NORWEGIAN UNIVERSITY OF SCIENCE AND TECHNOLOGY
DEPARTMENET OF CHEMISTRY

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EXAMINATION IN COURSE SIK 3038 / MNK KJ 253

CHROMATOGRAPHY

Thursday, 30th May 2002
5 hours, 09:00 - 14:00
(2,5 vekttall)

Language : English (four - 4 - pages)
Permitted aids: B1-type calculator with empty memory (as specified in NTNU's list of calculators approved for use in examinations at NTNU).
No other aids (i.e. printed or handwritten texts or notes) are allowed.
Deadline for the results to be anuounced : Wednesday 19th June 2002.

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1. (12p) Chromatography theory uses the following terms / abreviations:
k (or k'), α, t_R, R_S, N, N_{eff} and *HETP (H)*.

- Name each of these terms/abbreviations, and briefly explain what they express.
- For α and R_S, what are, by definition, the minimum numerical values of these terms ?
- List the terms that describe column properties only (i.e. their numerical values are not dependant on the analyte(s)).
  On increasing the values of the other terms listed above, which ones would lead to an increase R_S ?
- How would you express N_{eff} by making use of only the other parameters / terms listed above ?
- What is the relation between k (k'), t_R and t_0 ?

2. (8p) In order to analyse a mixture of N_2, CH_4, CO_2 and H_2S, suggest :
(i) a suitable chromatographic method of analysis (i.e. a chromatographic principle),
(ii) type of chromatographic column
(iii) the most suitable detector, and
(iv) the most suitable mobile phase(s).
Briefly comment on your choices.

3.

a) (3p) Name, and briefly describe, the three kinds of gradients that can be used in SFC (Supercritical Fluid Chromatography).

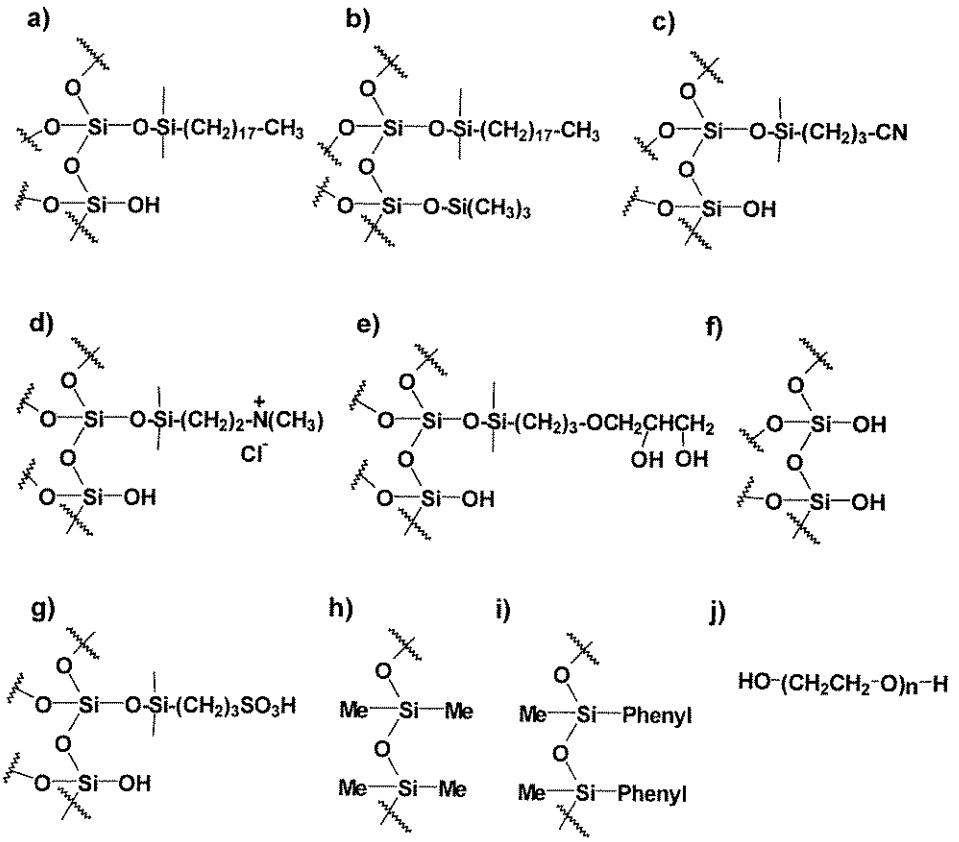
(3.)

b) (3p) Draw a diagram illustrating how density varies with the pressure and temperature for a supercritical fluid.

c) (3p) What are the three types of compounds SFC is especially suitable for.

d) (3p) List four detectors that may be used in SFC. For each of them, suggest where the flow restrictor has to be positioned with respect to the detector. Explain the reason for your positioning of the restrictor.

