Gel G" used in the Stahl modification (2) gives substantially better resolution and spot tightness with some lipid mixtures than does the Mallinckrodt silicic acid. However, the gypsum-bonded layers are extremely fragile on the glass strips employed. Thus its use in this form has been given only a cursory examination. It is felt that the hardness of the starch-bonded materials offers a substantial advantage, and work with similar silica gels with starch binder is in progress.

Acknowledgments

The authors gratefully acknowledge the help of many people who contributed to this work. R. A. Micheli and W. L. Stanley helped particularly with discussions and loans of materials and equipment. E. M. Bickoff generously supplied equipment and materials and Ann Gramps provided assistance for the preparation of the chromatostrips. William Murray did the photography, and Ronald G. Binder performed the GLC analyses.

REFERENCES

1. Stahl, E., Pharmazie, *11*, 633–637 (1956).
2. Stahl, E., Chemiker Zeit., *82*, 323–329 (1958).
3. Stahl, E., Parfümerie u. Kosmetik, *39*, 564 (1958).
4. Stahl, E., Arch. Pharm., Berlin, 292/64, 411–416 (1959).
5. Mangold, H. K., Fette, Seifen, Anstrichmittel, *61*, 877-881 (1959).
6. Mangold, H. K., and Malins, D. C., J. Am. Oil Chemists' Soc., *37*, 383–385 (1960).
7. Malins, D. C., and Mangold, H. K., J. Am. Oil Chemists' Soc., *37*, 576–578 (1960).
8. Gruger, E. J., Jr., Malins, D. C., and Gauglitz, E. J., Jr., J. Am. Oil Chemists' Soc., *37*, 214–217 (1960).
9. Morris, L. J., Holman, R. T., and Fontell, K., J. Am. Oil Chemists' Soc., *37*, 323–327 (1960).
10. Gauglitz, E. J., Jr., and Malins, D. C., J. Am. Oil Chemists' Soc., *37*, 425–427 (1960).
11. Jatzkewitz, H., and Mehl, E., Z. Physiol. Chem. *320*, 251–257 (1960).
12. Fontell, K., Holman, R. T., and Lambertsen, G., J. Lipid Res., *1*, 391–404 (1960).
13. Morris, L. J., Holman, R. T., and Fontell, K., J. Lipid Res., *1*, 412–420 (1960).
14. Kaufmann, H. P., and Makus, Z., Fette, Seifen, Anstrichmittel, *62*, 1014–1020 (1960).
15. Huhnstock, K., and Weicker, H., Klin. Wochschr. *38*, 1249–1250 (1960): Chem. Titles #4 2/20/61.
16. Barbier, M., Jäger, H., Tobias, H., and Wyss, E., Helv. Chim. Acta. *42*, 2440–2446 (1959).
17. Van Dam, M. J. D., DeKleuver, G. J., and DeHeus, J. G., J. Chromatog., *4*, 26–33 (1960).
18. Brenner, M., and Niederwieser, A., Experientia, *16*, 378–382 (1960).
19. Nicolaus, B. J. R., J. Chromatog., *4*, 384–390 (1960).
20. Gaenshirt, H., and Malzacher, A., Arch. Pharm., *293*, 925–932 (1960): Chem. Titles #6 (1960).
21. Gaenshirt, H., and Malzacher, A., Naturwissenschaften, *47*, 279–280 (1960).
22. Gaenshirt, H., and Morianz, K., Arch. Pharm., *293*, 1065–1075 (1960): Chem. Titles #3 (1961).
23. Dhont, J. H., and de Rooy, C., Analyst, *86*, 74–76 (1961).
24. Seiler, H., and Seiler, M., Helv. Chim. Acta, *43*, 1939–1941 (1960).
25. Kirchner, J. G., Miller, J. M., and Keller, G. J., Anal. Chem., *23*, 420–425 (1951).
26. Miller, J. M., and Kirchner, J. G., Anal. Chem., *24*, 1480–1482 (1952).
27. Miller, J. M., and Kirchner, J. G., Anal. Chem., *25*, 1107–1109 (1953).
28. Miller, J. M., and Kirchner, J. G., Anal. Chem., *26*, 2002 (1954).
29. Demole, E., J. Chromatog., *1*, 24–34 (1958): Chromatographic Rev., *1*, 1–10 (1959).
30. Stanley, W. L., Vannier, S. H., and Gentili, B., J. Assoc. Offic. Agr. Chemists, *40*, 282–286 (1957).
31. Stanley, W. L., and Vannier, S. H., J. Assoc. Offic. Agr. Chemists, *40*, 582–588 (1957).
32. Vannier, S. H., and Stanley, W. L., J. Assoc. Offic. Agr. Chemists, *41*, 432–435 (1958).
33. Stanley, W. L., Lindwall, R. C., and Vannier, S. H., J. Agr. Food Chem., *6*, 858–860 (1958).
34. Stanley, W. L., J. Assoc. Offic. Agr. Chemists, *42*, 643–646 (1959).
35. Stanley, W. L., J. Assoc. Offic. Agr. Chemists, in press.
36. Stanley, W. L., Ikeda, R. M., and Cook, S., Food Res., in press.
37. Horowitz, R. M., and Gentili, B., J. Org. Chem., *25*, 2183–2187 (1960).
38. Bickhoff, E. M., Lyman, R. L., Livingston, A. L., and Booth, A. N., J. Am. Chem. Soc., *80*, 3969–3971 (1958).
39. Lyman, R. L., Bickoff, E. M., Booth, A. N., and Livingston, A. L., Arch. Biochem. Biophys., *80*, 61–67 (1959).
40. Livingston, A. L., Bickoff, E. M., Guggolz, J., and Thompson, C. R., J. Agr. Food Chem., *9*(2), 135–137 (1961).
41. Guggolz, J., Livingston, A. L., and Bickoff, E. M., J. Agr. Food Chem., in press.
42. Micheli, R. A., unpublished results.
43. Jurd, L., J. Org. Chem., *24*, 1786–1787 (1959).
44. Hanford, W. E., Schreiber, R. S., and Gray, H. W. (E. I. du-Pont de Nemours & Company), U.S. 2,340,745 (1944).
45. Colonge, J., and Guyot, P., Bull. soc. chim. France, *1954*, 339–342.
46. Smith, C. R., Jr., Wilson, T. L., Melvin, E. H., and Wolff, I. A., J. Am. Chem. Soc., *82*, 1417–1421 (1960).

