Symposium on Thermal Oxidation and Polymerization in Fats

Madhu Sahasrabudhe

During recent years interest has increased in studies on chemical and nutritional aspects of heated fats. When a fat is heated, as in frying, chemical alterations occur. These can be grouped under two major classes: a) degradation of glycerides into peroxides, carbonyl compounds and hydroxy acids, and b) polymerization. Extent of these changes depends upon the presence or absence of air, and duration and temp of heating. Considerable work has been carried out on the nutritional aspects of heated fats. It has also been suggested that toxicity of these fats needs further critical examination. This symposium has been planned to bring together some of the leading scientists in the field to present more recent work on the subject.

Beginning on page 4 you will find a complete listing of the papers with abstracts. Some other prominent scientists in the field who are not presenting papers have very kindly agreed to sit on a panel for discussion at the end of the symposium. Among those who have already agreed to participate are C. M. Gooding, Corn Products Co., and E. G. Perkins, University of Illinois.

In spite of the short notice of the symposium the response has been very good. Some scientists in Europe and the Far East have indicated that they would participate if the symposium is extended to a future meeting.

All indications are that the symposium will be a success.

Food Chemical Codex Part I Now Available

Part I of *Food Chemicals Codex,* a new publication of the National Academy of Sciences, National Research Council, is now available upon a subscription basis, in loose-leaf form. Subsequent issues of the eight or ten parts will be available over the next two and one-half years at approximately four-month intervals.

Purpose of these provisional copies of this single source of standards for direct additives is to invite comments and criticisms relating to the provisional specifications before the Codex is published in a case bound book.

Part I is the initial result of a continuing effort to provide objective quality standards for chemicals added to foods to perform some desired function. Based on elements of safety and good manufacturing practice, these specifications are presented in a series of monographs supplemented by sections covering general analytical procedures and related subjects. It comprises about 100 pages, including General Provisions section, 25 monographs, and a major portion of the General Tests and related sections.

Codex is sponsored by the Food Protection Committee, and compiled by committees composed of industry, government and academic scientists. Subscription price for all parts is $25, including a double ring binder. For further information, write: J. R. Powers, Director, Food Chemicals Codex, National Academy of Sciences-National Research Council, Washington, D. C. 20418, or order directly from the Printing and Publishing office at the same address.

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GAS-LIQUID CHROMATOGRAPHY OF TRIMETHYLSILYL DERIVATIVES OF MONOGLYCERIDES AND HYDROXY FATTY ACIDS

R. D. Wood, P. K. Raju and Raymond Reiser

The use of gas chromatography for the analysis of monoglycerides and their acetylated derivatives has been limited due to their low volatility. An accurate, rapid method for the determination of a mixture of 1-mono and 2-monoglycerides so far has not been available, while the long retention time of the acetylated derivatives of long chain hydroxy fatty acids is undesirable for quantitative estimations.

A simple method for the rapid gas chromatographic analysis of isomeric monoglycerides and hydroxy fatty acids has been developed. The hydroxy compounds were converted to their respective trimethylsilyl derivatives and used directly for gas chromatographic analysis. The monoglycerides prepared from C-8,C-10,C-12,C-14,C-16 and C-18 fatty acids were resolved successfully. The isomeric 1-mono and 2-monoglycerides were also resolved. The retention time of methyl esters of hydroxy fatty acid-trimethylsilyl derivatives was one-sixth that of the untreated hydroxy esters and one-third that of the acetylated derivatives.

The advantages and applications of this new method for the rapid determination of isomeric glycerides and hydroxy fatty acids by gas chromatography in the realm of lipid research will be discussed.

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QUANTITATIVE DETERMINATION OF DOUBLE BOND POSITIONS IN UNSATURATED FATTY ACIDS

E. P. Jones and V. L. Davison

The position of unsaturation in natural fats, especially in partially hydrogenated fats, has become increasingly important to their edible and industrial utilization. The positional and geometric isomerization of bonds that occur during hydrogenation are usually estimated by cleavage of the molecules and analysis of the split products. Limitations of techniques have made it difficult to analyze all products quantitatively, and workers have usually reported only one of the cleavage products; i.e., the less volatile difunctional fragment.

By a combination of procedures, and with special attention to quantitation, we have developed a method to determine double bond positions in mono- and polyunsaturated acids. Key features include controlled oxidative cleavage, recovery of monobasic acids as salts, and their conversion to butyl esters for programmed gas-liquid chromatographic analysis. The wt of monobasic and dibasic acid found agree very closely with theory. However, at present neither malonic acid nor propionic acid may be quantitatively estimated because of the chemical instability of the former and the high water solubility and volatility of the latter.

Analyses are shown for high purity oleic, linoleic, and linolenic acids, and for conjugated and nonconjugated *cis,trans; trans,trans;* and *cis, cis* dienes. Analyses for *cis* and *trans* monoenes and for several trienes are also included.

The reproducibility and applicability of the procedure to a wide range of positional isomeric mixtures produced by partial homogeneous and heterogeneous hydrogenations of polyunsaturates are discussed.

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A RAPID METHOD FOR IDENTIFYING THE FATTY ACIDS OF DEGRAS

Nicholas Pelick and J. W. Shigley

A rapid method for identifying the fatty acids of a natural fat will be discussed. Degras or wool fat was chosen for analysis because of its unique distribution of neutral lipids. Thin layer chromatography, silver-ion chromatography and preparative TLC will be described in this method. The techniques of gas-liquid chromatography and the use of multiphase and programmed analysis are also used. These techniques of TLC and GLC were combined to achieve a rapid method for detecting and identifying over 90 fatty acid components in degras. Four homologous series of fatty acids were identified. They were normal straight chain acids, iso acids, anteiso acids and hydroxy fatty acids. All of the series included odd carbon member acids and unsaturated acids.

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A NEW FATTY ACID WITH UNSATURATION IN THE 3,4 POSITION

M. O. Bagby, W. O. Siegl and I. A. Wolff

Seed oil from *Calea urticaefolia* has ca. 35% isolated *trans* unsaturation. Gas-liquid chromatographic analyses indicate 31.2% of an unknown fatty acid. This new acid, isolated by countercurrent distribution, readily absorbs three moles of hydrogen to yield stearic acid. The unsaturated acid has one *trans* double bond; the other two are both *cis* and are methylene interrupted as shown by spectroscopy and lipoxidase isomerization. The usual treatment with alkali conjugates only two double bonds. Oxidative cleavage yields caproic, adipic, and malonic acids. Partial reduction of the triene with hydrazine yields a monoene mixture, from which *trans*-3-octadecenoic acid was isolated and characterized. The evidence indicates that the triene is the previously unknown *trans*-3,*cis*-9,*cis*-12-octadecatrienoic acid. Nuclear magnetic resonance spectroscopy confirmed the presence of β,γ-unsaturation.

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UREA FRACTIONATION IN THE ANALYSIS OF OILS

J. L. Iverson, Jerome Eisner and David Firestone

The methyl esters of butter, lard, olive oil, walnut oil, sesame oil, and lanolin were fractionated with urea to provide less complex fractions for examination by gas-liquid chromatography (GLC). Long chain saturated fatty acids are concn in the first fractions, while highly unsaturated fatty acids are concn in the last fractions. The procedure is simple, requires inexpensive reagents and equipment, is non-destruc-