

## Note

### Evaluation of new phases for the gas chromatography of dibasic acids

O. MLEJNEK and Ľ. CVEČKOVÁ

*Cables and Insulating Materials Research Institute, 891 23 Bratislava (Czechoslovakia)*

(Received March 14th, 1973)

The analysis of the methyl esters of dibasic acids by gas chromatography has been carried out by many workers. The separation of the three isomers of benzenedicarboxylic acids is difficult and requires the use of either special phases or very effective columns. Recently, Supelco Inc. (Bellefonte, Pa. 16823, U.S.A.) have developed new phases for the analysis of fatty acids: SP-222 for their methyl esters<sup>1</sup> and SP-216-PS for both the free acids and the methyl esters<sup>2</sup>. The advantages of these phases are the good resolution of saturated and unsaturated acids and the short elution times obtained.

We have found that the new phases are also suitable for the separation of dibasic acids, especially for the phthalic acid isomers. It can be seen from the chromatogram in Fig. 1 that terephthalic, isophthalic and *o*-phthalic acids are well separated in less than 10 min. In special instances, an isothermal run can be used (Fig. 2), which

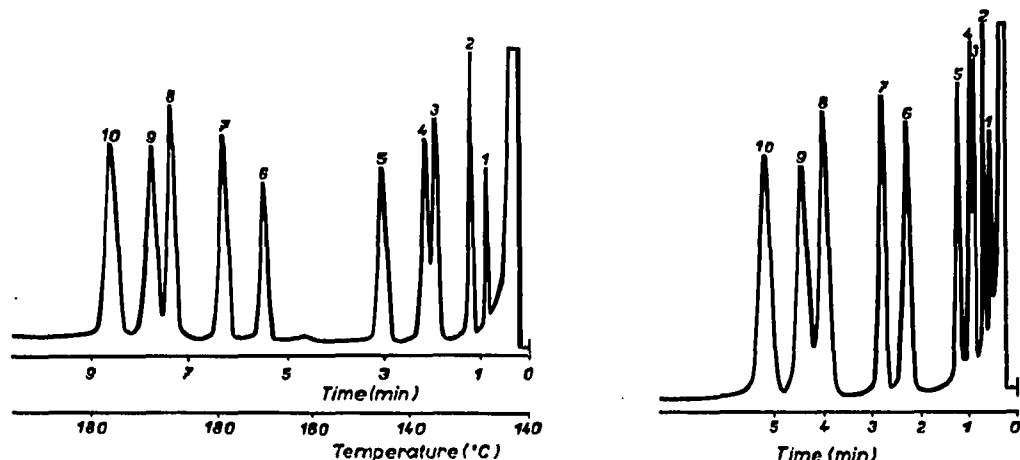


Fig. 1. Separation of the methyl esters of dibasic acids on 10% SP-216-PS. Initial temperature of the column 140°; after 2.5 min, programmed to 180° at the rate of 10°/min. Inlet pressure of nitrogen, 0.35 kp/cm<sup>2</sup>. The peaks are identified as follows: 1=oxalate; 2=fumarate; 3=itaconate; 4=maleate; 5=adipate; 6=azelaate; 7=sebacate; 8=terephthalate; 9=isophthalate; 10= *o*-phthalate.

Fig. 2. Isothermal separation of the methyl esters of dibasic acids on 10% SP-222-PSB at 190°. Inlet pressure of nitrogen, 0.4 kp/cm<sup>2</sup>. The peaks are identified as in Fig. 1.

shortens the analysis time by half. Also of interest is the short elution time of aliphatic dibasic acids, which permits sebacic acid to be eluted before the phthalic acid isomers.

We used Fractovap 2201 (Carlo Erba, Milan, Italy) with two columns 95 cm long and 3.5 mm I.D., filled with the original packings (10% on Supelcoport, 100–120 mesh). Methyl esters of the dibasic acids were prepared by the diazomethylation<sup>3</sup> of the free acids (technical grade) in dioxan at room temperature (saturated acids) or in diethyl ether at  $-60^{\circ}$  (unsaturated acids) in the presence of methanol. They were injected in amounts of 0.5–1  $\mu$ l into the injection port, which was heated to the same temperature as the flame ionization detector ( $210^{\circ}$ ).

#### ACKNOWLEDGEMENT

The authors are grateful to Dr. Walter Supina, President of Supelco Inc., for kindly providing the new packings for fatty acid analysis.

#### REFERENCES

- 1 Anonymus, *Chromatogr. Lipids*, 6 (1972) 2.
- 2 Anonymus, *Chromatogr. Lipids*, 7 (1973) 2.
- 3 O. Mlejnek, *J. Chromatogr.*, 70 (1972) 59.