[Received June 1, 1961]

• *Letter to the Editor*

Mass Spectrometry and Lipid Research

TWO DEVELOPMENTS in commercial equipment for mass spectrometry are of significance to lipid research: a) heated inlet and ion source systems for use with "high boilers" and b) rapid photographic registration of mass spectra. Preliminary results from our laboratory demonstrate the applicability of these advances to problems of determining the position of double bonds in monoenes and of identifying volatiles from reverted soybean oil.

Dinh-Nguyen, Ryhage, and Stenhagen have shown that the position of the double bond in methyl petroselinate and oleate may be located by first deuterating the double bond (1). This deuteration step is necessary since the initial position of the double bond (except for the α, β locations) is not reflected in the mass spectra of the nonoene isomers (2).

With the use of a heated (200°C.) sample inlet, ion source, and flight tube, the mass spectrum of methyl 6,7-dideutero stearate was also recorded in our laboratory but with a "Time-of-Flight"-type spectrometer. Shown in Fig. 6 of a paper appearing in this issue (3) are strip chart recordings of the spectra for methyl 6,7-dideutero stearate and for a mixture of stearate and dideutero stearate. Mass peaks at 298 and 300 (parent peaks) correspond to normal methyl stearate and methyl dideutero stearate ions, respectively, as well as do the fragment ions at 129 $[-(CH_2)_5COOCH_3^+]$ and 130 $[-CHD-(CH_2)_4\ COOCH_3^+]$ and at 143 $[-(CH_2)_6COOCH_3^+]$ and 145 $[-CHD-CHD-(CH_2)_4COOCH_3^+]$. The fragments differing from each other by one and two mass units are critical in determining the position of the deuterium and, hence, in ascertaining the original position of the double bond. These data confirm the prior observation of workers in Sweden, who used a magnetic scanning-type spectrometer (1).