4. What are the compounds a) - j) used for ? (*Cfr.* partial structures given below.)
(10x2p) Give a name and/or an expression for each of these compounds and comment on their properties.



5. Suggest an elution sequence, as well as detector(s) you would expect to be suitable for HPLC analyses of the compounds given in each of the examples a) to d), using the conditions mentioned in each of the examples, respectively.

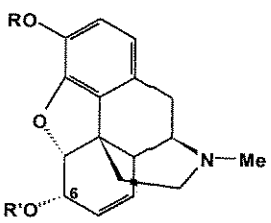
a) (2p) ethylbenzene, *p*-isopropyltoluene and 2-ethyltoluene; column/stationary phase: C18; eluent : acetonitrile/water (70/30);

(5.)

b) (2p) codeine, morphine and 6-acetylmorphine;
column/stationary phase: SiO_2;
eluent: hexane/dichloro-
methane/MeOH (75/20/5).

Structures:

codein; R = Me, R' = Ac
morfin; R og R' = H
6-Ac-morfin, R = H, R' = Ac



c) (2p) codeine, morphine and 6-acetylmorphine;
column/stationary phase: ODS (octadecylsilyl);
eluent: MeOH/acetonitrile.

d) (3p) OPA (orthophtalaldehyde) derivatives of *n*-decylamine, *n*-butylamine, *n*-octylamine and *n*-hexylamine;
column/stationary phase: C18; eluent: acetonitrile/water (25/75).
Draw the structural formula of one of (the four) OPA derivatives prepared and analysed here.

e) (4p) If the mobile phase velocity is increased by a factor of 2 in the analyses 5.a) - 5.d) above, how do these parameters derived from the chromatograms change :
retention time, t_R , peak width at half maximum, $t_{w0,5}$, retention factor, k (= capacity factor, k') and resolution, R_S ?

6.

a) (3p) Which desirable properties should a stationary phase for gas-liquid chromatography, GLC, have (list three of them) ?

b) (3p) Which carrier gases are suitable for the GLC analysis of propanone and propane-2-ol ?

c) (1p) Why is gas-solid chromatography, GSC, not suitable for the analysis of "common" organic chemical purposes ?

d) (2p) Why does capillary gas chromatography (High Resolution GC) use other injection systems than GC with packed columns ?

e) (3p) Sketch representative figures of chromatograms form GLC as obtained from a packed and from a capillary column, respectively, that clearly illustrate the differences between the two columns. Explain the differences.

Based on the information given below :

f) (2p) Calculate how many % of isopropylamine and of diethylamine are in the mobile phase.

g) (4p) Calculate resolution, R_S , for peaks 3 and 4. There are two different equations that can be used for the calculation of chromatographic resolution from a chromatogram. Calculate using both methods (the results may differ slightly).

h) (1p) Show how the value for t_0 , given i the table below, could be calculated from the other information available.

i) (2p) Explain why you would have expected the elution sequence found in the chromatogram shown below

j) (1p) Which is the chromatographic principle this analysis is an example of ?

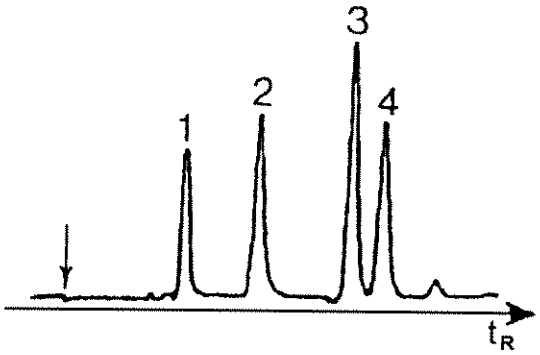
k) (2p) What are, generally speaking, the advantages and disadvantages of using nitrogen as a carrier gas ?

Data to problems 6.f) - 6.j) :

Primary, secondary, tertiary amines C_3-C_6

Separation of underivatized volatile amines on a wide-bore fused silica CP-Sil 5 CB column

Technique	: GC - capillary
Column	: 10 m × 0.53 mm fused silica WCOT CP-Sil 5 CB (5.0 μm) (Cat.no. 7645)
Temperature	: 50° C → 200° C, 5° C/min
Carrier gas	: N_2, 10 kPa (0.1 bar), 52 cm/s
Injector	: direct T = 300° C
Detector	: FID, 100 × 10^{-12} Afs T = 275° C
Sample size	: 0.2 μl
Solvent sample	: tetrachloroethene (perchloroethylene)



Peak identification:	Topp	t_R	$t_{w,0,5}$
(t_o)		(0,32 min)	
1. isopropylamine	1	1,12 min	6,7 sec
2. diethylamine	2	1,98 min	8,0 sec
3. diisopropylamine	3	2,97 min	8,2 sec
4. triethylamine	4	3,23 min	9,3 sec