DETERMINATION OF TRACE CONCENTRATIONS OF ALIPHATIC AMINES IN THE GAS PHASE AFTER PRECONCENTRATION ON A SOLID SORBENT

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The technique of trapping aliphatic amines from gaseous and liquid phases on solid polymeric organic sorbents followed by thermal desorption and quantitation by gas chromatography or, after conversion to fluorescent (UV) derivatives, by liquid chromatography was studied. The interaction of 8 primary aliphatic amines and 4 secondary amines with 3 sorbents, viz. Tenax GC, Chromosorb 103, and Separon SE, was examined. Analytical GC and HPLC procedures were developed for butylamine and subsequently tested on a $C_4 - C_7$ primary alkylamine mixture.

Low-molecular-weight primary and secondary aliphatic amines are potential components in the formation of carcinogenic nitrosamines, and so their determination in the working environment and in free air is gaining in importance. In addition to acid absorption solutions, solid sorbents are also used to trap and preconcentrate amines from air and water[1,2]. The assets of solid sorbents include a high preconcentration factor, rapid adsorption, and a suitable form of the trapped substances for subsequent sample handling, transport and quantitation.

The following factors must be taken into consideration when selecting a suitable sorbent[3]:

– Air sample volume that can be passed through the sorbent bed without analyte breakthrough.
– Simplicity, rapidity and completeness of desorption of the substances trapped.
– Sorbent background.
– Possibility and extent of sample decomposition during the preconcentration, storage and elution.
– Desorption.
– Affinity of the sorbent for water vapour.

The breakthrough volume (or maximum sample volume)[4,5], which depends on the retention volume and number of theoretical plates, is the principal criterion in assessing the sorbent capacity. This value is affected particularly by the following parameters[6]:

– Porosity, specific surface area, amount and homogeneity of sorbent and inertness of its surface with respect to sorbate.
– Concentrations and chemical structure of the components in sample.
– Velocity and temperature of gas phase passed through the sorbent.
– Shape and size of the sampling tube (sorbent bed).

Polymeric sorbents conventionally used in the chromatography of amines include Tenaxes (GC, TA), Chromosorbs (101, 103), Porapaks (N, T), and Separons (SE).

Derivatization procedures can be employed in chromatography to enable or improve detection and/or separation. In gas chromatography, derivatization is applied with a view to converting the substances into more volatile ones[7], whereas in liquid chromatography, derivatization reduces analyte volatility and increases detection sensitivity[8]. Conventional fluorescent derivatization agents for amines in liquid chromatography include dansyl chloride[8–11], *o*-phthalic dialdehyde[12], NDA/CN (naphthalene-2,3-dicarboxaldehyde)[13], fluorescamine[8,14], bansyl chloride[15], 1,2-NBI-6-SO_2Cl (1,2-naphthoylbenzymidazole-6-sulfochloride)[16], acridone and acridine derivatives[15], 9-FMOC (ref.[17]), 9-FMOC-Cl (9-fluorenylmethyl methanoate)[18], NBD-F and NBD-Cl (4-fluoro-7-nitrobenzo-2-oxa-1,3-diazole)[9], OPT (*o*-carboxybenzoic acid)[9], 1,2-naphthoquinone-4-sulfonate[19], FQCA (3-(2-furoyl)quinoline-2-carbaldehyde)[20], activated carbamates[21], etc. For UV detection, SNPA (*N*-succinimidyl-*p*-nitrophenyl acetate)[22], benzoylating agents[7], and DNBF (2,4-dinitrofluorobenzene)[7] are among suitable agents.