In the course of identifying hydrocarbons in volatiles of reverted soybean oils (4), a preliminary analysis was made on Phillips hydrocarbon mixture No. 37. This analysis was achieved by gas chromatography on a polypropylene glycol column at −20°C. (3), Fig. 11. Effluents were monitored by the mass spectrometer in a tandem arrangement described by Gohlke (5) in order to identify the components as they emerged. In the chromatogram of the Phillips mixture particular interest resides in the last peak eluted which appears to be a single component. Actually, it is composed of two components, *cis*-butene-2

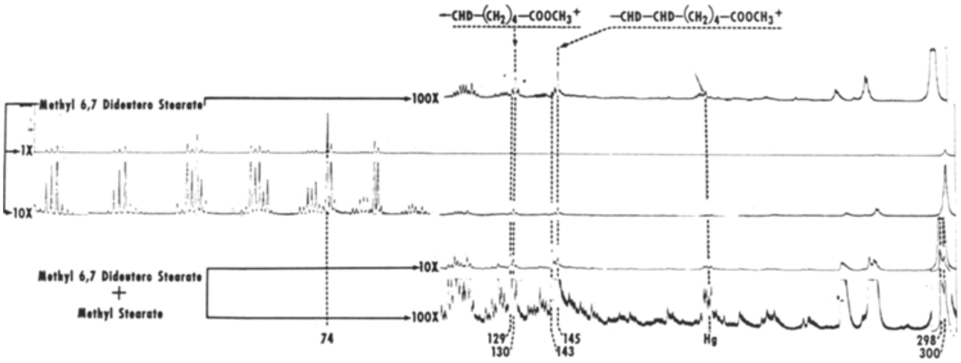

FIG. 1. Analog tracing of mass spectra for methyl 6,7-dideutero stearate (top 3 curves) and a mixture of methyl 6,7-dideutero stearate and methyl stearate.

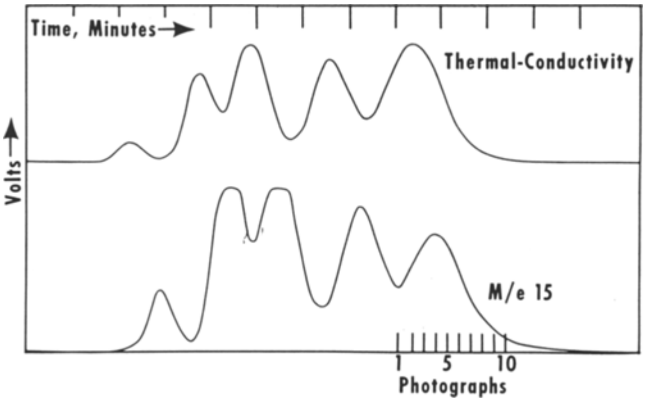

FIG. 2. Chart of simultaneous recording for thermal conductivity detector (upper tracing) and mass spectrometric detector set on M/e—15 (lower tracing).

and butadiene, as shown in the mass spectra (3), Fig. 10, photographed at about 15-sec. intervals during the emergence of the gas chromatographic peak. The parent ion at m/e=56 increases and decreases during its emergence at the left side of the chromatographic peak. The parent ion at m/e=54 on the right side of the chromatographic peak also increases and decreases. This method of photographic reproduction of mass spectra is noteworthy because of the rapidity with which the spectrum is recorded. The spectrometer is capable of displaying a mass spectrum or any portion of the spectrum (m/e=1 to about 4,000) on the oscillographic screen every 10^{-4} sec.

