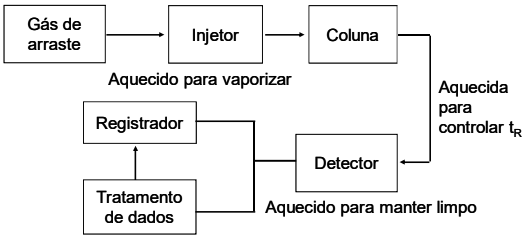


81



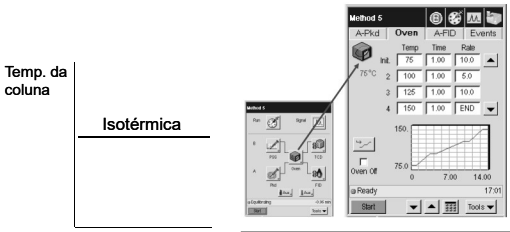
82

Zonas Aquecidas



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Isotérmica x Programação de temperatura

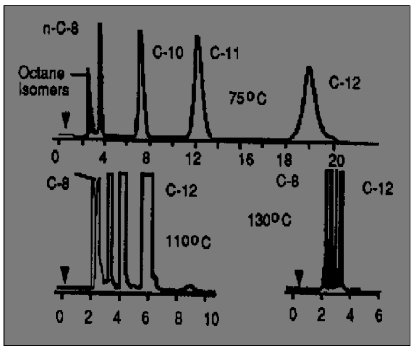


ISOTÉRMICA: amostras simples, poucos componentes
PE próximos (# menor 100ºC)
TEMP: 20 a 50º C abaixo PE dos analitos

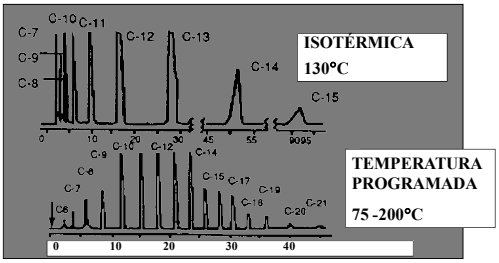
Programação Temp: amostras complexas, ampla faixa de ebulição
Ti: 50ºC abaixo PE mais volátil
Tf: acima PE menos volátil

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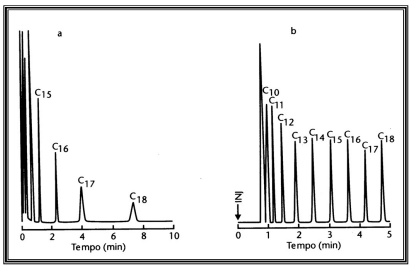
Efeito da Temperatura da Coluna

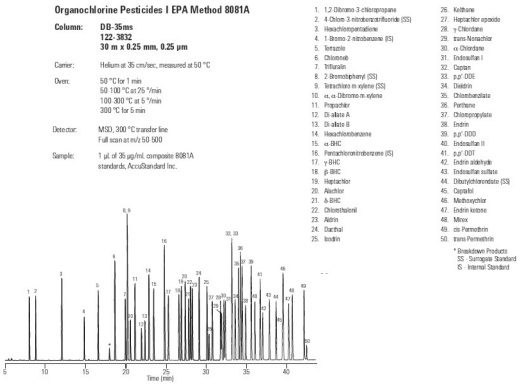


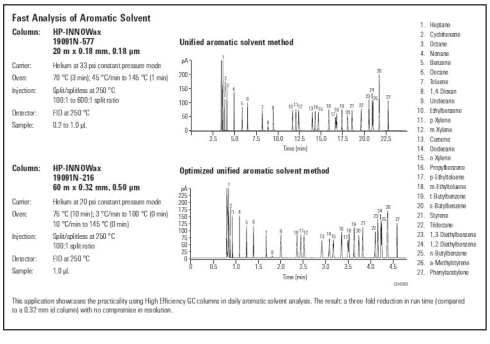
Efeitos da Temperatura

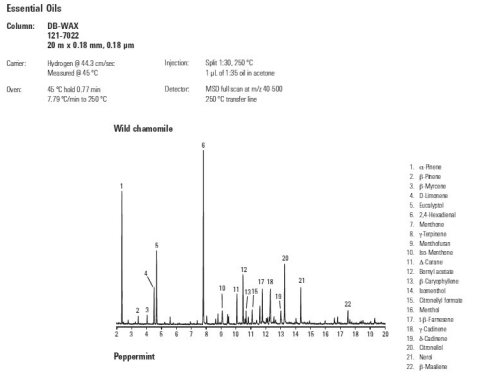


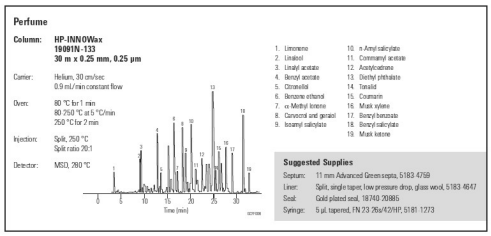
SEPARAÇÃO DE ALCANOS
(a) isotérmica (b) programada



