for the seqeustration of either metal ion would not turn out to be much different.

In contrast with results on calcium sequestration, tetrasodium pyrophosphate on a weight basis is found to be the most effective magnesium sequestrant among the polyphosphates. This can be explained from the fact that the thermodynamic stabilities of magnesium pyrophosphates and tripolyphosphates are not much different (19,20). Yet, the molecular weight of $Na_4P_2O_7$ is smaller than $Na_5P_3O_{10}$, so that on a weight basis it turns out to be a better magnesium sequestrant.

In most hard waters a mixture of calcium and magnesium is normally encountered. Although in mixtures calcium precipitates come out first because of their lower solubility, the magnesium ions consume polyphosphates in forming complexes. Therefore, it is not unrealistic to express total hardness as calcium. In practice, more sequestrant is usually required to dissolve a calcium precipitate than its magnesium analogue.

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Thermal Oxidation of Synthetic Triglycerides.
II. Analysis of the Volatile Condensable and Noncondensable Phases[1,2]

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Tripalmitin, 1-lauryl dipalmitin, 2-lauryl dipalmitin, 1-oleyl dipalmitin and 2-oleyl dipalmitin were subjected to thermal oxidation at 200 C in the presence of air. The volatile condensable products of oxidation collected after 3 and 24 hours were analyzed for carbonyl and carboxyl compounds. The volatile non-condensable products were quantitatively examined for carbon monoxide, carbon dioxide, and hydrogen. The results indicated that long chain carbonyls were formed in the initial stages of oxidation. When the oxidation was allowed to proceed for 24 hours, saturated aldehydes from C_1 to C_{16}, methyl ketones, and other ketones were found in the volatile oxidation products. A variety of fatty acids from C_4 to C_{16} were identified in the volatile fatty acids. The presence of dicarboxylic acids indicated that oxygen attacked the double bond of oleic acid in 1-oleyl dipalmitin. Possible mechanisms for the formation of the decomposition products were discussed.

THERMAL OXIDATION of a fat is known to produce a variety of decomposition products. A study of the types of carbonyl compounds and the fatty acids produced is of great importance in elucidation of the type of reactions taking place in a fat during thermal oxidation. The volatile condensable and noncondensable products in the thermal oxidation of synthetic triglycerides at 200C in the presence of air were therefore collected and characterized in order to gain a better insight into the mechanism of oxidation.

Experimental Procedure and Data

Tripalmitin, 1- and 2-lauryl dipalmitin, and 1- and 2-oleyl dipalmitin were thermally oxidized at 200C

in the presence of air. The synthetic triglycerides as well as the method of oxidation have been described in a preceding paper (1). The triglycerides were oxidized for periods of 3, 8, 15, and 24 hr. The condensable and noncondensable volatile products were collected for periods of 0–3, 3–8, 8–15, 15–24, and 0–24 hr. Upon completion of the reaction period, the absorption traps were detached and closed. The method for collection of volatile noncondensable products namely carbon monoxide, carbon dioxide. and hydrogen and their quantitative determination has been described in a previous paper (1). The volatile noncondensable decomposition products, namely carbon monoxide, carbon dioxide, and hydrogen, were calculated on a molar basis, and the amounts of carbon monoxide and hydrogen were calculated as equivalent to the carbon dioxide and water collected in the absorption tubes.

The cold traps containing the condensable products were removed from the −75C bath, the contents immediately taken up in carbonyl-free methanol, and the short connecting tube between the cold traps and the reaction flask rinsed with two 5 ml portions of carbonyl-free methanol. The condensate and washings were transferred to a 50 ml volumetric flask and made up to volume with carbonyl-free methanol; aliquots of this solution were used for further analysis.

