Identification of Some Volatile Products from Mildly Oxidized Pecan Oil

R.J. HORVAT and **S.D. SENTER**, Richard B. Russell Research Center,
Science and Education Administration, Agriculture Research, U.S.
Department of Agriculture, Athens, GA 30604

In order to establish reliable objective measurements that can be applied to surveillance testing of stored pecan kernels, it is necessary to understand the chemistry of their oxidative deterioration and its relation to development of off-flavors or aromas. It appears that the component in shelled pecan kernels most likely to limit their storage life is the natural pecan lipids. For this reason, pecan oil is being used as a model oxidation system. The major volatile compounds identified in this research from mildly autoxidized pecan oil are hereby reported. These compounds were identified by means of gas liquid chromatography-mass spectrometry (GLC-MS).

Pecan oil for these studies was pressed from kernels of the Stuart variety with a performated aluminum block and a Carver press. Fresh oil, peroxide value (PV) negligible, was allowed to stand at room temperature in a glass-stoppered Erlenmeyer flask until the PV reached 1.5 meq O_2/kg oil. Previous work done in this laboratory has shown that pecan oils at this level of oxidation prossessed a rancid off-odor (1).

Volatile products were distilled from ca. 25-g of the oxidized oil at room temperature under reduced pressure (10-20 Torr) and collected in a U-tube maintained at -78 C. Fifty-75 μl of condensate, primarily water, were collected and then extracted with two 25-μl volumes of isopentane and analyzed by GLC-MS. A Perkin-Elmer Model 900 gas chromatograph coupled by means of an effluent splitter to a DuPont 21-490B mass spectrometer was used. Forty percent of the effluent from the GLC column was introduced directly into the ion source of the mass spectrometer and 60% to the chromatograph's flame ionization detector. The 300-ft x 0.02-in. open tubular column used was coated with Carbowax 20 M. For each GLC-MS analysis, 0.8-1.0 μl of isopentane solution were used. Gas chromatographic conditions were: helium flow, 6.8 ml/min; injector, manifold, and flame ionization detector, 200 C; column programming, from 70-200 C at 2 C/min. Mass spectrometer conditions were: ion source temperature, 150 C; scan rate, 10 sec/decade; ionizing voltage, 70 eV; and ion source pressure, 4 x 10^{-6} Torr.

The compounds identified are given in Table I. Analysis of unoxidized pecan oil failed to show the presence of these components. The mass spectral data were compared with those of authentic compounds or with those in the literature. All compounds listed without parentheses in Table I were confirmed by both MS and GLC retention times. Most of the compounds identified in this investigation have been reported as being formed during the oxidation of oils containing oleic, linoleic, and linolenic acids as well as the methyl esters of these acids (1-4). The major unsaturated acids in pecan oil are those previously mentioned (5-10).

TABLE I

Volatile Compounds from Autoxidation of Pecan Oil

acetaldehyde
pentane
acetone
hexane
1-hexene
butanal
(2-butenal)
pentanal
pentanol
hexanal
toluene
n-propylbenzene
(ethylphenylketone)
heptane
benzaldehyde
octane
2-heptanone
heptanal
octanal
1-dodecene
decanal
1-tetradecene

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