EXPERIMENTAL

Chemicals

Alkylamines from methylamine through octylamine (Janssen Chimica, Beerse, Belgium), *N*-succinimidyl-*p*-nitrophenyl acetate (SNPA) (Pierce Chemical Company, Rockford, Illinois, U. S. A.), *N*,*N*-dimethyl-5-aminonaphthalene-1-sulfochloride (dansyl chloride), *o*-phthalic dialdehyde (OPA), and 2-mercaptoethanol (Fluka, Buchs, Switzerland); tetrahydrofuran stabilized (Laborchemie Apolda, Germany), acetone p.a., methanol, ethanol for UV, and 2-propanol p.a.; sodium carbonate decahydrate p.a., sodium hydrogencarbonate p.a., sodium tetraborate decahydrate p.a., sodium acetate p.a., sodium hydrogen phosphate p.a., and sodium hydroxide p.a. (Lachema, Brno, The Czech Republic); nitrogen from a pressure vessel and liquid nitrogen (VCHZ Synthesia, Pardubice, The Czech Republic), and solid carbon dioxide (Chemopetrol, Litvinov, The Czech Republic).

Tenax GC (60 – 80 mesh) and Chromosorb 103 (80 – 100 mesh) (Serva, Heidelberg, Germany), Separon SE (150 μm) (Tessek Praha, The Czech Republic).

Apparatus

An HP 1090M liquid chromatograph equipped with a diode array detector and an HP 1046A fluorescence detector, an HP 9000/310 data station (Hewlett–Packard, Avondale, PA, U.S.A.). A glass column 150 mm × 3 mm i.d. packed with Separon SGX C18 7 μm (Tessek Praha, The Czech Republic).

A Chrom 5 gas chromatograph with an FID (Laboratorni pristroje Praha, The Czech Republic), an ITG 309 integrator (Budoucnost Blatnice Cooperative, The Czech Republic), Apex v. 3.0 chromato-

graphic integrator (DataApex Praha, The Czech Republic), a glass column 1.2 m × 3 mm i.d. packed with Carbopack B + 4 wt.% PEG 20M + 0.8 wt.% KOH.

An U2C thermostat (Germany), a homemade thermostatted evaporating device and a thermodesorption facility.

The columns tested were U-shaped, 60 – 70 cm long, 3 mm i.d., each packed with 1 g of sorbent. Sorption tubes were made of glass, 62 mm long, 3 mm i.d. for gas chromatography, or 150 mm long, 7.5 mm i.d., with tapered ends for liquid chromatography (Table I).

Sorbents were conditioned in the glass columns or sorption tubes in the gas chromatographic thermostat for 6 h in a stream of helium (flow rate 5 ml min^{-1}); temperature was increased in a preprogrammed manner up to 200 °C for Chromosorb 103 and Separon SE, or up to 230 °C for Tenax GC. The freezing-out capillary was made of teflon, 70 cm long, 0.8 mm i.d., coiled in the central part.

Procedure

Gas chromatography. The specific retention volume and number of theoretical plates were determined as follows. The V_g values were plotted against $1/T$ and fitted by the equation

$$\log V_g = A/T + B \quad , \tag{1}$$

where A and B are constants. The maximum gas volume which can be passed through the tube was calculated[23–26] as

$$V_{max} = \frac{V_g w_L T_k 101.325}{p_b 273.15}\left(1 - \frac{2}{\sqrt{n}}\right), \tag{2}$$

where n is the number of theoretical plates of the packing, p_b is atmospheric pressure (in kPa), t_{max} is the maximum recommended temperature for desorption (in °C), T is temperature (in K), T_k is the suction temperature (in K), V_g is the specific retention volume (in l), and w_L is the packing weight (in g). The maximum sample volume (breakthrough volume) V_{max} is obtained in l. The gas chromatographic software GC-CHAR (ref.[27]) was employed to evaluate the interaction of sorbents with the amines.