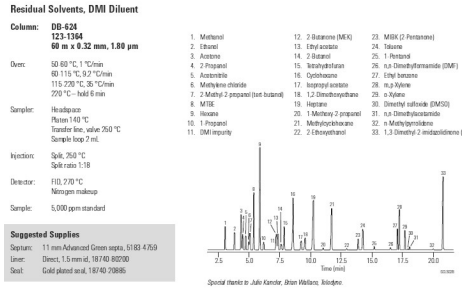






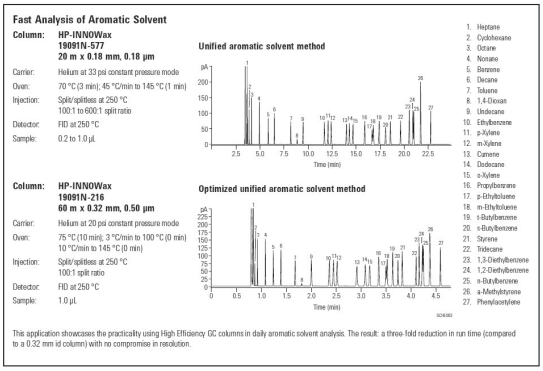


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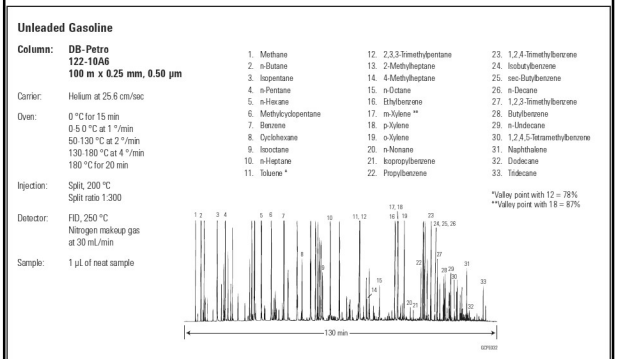
Special thanks to Julio Kanotar, Brian Wallace, Teledyne.

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This application showcases the practicality using High Efficiency GC columns in daily aromatic solvent analysis. The result: a three-fold reduction in run time (compared to a 0.32 mm id column) with no compromise in resolution.

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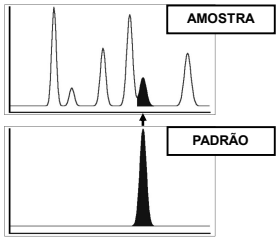
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ANÁLISE QUALITATIVA

Tempos de Retenção

Interações analito / FE
Pressão de vapor do analito
Condições operacionais (T_{COL}, F_C ...)

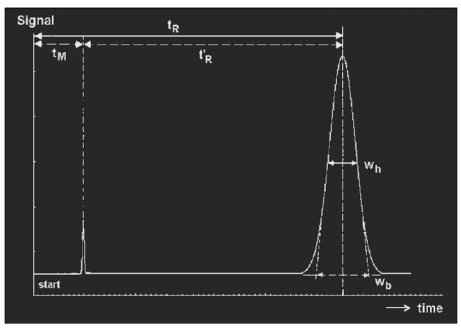
Fixas as condições operacionais, o tempo de retenção ajustado de um analito é uma constante



Comparação de cromatogramas da amostra e de uma solução padrão do analito suspeito

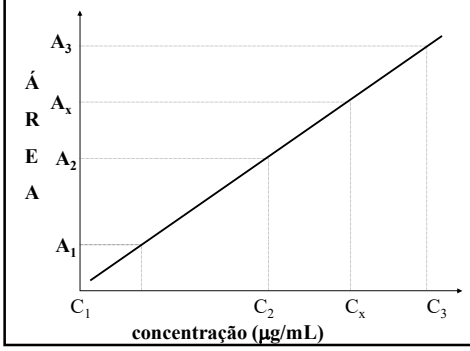
95

Análise quantitativa



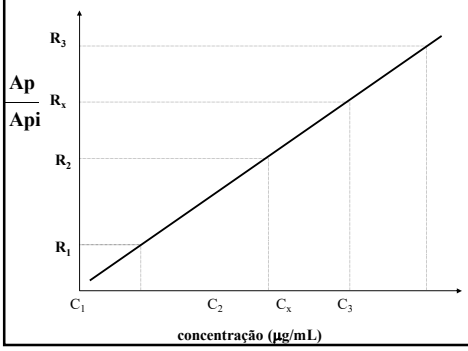
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MÉTODO CALIBRAÇÃO EXTERNA



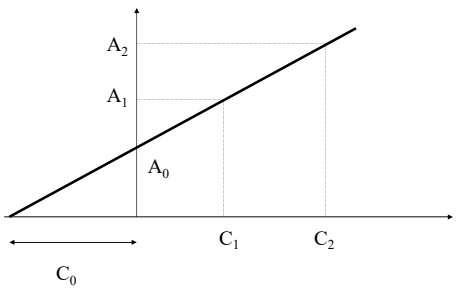
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MÉTODO PADRONIZAÇÃO INTERNA



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MÉTODO DA ADIÇÃO DO ANALITO
Extrapolação linear por adição de analito



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