



TSK-GEL 色谱柱应用数据集 (2)

— 医药品和化妆品 —



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1. 关于TSK-GEL反相色谱分析柱

硅胶基质反相色谱柱

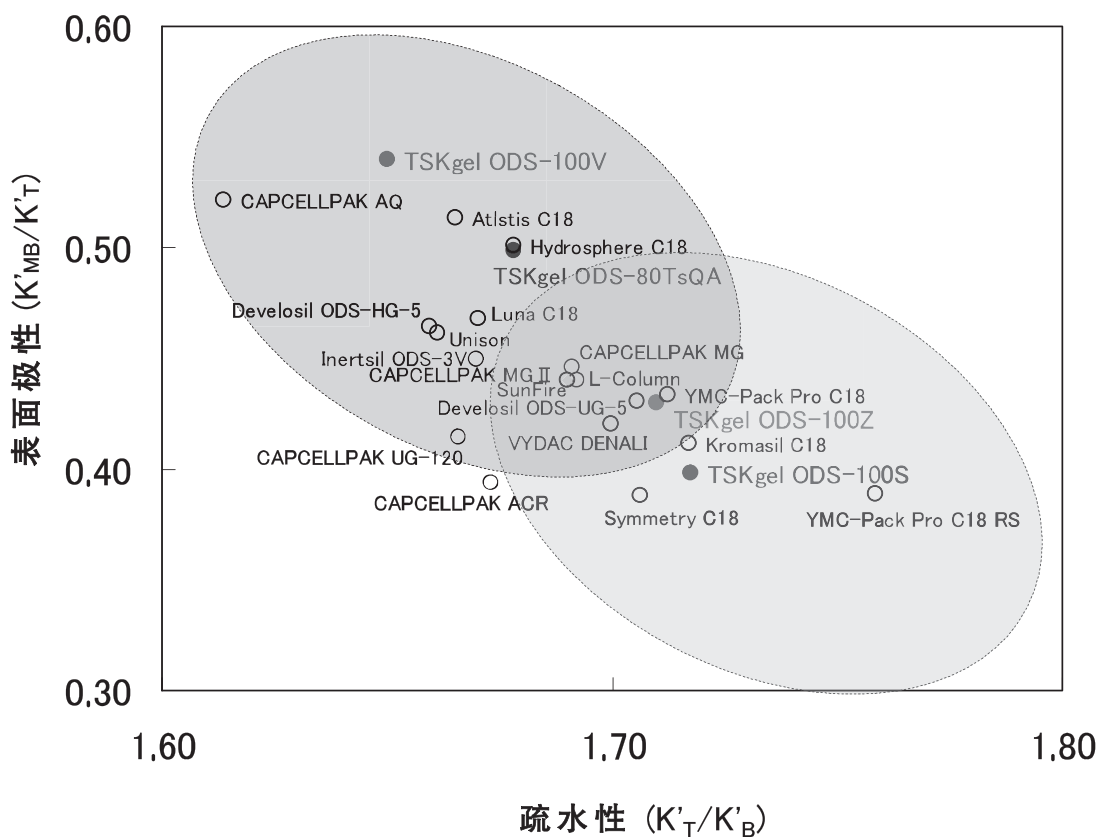
色谱柱	官能团/键合方式	封端	含碳量 (%)	粒径 (μm)	孔径 (nm)	特长
ODS-100Z	C18alkyl, monomeric	Y	20	5,3	10	首选色谱柱
ODS-100V	C18alkyl, monomeric	Y	15	5,3	10	首选色谱柱
ODS-120T	C18alkyl, polymeric	Y	22	5	12	
ODS-120A	C18alkyl, polymeric	N	22	5	12	芳香族系样品
ODS-100S	C18alkyl, polymeric	Y	19	5	10	耐碱能力强 (适用 pH 范围宽)
ODS-80Ts	C18alkyl, monomeric	Y	15	5	8	
ODS-80Ts QA	C18alkyl, monomeric	Y	15	5	8	适用于认证实验的色谱柱
ODS-80T _M	C18alkyl, monomeric	Y	15	5	8	
Super-ODS	C18alkyl, polymeric	Y	8	2	10	高速高分辨率 ODS 色谱柱
OligoDNA RP	C18alkyl, monomeric	N	11	5	25	寡聚核酸分析用 ODS 色谱柱
Octyl-80Ts	C8alkyl, monomeric	Y	10	5	8	
Super-Octyl	C8alkyl, polymeric	Y	5	2	10	高速高分辨率 C8 色谱柱
Super-Phenyl	phenyl, polymeric	Y	3	2	10	高速高分辨率苯基色谱柱 与 C18, C8 有相同的分离选择性
CN-80Ts	CN, monomeric	Y	9	5	8	氰丙酰基反相色谱柱
TMS-250	C1alkyl, monomeric	Y	5	10	25	蛋白质分离用反相色谱柱

聚合物基质反相色谱柱

色谱柱	官能团/键合方式	封端	含碳量 (%)	粒径 (μm)	孔径 (nm)	特长
Octadecyl-2PW	C18alkyl, monomeric	—	—	5	10-20	多肽分离
Octadecyl-4PW	C18alkyl, monomeric	—	—	7	50	高分子量多肽分离
Phenyl-5PW RP	C18alkyl, monomeric	—	—	10	100	蛋白质分离
Octadecyl-NPR	C18alkyl, monomeric	—	—	2.5	nonporous	无孔型 (或非多孔型)

ODS 反相色谱柱比较示意图

(疏水性及表面极性)



K'_B : 苯的容量因子

K'_T : 甲苯的容量因子

K'_{MB} : 甲基苯甲酸的容量因子

2. 医药品分析应用数据

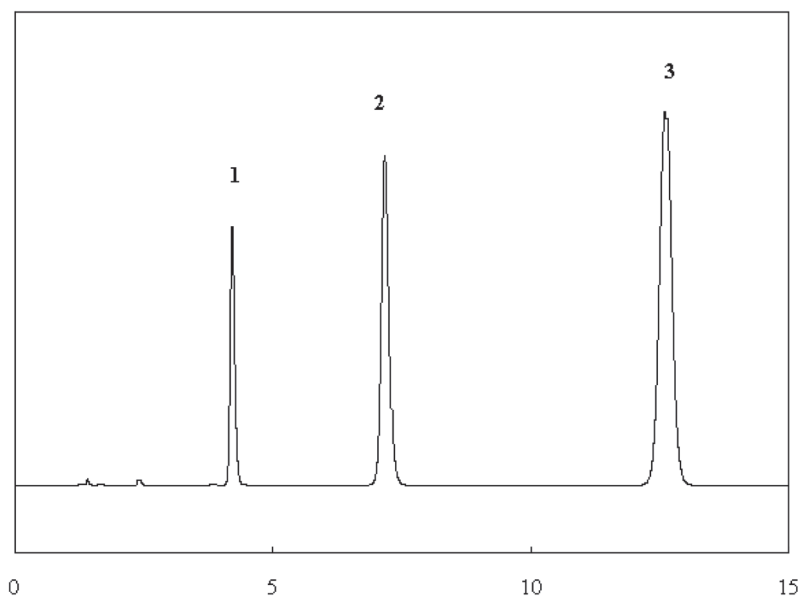
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2 - 1 精神神经症药物的分离(1)



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)

Eluent : 50 mmol/L
sodium phosphate buffer (pH2.2)
/CH₃CN = 60/40

Flow rate : 1.0 mL/min

Detection : UV (230 nm)

Temperature : 40 °C

Inj. volume : 10 μ L

Concentration: 10 mg/L

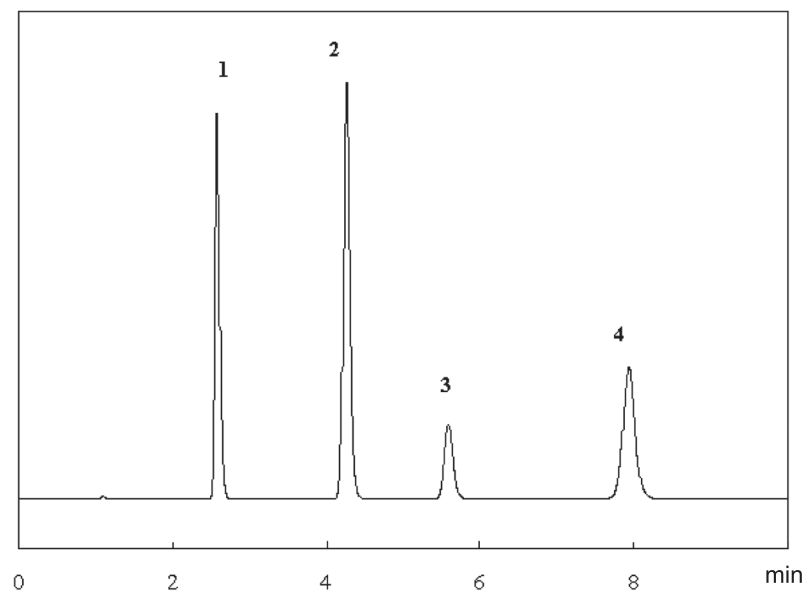
Samples

1 : haloperidol

2 : reserpine

3 : diazepam

2 - 2 精神神经症药物的分离(2)



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)

Eluent : 25 mmol/L sodium phosphate
buffer (pH7.5) /CH₃CN = 60/40

Flow rate : 1.0 mL/min

Detection : UV (230 nm)

Temperature : 40 °C

Inj. volume : 10 μ L

Samples

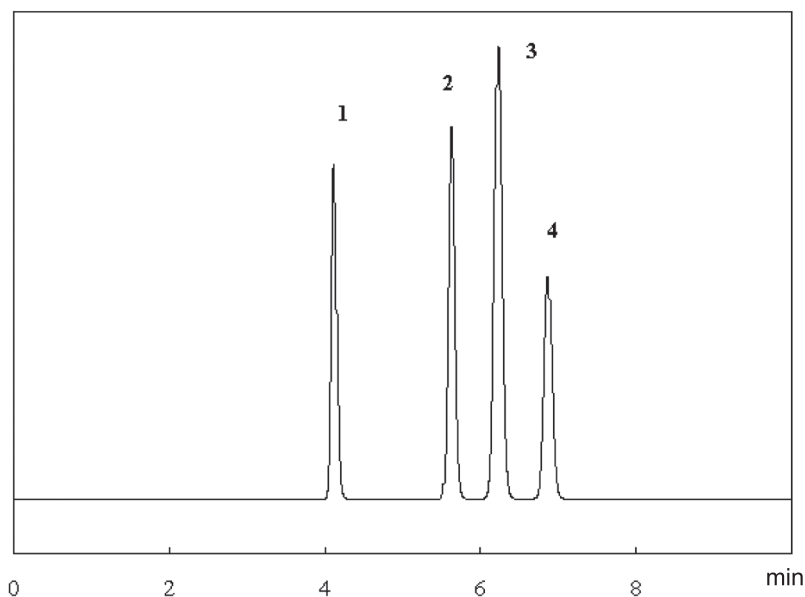
1 : nitrazepam (10 mg/L)

2 : reserpine (10 mg/L)

3 : haloperidol (3 mg/L)

4 : diazepam (3 mg/L)

2 - 3 非类固醇类抗炎症药物的分离

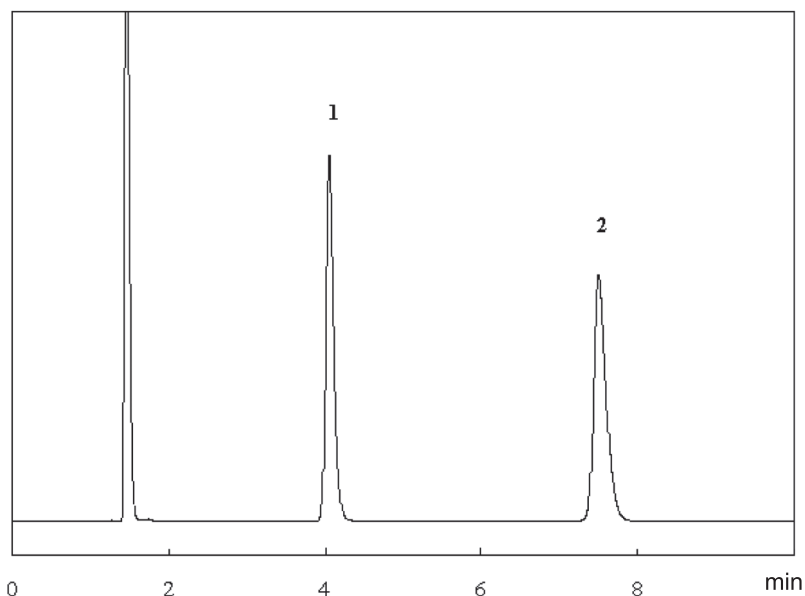


Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 25 cm)
Eluent : H₃PO₄ in (CH₃CN/H₂O = 60/40),
pH 2.2
Flow rate : 1.0 mL/min
Detection : UV (230 nm)
Temperature : 40 °C
Inj. volume : 10 μ L
Concentration: 50 mg/L

Samples

- 1 : ketoprofen
- 2 : flurbiprofen
- 3 : indometacin
- 4 : ibuprofen

2 - 4 抗组胺药物的分离

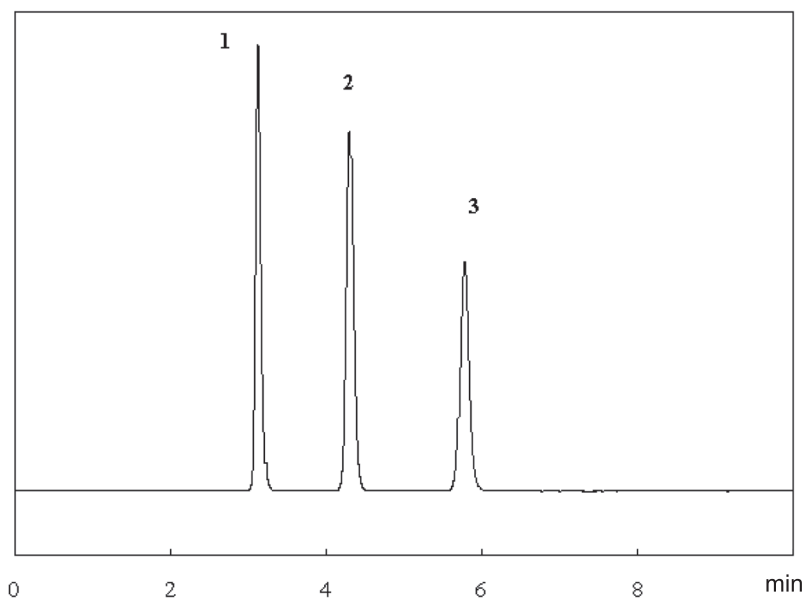


Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : 5 mmol/L sodium octanesulfonate
in (H₃PO₄ in (CH₃CN/H₂O =
60/40), pH 2.2)
Flow rate : 1.0 mL/min
Detection : UV (220 nm)
Temperature : 40 °C
Inj. volume : 10 μ L

Samples

- 1 : chlorpheniramine maleate
- 2 : diphenhydramine

2 - 5 黄嘌呤衍生物的分离

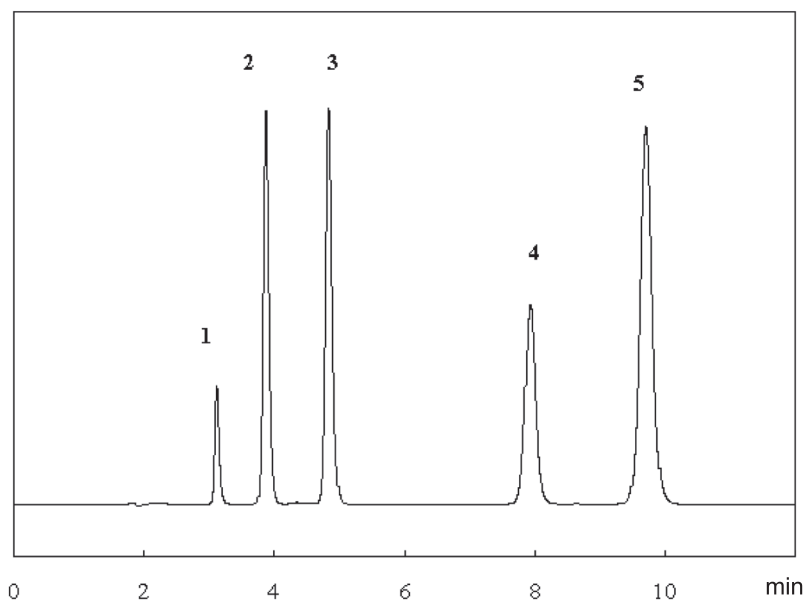


Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : CH₃OH/H₂O = 30/70
Flow rate : 1.0 mL/min
Detection : UV (254 nm)
Temperature : 40 °C
Inj. volume : 10 μ L
Concentration: 100 mg/L

Samples

- 1 : theobromine
- 2 : theophylline
- 3 : caffeine

2 - 6 抗癫痫药物的分离

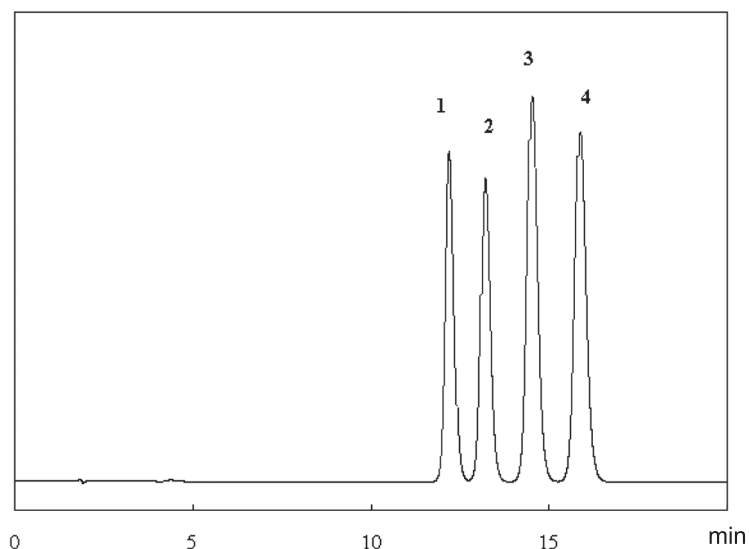


Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : 0.1 mol/L
sodium phosphate buffer
(pH2.2) /CH₃OH = 50/50
Flow rate : 1.0 mL/min
Detection : UV (220 nm)
Temperature : 40 °C
Inj. volume : 10 μ L