Simulation of exposure of sorbents from the gas phase was performed from an evaporation vessel thermostatted at 40 °C (Fig. 1). The carrier gas flow rate was 20 ml min^{-1}. Subsequently, solution of the amine in methanol was injected. The time of suction was 15 min, during which approximately

TABLE I
Parameters of preconcentration tubes for butylamine

Sorbent	t_{max}, °C	V_{max}, l/g	w_L(GC), g	V_{max}(GC) l/tube	w_L(HPLC), g	V_{max}(HPLC), l/tube
Tenax GC	300	7.14	0.0478	0.34	0.3652	2.6
Chromosorb 103	190	7.60	0.1240	0.94	0.7004	5.3
Separon SE	190	13.72	0.1393	1.91	0.7445	10.2

300 ml of the gas mixture was passed through. The exposed sorption tubes were desorbed in the homemade thermodesorption device (Fig. 2) attached to the gas chromatographic injection section. The thermodesorption temperature was about 180 °C, heating time was 3 min.

Derivatization and HPLC. This part of the experiment called as sorption derivatization technique was based on the following sequence: trapping on sorbent – thermal desorption – derivatization[28] – HPLC. The preparation and exposure of the sorption tubes is described above. Desorption was performed in a device shown in Fig. 3. The exposed sorption tube was placed in a furnace, one end was

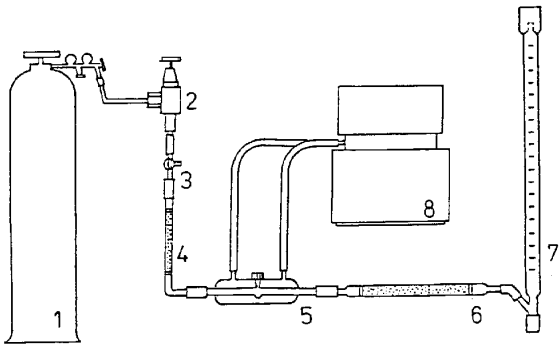

FIG. 1
Sorption (exposure) apparatus. 1 Nitrogen pressure vessel, 2 needle valve, 3 three-way valve, 4 carbonaceous molecular sieve, 5 proportioning evaporating vessel, 6 sorption tube, 7 bubble flowmeter, 8 thermostat

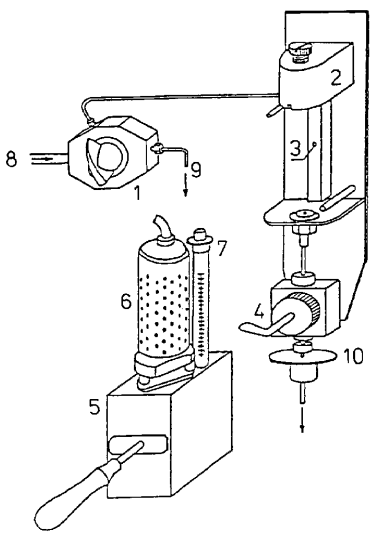

FIG. 2
Thermal desorption device for gas chromatographic analysis. 1 Two-way valve, 2 sorption tube clamping, 3 resistance thermometer sensor, 4 one-way valve, 5 heating block, 6 heating element, 7 Vertex thermometer, 8 carrier gas feed, 9 carrier gas inlet capillary for GC, 10 chromatographic injection head

attached to helium feed at 10 ml min^{-1}, the other end was attached to a freezing-out capillary which had been cooled in liquid nitrogen. The temperature in the furnace was adjusted to 180 °C, and desorption was allowed to proceed for 10 min.

Three reagents were used for derivatization: dansyl chloride, OPA, and SNPA.

Derivatization with dansyl chloride[8]. After terminating the thermal desorption of amine from the exposed enrichment tube, the freezing-out tube was removed from the system, its end was attached to a syringe containing 3 ml of solution of the reagent in acetone (0.1 g l^{-1}), and the frozen-out amine was eluted into a volumetric flask which contained 1 ml of $NaHCO_3$ solution (c = 0.1 mol dm^{-3}) for pH adjustment roughly to 8.5. The reaction mixture was agitated and the vessel was placed in a thermostat at 40 °C for 10 min.

