Liquid Chromatography of some Arene Tricarbonylchromium Complexes

BY HANS VEENING,* JACK M. GREENWOOD, WILLIAM H. SHANKS, and BENNETT R. WILLEFORD

(*Department of Chemistry, Bucknell University, Lewisburg, Pennsylvania* 17837)

Summary The first separation of metal π-complexes by liquid chromatography has been achieved.

RECENTLY we reported[1] the first systematic study of the gas chromatographic behaviour of several volatile arene tricarbonylchromium complexes. This g.l.c. work provides a very rapid and useful procedure for the analysis of mixtures of these compounds. It is limited, however, to those complexes which are volatile and stable at the elevated temperatures used in g.l.c. work. The potential value of liquid chromatography (l.c.) for the separation and quantitative determination of the less volatile and generally less stable complexes of this type became apparent. Previous work has shown that liquid chromatography with u.v.-detection is very suitable for the separation of metal chelates.[2]

We report here the first separation of metal π-complexes by l.c. The Figure shows the separation under the conditions chosen (Jeolco JLC-2A constant flow pump, 1·85 ml./min.; 30°; 55 cm. × 3·5 mm., i.d., glass column packed with 36—75 μ Carbowax-400/Porasil-C; 2,2,4-trimethylpentane as moving phase; Beckman DB spectrophotometer at 320 nm. as detector; benzene solution 10^{-3}M in each complex; 10 μl. sample) of a four-component mixture of benzene-, toluene-, *m*-xylene-, and mesitylene-tricarbonylchromium. It is interesting to note that the order of elution of the components in l.c. is the inverse of the observed in g.l.c.[1] Apparently, as the number of substituted methyl groups on the ligands increases, the partition ratio (k) becomes smaller because of lower solubility of the methylated derivatives in the stationary phase.

Procedures for the separation and determination by l.c. of other π-complexes, including isomeric mixtures, are now being developed. These analytical methods will be very useful in connection with kinetic and equilibrium studies of this type.

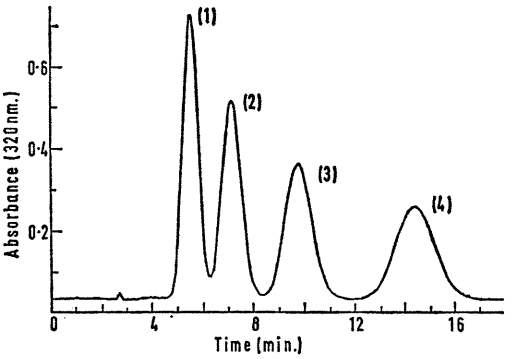

FIGURE. *Liquid chromatographic separation of mesitylenetricarbonylchromium* (1), m-*xylenetricarbonylchromium* (2), *toluenetricarbonylchromium* (3), *and benzenetricarbonylchromium* (4).

We gratefully acknowledge financial support from the Petroleum Research Fund and the National Science Foundation.

(*Received, September 8th,* 1969; *Com.* 1352.)

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[2] J. F. K. Huber, J. C. Kraak, and H. Veening, unpublished work.