Samples

- 1 : ethosuximide (250 mg/L)
- 2 : primidone (50 mg/L)
- 3 : phenobarbital (50 mg/L)
- 4 : phenytoin (25 mg/L)
- 5 : carbamazepine (25 mg/L)

2 - 7 三环类抗抑郁症药物的分离

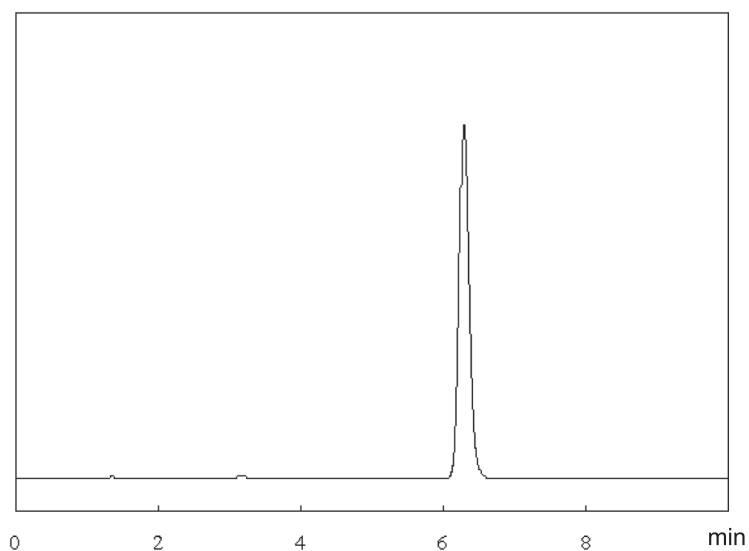


Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : 0.1 mol/L sodium phosphate
buffer (pH 2.2) / CH₃OH = 50/50
Flow rate : 1.0 mL/min
Detection : UV (220 nm)
Temperature : 40 °C
Inj. volume : 10 μ L
Concentration: 50 mg/L

Samples

1 : desipramine
2 : imipramine
3 : nortriptyline
4 : amitriptyline

2 - 8 镇静剂有效成分 (苯海拉明) 的分离

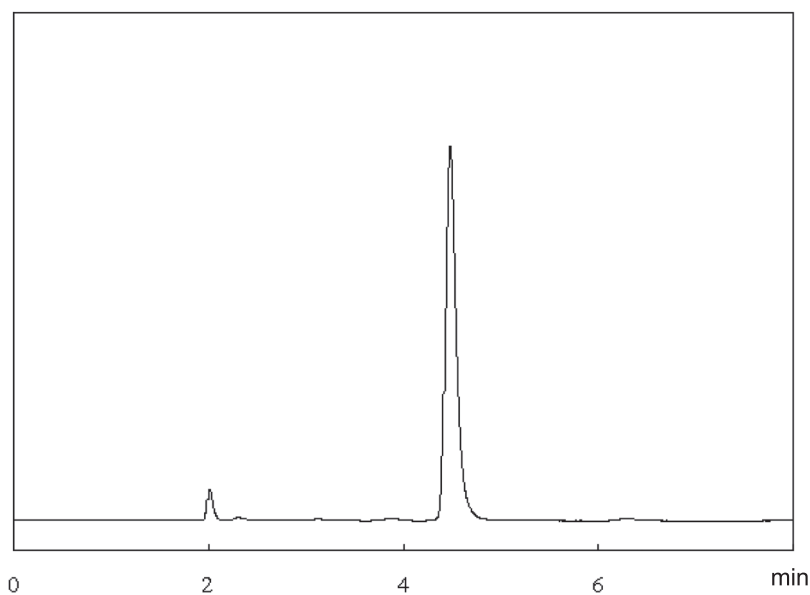


Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : 10 mmol/L SDS in (H₂O/CH₃CN
= 40/60)
Flow rate : 1.0 mL/min
Detection : UV (220 nm)
Temperature : 40 °C
Inj. volume : 10 μ L
Concentration: 50 mg/L

Sample

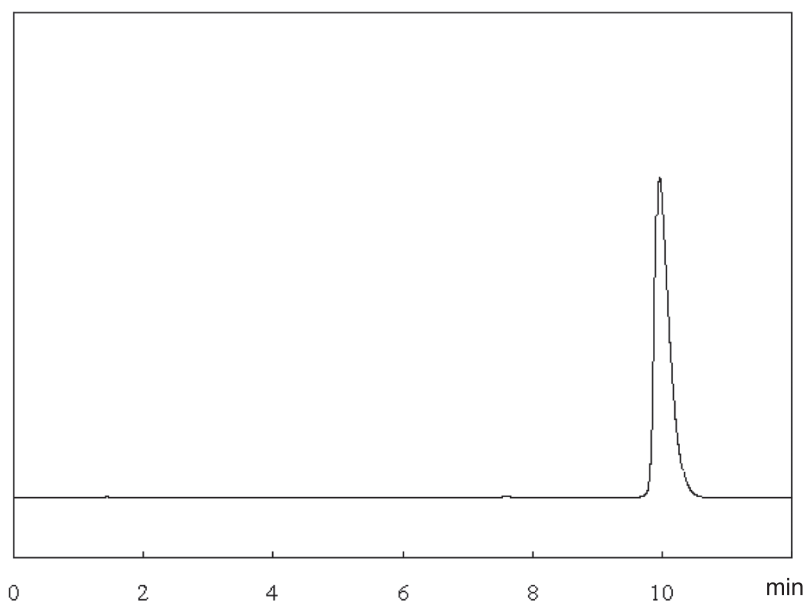
diphenhydramine

2-9 西吡氯铵的分离



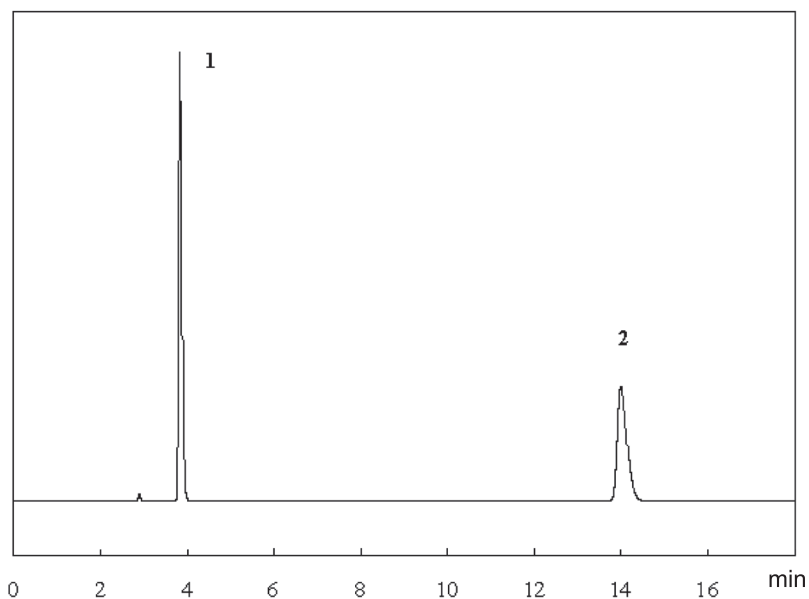
Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : 0.3 mol/L NH_4Cl in ($\text{CH}_3\text{OH}/\text{H}_2\text{O}$
= 90/10)
Flow rate : 1.0 mL/min
Detection : UV (254 nm)
Temperature : 40 $^\circ\text{C}$
Inj. volume : 10 μL
Concentration: 0.1 mg/L
Sample
cetylpyridinium chloride

2-10 抗晕动病药物有效成分 (盐酸氯苯苄嗪) 的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : 20 mmol/L sodium phosphate
buffer (pH 3.5) / CH_3CN = 50/50
Flow rate : 1.0 mL/min
Detection : UV (230 nm)
Temperature : 40 $^\circ\text{C}$
Inj. volume : 10 μL
Concentration: 50 mg/L
Sample
meclizine dihydrochloride

2 -11 抗晕动病药物有效成分 (乘晕宁) 的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 25 cm)

Eluent : 10mmol/L sodium
hexanesulfonate in
H₂O/CH₃CN = 65/35 (pH 2.2,
H₃PO₄)

Flow rate : 1.0 mL/min

Detection : UV (230 nm)

Temperature : 40 °C

Inj. volume : 10 μ L

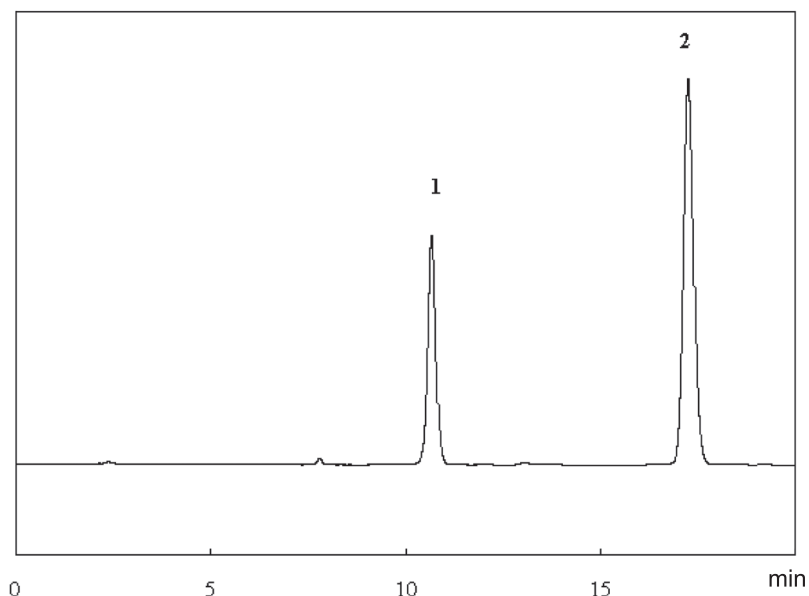
Concentration: 0.1 mg/L

Samples

1 : 8-chlorotheophylline

2 : diphenhydramine

2 -12 牙周病治疗药物有效成分的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 25 cm)

Eluent : 5mmol/L SDS in
H₂O/CH₃CN = 35/65 (pH 2.2,
H₃PO₄)

Flow rate : 1.0 mL/min

Detection : UV (248 nm)

Temperature : 40 °C

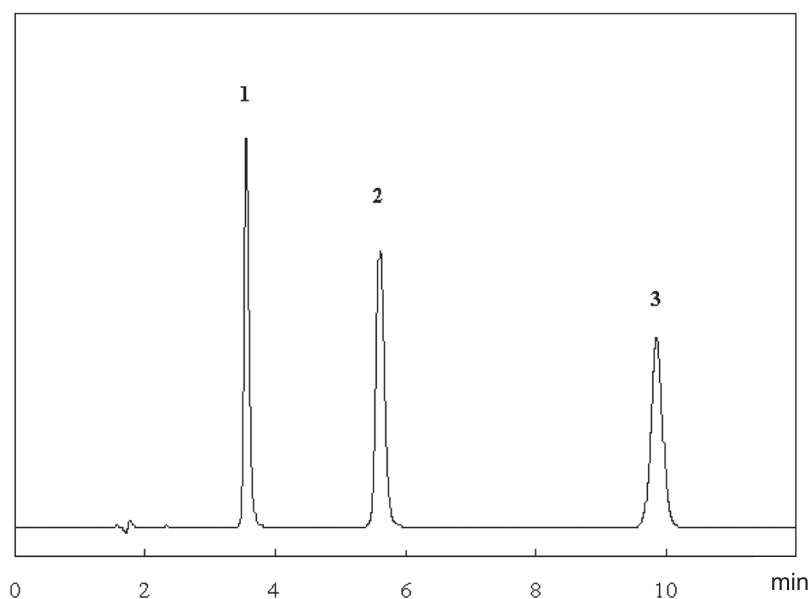
Inj. volume : 10 μ L

Samples

1 : chlorhexidine hydrochloride (0.1 g/L)

2 : β -glycyrrhetic acid (0.2 g/L)

2 -13 普鲁卡因酰胺的分离



Column : TSKgel ODS-100V 5 μm
(4.6 mm I.D. × 15 cm)

Eluent : 25 mmol/L sodium phosphate
buffer (pH7.0)
/CH₃CN/CH₃OH = 80/10/10

Flow rate : 1.0 mL/min

Detection : UV (210 nm)

Temperature : 40 °C

Inj. volume : 10 μL

Concentration: 50 mg/L

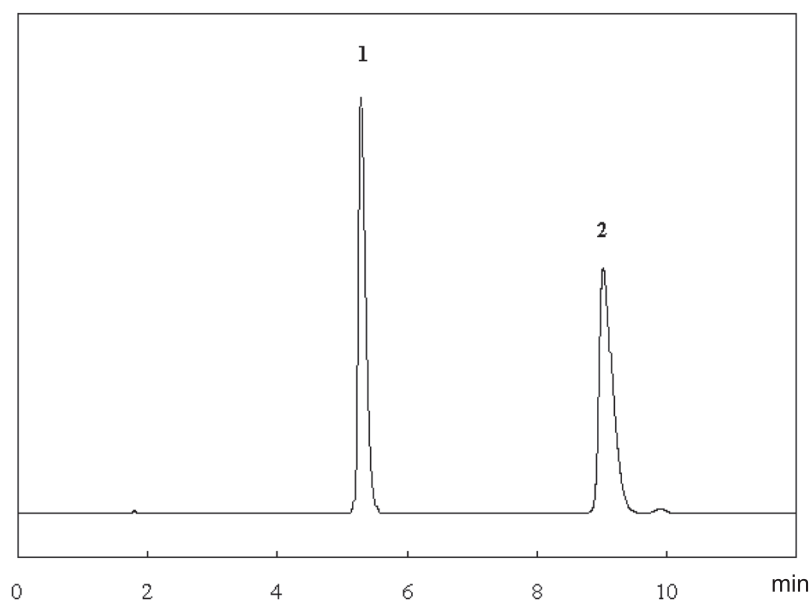
Samples

1 : procainamide

2 : N-acetylprocainamide

3 : phenol

2 -14 阿托品、东莨菪碱的分离



Column : TSKgel ODS-100V 5 μm
(4.6 mm I.D. × 15 cm)

Eluent : 10 mmol/L
sodium phosphate buffer
(pH2.0) /CH₃CN = 85/15

Flow rate : 1.0 mL/min

Detection : UV (210 nm)

Temperature : 40 °C

Inj. volume : 10 μL

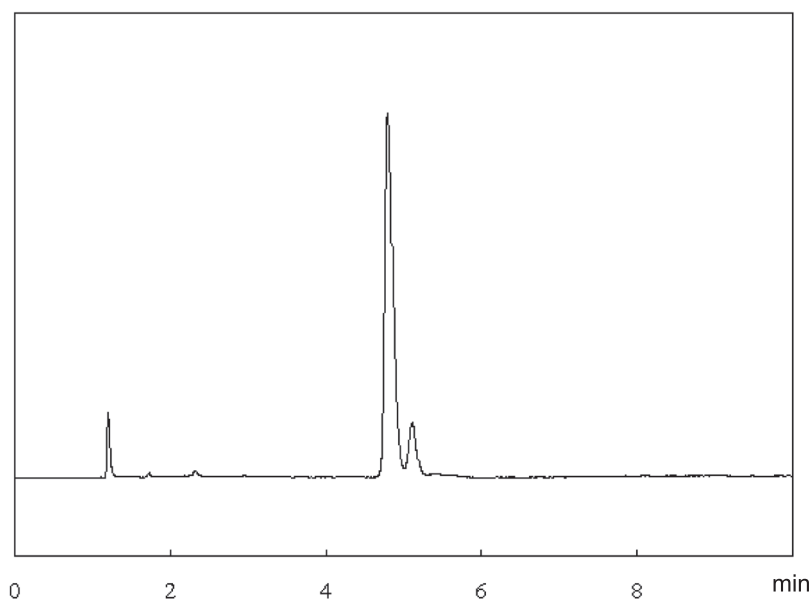
Concentration: 0.1 mg/L

Samples

1 : scopolamine

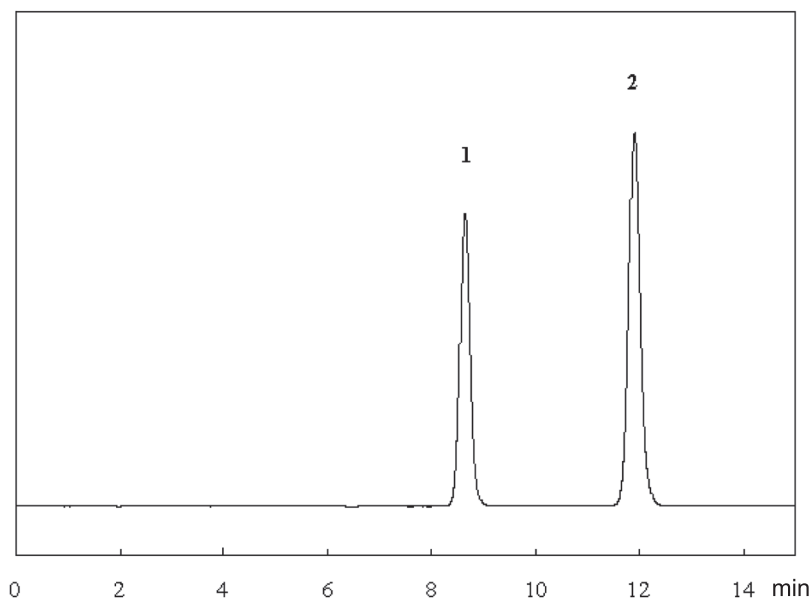
2 : atropine

2 -15 肉碱盐酸盐的分离



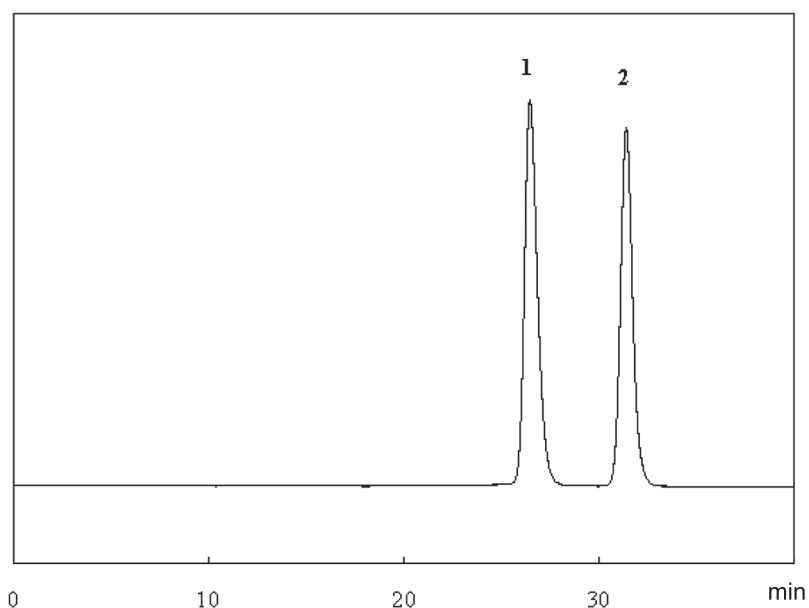
Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : 0.1 % H₃PO₄, 5 mmol/L sodium octanesulfonate, H₂O/CH₃CN = 85/15
Flow rate : 1.0 mL/min
Detection : UV (230nm)
Temperature : 40 °C
Inj. volume : 5 μ L
Concentration: 0.5 mg/L
Samples : carnitine hydrochloride