Thirty ml of the methanolic solution which contained the condensable phase was neutralized with 0.1 N NaOH to the phenolphthalein end point, and extracted with three 20 ml. portions of ethyl ether. The ether extracts were combined and divided into two equal portions. Twenty ml of 2,4-dinitrophenylhydrazine reagent prepared in carbonyl-free methanol was added to one portion of the ether solution. The ether was allowed to evaporate slowly and the volume

TABLE I
Thermal Oxidation of Some Synthetic Triglycerides

Time in hr	Tripalmitin g/hr/g-mole			1-Lauryl, 2–3 dipalmitin g/hr/g-mole			2-Lauryl, 1–3 dipalmitin g/hr/g-mole			1-Oleyl. 2–3 dipalmitin g/hr/g-mole			2-Oleyl, 1–3 dipalmitin g/hr/g-mole		
	CO_2	CO	H_2	CO_2	CO	H_2	CO_2	CO	H_2	CO_2	CO	H_2	CO_2	CO	H_2
0– 3	1.41	2.45	0.120	1.54	1.34	0.111	0.85	0.92	0.081	0.52	0.74	0.065	0.67	0.60	0.048
3– 8	0.59	0.89	0.066	0.87	0.83	0.041	0.67	0.59	0.041	0.61	1.13	0.066	0.72	0.72	0.050
8–15	0.55	1.07	0.072	0.49	0.30	0.141	0.62	0.38	0.035	0.75	0.76	0.048	0.48	0.27	0.030
15–24	0.68	0.41	0.040	0.61	0.21	0.011	0.62	0.18	0.020	0.78	0.57	0.041	0.47	0.18	0.025
0–24	0.58	0.70	0.075	0.72	0.47	0.072	0.86	0.64	0.043	0.42	0.54	0.039	0.70	0.49	0.045

TABLE II
Analysis of Carbonyls from Volatile Condensable Oxidative Products of
Synthetic Triglycerides

Carbon number on UCON	Per Cent Composition Based on UCON Column									
	Tripalmitin		1-Lauryl dipalmitin		2-Lauryl dipalmitin		1-Oleyl dipalmitin		2-Oleyl dipalmitin	
	0–3 hr	0–24 hr	0–3 hr	0–24 hr	0–3 hr	0–24 hr	0–3 hr	0–24 hr	0–3 hr	0–24 hr
5.0	......	0.6	0.3	2.0	......	0.5	0.8	0.8	0.4	1.1
5.6	......	......	......	0.7	......	......	0.3	0.3	......	......
6.0	2.3	0.6	0.3	5.9	3.2	0.5	0.8	0.8	0.4	0.5
6.7	......	......	0.3	2.2	......	0.3	0.5	0.5	......	......
7.0	......	0.6	0.7	8.8	......	0.5	2.1	2.1	1.2	1.7
7.6	......	0.6	1.0	4.6	......	0.2	0.8	0.8	0.9	1.1
8.0	......	0.6	2.0	6.8	......	0.5	7.4	7.3	4.2	5.0
8.6	......	1.2	2.0	3.5	......	0.5	2.6	3.1	2.1	3.2
9.0	......	1.8	2.3	4.3	......	1.0	16.2	19.8	10.8	15.6
9.3	......	1.2	2.0	1.1	......	5.6	1.0	1.3	......	0.5
9.7	......	3.1	2.7	2.2	......	2.0	3.8	4.2	2.1	4.4
10.0	......	3.7	4.0	8.2	......	3.5	5.9	7.6	5.8	7.8
10.3	......	3.1	......	1.5	......	3.0	1.3	2.3	......	1.1
10.7	......	7.0	3.0	1.8	......	8.0	1.3	1.3	1.6	1.7
11.0	......	8.0	6.0	10.0	......	13.0	12.8	12.8	11.3	13.9
11.3	......	4.9	......	......	......	6.0	......	......	......	......
11.7	......	25.0	......	5.8	......	9.0	2.3	2.1	2.4	2.8
12.0	26.4	6.8	8.0	4.4	52.8	4.1	10.8	10.4	16.9	12.2
12.3	......	4.3	......	5.5	......	5.0	......	......	......	......
13.0	39.7	15.4	40.0	11.1	24.8	22.0	19.2	10.1	25.6	6.1
14.0	33.6	6.2	25.0	2.2	16.0	4.0	10.3	6.5	14.3	7.8
15.0	......	5.0	......	7.3	......	10.6	......	5.9	......	12.2

of the methanolic solution reduced to approximately 5 ml on a steam bath. The solution was made slightly turbid with distilled water, cooled overnight in a refrigerator, the 2,4-dinitrophenylhydrazones removed by filtration, washed with cold water, and dried under vacuum. The hydrazones were then dissolved in 2 ml of carbonyl-free methanol, and the solution was subjected to paper chromatography (2).

