

CHROM. 10,312

## Note

### Behaviour of C-14 labelled methyl palmitate during chromatography of petroleum hydrocarbons on the SARA column

ROHAN H. WICKRAMASINGHE

Laboratory of Chemical Evolution, Department of Chemistry, University of Maryland, College Park, Md. 20742 (U.S.A.)

AARON L. MILLS and RITA R. COLWELL\*

Department of Microbiology, University of Maryland, College Park, Md. 20742 (U.S.A.)

and

CYRIL PONNAMPERUMA

Laboratory of Chemical Evolution, Department of Chemistry, University of Maryland, College Park, Md. 20742 (U.S.A.)

(Received June 13th, 1977)

Petroleum residuals may be characterized by the "SARA" (saturated-aromatic-resin-asphaltene) method as described by Jewell *et al.*<sup>1</sup>. This chromatographic method employs a column packed in sequence with (a) Amberlyst 15 H<sup>+</sup> ion exchanger, (b) Amberlyst A-29 OH<sup>-</sup> resin, (c) FeCl<sub>3</sub>-coated clay and finally (d) Amberlyst A-29 OH<sup>-</sup> resin beds. Use of the technique provides three fractions: asphaltenes, "oils" and resins. In connection with investigations on oil pollution of the marine environment and the microbial degradation of spilled oil<sup>2-3</sup>, it was necessary to identify the chromatographic behaviour of an ester when using this technique. For this purpose we utilized (U-C-14) methyl palmitate to monitor the behaviour of these fractions of crude oil.

## EXPERIMENTAL

The apparatus used was the preparative (recycle) SARA column of the dimensions described by Jewell *et al.*<sup>1</sup>. The materials for packing the column were a generous gift of Dr. Jewell. ICN(U-C-14) methyl palmitate of specific activity 305 mCi/mmole and high-quality benzene, *n*-pentane and chloroform (Burdick and Jackson, Muskegon, Mich., U.S.A.) were used. The methyl palmitate was mixed with a sample of the light Arabian crude oil which was spilled from the Metula supertanker in the Straits of Magellan in 1974, with 0.42-0.82 g used for each of three determinations. Chromatography was performed according to the procedure of Jewell *et al.*<sup>1</sup>. Refluxing with pentane or chloroform was performed for a minimum of 24 h each.

Aliquots from each fraction obtained were mixed with 10 ml of scintillation fluid and counted for 10 min in an ABAC SL 40 spectrometer. The scintillation fluid used was 1 g Omnifluor (NEN) in 250 ml toluene (scintanalyzed). Minimal amounts of each fraction were used to minimize the effects of quenching.

## RESULTS AND DISCUSSION

Table I shows the results obtained from a typical experiment, with three determinations made. In terms of specific activity, as well as distribution of radioactivity between fractions, the (U-C-14) methyl palmitate chromatographed in the "oils" fraction. Assuming that the chromatographic behaviour of methyl palmitate is representative of that of a typical ester, it is concluded that these compounds may generally be found in the "oils" fraction, with the saturated and aromatic hydrocarbons and nonpolar non-hydrocarbons, such as ethers and thioethers<sup>1</sup>.

TABLE I

## PASSAGE OF C-14 METHYL PALMITATE THROUGH THE SARA COLUMN

Total recovery of asphaltenes, "oils" and resins, after chromatography and drying of each fraction to constant weight, amounted to 70.4% of the crude oil sample applied to the column. The "blank" placed in the scintillation counter together with these samples gave a reading of 10.9 cpm, with a standard error of 25.3 cpm, which could account entirely for the raw counts recorded for the asphaltenes sample and a proportion of those for the resins.

	<i>Recovery (wt.%)</i>	<i>Activity (cpm/mg)</i>	<i>Distribution of radioactivity per fraction (%)</i>
Asphaltenes	1.5	87.1	0.04
"Oils"	91.4	3791.1	99.46
Resins	7.1	245.9	0.50

## ACKNOWLEDGEMENT

We thank Dr. D. M. Jewell for helpful advice on the technique and a gift of the chromatography bed materials.

## REFERENCES

- 1 D. M. Jewell, E. W. Albaugh, B. E. Davis and R. G. Ruberto, *Ind. Eng. Chem., Fundam.*, 13 (1974) 278.
- 2 J. D. Walker, R. R. Colwell, M. C. Hamming and H. T. Ford, *Bull. Environ. Contam. Toxicol.*, 13 (1975) 245.
- 3 R. H. Wickramasinghe, A. L. Mills, J. D. Walker, R. R. Colwell, A. Shimoyama and C. Ponnampereuma, *Abstracts, 11th Middle Atlantic Regional Meeting, Amer. Chem. Soc., Newark, Del., 1977*, EN 10.