2 -16 盐酸普鲁卡因的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : 0.1 % sodium pentanesulfonate, 50 mmol/L potassium phosphate buffer (pH3.0) /CH₃OH = 80/20
Flow rate : 1.0 mL/min
Detection : UV (254 nm)
Temperature : 40 °C
Inj. volume : 5 μ L
Concentration: 0.1 mg/L
Samples
1 : procaine hydrochloride
2 : caffeine

2-17 卡络磺钠的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)

Eluent : 10 mmol/L
ammonium phosphate buffer
(pH 3.0) / Ethanol = 92.5/7.5

Flow rate : 1.0 mL/min

Detection : UV (360nm)

Temperature : 40 $^{\circ}$ C

Inj. volume : 10 μ L

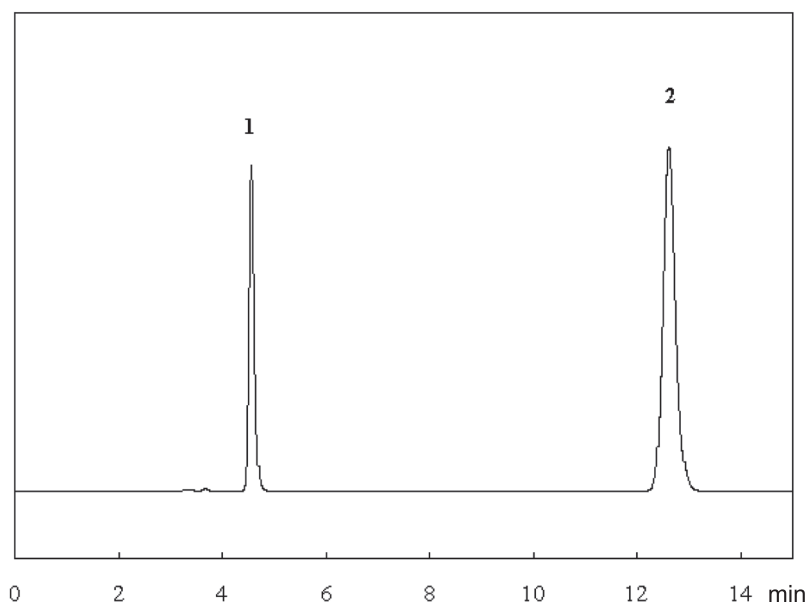
Concentration: 50 mg/L

Samples

1 : carbazochrome sodium sulfonate

2 : carbazochrome

2-18 氢化可的松琥珀酸的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)

Eluent : 20 mmol/L sodium acetate
buffer (pH 4.0) / CH₃CN = 60/40

Flow rate : 1.0 mL/min

Detection : UV (254 nm)

Temperature : 40 $^{\circ}$ C

Inj. volume : 10 μ L

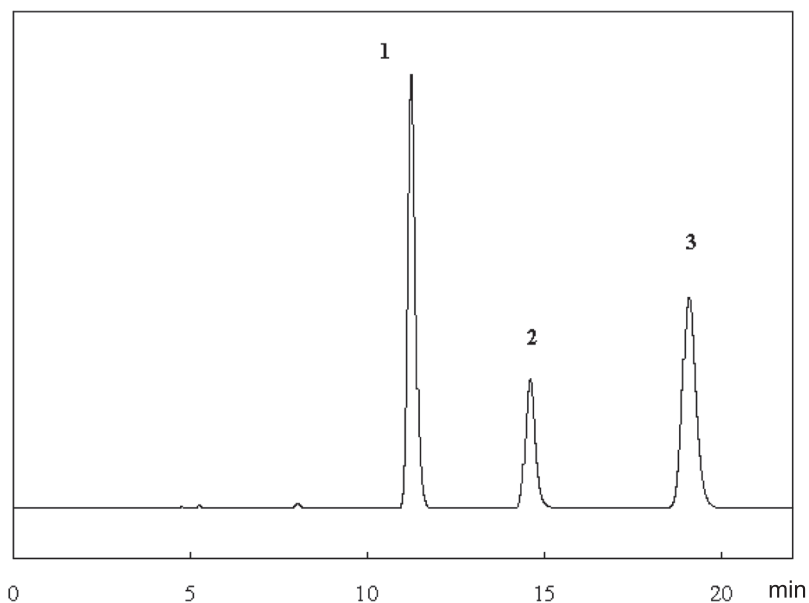
Concentration: 0.1 mg/L

Samples

1 : hydrocortisone succinate

2 : butyl p-hydroxybenzoate

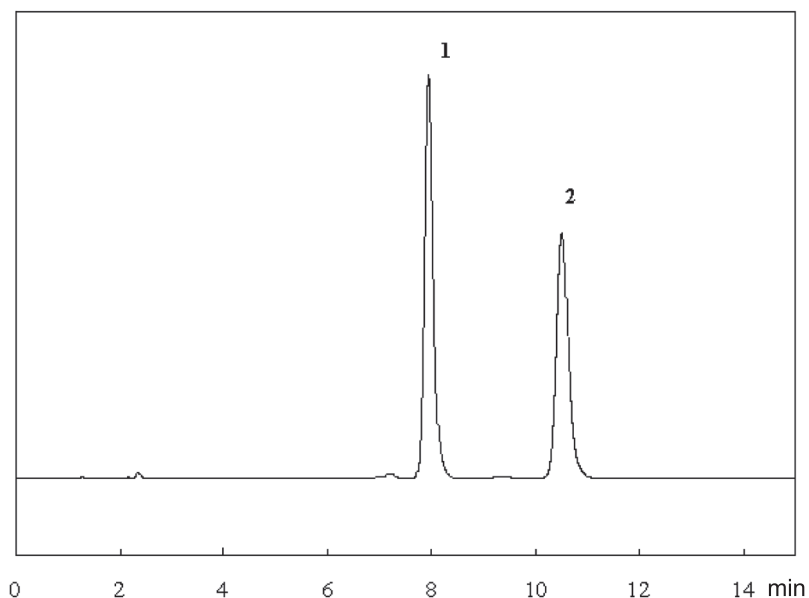
2 -19 醋酸可的松的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : H₂O/CH₃CN = 65/35
Flow rate : 1.0 mL/min
Detection : UV (254 nm)
Temperature : 40 $^{\circ}$ C
Inj. volume : 10 μ L
Concentration: 50 mg/L

Samples
1 : propyl p-hydroxybenzoate
2 : butyl p-hydroxybenzoate
3 : cortisone acetate

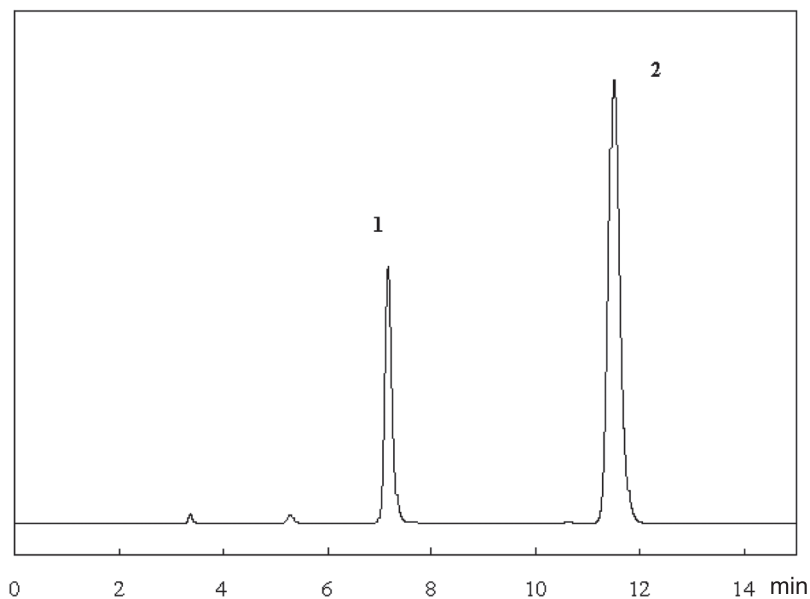
2 -20 维生素 E 醋酸酯的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : H₂O/CH₃OH = 2/98
Flow rate : 1.0 mL/min
Detection : UV (284 nm)
Temperature : 40 $^{\circ}$ C
Inj. volume : 20 μ L
Concentration: 0.1 g/L

Samples
1 : d- α -tocopherol
2 : tocopherol acetate

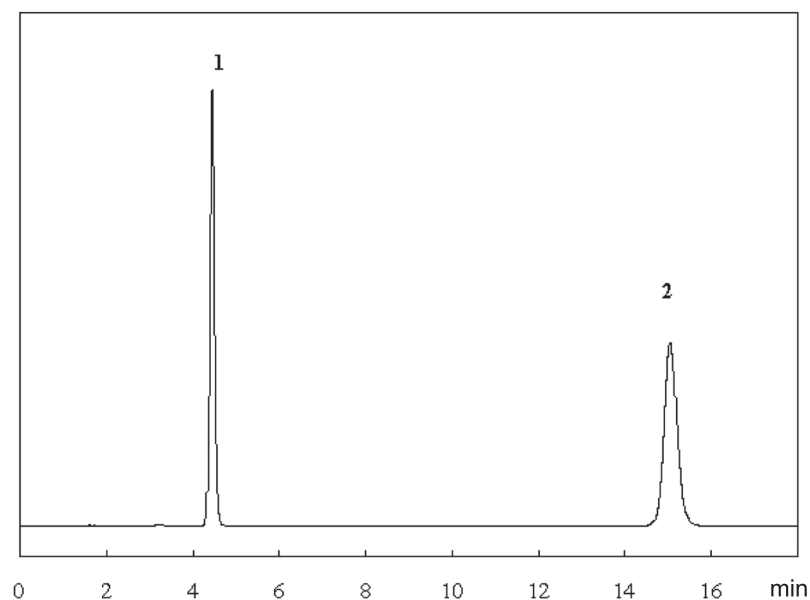
2 -21 醋酸泼尼松龙的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : H₂O/CH₃CN = 60/40
Flow rate : 1.0 mL/min
Detection : UV (254 nm)
Temperature : 40 °C
Inj. volume : 10 μ L
Concentration: 0.1 g/L

Samples
1 : prednisolone acetate
2 : butyl p-hydroxybenzoate

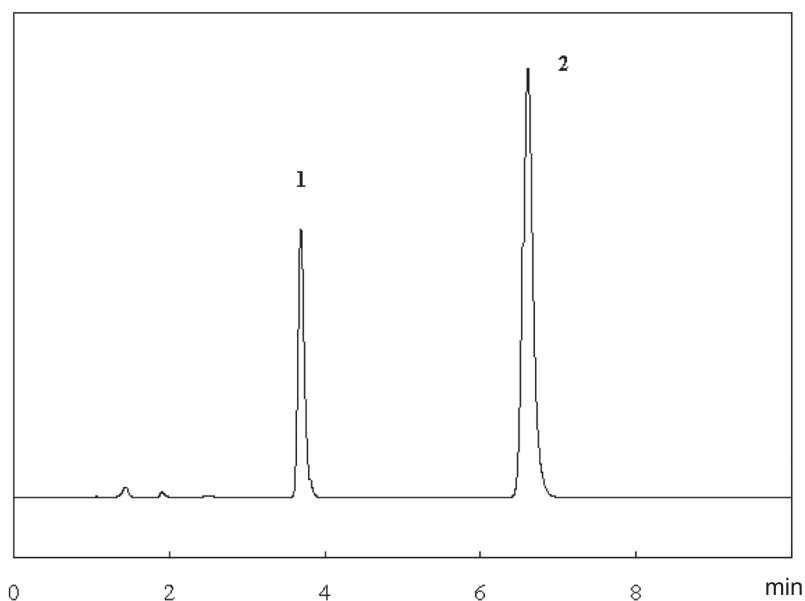
2 -22 醋酸地塞米松的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : H₂O/CH₃OH = 65/35
Flow rate : 1.0 mL/min
Detection : UV (284 nm)
Temperature : 40 °C
Inj. volume : 10 μ L
Concentration: 0.1 g/L

Samples
1 : dexamethasone acetate
2 : progesterone

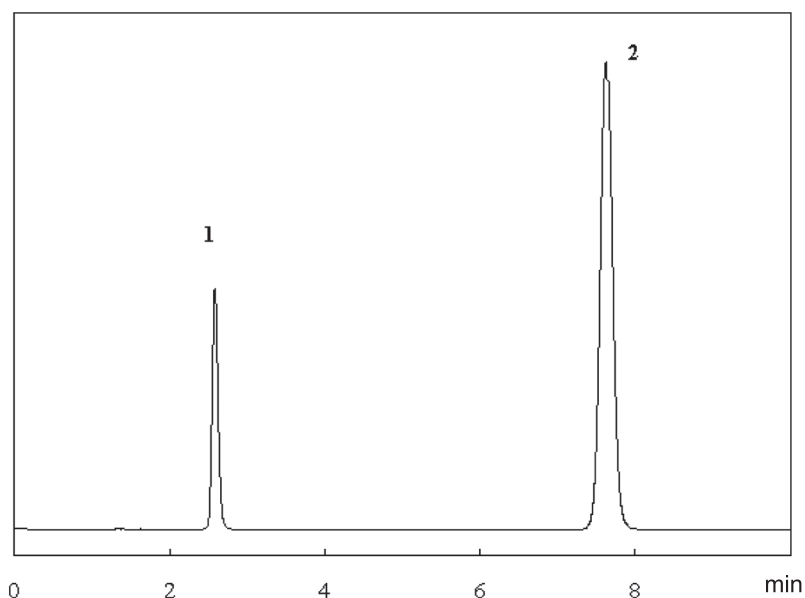
2 -23 环扁桃酯的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : H₂O/CH₃CN = 20/80
Flow rate : 1.0 mL/min
Detection : UV (228 nm)
Temperature : 40 °C
Inj. volume : 10 μ L
Concentration: 0.1 g/L

Samples
1 : cyclandelate
2 : dicyclohexyl phthalate

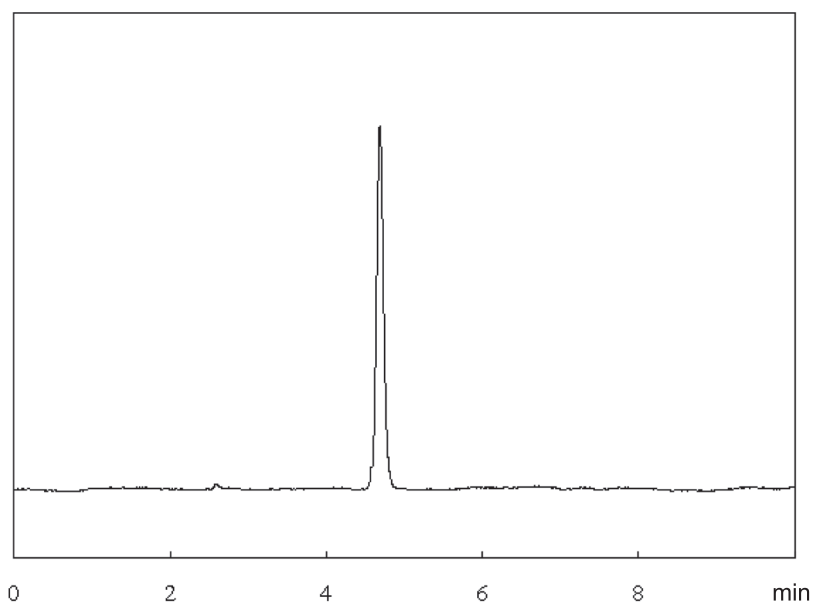
2 -24 双嘧达莫的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : 0.1 % KH₂PO₄/CH₃OH = 20/80
Flow rate : 1.0 mL/min
Detection : UV (280 nm)
Temperature : 40 °C
Inj. volume : 20 μ L

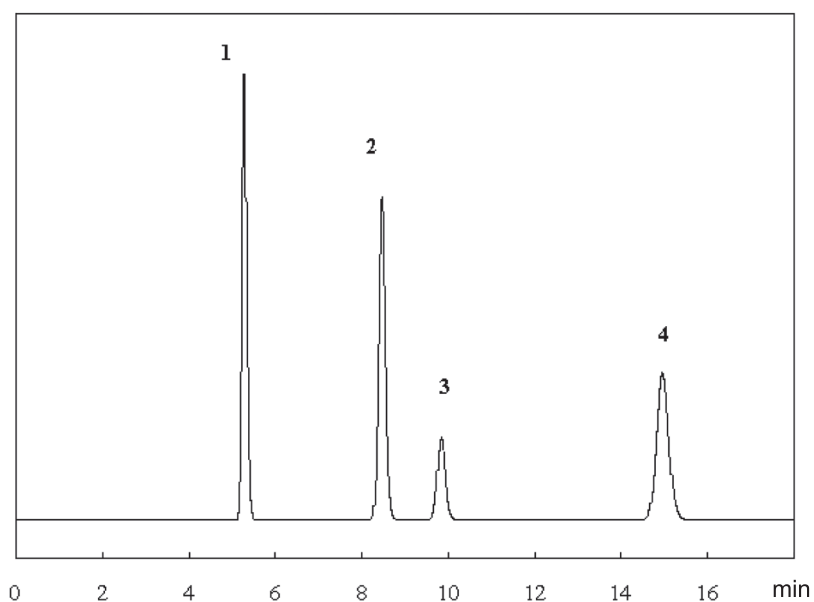
Samples
1 : dipyrindamole (25 mg/L)
2 : p-terphenyl (400 mg/L)