Derivatization with *o*-phthalic dialdehyde[8]. First, three solutions were prepared as follows: solution I contained 50 mg of *o*-phthalic dialdehyde in 5 ml of ethanol, solution II contained 22.5 ml of 2-mercaptoethanol in ethanol, and solution III contained 1.7 g of sodium tetraborate decahydrate and 1.6 g of sodium carbonate decahydrate in 200 ml of water (buffer pH 10.5). The solution for derivatization contained 1.5 ml of solution I, 1.5 ml of solution II, and 90 ml of solution III. The frozen-out amine was taken up in 3 ml of this solution and allowed to stand for 5 min at room temperature.

Derivatization with SNPA (ref.[22]). The frozen-out amine was eluted from the capillary with 2 ml of tetrahydrofuran and collected in a vessel containing 1.5 ml of distilled water. SNPA (10 mg) was added, and the reaction mixture was allowed to stand at room temperature for 30 min. The excess reagent was decomposed with 0.2 ml of saturated aqueous sodium carbonate solution.

Analysis by HPLC. Derivatization was carried out in 5 ml volumetric flasks, which were made up to the mark with methanol, and 15 μl aliquots were injected into the liquid chromatograph. For all derivatives, their UV and fluorescence spectra were measured and the optimum detection wavelengths identified.

Analysis of dansyl derivatives. Dansyl derivative of butylamine was analyzed by using a methanol–water 70 : 30 mobile phase at a flow rate of 1 ml min^{-1}. Isocratic elution was applied to the mixture of butylamine through heptylamine; methanol–water 70 : 30 mobile phase, gradient elution

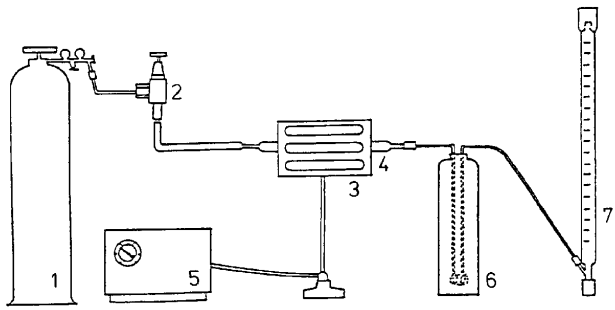

FIG. 3

Desorption apparatus for derivatization. 1 Helium pressure vessel, 2 needle valve, 3 furnace, 4 sorption tube, 5 furnace control, 6 Dewar vessel with liquid nitrogen and freezing-out capillary, 7 bubble flowmeter

with 68 – 100 vol.% methanol in water within 7 min. Fluorescence detection using λ_{ex} = 240 nm, λ_{em} = 500 nm.

Analysis of OPA derivatives. The butylamine derivative was analyzed using a methanol–water 70 : 30 mobile phase at a flow rate of 1 ml min^{-1}. The butylamine through heptylamine mixture was eluted with a gradient of 70 – 90 vol.% methanol in water within 10 min. Fluorescence detection: λ_{ex} = 223 nm, λ_{em} = 435 nm.

Analysis of SNPA derivatives. The butylamine derivative was analyzed using a methanol–water 50 : 50 mobile phase at a flow rate of 1 ml min^{-1}. The butylamine through heptylamine mixture was separated by gradient elution with 45 – 70 vol.% methanol in water within 10 min. Absorption detection at λ_{max} = 280 nm.

RESULTS AND DISCUSSION

This work was part of a complex study[29,30] dealing with the determination of low concentrations of aliphatic amines in gaseous and liquid phases.