Chromatograms were run on a 7.5 x 40 cm strip of Whatman No. 1 filter paper which had been exposed to a solution containing 20% ethylene glycol and 80% carbonyl-free methanol (v/v) and dried in a draft of air for 1 hr. The paper strips were spotted with a standard and an unknown solution, and the strips allowed to equilibrate in a chromatographic jar overnight. Fifteen ml. of n-heptane saturated with methanol was then added to the solvent trough, and the hydrazones allowed to separate by descending chromatography.

The second half of the ether extract was dried over sodium sulfate, the solvent removed with a stream of nitrogen, and the residue subjected to gas liquid chromatography in an Aerograph A-90-C instrument. The carbonyls in the residues were separated on a 5 ft ¼ in copper column packed with Ucon, nonpolar substrate, supported on 30–60 mesh fire brick. Column temperature was maintained at 170C with a helium pressure of 10 psig, and filament current was kept at 200 ma. A standard containing equal volumes of saturated normal aliphatic aldehydes from C_3 to C_{10} was run on the gas chromatogram. A plot of retention times versus chain length gave a straight line on the semilog paper; a standard curve was used to identify peaks in the unknown sample.

The methanolic solution which had been neutralized with 0.1 N NaOH and freed of carbonyls was acidified with dilute HCl, and extracted with three 10 ml portions of ethyl ether. The ether extracts were washed free of mineral acid with distilled water, dried over anhydrous sodium sulfate, the solvent removed in a stream of nitrogen, and the methyl esters prepared according to the method of Radin et al. (3). The methyl esters were analyzed by gas liquid chromatography as previously described (1).

Results and Discussion

A gain in weight of the absorption tubes was noted indicating the formation of carbon dioxide, carbon monoxide, and hydrogen during thermal oxidation of the glycerides (Table I). The evolution of hydrogen indicated that dehydrogenation of the glyceride molecule may have occurred; the evolution of carbon monoxide was equal to or more than that of carbon dioxide in the early and middle stages of thermal oxidation. After 15 hr, carbon dioxide was evolved more readily. The evolution of carbon monoxide could have occurred through the elimination of a carbonyl group from a nonterminal position in the glyceride molecule. Either of the following types of carbonyl compounds can undergo such a reaction:

$$RCOCH_2COCOOR \longrightarrow RCOCH_2COOR + CO$$
$$RCOCOCOR \longrightarrow RCOCOR + CO$$
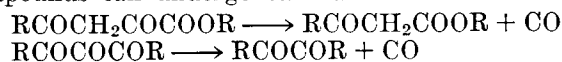
It has been observed that such compounds will lose carbon monoxide readily. In such compounds the carbonyl group to be eliminated is held loosely because of the electron impoverishment of the adjacent atoms. It might be assumed that oxygen attacks in a random manner and, as such, formation of such compounds is a definite possibility.

The presence of formaldehyde, acetaldehyde, and propionaldehyde indicated the formation of short chain compounds even in the early stages of oxidation. There were also a number of other spots on the paper, which were difficult to identify. The carbonyls were, therefore, analyzed by gas liquid chromatography us-

TABLE III

Composition of Fatty Acids from Condensable Volatile Decomposition
Products—Thermally Oxidized Triglycerides