2 -25 司可巴比妥钠的分离



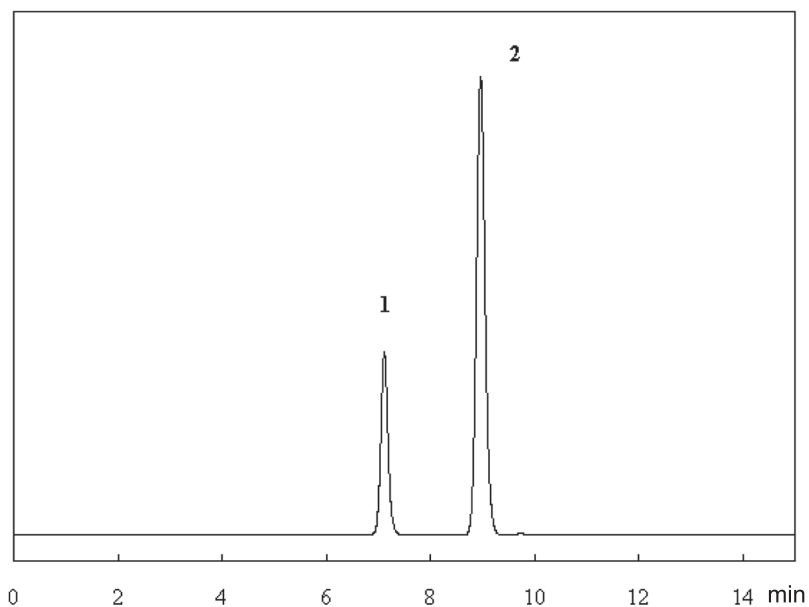
Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : 10 mmol/L sodium octanesulfonate,
0.1 mol/L sodium phosphate
buffer (pH 6.8)/CH₃CN = 60/40
Flow rate : 1.0 mL/min
Detection : UV (254 nm)
Temperature : 40 °C
Inj. volume : 5 μ L
Concentration: 50 mg/L
Samples : secobarbital sodium

2 -26 地塞米松的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : H₂O/CH₃CN = 60/40
Flow rate : 1.0 mL/min
Detection : UV (254 nm)
Temperature : 40 °C
Inj. volume : 10 μ L
Concentration: 50 mg/L
Samples
1 : methyl p-hydroxybenzoate
2 : ethyl p-hydroxybenzoate
3 : dexamethasone
4 : propyl p-hydroxybenzoate

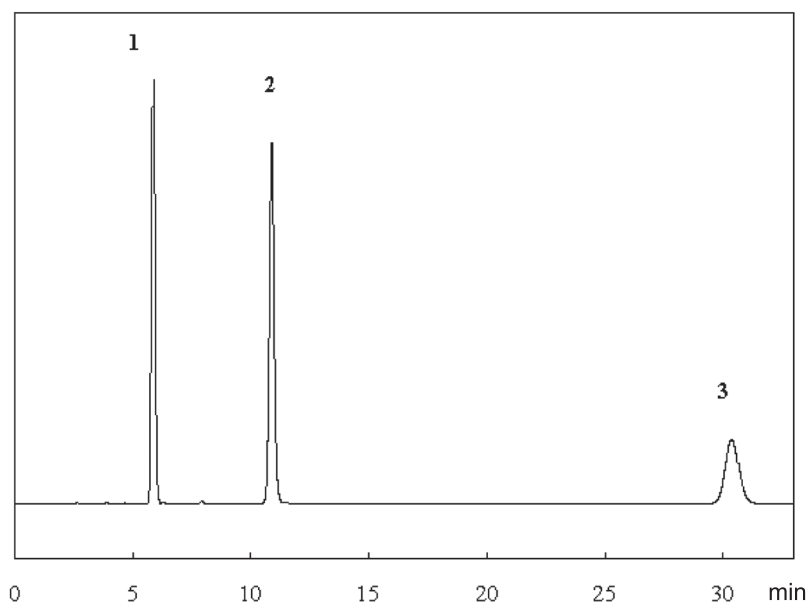
2 -27 去炎松的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : H₂O/CH₃CN = 75/25
Flow rate : 1.0 mL/min
Detection : UV (254 nm)
Temperature : 40 $^{\circ}$ C
Inj. volume : 10 μ L
Concentration: 100 mg/L

Samples
1 : triamcinolone
2 : methyl p-hydroxybenzoate

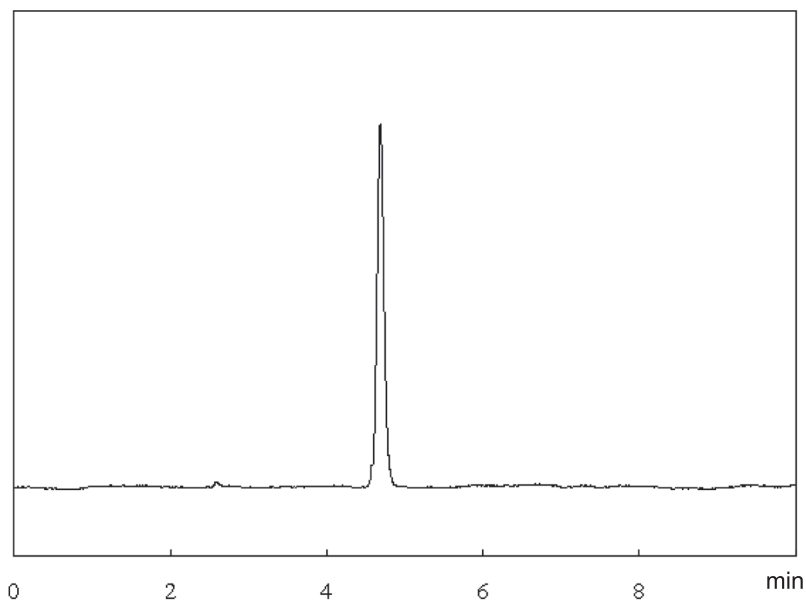
2 -28 曲安奈德的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : H₂O/CH₃CN = 75/25
Flow rate : 1.0 mL/min
Detection : UV (240 nm)
Temperature : 40 $^{\circ}$ C
Inj. volume : 10 μ L
Concentration: 125 mg/L

Samples
1 : triamcinolone
2 : prednisolone
3 : triamcinolone acetonide

2 -29 烟酸的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)

Eluent : 1.1g sodium octanesulfonate in
1000 mL + (50mmol/L sodium
phosphate buffer (pH3.0)
/CH₃OH = 80/20)

Flow rate : 1.0 mL/min

Detection : UV (260 nm)

Temperature : 40 °C

Inj. volume : 10 μ L

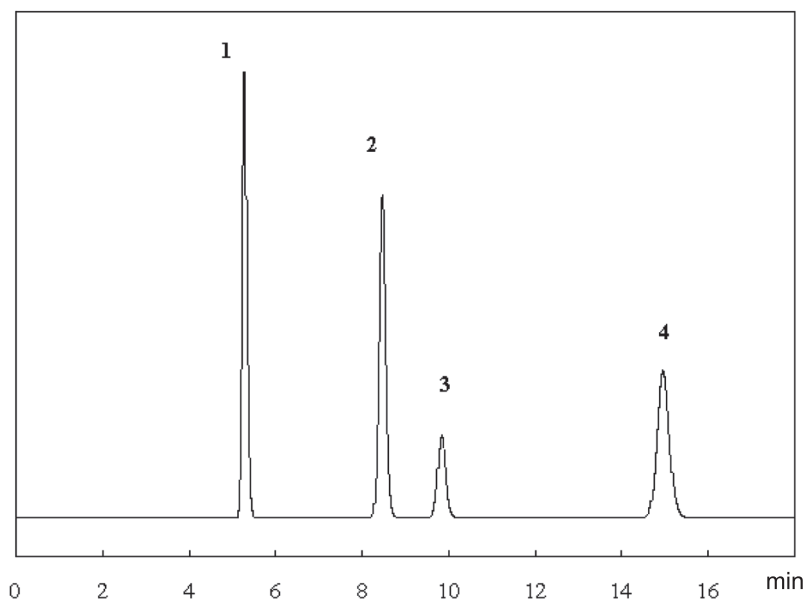
Concentration: 100 mg/L

Samples

1 : nicotinic acid

2 : caffeine

2 -30 氟米龙的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)

Eluent : H₂O/CH₃OH = 30/70

Flow rate : 1.0 mL/min

Detection : UV (254 nm)

Temperature : 40 °C

Inj. volume : 20 μ L

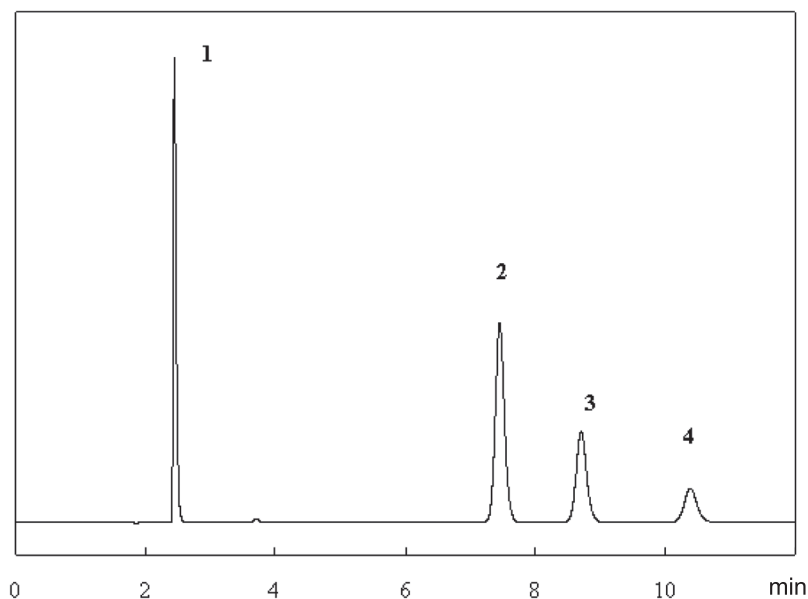
Concentration: 50 mg/L

Samples

1 : fluorometholone

2 : butyl p-hydroxybenzoate

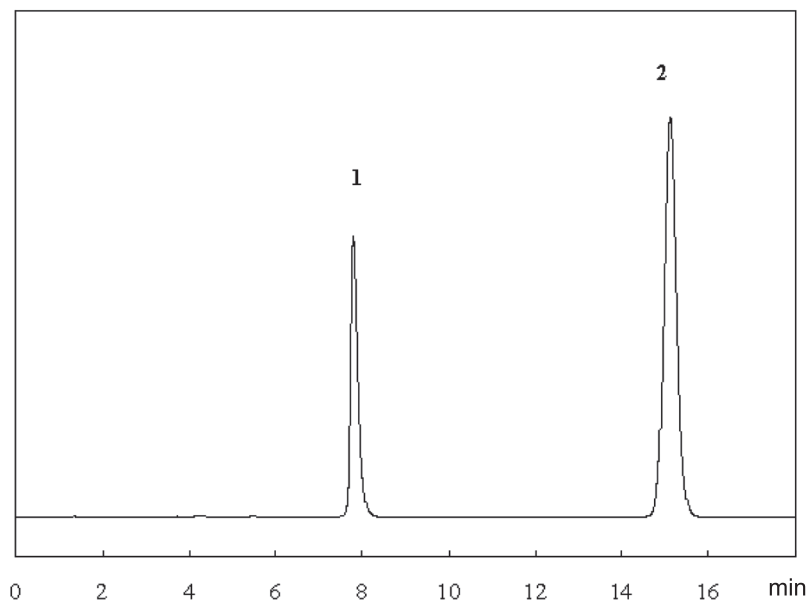
2 -31 碘、水杨酸、苯酚酯剂的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : 0.1 mol/L potassium phosphate
buffer (pH2.5)/CH₃CN = 75/25
Flow rate : 1.0 mL/min
Detection : UV (270 nm)
Temperature : 40 °C
Inj. volume : 10 μ L

Samples
1 : theophylline (12.5 mg/L)
2 : phenol (62.5 mg/L)
3 : benzoic acid (62.5 mg/L)
4 : salicylic acid (62.5 mg/L)

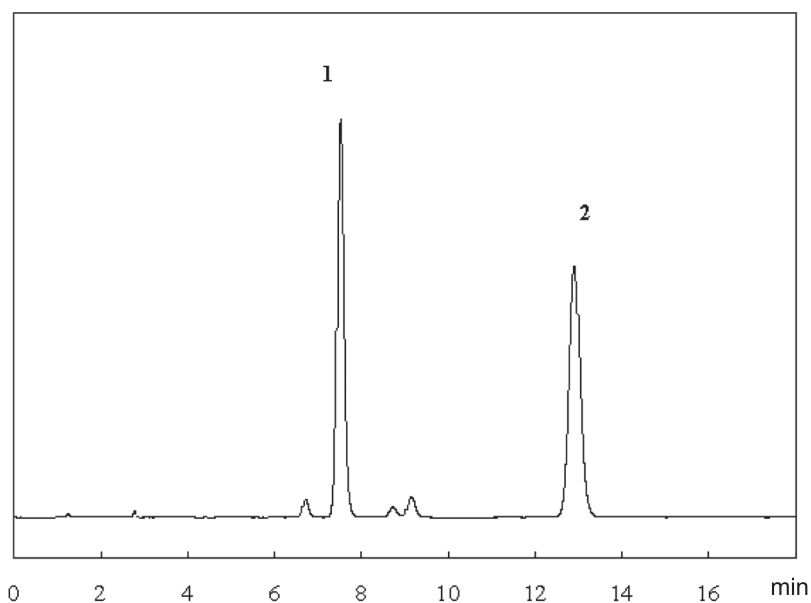
2 -32 利血平的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : 50 mmol/L sodium phosphate
buffer (pH2.2)/CH₃CN = 60/40
Flow rate : 1.0 mL/min
Detection : UV (268 nm)
Temperature : 40 °C
Inj. volume : 20 μ L
Concentration: 50 mg/L

Samples
1 : reserpine
2 : butyl p-hydroxybenzoate

2 -33 利多卡因的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)

Eluent : 2.88 g SDS in 1000 mL
(20 mmol/L sodium phosphate
buffer (pH3.0)
/CH₃CN = 55/45)

Flow rate : 1.0 mL/min

Detection : UV (254 nm)

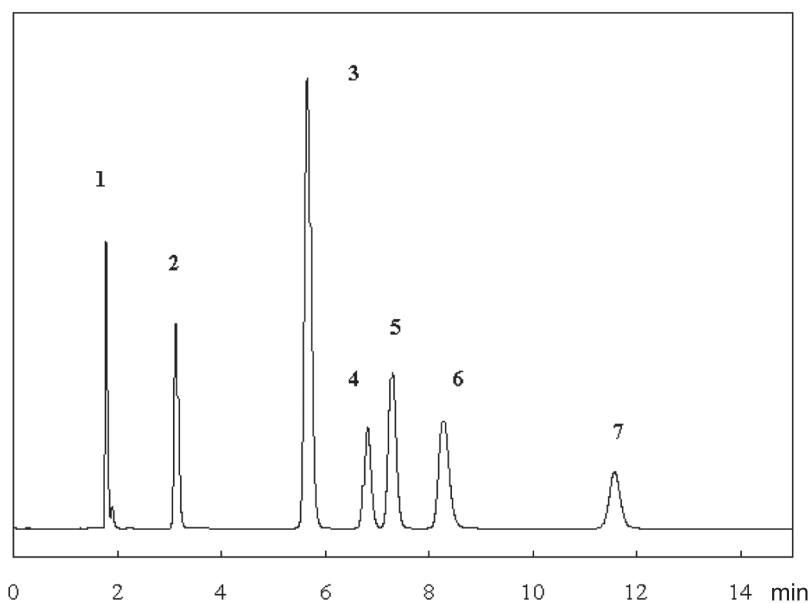
Temperature : 40 °C

Inj. volume : 5 μ L

Samples

1 : lidocaine hydrochloride (400 mg/L)
2 : benzophenone (25 mg/L)

2 -34 感冒药物成分的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)

Eluent : 50 mmol/L sodium phosphate
buffer (pH2.5)/CH₃OH = 70/30

Flow rate : 1.0 mL/min

Detection : UV (280 nm)

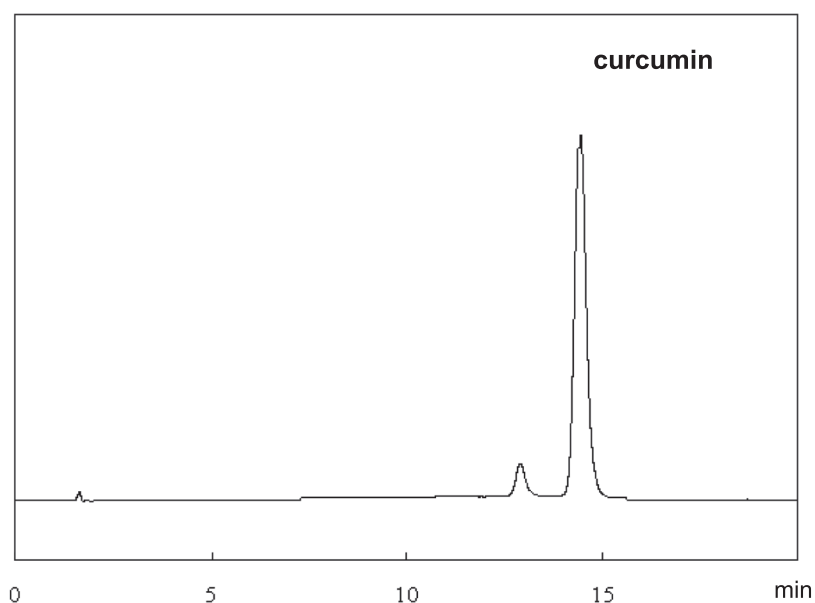
Temperature : 40 °C

Inj. volume : 5 μ L

Samples

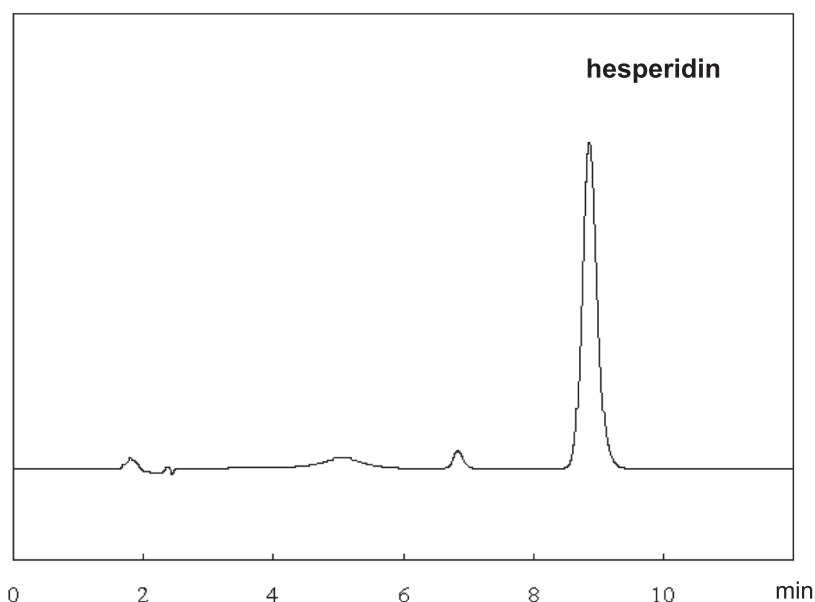
1 : potassium guaiacolsulfonate (50 mg/L)
2 : acetaminophen (50 mg/L)
3 : caffeine (50 mg/L)
4 : salicylamide (50 mg/L)
5 : phenol (100 mg/L)
6 : chlorpheniramine maleate (100 mg/L)
7 : aspirin (100 mg/L)

