Acknowledgement

This work was supported in part by a grant-in-aid from the Ministry of Education (Japan), which is gratefully acknowledged.

Pharmaceutical Institute, Tohoku University, School of Medicine, Sendai (Japan)

TOSHIO NAMBARA
TOSHIHIRO KUDO
HARUKO IKEDA

1 T. NAMBARA AND R. IMAI, *J. Chromatog.*, 25 (1966) 248.
2 T. NAMBARA, T. KUDO, H. HOSODA AND S. GOYA, *J. Chromatog.*, 31 (1967) 210.
3 T. NAMBARA, T. KUDO, H. HOSODA, K. YAMANOUCHI AND S. GOYA, *J. Chromatog.*, 31 (1967) 535.
4 C. C. SWEELY, R. BENTLEY, M. MAKITA AND W. W. WELLS, *J. Am. Chem. Soc.*, 85 (1963) 2497.
5 W. J. A. VANDENHEUVEL AND E. C. HORNING, *Biochim. Biophys. Acta*, 74 (1963) 560.
6 H. M. FALES AND T. LUUKAINEN, *Anal. Chem.*, 37 (1965) 955.
7 W. J. A. VANDENHEUVEL AND E. C. HORNING, *Biochim. Biophys. Acta*, 64 (1962) 416.

Received February 5th, 1968

J. Chromatog., 34 (1968) 526–529

CHROM. 3417

Gas chromatographic separation of pyridine homologues, chloroanilines and toluidines

During the course of studies on the separation of isomers by solvent extraction, GLC techniques have been used for quantitative analysis of the isomers present in the solvent phases. Several liquid phases have been studied and it has been found that glycerol, diglycerol and polyphenyl ether (5 ring-OS-124) give good resolution of pyridine bases, *o*- and *p*-chloroanilines and *o*- and *p*-toluidines.

Glycerol and diglycerol have previously been used[1,2] for the analysis of pyridine bases but have not been reported for *o*- and *p*-chloroanilines or *o*- and *p*-toluidines. Of these two stationary phases, diglycerol gave very good results. It was found with glycerol that, whilst it gave good resolution, its efficiency decreased with time for the chloroanilines and the toluidines. The resolution obtained for *o*- and *p*-chloroanilines and for *o*- and *p*-toluidines is given in Table I.

It is believed that the use of polyphenyl ether for the separation of pyridine bases, *o*- and *p*-chloroaniline and *o*- and *p*-toluidines has not previously been reported.

Experimental

Instrumentation. The Pye "Series 104" Dual Flame Ionisation, Temperature Programmed Chromatograph Model 24 was used.

TABLE I

RETENTION TIMES AND CONDITIONS

Conditions		*Compound*	*Retention time (min)*
Stationary phase and column length	*Temperature (°C)*		
20% polyphenyl ether; 9 ft.	110	Pyridine	14.5
		α-Picoline	17.3
		2,6-Lutidine	20.5
		β-Picoline	30.5
		γ-Picoline	33.7
		2,5-Lutidine	35.0
		2,4-Lutidine	40.5
	135	*o*-Toluidine	70.0
		p-Toluidine	75.0
	178	*o*-Chloroaniline	25.0
		p-Chloroaniline	30.0
12% diglycerol; 5 ft.	90	*o*-Toluidine	3.0
		p-Toluidine	4.0
	115	*o*-Chloroaniline	1.5
		p-Chloroaniline	2.5
20% glycerol; 5 ft.	90	*o*-Toluidine	3.0
		p-Toluidine	4.0
	115	*o*-Chloroaniline	1.5
		p-Chloroaniline	2.5

Solid support preparation. Chromosorb P of 80–100 mesh was treated with alcoholic sodium hydroxide (6% NaOH on dried Chromosorb P), the alcohol subsequently being evaporated.

Postgraduate School of Chemical Engineering,
University of Bradford,
Bradford, 7 (Great Britain)

M. ANWAR
C. HANSON*
A. N. PATEL**

1 W. J. MURRAY AND A. F. WILLIAMS, *Chem. Ind. (London)*, (1956) 1020.
2 J. S. FITZGERALD, *Australian J. Appl. Sci.*, 12 (1961) 51.

First received December 29th, 1967; modified January 31st, 1968

* Currently Visiting Associate Professor of Chemical Engineering, Purdue University, Lafayette, In., U.S.A.
** Currently Senior Lecturer in Chemical Technology, University of Science and Technology, Kumasi, Ghana.