Carbon number on DEGS	Carbon number on Apiezon L	Tentative identification	Per Cent Composition Based on DEGS									
			Tripalmitin		1-Lauryl, 2-3 Dipalmitin		2-Lauryl, 1-3 Dipalmitin		1-Oleyl, 2-3 Dipalmitin		2-Oleyl, 1-3 Dipalmitin	
			0-3 hr	0-24 hr	0-3 hr	0-24 hr	0-3 hr	0-24 hr	0-3 hr	0-24 hr.	0-3 hr	0-24 hr
4.0	4.0	$C_4 S^a$	......	......	......	......	......	......	......	5.1	......	......
5.0	5.0	$C_5 S$	......	......	......	......	......	......	33.4	13.7	......	......
6.0	6.0	$C_6 S$	2.4	......	......	3.8	......	......	4.6	......	10.0	......
6.5	......	U^b	2.2	......	......	......	......	......	......	10.9	......	......
7.0	7.0	$C_7 S$	4.8	Trace	......	3.1	0.8	Trace	......	......	......	4.9
7.6	......	U	3.9	......	......	......	......	......	9.4	5.8	......	......
8.0	8.0	$C_8 S$	Trace	0.4	......	7.7	0.9	1.2	5.2	13.9	20.0	16.8
8.5	......	U	8.4	6.4	......	......	......	......	......	0.9	......	......
9.0	9.0	$C_9 S$	Trace	6.4	......	4.3	1.0	3.8	......	3.3	20.4	25.2
9.7	......	U	7.8	......	......	......	......	......	3.5	2.7	......	......
10.0	10.0	$C_{10} S$	Trace	6.4	1.4	5.5	2.3	8.8	......	......	8.6	4.9
10.7	......	U	2.2	......	......	......	......	......	1.0	......	......	5.2
11.0	11.0	$C_{11} S$	Trace	6.4	3.2	7.5	7.3	13.7	0.7	3.8	6.1	3.2
11.5	6.7	$C_4 D^c$	......	......	......	......	......	......	1.7	4.7	......	......
12.0	12.0	$C_{12} S$	3.6	9.5	10.1	22.6	48.7	31.5	1.2	1.5	15.3	14.3
12.6	......	U	......	......	......	......	......	......	0.7	3.6	5.5	6.8
13.0	13.0	$C_{13} S$	3.9	5.8	1.4	1.6	6.3	6.2	2.8	......	......	1.4
13.5	12.4	HA^d	......	11.3	61.8	6.8	16.0	7.2	......	......	......	......
13.5	8.8	$C_6 D$	......	......	......	......	......	......	......	8.0	......	......
14.0	14.0	$C_{14} S$	26.0	2.1	4.1	4.0	4.9	2.1	5.6	......	11.3	5.0
14.3	......	U	6.5	1.0	......	0.2	......	1.1	......	2.6	......	4.1
15.0	15.0	$C_{15} S$	1.8	4.2	4.1	4.3	3.0	6.1	0.4	1.8	1.6	2.5
15.4	10.6	$C_8 D$	1.1	1.2	......	......	......	1.2	......	......	......	......
16.0	16.0	$C_{16} S$	25.6	26.5	14.0	22.9	5.0	10.2	3.2	12.3	1.0	3.3
16.5	11.7	$C_9 D$	Trace	1.7	......	......	......	......	0.8	1.4	......	......
17.0	......	U	......	2.5	......	3.1	1.9	1.3	2.9	1.5	......	1.7
17.6	......	C	......	0.5	......	0.2	......	1.6	......	......	......	......
18.0	18.0	$C_{18} S$	......	0.8	......	......	1.9	Trace	......	......	......	......
18.3	17.6	$C_{18} M^e$	......	......	......	......	......	......	......	......	......	1.4
18.8	......	U	......	4.4	......	......	......	2.3	......	4.0	......	......
19.8	......	U	......	Trace	......	......	......	......	......	......	......	......
20.8	......	U	......	2.5	......	......	......	......	......	Trace	......	......

a Saturated.　b Unidentified.　c Dicarboxylic.　d Hydroxy acid.　e Monoenoic.

ing authentic aldehydes as standards. Some important differences in the condensable carbonyls were observed in the first 3 hr of oxidation (Table II). In the case of tripalmitin and 2-lauryl dipalmitin, the major fraction was found to be composed of C_{13} and C_{14} aldehydes. However, the volatile condensable products from 1-lauryl dipalmitin and 1- and 2-oleyl dipalmitin were found to contain a whole spectra of saturated aldehydes from C_5 to C_{15} in addition to a number of compounds which were not identified. When the oxidation was continued for more than 3 hr, there was practically no difference in the volatile decomposition carbonyl compounds from different triglycerides. The results indicated that the attack of oxygen in the early stages of oxidation probably took place near the ester linkage in the glyceride molecule with the formation of hydroperoxides and of scission to long chain aldehydes, namely C_{13} and C_{14}. However, on further heat treatment the attack of oxygen took place at random with the formation of short and long chain carbonyl compounds.

