

Notes

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Gas chromatographic separation of cresol, xlenol and chloroaniline isomers

In a previous communication¹, work was reported on the gas chromatographic separation of pyridine homologues, chloroanilines and toluidines. This was employed as an analytical technique during studies on separation of the isomers of these compounds by solvent extraction. Since then, work on the use of solvent extraction for such separations has been extended to the cresol and xlenol isomers. This necessitated a method of quantitative analysis for the isomers present in the solvent phases. Several previously described liquid phases were studied for the gas chromatographic separation of the isomers but none was found to be satisfactory.

TABLE I
RETENTION TIMES AND CONDITIONS

<i>Condition</i>		<i>Compound</i>	<i>Retention time (min)</i>
<i>Stationary phase and column length</i>	<i>Temperature (°C)</i>		
30% Dispersol CWL; 5 ft.	180	<i>o</i> -cresol	9.0
		<i>p</i> -cresol	12.0
		<i>m</i> -cresol	15.0
		2,6-xlenol	5.5
		2,4-xlenol	10.0
		2,5-xlenol	10.0
		2,3-xlenol	12.0
		3,4-xlenol	16.0
		3,5-xlenol	16.0
20% Carbowax 1500; 5 ft.	180	<i>o</i> -chloroaniline	4.0
		<i>p</i> -chloroaniline	7.0

The possible use of Dispersol CWL (a product of ICI Ltd.) as a liquid phase for GLC was studied and it was found to give good resolution of the cresol and xlenol isomers. The use of this material as a liquid phase for GLC has not previously been reported.

In our previous communication¹, separation of the chloroaniline isomers was described using polyphenyl ether, diglycerol and glycerol as liquid phases. During subsequent work, it has been found that Carbowax 1500 is superior to any of these phases for this separation.

Experimental

Instrumentation. A Pye Series 104 dual flame ionisation, temperature-programmed chromatograph Model 24 was used.

Column preparation. Two parts by weight of Dispersol CWL and one part by weight of solid potassium hydroxide were dissolved in aqueous methanol and applied to Chromosorb W (80-100 mesh) as solid support. Celite was employed as solid support for the Carbowax 1500.

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