2 -35 姜黄色素的分离 (姜黄萃取物)



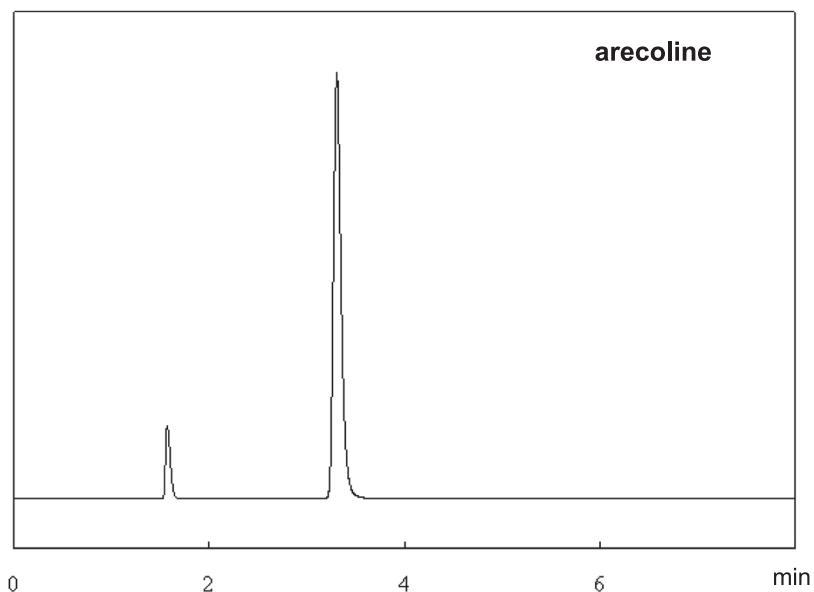
Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : 0.1 % TFA/CH₃CN = 55/45
Flow rate : 1.0 mL/min
Detection : UV (420 nm)
Temperature : 40 °C
Inj. volume : 5 μ L
Concentration: 0.1 g/L
Sample : curcumin

2 -36 橘皮苷的分离 (香橙萃取物)



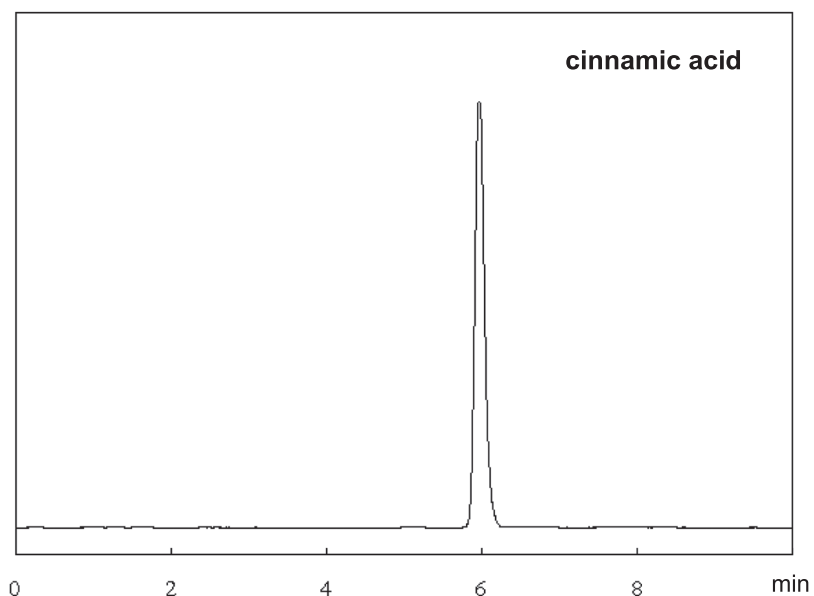
Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : CH₃COOH/H₂O/CH₃CN
= 1/80/20
Flow rate : 1.0 mL/min
Detection : UV (280 nm)
Temperature : 40 °C
Inj. volume : 10 μ L
Concentration: 50 mg/L
Sample : hesperidin

2 -37 檳榔碱的分离



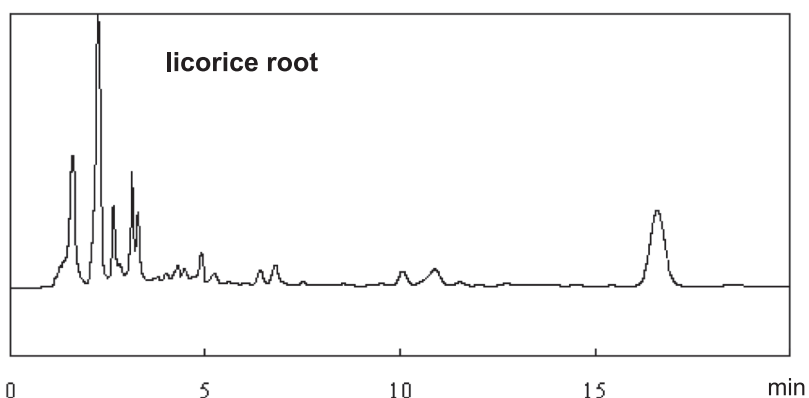
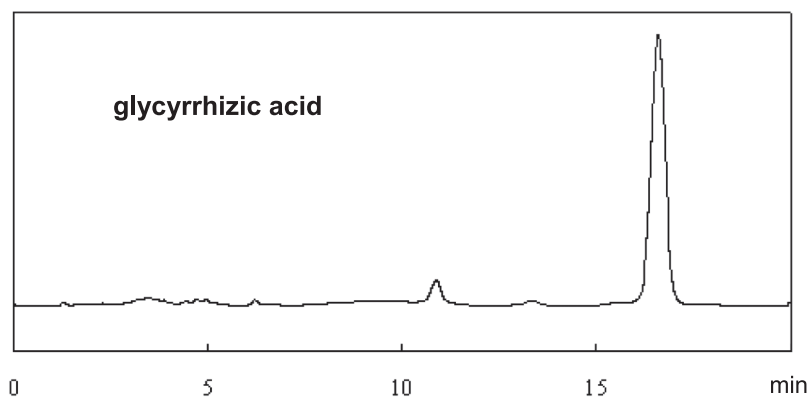
Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : (20 mmol/L NaH₂PO₄ +
100 mmol/L NaClO₄)/CH₃OH
= 90/10
Flow rate : 1.0 mL/min
Detection : UV (215 nm)
Temperature : 40 °C
Inj. volume : 20 μ L
Concentration: 100 mg/L
Sample : arecoline

2 -38 肉桂酸的分离 (桂皮萃取物)



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : CH₃COOH/H₂O/CH₃CN
= 1/65/35
Flow rate : 1.0 mL/min
Detection : UV (275 nm)
Temperature : 40 °C
Inj. volume : 5 μ L
Concentration: 50 mg/L
Sample : cinnamic acid

2 -39 甘草中的甘草酸的分离



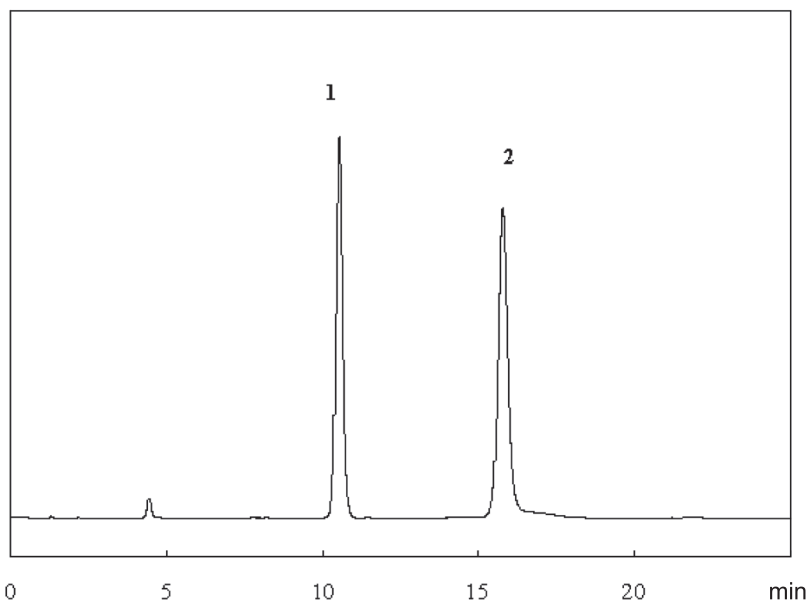
Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : H₃PO₄/H₂O/CH₃CN = 1/650/350
Flow rate : 1.0 mL/min
Detection : UV (254 nm)
Temperature : 40 °C
Inj. volume : 10 μ L
Concentration: 25 mg/L (std.)

Samples : glycyrrhizic acid (std.)
licorice root

Pretreatment

1. Add 7 mL ethanol to 50 g licorice root, mix, then centrifuge and remove the supernatant.
2. Add 2.5 mL ethanol to the residue; repeat the process twice.
3. Combine all extracts and make up to 20 mL to prepare sample for analysis.

2 -40 东莨菪浸膏的分离

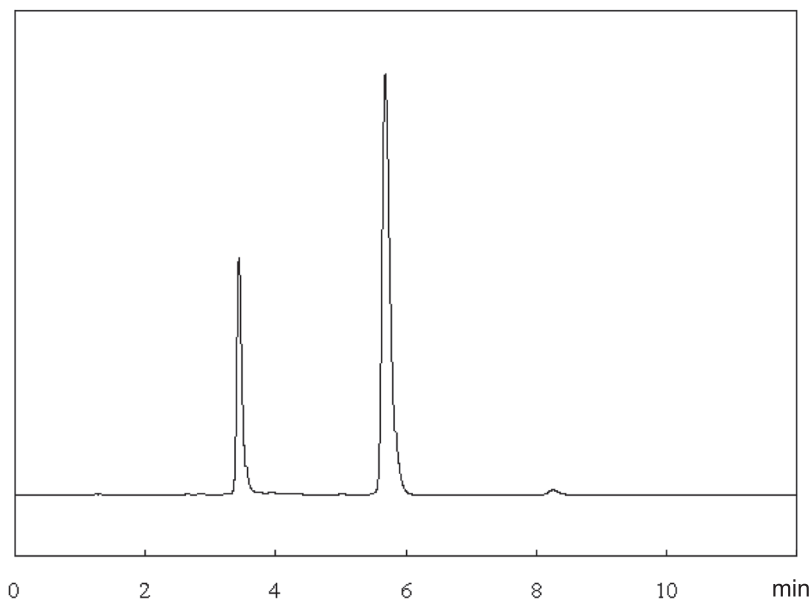


Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : 0.1 % sodium octansulfonate,
10 mmol/L potassium phosphate
buffer (pH2.5)/CH₃OH = 60/40
Flow rate : 1.0 mL/min
Detection : UV (210 nm)
Temperature : 40 °C
Inj. volume : 10 μ L
Concentration: 0.1 g/L

Samples

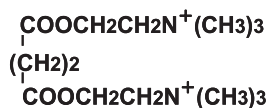
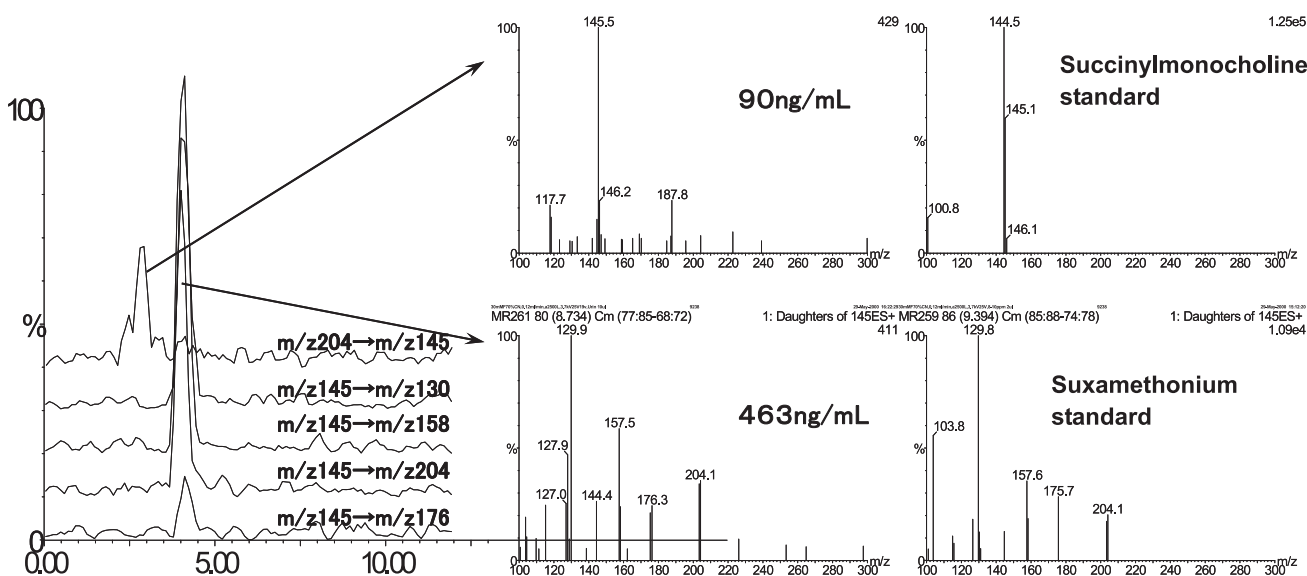
- 1 : scopolamine
- 2 : atropine

2 -41 乌头碱的分离



Column : TSKgel ODS-100V 5 μ m
 (4.6 mmI.D. \times 15 cm)
 Eluent : 50 mmol/L potassium phosphate
 buffer (pH2.5)/CH₃CN = 68/32
 Flow rate : 1.0 mL/min
 Detection : UV (254 nm)
 Temperature : 40 °C
 Inj. volume : 10 μ L
 Concentration: 0.5 g/L
 Samples : aconitine

2 -42 服用肌松弛药物琥珀胆碱自杀者尿液的 LC/MS/MS 分析



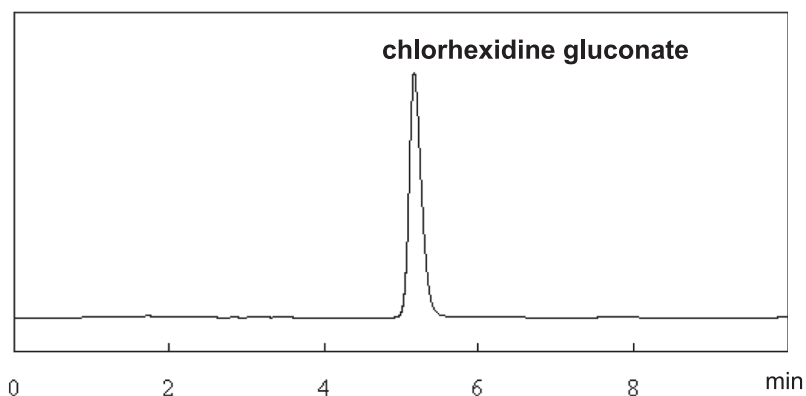
Inst. : QuatroLC (Micromass)
 Column : TSKgel VMPak-25
 Eluent : 30 mmol/L HCOONH₄/CH₃CN = 30/70

3. 化妆品分析应用数据

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3 - 1 护肤霜中葡萄糖酸洗必太的分离

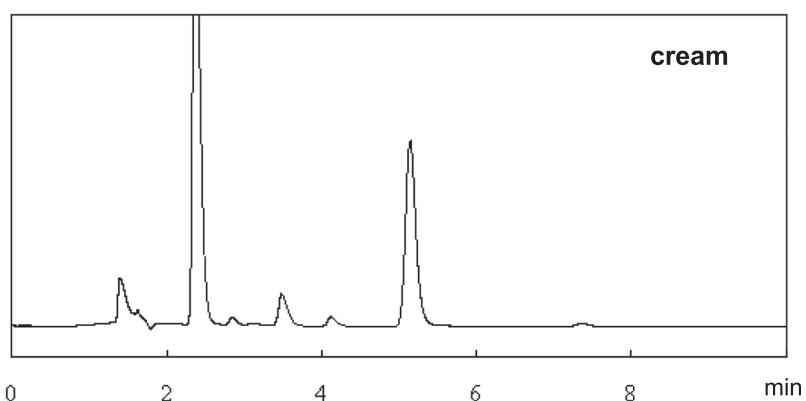


Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : (sodium dodecylsulfate 1g +
CH₃COOH 2 g) in (H₂O/CH₃CN/
THF = 400/200/200 (mL))
Flow rate : 1.0 mL/min
Detection : UV (258 nm)
Temperature : 40 °C
Inj. volume : 10 μ L
Concentration: 20 mg/L (std.)

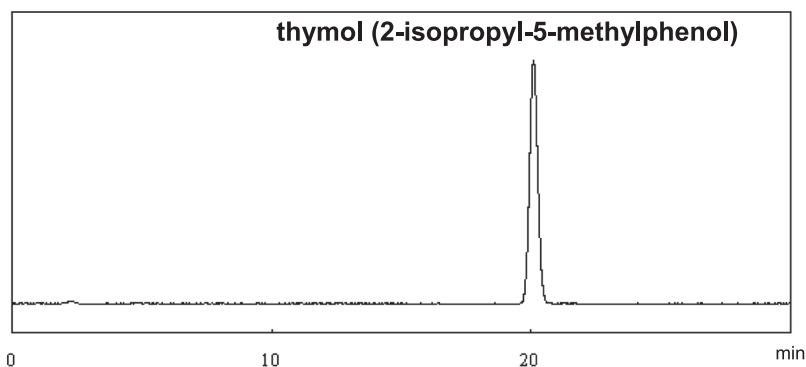
Samples : chlorhexidine gluconate (std.)
cream

Pretreatment

1. Add 10 mL methanol to 2 g face washing foam, and shake.
2. Centrifuge (5000 rpm, 10 min) and use supernatant as sample for analysis.