The unidentified peaks on the gas chromatogram might have been due to various ketonic compounds and probably unsaturated aldehydes. In all cases the carbonyl fractions gave a positive iodoform reaction indicating the presence of methyl ketones. As saturated aldehydes and methyl ketones of the same chain length had almost the same retention times, it was impossible to differentiate between the two. The carbonyl fractions were therefore oxidized with 3% hydrogen peroxide (4). The aldehydes were converted to the corresponding acids with the other carbonyls remaining unreacted. The acids were recovered and analyzed by gas chromatography after converting them into methyl esters. In general, saturated aldehydes from C_5 to C_{14} were detected. If the carbonyl group was shifted nearer to the middle of the chain, it was observed that such a compound had a lower retention time on a Ucon column as compared to the aldehyde of the same chain length. It appeared from this observation that some of the unidentified peaks might have been due to ketones of the same chain length as an aldehyde indicated by the next peak on the gas chromatogram. Some of the unidentified peaks might also be due to monocarbonyl enals, as the formation of such carbonyls on mild oxidation of methyl oleate and triolein has been shown by Gaddis et al. (5).

With an increase in the time of oxidation, there was a gradual increase in the acidity of the volatile condensable phase indicating the increased evolution of fatty acids. The volatile phase was found to contain a variety of fatty acids (Table III). In the case of tripalmitin, myristic, and palmitic acids were the main fractions, the remainder being made up of fatty acids from C_6 to C_{15}. Lauric and palmitic acids were the main fractions obtained during the oxidation of 1- and 2-lauryl dipalmitin. These fatty acids were probably hydrolyzed from the glyceride molecules as reported earlier (1), and swept into the traps from the reaction flask. The hydroxy acid with a carbon number of 13.5 on diethylene glycol succinate column was also found in the volatile condensation products. Saturated fatty acids from C_4 to C_{16} in chain length were found in the volatile phase after oxidation of 1- and 2-oleyl dipalmitin. However, the major portion consisted of the short chain fatty acids. C_4, C_6, C_9, and C_{10} dicarboxylic fatty acids were found in the volatile products from 1-oleyl dipalmitin. The identification of dicarboxylic acids definitely indicated that there is a shifting of the double bond from the usual 9:10 position in oleic acid during heating, and that the attack of oxygen takes place at the double bond with the formation of dicarboxylic acids. The high percentage of C_8 and C_9 saturated acids, and the absence of dicarboxylic acids in the case of 2-oleyl dipalmitin, indicated that oxidation of the double bond did not lead to a mono- and dicarboxylic acid formation. In the usual reaction when a double bond is ruptured by oxidation, the ends of both the fragments are oxidized to carboxyl groups. In the case of
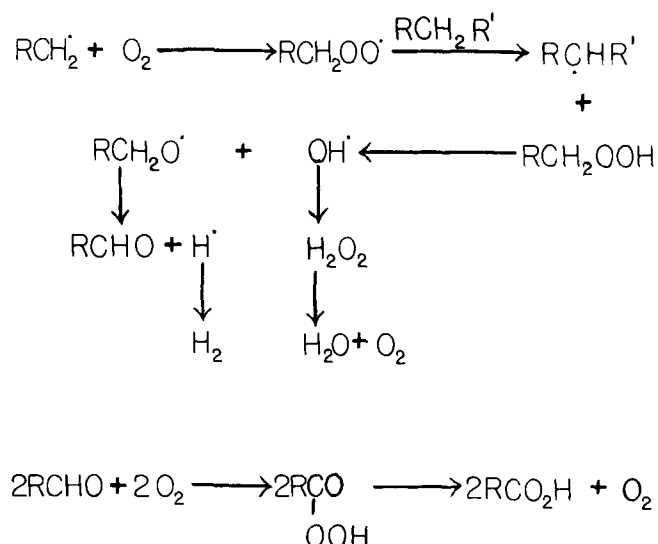
$$RCH_2^{\cdot} + O_2 \longrightarrow RCH_2OO^{\cdot} \xrightarrow{RCH_2R'} R\overset{\cdot}{C}HR'$$

$$+$$

$$RCH_2O^{\cdot} + \overset{\cdot}{O}H \longleftarrow RCH_2OOH$$

$$\downarrow \qquad\qquad \downarrow$$

$$RCHO + H^{\cdot} \qquad H_2O_2$$

$$\downarrow \qquad\qquad \downarrow$$

$$H_2 \qquad\qquad H_2O + O_2$$

$$2RCHO + 2O_2 \longrightarrow 2R\underset{OOH}{\overset{|}{C}O} \longrightarrow 2RCO_2H + O_2$$

FIG. 1. Reaction scheme for the formation of water and oxygen.

