

4-*cis*-Heptenal in Autoxidized Soybean and Linseed Oils?

Hammond and Seals (1) tentatively identified 4-*cis*-heptenal in strongly autoxidized soybean and linseed oils. In connection with this abstract, and because in previous experiments we did not detect 4-*cis*-heptenal in autoxidized soybean oils, we analyzed autoxidized methyl linolenate (prepared by J. B. A. Stroink).

Two samples of ~2.5 g of methyl linolenate (purity ~100%, gas liquid chromatography (GLC)) were stored for five days in air in diffuse daylight. The samples with peroxide values (PO values) of 1311 and 1035 were heated in a closed system under nitrogen at 100 C for 1 hr to decompose the peroxides, and subsequently degassed (2) at 40 C and $<10^{-5}$ mm Hg for 5 hr. The volatile carbonyl compounds obtained were analyzed as follows:

Sample 1. The compounds were taken up in carbonyl-free hexane, converted into their 2,4-dinitrophenylhydrazones (2,4-DNPHs) (3) and analyzed by thin layer chromatography (4), successively on 33.3% Carbowax-400/Kieselguhr G, Silica Gel G and 30% AgNO₃/Silica Gel G, with authentic 4-*cis*- and 4-*trans*-heptenal-DNPHs as model substances. Of the original DNPH mixture in question, the small band of DNPHs, occurring between the pentanal- and hexanal-DNPHs on the Carbowax plate (5), was collected. This band was separated on a silica chromatoplate into six new bands. One band with the same R_f value as that of authentic 4-heptenal-DNPH (*cis* and *trans*) was scraped from the plate and exhaustively extracted with diethyl ether. Chromatography of the band on 30% AgNO₃/SiO₂ resulted in five new bands. None of those bands migrated at the same rate as that of the above-mentioned model compounds (6). With this sequence of analyses, resulting in a total recovery of 90%, not even traces of 4-*cis*-heptenal could be detected, although the detection limits in the three systems used are as low as 0.2–1 μg.

Sample 2. The compounds were analyzed directly by means of GLC (immobile phase 10% PEGA on Gasechrom Q; column length 200 cm; column diameter 4 mm; flame-ionization detector; temperature programming 60–180 C, 2C/min). In the peak emerging at the same retention time as that of authentic 4-*cis*-heptenal, no 4-*cis*-heptenal could be identified by means of micro IR spectroscopic analysis (7).

To assess whether methyl linoleate induces the formation of 4-*cis*-heptenal from autoxidized methyl linolenate, two mixtures of methyl linolenate and methyl linoleate (ratios by weight 2:1 and 9:1, respectively; PO values 1035 and 450, respectively) were subjected to the above experimental procedure. Also in this case no 4-*cis*-heptenal could be detected.

The detection by Hammond and Seals of 4-*cis*-heptenal in autoxidized soybean and linseed oil can only be explained by the presence of an unknown precursor in their oil samples. The oxidation mechanism given by Loury (8) according to which linolenic acid might be the precursor of 4-*cis*-heptenal, becomes very improbable in the light of our findings.

P. W. MEIJBOOM¹

G. A. JONGENOTTER

A. MAARLEVELD

Unilever Research Laboratory
Vlaardingen, The Netherlands

¹ Present address: Olivier van Noortlaan 120, Vlaardingen, The Netherlands.

REFERENCES

1. Hammond, E. G., and R. Seals, AOCs 42nd Fall Meeting, New York, October 1968, Abstract No. 87.
2. De Bruijn, J., and J. C. M. Schogt, JAOCS 38, 40–44 (1961).
3. Schwartz, D. P., and O. W. Parks, Anal. Chem. 33, 1396–1398 (1961).
4. Meijboom, P. W., Fette, Seifen, Anstrichm. 70, 477–481 (1968).
5. Meijboom, P. W., J. Chromatog. 24, 427–431 (1966).
6. Meijboom, P. W., and G. Jurriens, J. Chromatog. 18, 424–426 (1965).
7. Bus, J., and Th. J. Liefkens, Z. Anal. Chem. in press.
8. Loury, M., Rev. Fr. Corps Gras 8, 686–699 (1961).

[Received April 24, 1970]

• Letter to the Editor

Determination of Aflatoxin in Peanut and Cottonseed Soapstocks

Sir:

With reference to the article entitled "Determination of Aflatoxin in Peanut and Cottonseed Soapstocks" by Alva F. Cucullu, Louise S. Lee, W. A. Pons, Jr., L. A. Goldblatt that appeared in the June 1970 issue of JAOCS, the authors state that the use of water vapor in thin layer chromatography (TLC) development of aflatoxins greatly improves their separation. Further, that this adaptation was suggested from the article by Stubblefield et al., July 1969 issue of Journal of the Association of Official Analytical Chemists (52, 669–672, 1969).

May I call the attention of the authors to an article that I published in the February 1969 issue of JAOCS and to a paper which I presented at the Joint Meeting of the AOCS-AACC, Washington, D.C., April, 1968, at a session chaired by W. A. Pons, Jr., that the use of water in the TLC solvent system of aflatoxins greatly improves their separation. This was acknowledged by Stubblefield et al., in their paper cited above.

JAMES VELASCO

Agricultural Research Center
Beltsville, Maryland 20705

[Received July 21, 1970]