3 - 2 护手霜中麝香草酚的分离

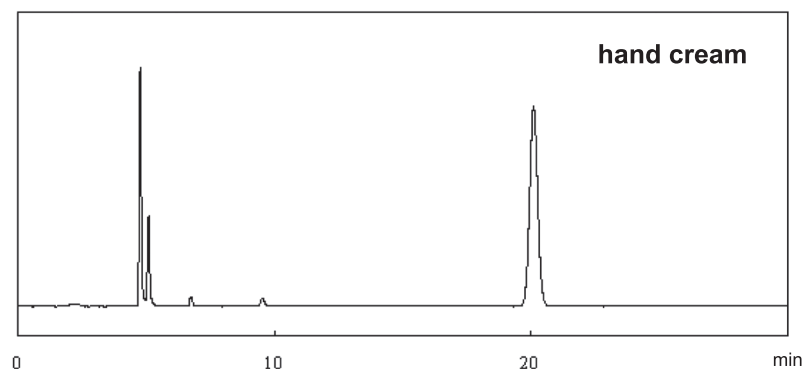


Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 25 cm)
Eluent : H₂O/CH₃CN = 50/50
Flow rate : 1.0 mL/min
Detection : FL (ex ; 280 nm, em ; 305 nm)
Temperature : 40 °C
Inj. volume : 10 μ L
Concentration: 0.5 mg/L (std.)

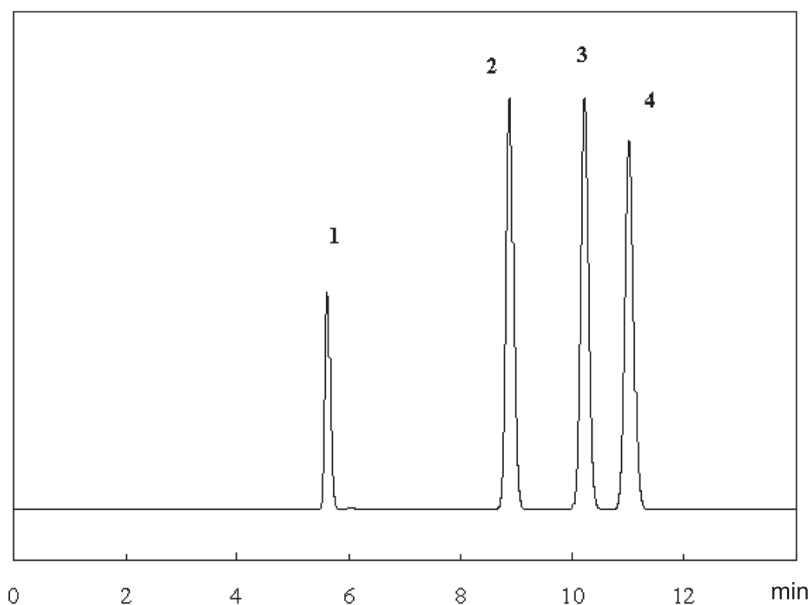
Sample : thymol (std.)
hand cream

Pretreatment

- Add methanol to 10 g of hand cream, and agitate using ultrasound.
Make up to 100 mL with methanol then filter (PTFE, 0.5 μ m). Dilute filtrate with methanol.



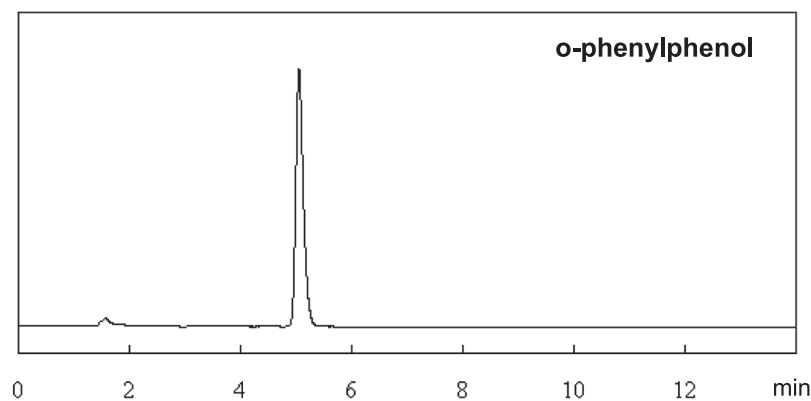
3 - 3 氯化钾酚的分离



Column : TSKgel ODS-100V 5 μm
(4.6 mm I.D. × 25 cm)
Eluent : H₂O/CH₃OH = 40/60
Flow rate : 1.0 mL/min
Detection : UV (227 nm)
Temperature : 40 °C
Inj. volume : 20 μL
Concentration: 50 mg/L

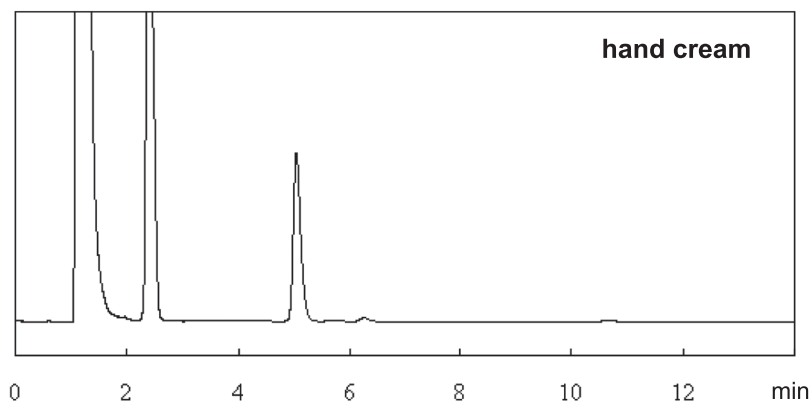
Samples
1 : methyl p-hydroxybenzoate
2 : 2-chloro-5-methylphenol
3 : 4-chloro-2-methylphenol
4 : 4-chloro-3-methylphenol

3 - 4 护手霜中联苯的分离



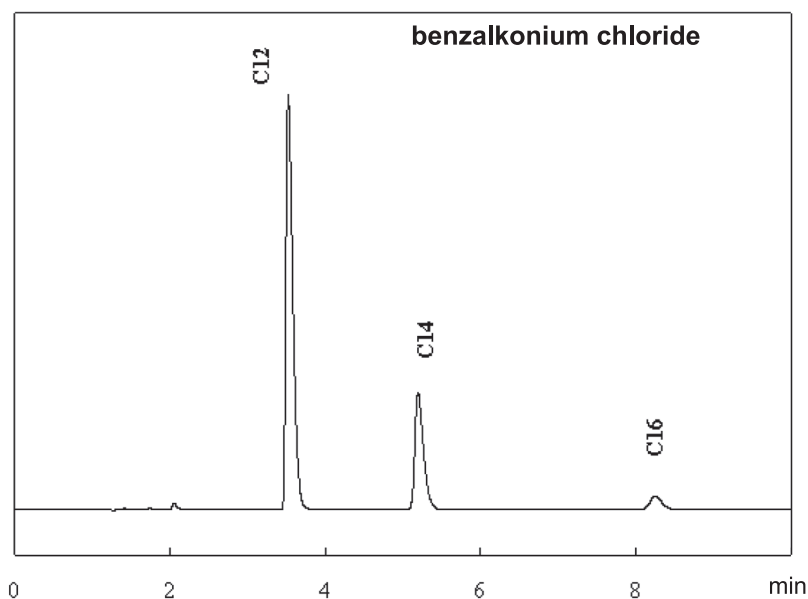
Column : TSKgel ODS-100V 5 μm
(4.6 mm I.D. × 15 cm)
Eluent : H₂O/CH₃CN = 45/55
(pH 2.5, H₃PO₄)
Flow rate : 1.0 mL/min
Detection : UV (247 nm)
Temperature : 40 °C
Inj. volume : 10 μL
Concentration: 5 mg/L (std.)

Sample : o-phenylphenol (std.)
hand cream



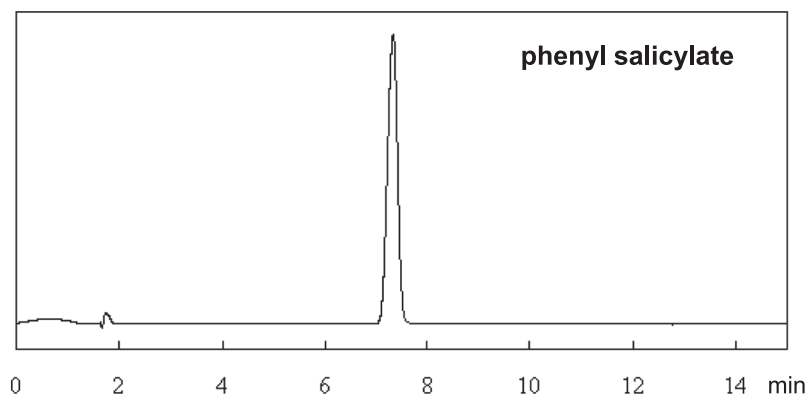
Pretreatment
Add methanol to 1 g of hand cream, and agitate using ultrasound.
Make up to 100 mL with methanol then filter (PTFE, 0.5 μm). Dilute filtrate with methanol.

3 - 5 洁尔灭 (杀藻胺 ; 苯扎氯铵) 的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : 50 mmol/L NaClO₄ in
(H₂O/CH₃CN = 20/80)
Flow rate : 1.0 mL/min
Detection : UV (265 nm)
Temperature : 40 °C
Inj. volume : 10 μ L
Concentration: 50 mg/L
Samples : benzalkonium chloride

3 - 6 护手霜中水杨酸苯酯的分离

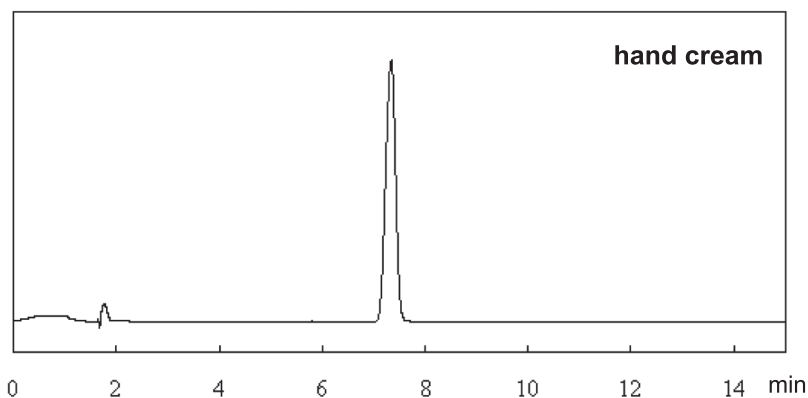


Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : H₂O/CH₃CN = 40/60
Flow rate : 0.8 mL/min
Detection : UV (310 nm)
Temperature : 40 °C
Inj. volume : 10 μ L
Concentration: 40 mg/L (std.)

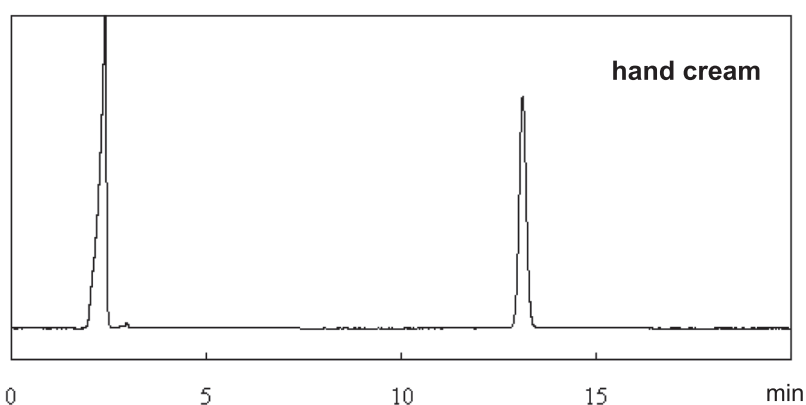
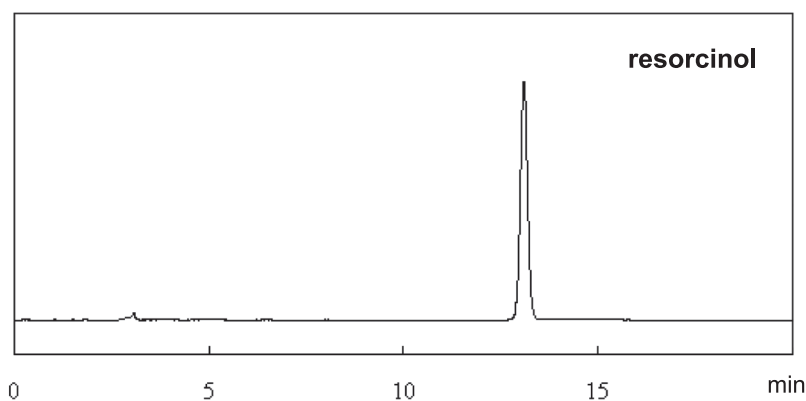
Sample : phenyl salicylate (std.)
hand cream

Pretreatment

1. Measure 1 g of cream; add 40 mL of THF; shake and dissolve.
2. Make up to a total of 50 mL with THF, and filter through 0.45 μ m filter (PTFE).
3. Use filtrate as sample for analysis.



3 - 7 护手霜中间苯二酚的分离



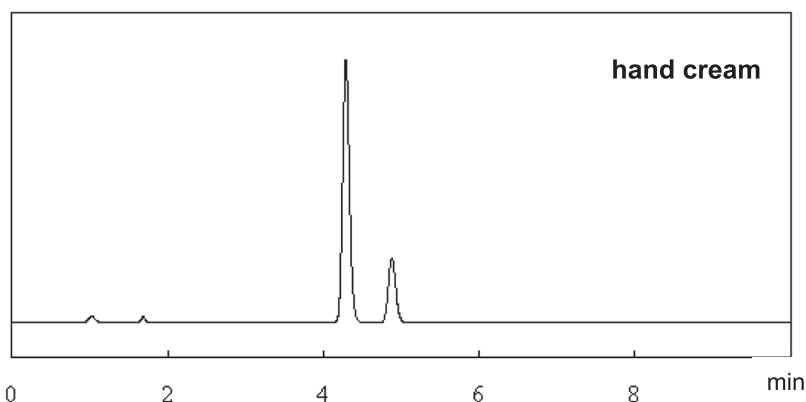
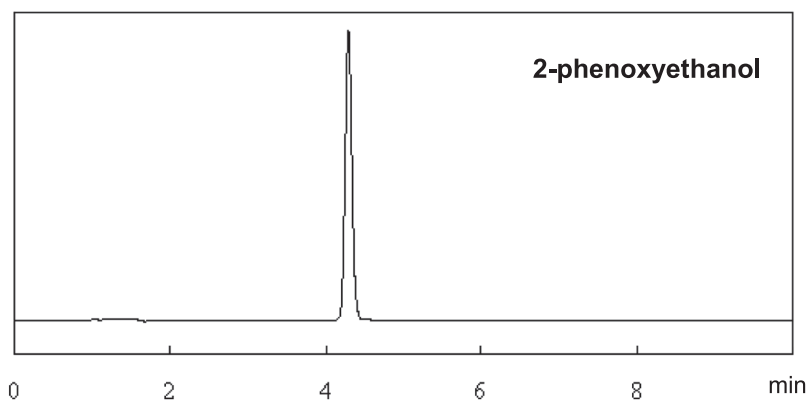
Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 25 cm)
Eluent : H₂O/CH₃OH = 90/10
Flow rate : 1.0 mL/min
Detection : UV (275 nm)
Temperature : 40 °C
Inj. volume : 10 μ L
Concentration: 20 mg/L

Sample : resorcinol (std.)
hand cream

Pretreatment

1. Measure 1 g of cream, add solvent, shake and dissolve.
2. Make up to a total of 50 mL with the solution, and filter through 0.45 μ m filter (PTFE).
3. Use filtrate as sample for analysis.

3 - 8 护手霜中苯氧乙醇的分离



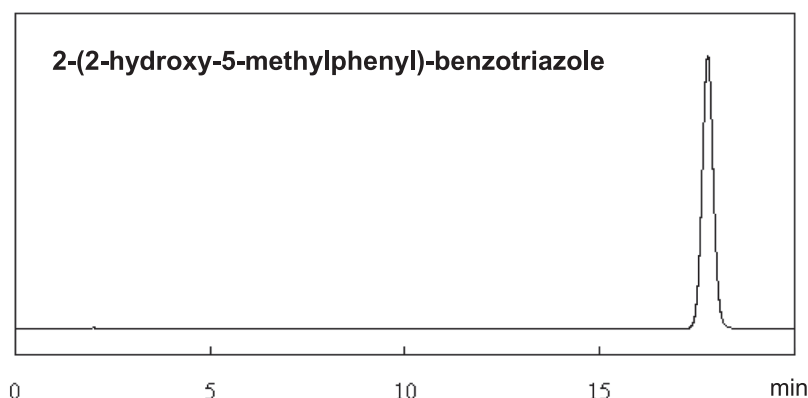
Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : H₂O/CH₃CN = 70/30
Flow rate : 1.0 mL/min
Detection : UV (270 nm)
Temperature : 40 °C
Inj. volume : 20 μ L
Concentration: 100 mg/L (std.)

Sample : 2-phenoxyethanol (std.)
hand cream

Pretreatment

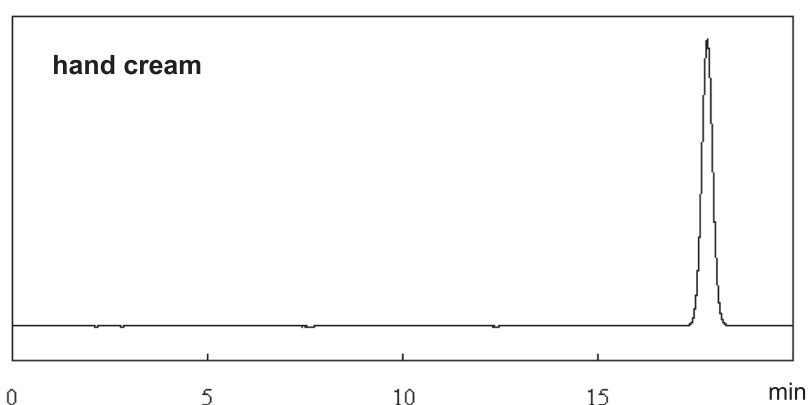
1. Measure 100 mg of cream, add water/methanol at 1/1 ratio; shake and dissolve.
2. Make up to a total of 50 mL with the solution, and filter through 0.45 μ m filter (PTFE).
3. Use filtrate as sample for analysis.