2-oleyl dipalmitin the other fragment is probably oxidized to a semialdehyde. There is also a possibility that the dicarboxylic acid undergoes a beta oxidation and decarboxylation to form a methyl ketone at one end of the molecule.

The appearance of short chain carbonyls and fatty acids in the volatile condensable products indicated that oxygen attacked the fatty acids of the glyceride molecules in the initial stages of oxidation, which resulted in the formation of hydroperoxides and free radicals followed by cleavage. Several workers (6,7,8) have proposed mechanisms by which hydroperoxides break down with simultaneous cleavage of the adjacent carbon to carbon bond to form an aldehyde and some unidentified fragment. The specific aldehyde which is formed depends upon the position of the hydroperoxide group in the chain, and the length of the carbon chain which is cleaved from the parent molecule.

It is possible that ketones are preferentially formed from peroxide radicals and peroxides. These peroxides are at methylene carbon atoms, and probably occur more or less at random. Further oxidation of the ketone can cause cleavage of the carbon chain and yield acids or aldehydes.

According to Fritsch and Deatherage (8) an alkyl free radical is initially formed during thermal oxidation. Such a radical could form through decarboxylation of free acids during thermal oxidation (1). The alkyl free radical could react with oxygen to yield a primary hydroperoxide. The hydroperoxide at the high temperature of thermal oxidation would decompose into an alkoxy and a hydroxy radical. The highly unstable alkoxy radical would produce an aldehyde, and the aldehyde could be oxidized to an acid. The hydroxy radical could dimerize into hydrogen peroxide which would rapidly decompose into water and oxygen. Reactions involved are shown in Figure 1.

The chemistry of oils oxidized at high temperature is more complex than that of either thermal or oxidative polymerization, since both heat and oxygen are involved. It appears that hydrolysis of the ester linkage occurs. Formation of a free acid with subsequent decarboxylation results in a molecule which can easily enter into the series of reactions as proposed by Fritsch and Deatherage. The end products of this series would be aldehydes, lower molecular weight acids, water, hydrogen, and oxygen. All these products, with the exception of oxygen, were detected as end products of thermal oxidation.

Oxygenation of the glyceride molecule can activate the molecule so as to make the molecule more susceptible to oxidation. Breakdown products of such activated molecules can undergo oxidation, be removed from the oxidation system, or individually undergo further oxidation and breakdown.

Esters containing a beta hydrogen (Fig. 2) are believed to undergo thermal decomposition via a quasi
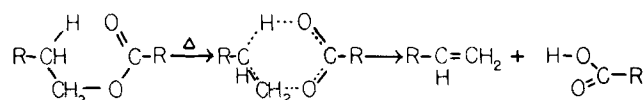

FIG. 2. Thermal decomposition of ester via a quasi six-membered ring.

six-membered ring (9). Thus, in a purely thermal system, as an organic compound is heated to higher and higher temperatures, the vibrational energy distributed among the bonds of the molecule increases. In the simple case of a diatomic molecule, when vibrational energy becomes equal to bond strength, the molecule will rupture. In more complex molecules, the bond strength of the weakest bond determines the upper limit of thermal stability, if there are no low energy decomposition pathways. When thermal effects are coupled with oxidative effects, the reactions become more complex. Each type of reaction requires activation. The reaction which requires the lowest activation energy occurs first and those requiring higher activation energies later. The series of reactions are not simple, nor are they in any way definitive, since low energy decomposition pathways may result due to fixation of oxygen in the molecule or to partial decomposition of the molecule. Such changes in molecular structure will undoubtedly alter the energy distribution of a molecule, thereby changing the type of reaction that might occur.

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