Investigation of Sorbents

The dependence of the specific retention volume on temperature was measured for 8 primary and 4 secondary aliphatic amines using 3 different sorption packings. The retention and breakthrough data gave evidence that the 3 sorbents, viz. Tenax GC, Chromosorb 103, and Separon SE, are all well suited to the trapping and preconcentration of primary and secondary aliphatic amines (Figs 4*a*, 4*b*, Table II). The sorbents

TABLE II
V_{max} values (1/g packing) at 20 °C/101.325 kPa: numbers of theoretical plates are given in parentheses

Amine	Tenax GC	Chromosorb 103	Separon SE
Methylamine	0.04 (46)	0.05 (52)	0.18 (94)
Ethylamine	0.25 (60)	0.18 (73)	0.63 (148)
Propylamine	1.19 (227)	1.09 (190)	2.96 (208)
Butylamine	7.14 (257)	7.60 (303)	13.72 (315)
Pentylamine	41.28 (340)	28.52 (375)	75.61 (360)
Hexylamine	322.8 (530)	131.7 (390)	214.1 (614)
Heptylamine	1 095 (1 190)	686.5 (520)	660.7 (900)
Octylamine	4 168 (4 000)	2 405 (1 150)	2 574 (2 400)
Dimethylamine	0.20 (90)	0.07 (220)	0.27 (113)
Diethylamine	6.05 (250)	2.62 (366)	4.92 (200)
Dipropylamine	167.1 (870)	35.08 (490)	52.80 (288)
Dibutylamine	2 803 (2 650)	1 477 (670)	895.4 (1 810)

exhibit relatively high breakthrough volumes, a good stability for the thermal desorption, and a good reproducibility.

The observed and calculated breakthrough volumes (Table II) are in a good agreement with recent published data, e.g. refs[4,31]. Brown and Purnell[4] claim that due to its values, methylamine is not applicable. The V_{max} volumes obtained by us for methylamine and ethylamine are lower than 0.65 l g^{-1}, hence, too low for practical application. Propylamine can only be treated by using Separon SE. Therefore, alkylamines starting from butylamine were used in this study.

The log V_g vs boiling temperature plot for the homologous series (Fig. 5) allows us to predict the specific retention volumes of the missing members or to check the observed and calculated data with a sufficient accuracy.

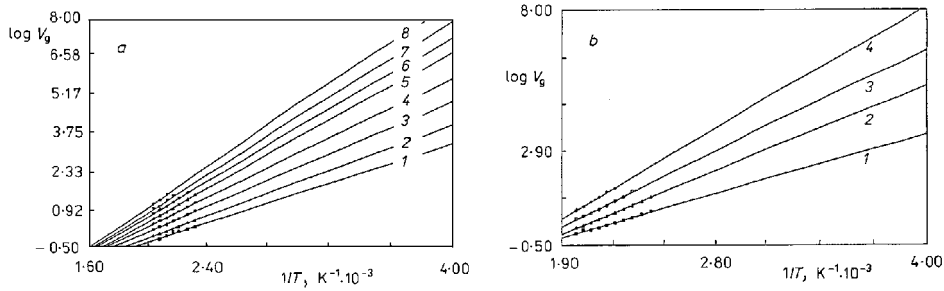

FIG. 4

Dependence of V_g on $(1/T) \cdot 10^3$. (*a*) Primary amines on Separon SE: *1* methylamine, *2* ethylamine, *3* propylamine, *4* butylamine, *5* pentylamine, *6* hexylamine, *7* heptylamine, *8* octylamine. (*b*) Secondary amines on Separon SE: *1* dimethylamine, *2* diethylamine, *3* dipropylamine, *4* dibutylamine

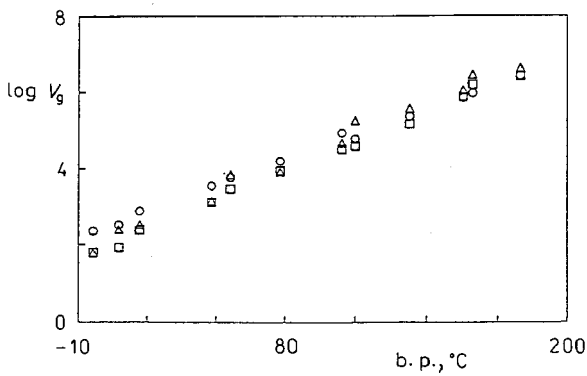

FIG. 5

Dependence of log V_g of primary and secondary amines at 20 °C on their boiling temperatures. Sorbent: Δ Tenax GC, ❒ Chromosorb 103, ❍ Separon SE

Care must be exercised in preparing the testing columns and sorption tubes. The material must have a well-defined weight and must be packed and conditioned in a reproducible manner. It is particularly poor conditioning that can deteriorate the quality of the sorption packing.