3-9 护手霜中紫外线吸收剂的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 25 cm)
Eluent : H₂O/CH₃OH = 3/17
Flow rate : 1.0 mL/min
Detection : UV (335 nm)
Temperature : 40 °C
Inj. volume : 20 μ L
Concentration: 40 mg/L (std.)

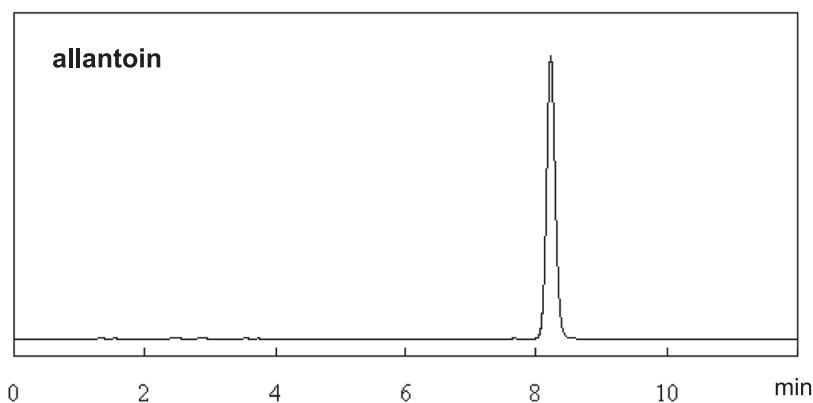
Sample : 2-(2-hydroxy-5-methylphenyl)-benzotriazole (std.)
hand cream



Pretreatment

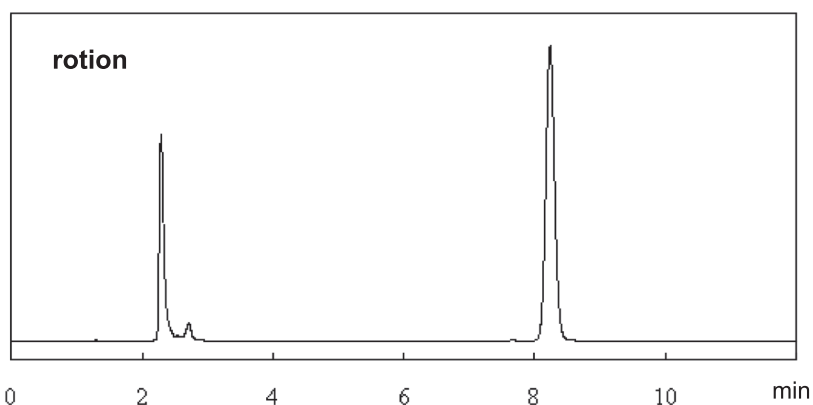
1. Measure out 0.2 g of cream into a beaker, add 2 mL of THF; shake and dissolve.
2. Transfer to 50 mL graduated flask and wash with methanol.
3. Add 2 mL of THF to the same beaker, wash, and add to graduated flask together with dissolved solution.
4. Make up to 50 mL with methanol and use this for sample analysis.

3-10 乳液中尿囊素的分离



Column : TSKgel Amide-80 5 μ m
(4.6 mm I.D. \times 25 cm)
Eluent : H₂O/CH₃CN = 15/85
Flow rate : 1.0 mL/min
Detection : UV (210 nm)
Temperature : 40 °C
Inj. volume : 20 μ L
Concentration: 75 mg/L (std.)

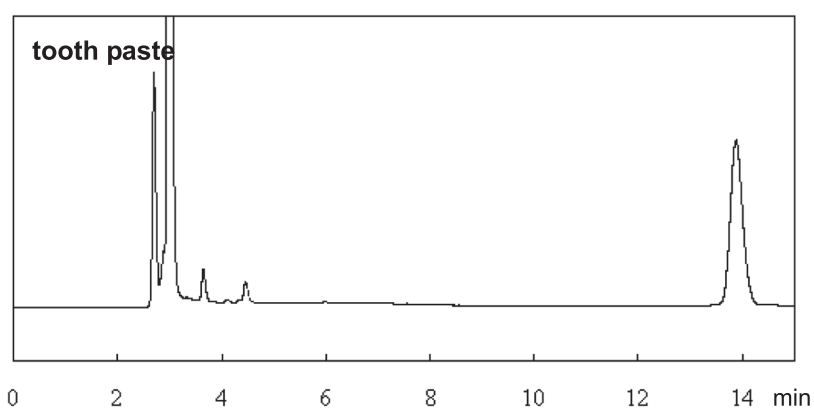
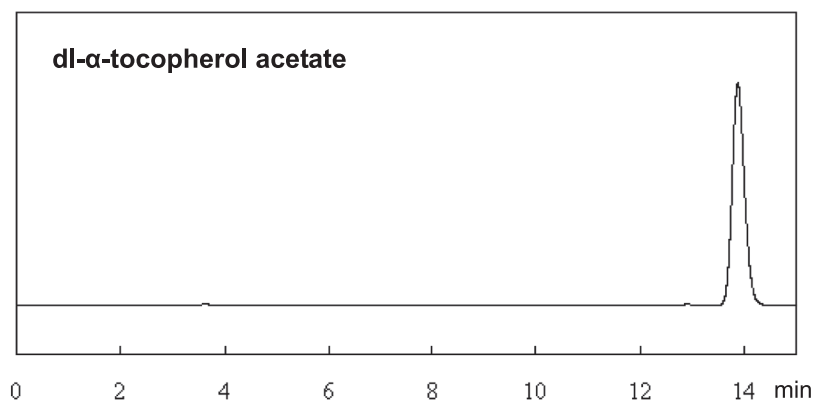
Samples : allantoin (std.)
rotion



Pretreatment

1. Measure 1 g of cream, add 10 mL of solvent; shake and dissolve.
2. Make up to a total of 20 mL with the solution, and filter through 0.45 μ m filter (PTFE).
3. Use filtrate as sample for analysis.

3 -11 牙膏中醋酸盐维生素 E 的分离



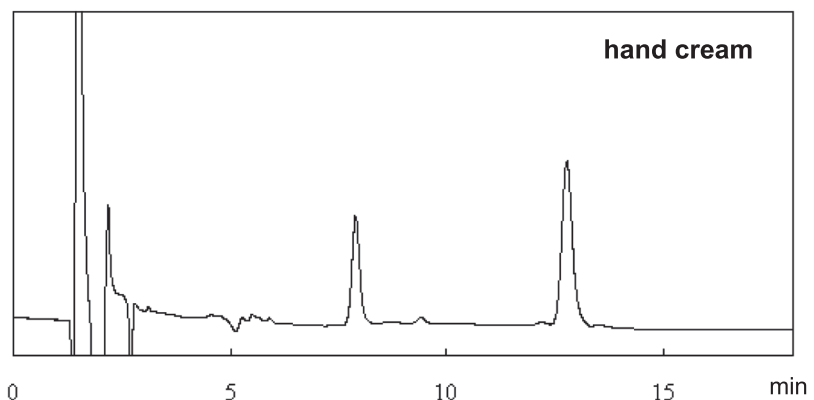
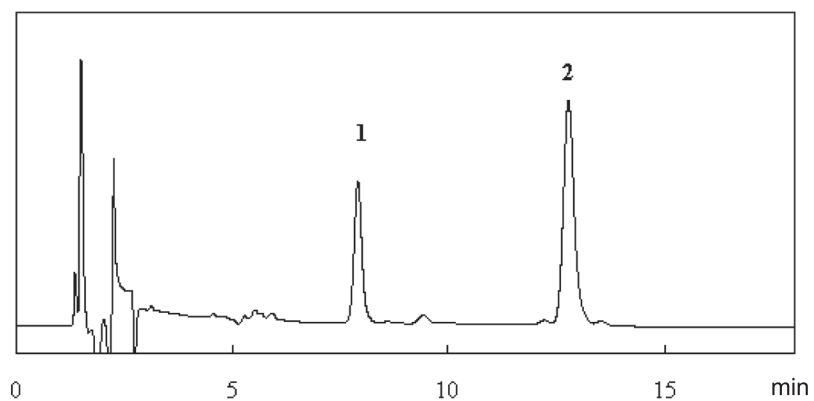
Column : TSKgel ODS-100V 5 μm
(4.6 mmI.D. × 25 cm)
Eluent : CH₃CN/2-propanol = 90/10
Flow rate : 1.0 mL/min
Detection : UV (280 nm)
Temperature : 40 °C
Inj. volume : 20 μL
Concentration: 50 mg/L (std.)

Samples : dl-α-tocopherol acetate (std.)
tooth paste

Pretreatment

1. Add 10 mL of solvent to 1 g of tooth polishing powder, and dissolve using ultrasound.
2. Filter through 0.45 μm filter (PTFE).
3. Use filtrate as sample for analysis.

3 -12 护手霜中甘草酸、甘草次酸的分离



Column : TSKgel ODS-100V 5 μm
(4.6 mmI.D. × 15 cm)
Eluent : (tri-n-octylamine 1.5g +
CH₃COOH 4g + CH₃CN650mL)
+ (EDTA2Na 1g + H₂O 350mL)
Flow rate : 1.0 mL/min
Detection : UV (250 nm)
Temperature : 40 °C
Inj. volume : 10 μL
Concentration: 50 mg/L (std.)

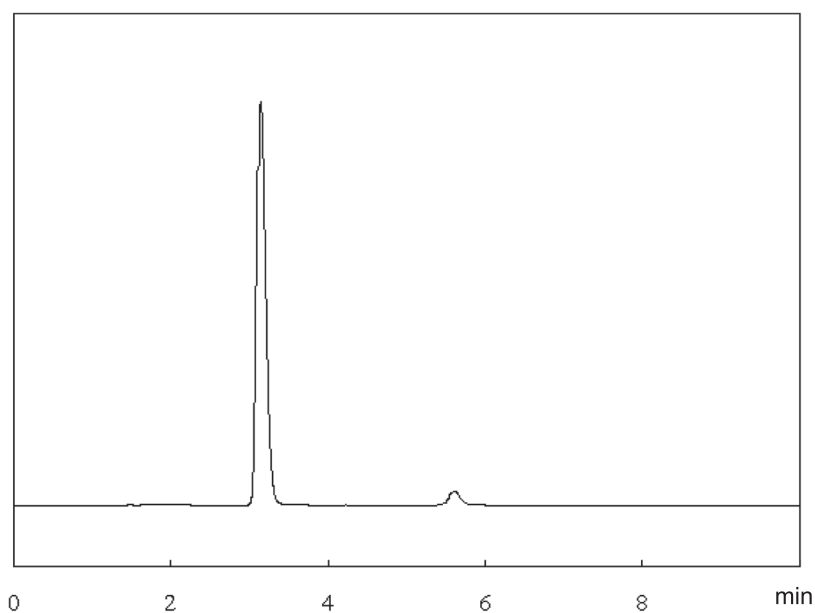
Samples

- 1 : glycyrrhizic acid (std.)
- 2 : glycyrrhetic acid (std.)
hand cream

Pretreatment

1. Dissolve 1 g sample in 10 mL of THF.
2. Pass 4 mL of this solution through a Sep-Pak NH₂ cartridge, and wash with 10 mL of methanol.
3. Elute with elution solvent (2.67% KH₂PO₄/CH₃CN = 50/50) to prepare sample for analysis.

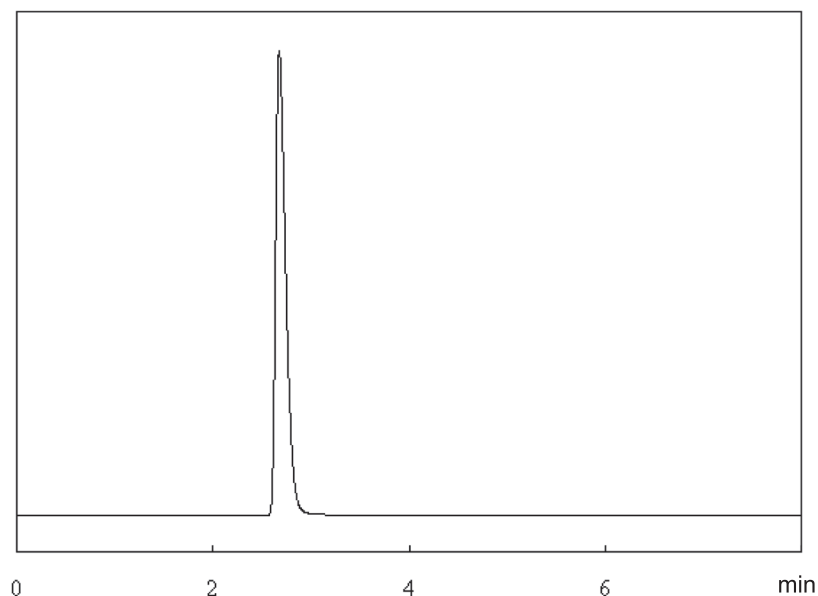
3 -13 巯基乙酸钙的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : 0.2 % H₃PO₄/CH₃OH = 85/15
Flow rate : 1.0 mL/min
Detection : UV (220 nm)
Temperature : 50 °C
Inj. volume : 20 μ L
Concentration: 200 mg/L

Sample : calcium thioglycolate

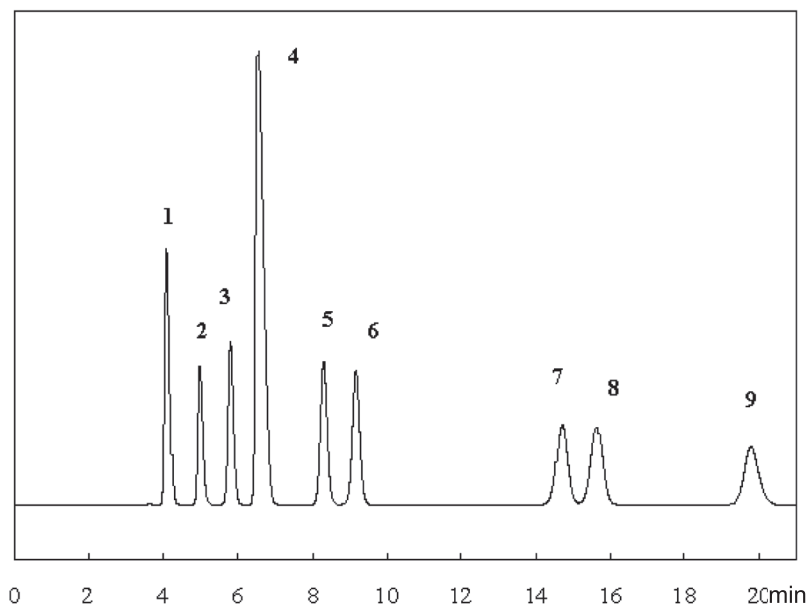
3 -14 苯酚磺酸锌的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : 0.1 mol/L potassium phosphate
buffer (pH 2.3)/CH₃OH = 97/3
Flow rate : 1.0 mL/min
Detection : UV (230 nm)
Temperature : 40 °C
Inj. volume : 10 μ L
Concentration: 20 mg/L

Sample : zinc phenolsulfonate

3 -15 防腐剂的分离(1)

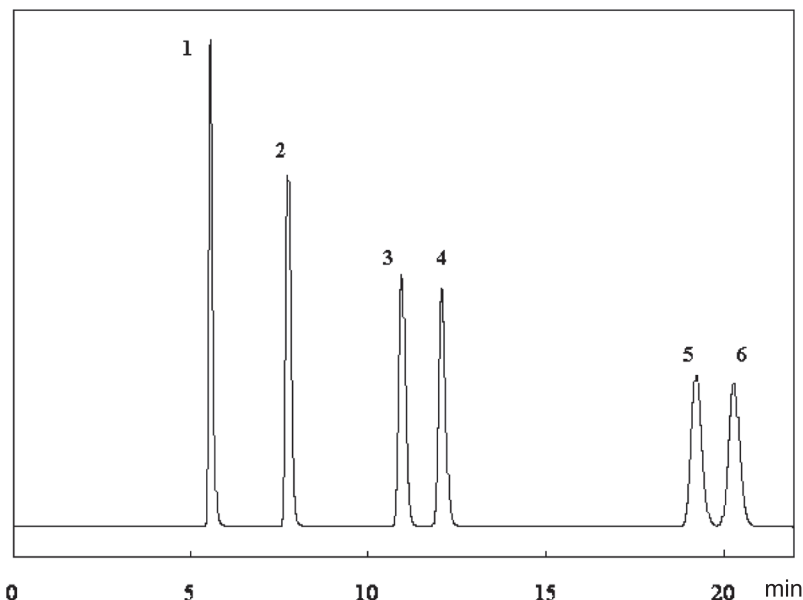


Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : (50 mmol/L NaH₂PO₄/
CH₃OH/CH₃CN
= 50/35/15) + 2 mmol/L
hexadecyltrimethylammonium
chloride (pH5.2)
Flow rate : 1.0 mL/min
Detection : UV (235 nm)
Temperature : 40 $^{\circ}$ C
Inj. volume : 10 μ L
Concentration: 50 mg/L

Samples

- 1 : methyl p-hydroxybenzoate
- 2 : dehydroacetic acid sodium salt
- 3 : ethyl p-hydroxybenzoate
- 4 : sorbic acid sodium salt
- 5 : iso-propyl p-hydroxybenzoate
- 6 : n-propyl p-hydroxybenzoate
- 7 : iso-butyl p-hydroxybenzoate
- 8 : n-butyl p-hydroxybenzoate
- 9 : salicylic acid sodium salt

3 -16 防腐剂的分离(2)

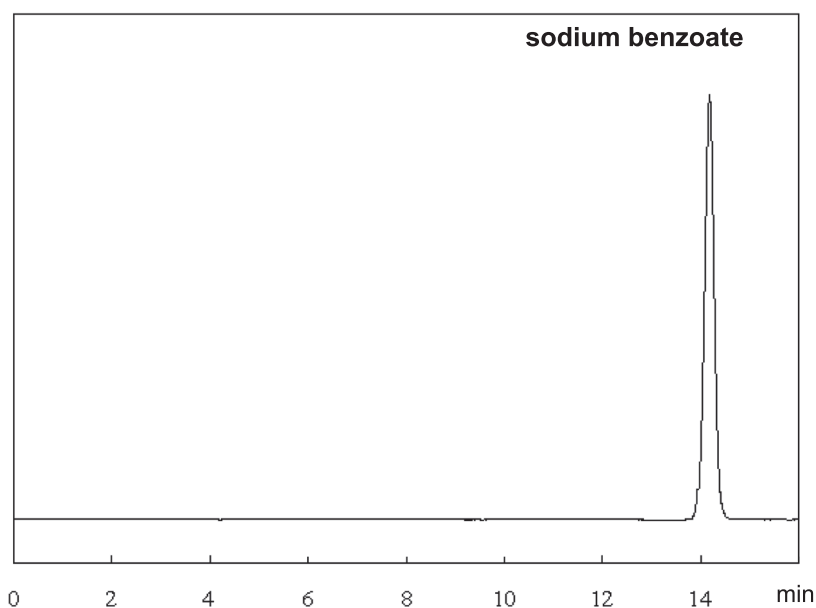


Column : TSKgel ODS-100Z 5 μ m
(4.6 mm I.D. \times 25 cm)
Eluent : 50 mmol/L sodium phosphate
buffer (pH6.8)/CH₃OH = 40/60
Flow rate : 1.0 mL/min
Detection : UV (270 nm)
Temperature : 40 $^{\circ}$ C
Inj. volume : 5 μ L
Concentration: 50 mg/L

