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Note

"C_{22}"-A superior bonded silica for use in reversed-phase high-performance liquid chromatography

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We have recently shown[1] the importance of the screening of several bonded supports prior to the development of a separation involving reversed-phase high-performance liquid chromatography (HPLC). This study revealed that resolution improved with increasing chain length and that the C_{18} (*n*-octadecyl)-bonded silica was the best of the then existing materials. The only exceptions to this generalisation appear to be those applications involving ion-exchange and ion-pair interactions with the support.

As a significant development of this work, we have now extended the length of the carbon chain to C_{22} (dococyl). Details of the superior performance of this new bonded support are now reported.

EXPERIMENTAL

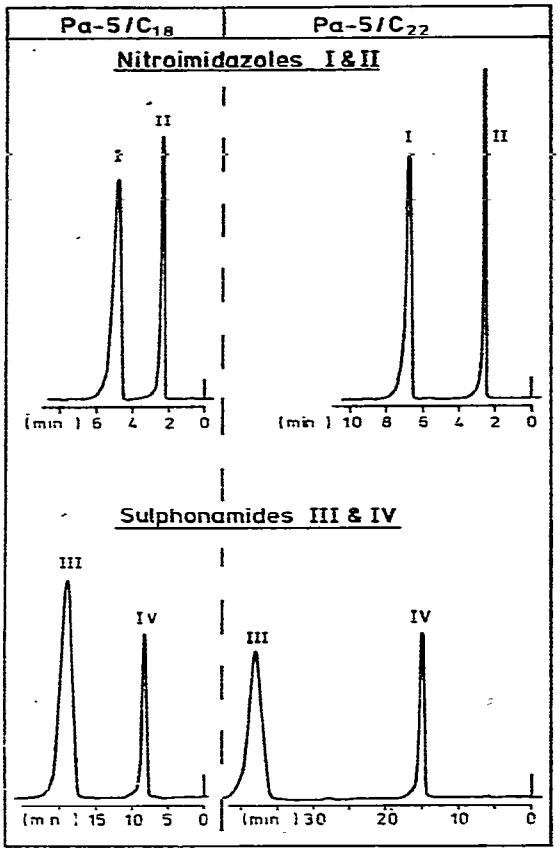
All conditions, reagents, equipment and chemical structures relating to this communication are as previously reported[1].

RESULTS AND DISCUSSION

Our previous study[1] indicated that a linear relationship existed between resolution and alkyl chain length up to C_{18}. We now have established that this linearity extends to at least C_{22} and that substantially better separations have been observed for each of the applications used in our column screen. These comparisons were carried out under identical experimental conditions. Typical results are shown in Figs. 1 and 2.

It is probable that the capacity of preparative, reversed-phase HPLC is greater with C_{22}-bonded silica. This aspect is currently being investigated.

In order to achieve a separation on Pa-5/C_{22} in a time equivalent to that achieved on Pa-5/C_{18}, the level of organic solvent in the eluent must be increased.



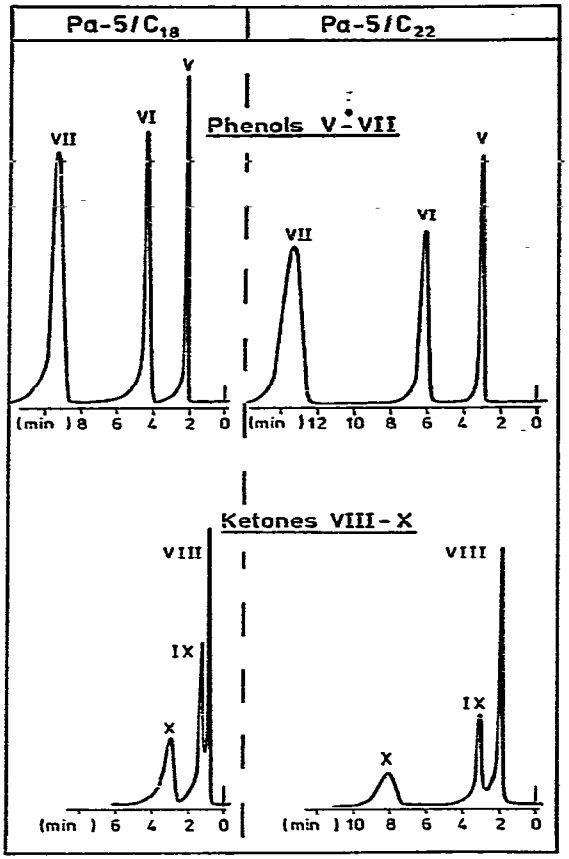


Fig. 1. Chromatograms of the nitroimidazole "misonidazole" I and its metabolite II and of the sulphonamide "mefruside" III and its metabolite IV on Pa-5/C_{18} and Pa-5/C_{22} columns. Eluent compositions for these separations were: methanol–water (1:9) for nitroimidazole separation; methanol–water (2:8) for sulphonamide separation.

Fig. 2. Chromatograms of phenol and ketone mixtures on Pa-5/C_{18} and Pa-5/C_{22} columns. Eluent compositions for these separations were: methanol–water (2:8) for phenols; methanol–water (4:6) for ketones.

TABLE I

IDENTIFICATION OF THE COMPOUNDS SEPARATED IN FIGS. 1 AND 2

Peak No.	*Identification*
I	Misonidazole
II	Metabolite of I
III	Mefruside
IV	Metabolite of III
V	Phenol
VI	4-Methyl phenol
VII	2,4-Dimethyl phenol
VIII	Acetone (dimethyl ketone)
IX	Diethyl ketone
X	Di-*n*-propyl ketone

This is of significance when the compounds to be separated are only sparingly soluble in water. In this category, the sulphonamide "mefruside" III and its metabolite IV are good examples.

The structures of all of the compounds separated can be found in our previous study[1]. An identification of the peaks is given in Table I.

Work designed to extend the investigation to even larger chain lengths and to a much wider range of compounds is now in progress and will be published in due course.

REFERENCE

1 C. J. Little, A. D. Dale and M. B. Evans, *J. Chromatogr.*, 153 (1978) 381; also presented at the 4th SAC Conference, July 1977, Birmingham University.