The attainable detection limit is the principal criterion of usability of sorbents. This is given by the V_{max} value, which is most favourable with Separon SE for lower primary amines (up to pentylamine), and with Tenax GC for amines starting with hexylamine. Tenax is also the most suitable material for secondary amines. Moreover, Tenax GC exhibits a high thermal stability, owing to which the tube packing has a long service time.

Sorption and Desorption of Amines

The following criteria apply to the choice of sorbent: the specific (breakthrough) volume at exposure temperature, desorption recovery, and overall efficiency, which involves, in addition to the completeness of sorption during sampling and desorption recovery, some additional parameters such as the stability of the compound on the sorbent.

For determining the desorption recovery, the overall efficiency of the procedure, and sorbent capacity, it is convenient to bring sorbate in contanct with sorbent in the gas phase. The conventional direct injection of the liquid sorbate or its solution in a volatile solvent[32] is simple but does not correspond to the actual sampling conditions.

When selecting a sorbent for gas phase sampling it is usually sufficient to introduce a known amount of substance (amines) in the gas phase, irrespective of the stability of concentration in the passed-through air. A simple discontinuous method was applicable: a thermostatted evaporating device into which the sample of amine in methanol was injected was used to simulate exposure. In fact, methanol is only weakly retained and its competition is virtually nil in an open system[5].

Gas Chromatographic Analysis of Amines

The linearity of the calibration curve of butylamine was examined for the 3 sorbents. The correlation coefficients for analyte amounts of 10^{-8} to 10^{-7} moles were from 0.9911 to 0.9978.

It is an asset of the thermal desorption technique employed to transfer the amine from the preconcentration tube to the gas chromatograph that all the trapped quantity is analyzed at once. The thermal desorption recovery depends on the nature of the amine, on the sorbent, thermodesorption facility, and way of transferring the released deposit on the analytical column. A desorption temperature of 180 °C proved to be sufficient for all the amines, as evidenced by repeated desorption and analysis of the preconcentration tube. The number of desorption steps necessary to attain the blank value

depends on the packing exposure, and is typically 2 to 3. At about 200 °C, which is the thermal stability value of the majority of polymeric organic sorbents, the number of desorption steps decreases but the sorbents, including Tenax, suffer rapid degradation. The presence of oxygen, even in small amounts, has an adverse effect.

If no breakthrough occurs during the sampling and the sorbate trapped is stable, the overall efficiency of the procedure corresponds to the desorption recovery and is typically 93 – 97%.

If amines were to be identified in real samples, a selective detector would have to be employed, or some of the reaction chromatography methods[33] would have to be applied.

Derivatization and HPLC Analysis of the Derivatives

The amine preconcentration method was tested on the 3 sorbents. Since the volumes passed were invariably lower than V_{max}, the results obtained by using the same injected amount of butylamine and the same derivatization agents were virtually identical for the 3 sorbents.

The suitability of the reagents can be assessed from two points of view, viz. with respect to the detection limit of the derivatives and with respect to the reaction rate. The calibration plot of the peak areas, representing the detector responses to the fluorescence or UV absorption of the derivatives, was measured for butylamine compounds with dansyl chloride, OPA, and SNPA, following sorption on Tenax GC. For the fluorescent reagents, the calibration plots were linear with correlation coefficients of 0.9945 to 0.9980 for amounts of 0.01 to 5 μmol. For the SNPA derivatives, the calibration dependence was linear over the region of 0.6 to 20 μmol butylamine with a correlation coefficient of 0.9999. The response of the derivative prepared by direct reaction revealed that the overall recovery was 88 – 95%.

