

CHROM. 14,695

Note

Glass capillary gas chromatography of C₂–C₅ 2-nitro-1-alkanols

S. BONEVA*

High Chemical Technological Institute, Burgas 8010 (Bulgaria)

and

N. DIMOV

Chemical Pharmaceutical Research Institute, Sofia 1156 (Bulgaria)

(Received December 29th, 1981)

The separation of C₂–C₅ 2-nitro-1-alkanols on a Carbowax 20M packed column was reported earlier¹. The analysis time was 40 min and the resolution, R_s , was not always greater than 1. We have therefore studied the separation of these compounds on Carbowax 20M wall-coated open tubular (WCOT) glass capillary columns. The influence of the analysis temperature, gas hold-up determination method (with methane or according to Peterson and Hirsch² with different groups of *n*-alkanes) and the nature of the standards used (*n*-alkanes and *n*-alkanols) was investigated in order to obtain retention data that would permit an accurate identification.

EXPERIMENTAL

Preparation of the glass capillary column

A SIMAX borosilicate glass tube, 7–8 mm O.D. and 2–3 mm I.D., was used for drawing a capillary of 0.25–0.30 mm I.D. The inside surface of the capillary was then coated with isopropyl 1,2-difluoro-2-chloroethyl ether (TEE), which was first passed through one end of the column for 3 h in one direction and then for an additional 3 h through the other end in the opposite direction. Both ends of the capillary were sealed and the column was heated at 623°K for 24 h. After purging the capillary with nitrogen it was coated dynamically with Carbowax 20M using 2.5–3 ml of a 4% solution in chloroform at a velocity of 0.3–0.5 cm/sec. The column was purged again with nitrogen overnight and then conditioned using temperature programming from 313 to 513°K at a rate of 1°K/min and finally heated for 3 h to the maximum operating temperature.

GC conditions

A Carlo Erba Fractovap 2407T gas chromatograph equipped with a flame-ionization detector (FID) was used. Retention time and peak area measurements were carried out with an Autolab Model 6300 integrator.

Optimal results were obtained when using 24-m capillary column at 413°K. Nitrogen was used as the carrier gas at a flow-rate of 0.6 cm³/min; the splitting ratio was 1:150, the injection port temperature 523°K and the sample size 0.1 μl.

RESULTS AND DISCUSSION

The separation of C_2 - C_5 2-nitro-1-alkanols is shown in Fig. 1. Elution from the column required only 12 min. The separation of all peaks is very good and the resolution, R_s , is always greater than 1.

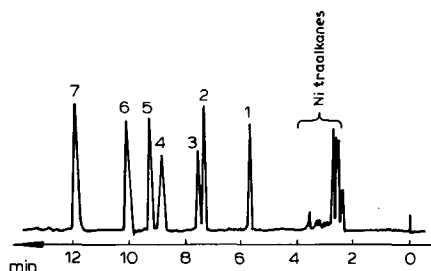


Fig. 1. Chromatogram of C_2 - C_5 2-nitro-1-alkanols on a Carbowax 20M WCOT glass capillary column at 413°K. Peaks: 1 = 2-nitro-2-methyl-1-propanol; 2 = 2-nitro-1-propanol; 3 = 2-nitro-2-methyl-1-butanol-1; 4 = nitroethanol; 5 = 2-nitro-1-butanol; 6 = 2-nitro-3-methyl-1-butanol; 7 = 2-nitro-1-pentanol.

The retention indices were calculated at different temperatures. The gas hold-up time was determined with methane and according to Peterson and Hirsh with C_{15} , C_{16} , C_{17} and C_{18} *n*-alkanes. The retention indices were calculated using different standards (*n*-alkanes and *n*-alkanols). The dispersions between the experimental retention indices were compared by the Fisher and Gohren³ criteria. It was established that the dispersions were always homogeneous but with *n*-alkanols as standards they were smaller. On this basis the mean values of the retention indices were calculated and the results are given in Table I.

TABLE I

RETENTION INDICES OF C_2 - C_5 2-NITRO-1-ALKANOLS ON A CARBOWAX 20M GLASS CAPILLARY COLUMN

Compound	Standard	
	<i>n</i> -Alkanes (403-423°K)	<i>n</i> -Alkanols (413-423°K)
2-Nitro-2-methyl-1-propanol	1898 ± 5	1095 ± 1
2-Nitro-1-propanol	1990 ± 5	1184 ± 1
2-Nitro-2-methyl-1-butanol	2000 ± 5	1193 ± 1
Nitroethanol	2045 ± 5	1244 ± 1
2-Nitro-1-butanol	2067 ± 5	1256 ± 1
2-Nitro-3-methyl-1-butanol	2093 ± 5	1281 ± 1
2-Nitro-1-pentanol	2140 ± 5	1334 ± 1

The data in Table I show that better reproducibility of the retention indices is obtained when *n*-alkanols are used as standards.

CONCLUSIONS

The separation of C₂–C₅ 2-nitro-1-alkanols on a Carbowax 20M WCOT glass capillary column is better than that on a Carbowax 20M packed column; the resolution is greater than 1 for all of the peaks and the analysis time is shorter. Retention indices calculated using *n*-alkanols as standards have better reproducibility.

REFERENCES

- 1 N. Dimov and S. Boneva, *J. Chromatogr.*, 206 (1981) 549.
- 2 M. L. Peterson and J. Hirsch, *J. Lipid Res.*, 1 (1959) 132.
- 3 R. A. Fisher and F. Yater, *Statistical Tables for Biological, Agricultural and Medical Research*, Oliver and Boyd, Edinburgh, 5th ed., 1957.