Samples

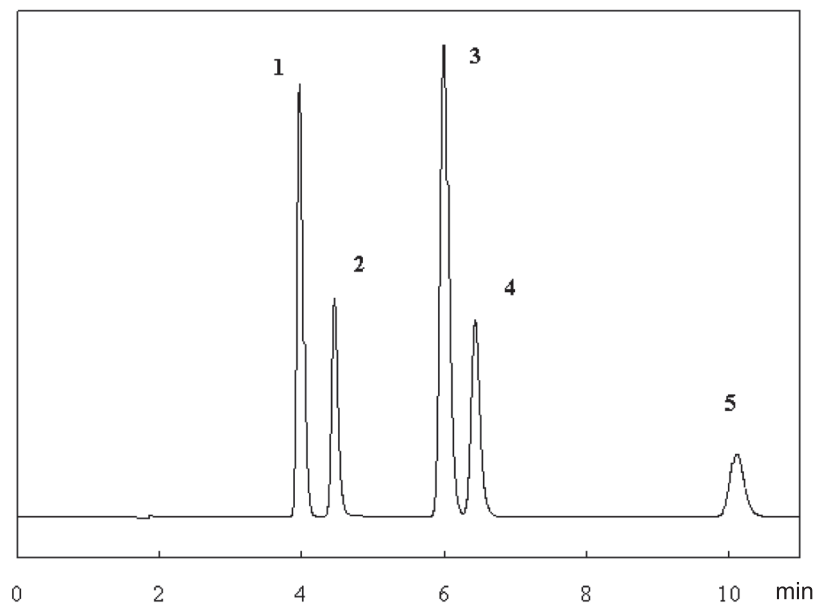
- 1 : methyl p-hydroxybenzoate
- 2 : ethyl p-hydroxybenzoate
- 3 : iso-propyl p-hydroxybenzoate
- 4 : n-propyl p-hydroxybenzoate
- 5 : iso-butyl p-hydroxybenzoate
- 6 : n-butyl p-hydroxybenzoate

3-17 安息香酸钠的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 25 cm)
Eluent : 50 mmol/L sodium phosphate
buffer (pH5.5)/CH₃CN = 97/3
Flow rate : 1.0 mL/min
Detection : UV (227 nm)
Temperature : 40 °C
Inj. volume : 20 μ L
Concentration: 10 mg/L
Sample : sodium benzoate

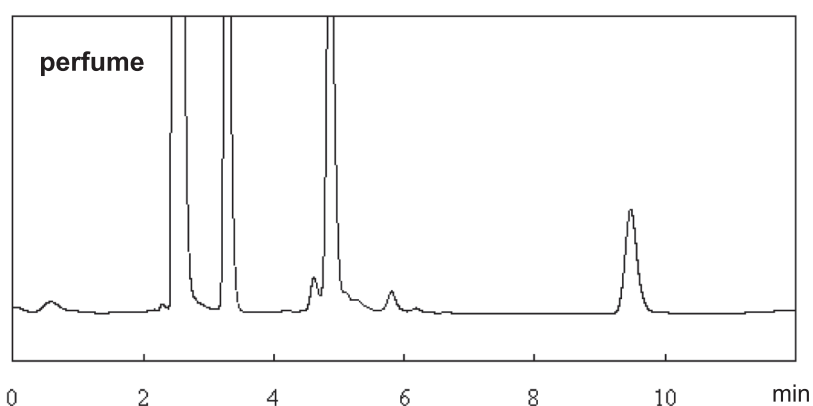
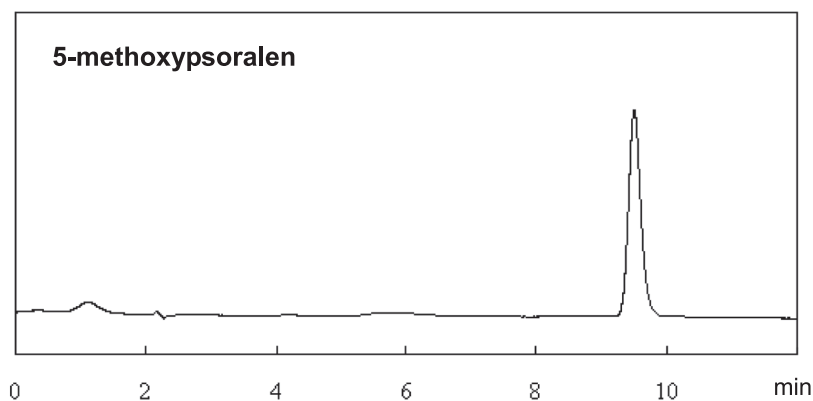
3-18 苯甲酸酯的分离



Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 15 cm)
Eluent : 50 mmol/L sodium phosphate
buffer (pH5.5) + 5 mmol/L
hexadecyltrimethylammonium
chloride/CH₃CN = 60/40
Flow rate : 1.0 mL/min
Detection : UV (227 nm)
Temperature : 40 °C
Inj. volume : 20 μ L
Concentration: 10 mg/L

Sample
1 : 2-phenoxyethanol
2 : methyl p-hydroxybenzoate
3 : sodium benzoate
4 : ethyl p-hydroxybenzoate
5 : n-propyl p-hydroxybenzoate

3 -19 香水中的佛手柑内酯的分离



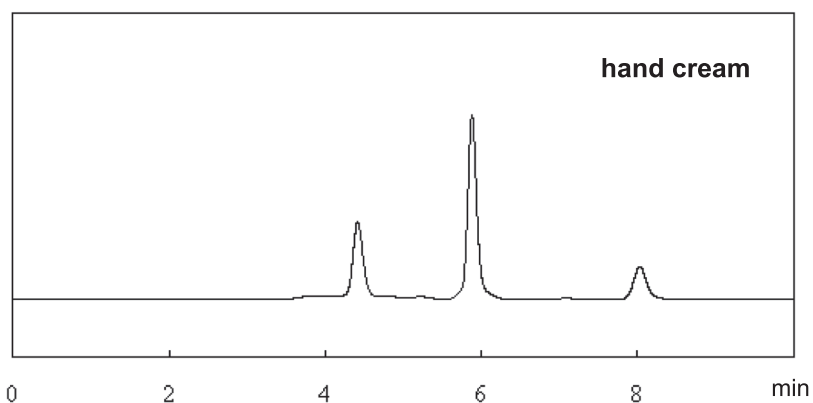
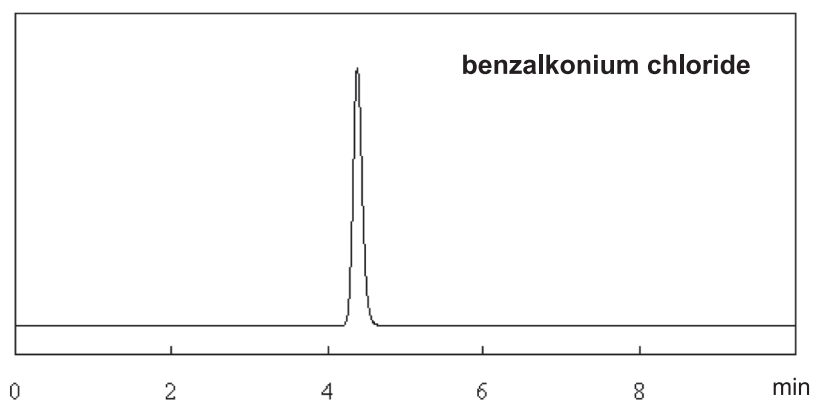
Column : TSKgel ODS-100V 5 μ m
(4.6 mm I.D. \times 25 cm)
Eluent : H₂O/CH₃CN/CH₃OH = 2/1/1
Flow rate : 1.0 mL/min
Detection : UV (268 nm)
Temperature : 40 °C
Inj. volume : 10 μ L
Concentration: 0.5 mg/L (std.)

Samples : 5-methoxypsoralen (std.)
perfume

Pretreatment

1. Measure 0.4 g of sample, and using a 100-mL separatory funnel, add 30 mL water, 5 g sodium chloride, and 30 mL ether; shake for 10 minutes, and isolate the ether layer.
2. Add an additional 20 mL ether to the water layer and extract.
3. Combine the ether layers, dry with 10 g anhydrous sodium sulfate, blow dry with dry nitrogen, and condense to approximately 1 mL.
4. Load condensate onto a florisil column using ether/petroleum ether (7/3), and inject 20 mL of ether/petroleum ether (7/3).
5. Elute with 10 mL of ether/acetone (1/1) and condense eluate to approximately 1 mL using dry nitrogen.
6. Add solvent to condensate to prepare 2-mL sample for analysis.

3 -20 护手霜中杀藻胺的分离



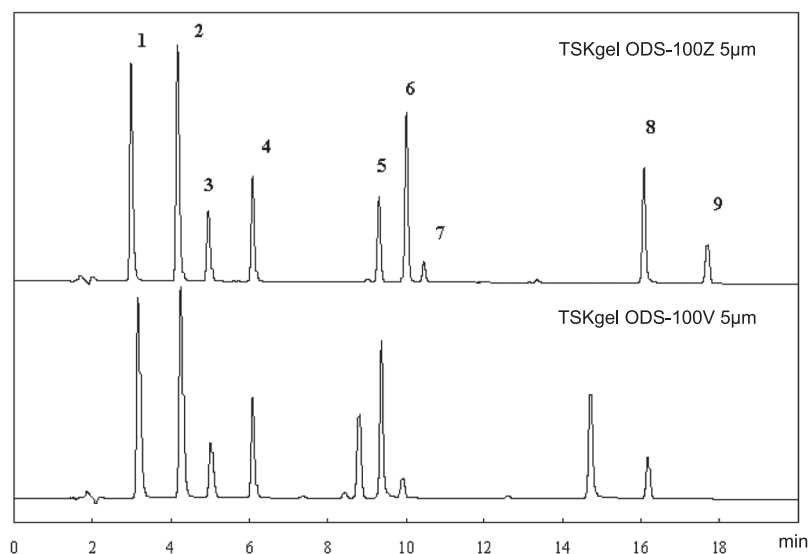
Column : TSKgel SuperAW2500
(4.6 mm I.D. \times 15 cm)
Eluent : 50 mmol/L NaClO₄ in
(H₂O/CH₃CN = 50/50)
Flow rate : 0.6 mL/min
Detection : UV (265 nm)
Temperature : 40 °C
Inj. volume : 10 μ L
Concentration: 1 g/L (std.)

Sample : benzalkonium chloride (std.)
hand cream

Pretreatment

1. Measure 4 g of cream, dissolve in methanol to make up 100 mL.
2. Filter through a 0.45- μ m (cellulose acetone) filter.
3. Dilute filtrate 4-fold with distilled water to prepare sample for analysis.

3 -21 9种苯酚类抗氧化剂的分离



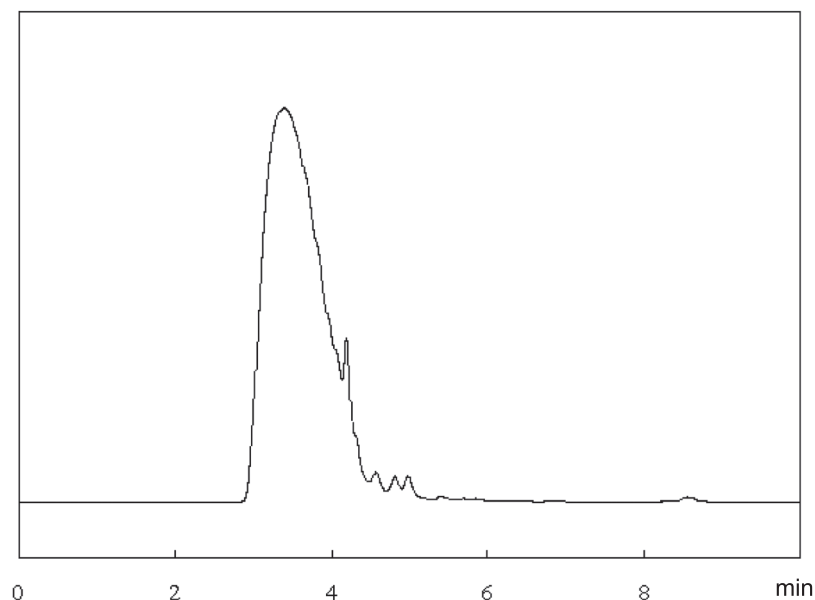
Column : TSKgel ODS-100Z 5 µm
(4.6 mm I.D. × 15 cm)
TSKgel ODS-100V 5 µm
(4.6 mm I.D. × 15 cm)

Eluent A : 5 % CH₃COOH
B : CH₃CN
Gradient : 0min (B 40 %) →
15min (B 90 %) →
25 min (B 90 %)

Flow rate : 1.0 mL/min
Detection : UV 280nm
Inj. volume : 10 µL
Concentration: 50 mg/L

Samples
1 : PG (propyl gallate)
2 : THBP (2,4,5-trihydroxybutyrophenone)
3 : TBHQ (tert-butyl hydroquinone)
4 : NDGA (nordihydroguaiaretic acid)
5 : BHA (butylated hydroxyanisole)
6 : OG (octyl gallate)
7 : HMBP (4-hydroxymethyl-2,6-di-tert-butylphenol)
8 : DG (dodecyl gallate)
9 : BHT (butylated hydroxytoluene)

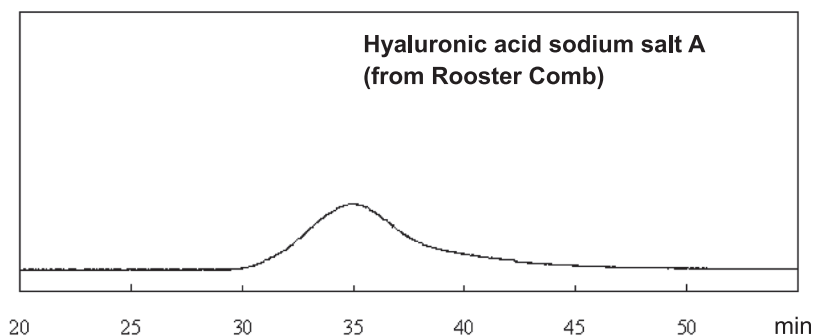
3 -22 聚氨基丙基双胍的分离



Column : TSKgel SuperAW3000
(6.0 mm I.D. × 15 cm)
Eluent : 1.0 % H₃PO₄
Flow rate : 0.6 mL/min
Detection : UV (235 nm)
Inj. volume : 5 µL
Temperature : 40 °C
Concentration: 2 g/L

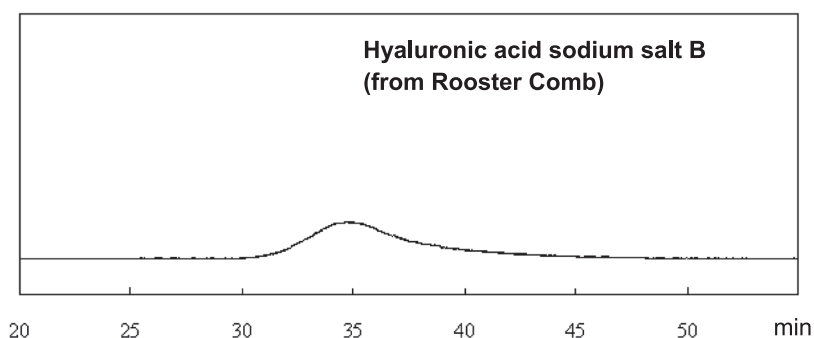
Samples : polyaminopropyl biguanide

3 -23 透明质酸的分离

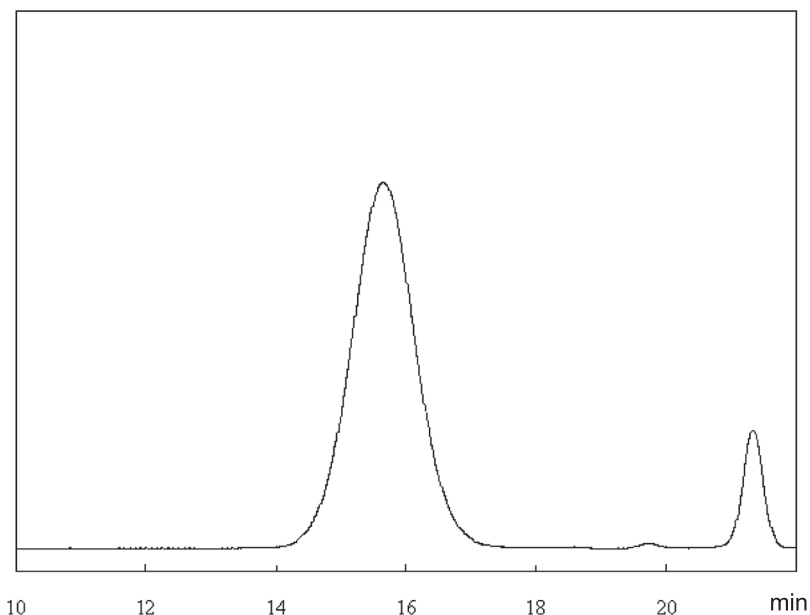


Column : TSKgel G6000PW + G5000PW
(7.5 mmI.D. × 30 cm × 2)
Eluent : 0.1 mol/L NaNO₃
Flow rate : 0.3 mL/min
Detection : RI
Temperature : 40 °C
Inj. volume : 100 μL
Concentration: 0.1 g/L

Sample : hyaluronic acid sodium salt



3 -24 硫酸软骨素的分离



Column : TSKgel GMPW_{xL} × 2
(7.8 mmI.D. × 30 cm × 2)
Eluent : 0.1 mol/L NaNO₃
Flow rate : 1.0 mL/min
Temperature : 40 °C
Inj. volume : 100 μL
Detection : RI
Concentration: 1.0 g/L

Sample : chondroitin sulfate

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