The use of the procedure with a model mixture of pentylamine, hexylamine and heptylamine (Figs 6 and 7) gave evidence that the method is applicable to higher primary alkylamines, which can be well separated by HPLC if a suitable mobile phase gradient is adjusted. The lowest detectable concentrations (Table III) are comparable with those attained by other authors concerned with the analysis of amines in air, which were on the order of 10^{-9} mol m^{-3} (ref.[34]) or 10^{-6} mol m^{-3} (ref.[35]).

The detection limit can also be reduced by using a larger volume of gas sucked through a larger amount of sorbent and, in the case of derivatization, by reducing the volume of eluate from the preconcentration step; computerized accumulation of repeated chromatograms[36] is convenient as well. The major asset of the sorption–desorption–derivatization system is in the fact that while all organic substances are sorbed and desorbed, only amines are derivatized and detected.

TABLE III

Lowest detectable concentration of butylamine (in moles per m^3 gas) and the tube packing weight (Table I)[a]

Sorbent	Dns-Cl	OPA	SNPA	GC
Tenax GC	$2 . 10^{-6}$	$2 . 10^{-6}$	$2 . 10^{-4}$	$2 . 10^{-6}$
Chromosorb 103	$8 . 10^{-7}$	$8 . 10^{-7}$	$1 . 10^{-4}$	$1 . 10^{-6}$
Separon SE	$4 . 10^{-7}$	$4 . 10^{-7}$	$6 . 10^{-5}$	$5 . 10^{-7}$

[a] For passing a volume identical with the maximum retention volume for the given sorbent (Table II).

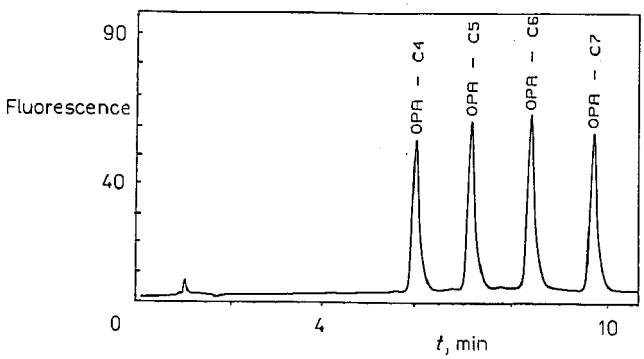

FIG. 6

Chromatogram of a mixture of OPA derivatives of butylamine, pentylamine, hexylamine, and heptylamine. Mobile phase: gradient of 70 – 90 vol.% methanol in water, time 10 min, flow rate 1 ml min^{-1}, fluorescence detection with λ_{ex} = 223 nm, λ_{em} = 435 nm

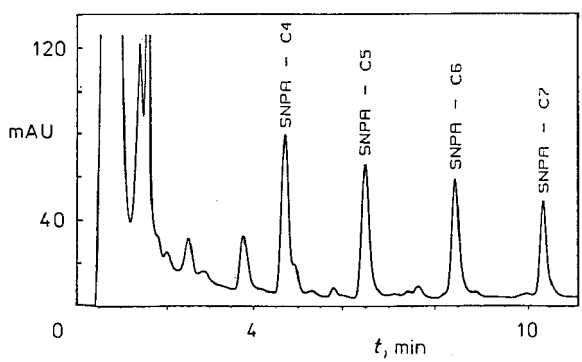

FIG. 7

Chromatogram of a mixture of SNPA derivatives of butylamine, pentylamine, hexylamine, and heptylamine. Mobile phase: gradient of 45 – 70 vol.% methanol in water, time 10 min, flow rate 1 ml min^{-1}, absorption detection at λ_{max} = 280 nm

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