The mass spectrometer can be used as a detector for gas-liquid chromatography by setting the controller analog chassis to monitor the m/e = 15 peak, and its recorded output indicates the concentration of the methyl (CH_3^+) ion. Its curve resembles, with a slight lag, the chromatographic curve of the thermal conductivity detector and attests to the rapid clearance of successive components from the mass spectrometer. If set up in this manner as a "detector" for gas chromatography, the mass spectrometer is about a thousand times more sensitive than the thermal conductivity detector feeding a 10-millivolt recorder.

Monitoring of high-temperature chromatography has also been performed on fatty acid methyl esters, and rapid clearance of successive components has been observed.

This combination of the two complementary techniques exploits the high resolving and quantitative characteristics of gas chromatography and the unusual identification capacity of mass spectrometry and should constitute a new tool of wide applicability in lipid research.

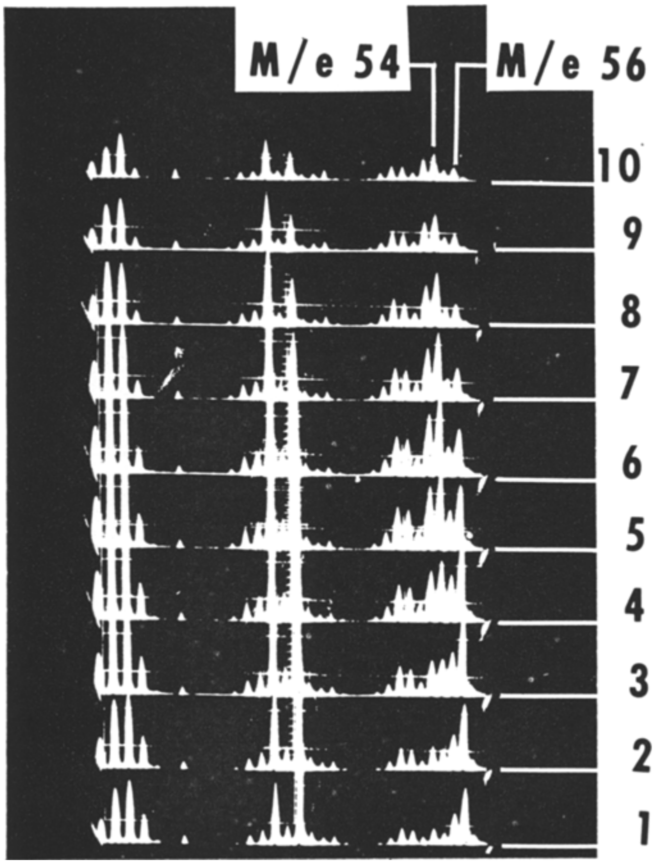

FIG. 3. Photograph of the mass spectrum for the *cis*-butene-2 and butadiene peak of the Phillips No. 37 mixture taken at 15-sec. intervals as indicated on the lower section in Fig. 2.

E. SELKE, C. R. SCHOLFIELD,
C. D. EVANS, and H. J. DUTTON,
Northern Regional Research
Laboratory, Peoria, Ill.

REFERENCES

1. Dinh-Nguyen, N., Ryhage, R., Ställberg-Stenhagen, Stina, Arkiv Kemi, *15*, 433–438 (1959).
2. Hallgren, B., Ryhage, R., Stenhagen, E., Acta Chem. Scand., *13*, 845–847 (1959).
3. Dutton, H. J., J. Am. Oil Chemists' Soc., *38*, (this issue) (1961).
4. Frankel, E. N., Nowakowska, Janina, and Evans, C. D., J. Am. Oil Chemists' Soc., *38*, 161–162 (1961).
5. Gohlke, R. S., Anal. Chem., *31*, 535–541 (1959).

[Received September 27, 1961]