

• Technical

The Isolation and Characterization of the Polymers Formed During the Thermal Oxidation of Corn Oil¹

E. G. PERKINS² and F. A. KUMMEROW, Department of Food Technology, University of Illinois, Urbana, Illinois

THE CHEMISTRY of thermally oxidized oils has not been extensively explored. The oxidation of linseed oil at high temperatures (1) indicated that this oxidation involved free radical mechanisms, such as those proposed for autoxidation (2, 3, 4, 5, 6). Hess and O'Hare have indicated that hydroxyl groups may be initially formed during thermal oxidation and that these hydroxyl groups could then react to form other products (1). Moreno and Lopez oxidized cottonseed and olive oil at 120°C. with air and manganese dioxide as catalyst and produced hydroxylated compounds (7) while Skellon and Taylor oxidized brassidic acid at 120°C. and isolated dihydroxybehenic and keto-hydroxy acids from the reaction mixture (8). In general, the literature concerning thermal oxidation suggested that the high-temperature oxidation of long-chain fatty acids resulted in hydroxylated materials of varying molecular weight. Since edible oils are subjected to temperatures of about 200°C. in the presence of air during commercial food-frying operations, it is of interest to examine more closely the types of products which could be formed in such oils under controlled laboratory conditions. In the present study corn oil was thermally oxidized at 200°C., the oil was separated into various fractions, and the fractions were characterized.

Experimental

Preparatory and Analytical Procedures. The thermal oxidation of corn oil was carried out as in previous studies (9). In the present study approximately 1 kg. of corn oil was oxidized at 200°C. \pm 5°C. for 48 hrs. Air was bubbled through the hot oil at the rate of 200 ml./min./kg. The fatty acids were prepared by saponification of the thermally oxidized oil with potassium hydroxide in 95% ethanol; the amount of base used was calculated from the saponification value of the oil. The methyl esters were prepared from the acidified soaps by esterification with methanol, using a ratio of 1 g. of fatty acid to 10 ml. of methanol.

The fatty acid composition of the thermally oxidized oils and isolated polymer fractions was determined by the spectrophotometric method of Brice *et al.* (10). The Wijs iodine values were determined according to the Official A.O.C.S. method (11). Neutralization and saponification equivalents were determined with the aid of the micro method of Pregl (12). The molecular weight determinations were carried out according to a modification of the original Rast method (13). Hydroxyl groups were determined

by the method of Smith and Shriner (14) except that the heating was done on a steam bath for 30 min.

Fractionation of the Fatty Acids from Thermally Oxidized Corn Oil. The fatty acids from thermally oxidized corn oil were subjected to urea fractionation as previously described (15). The nonurea adduct-forming fraction (NAFM) was distilled at 150°C. at 1–3 μ in a short-path, falling-film molecular still of our own design. The nondistillable material was fractionated with Skellysolve F and cyclohexane. The solvent fractionation procedures and the fractions obtained are outlined in Figure 1. The polymeric fractions were exhaustively extracted with Skellysolve F and cyclohexane; the ratios of solvent to polymer fractions employed were in the order of 10 to 1. The fractions obtained were completely soluble in toluene, carbon tetrachloride, chloroform, diethyl ether, and ethanol. Attempts to fractionate and purify these materials by further solvent fractionation, or by paper and column chromatography proved fruitless.

Results

The thermal oxidation of corn oil for 48 hrs. at 200°C. led to the formation of an oil which differed both chemically and nutritionally from fresh corn oil. The differences in chemical composition are presented in this paper; the differences in nutritional properties have been presented elsewhere (16). As previously reported (17), an increase in viscosity and oxygen content and a decrease in unsaturation were observed in thermally oxidized corn oil as compared with fresh corn oil.

A comparison of the fatty acid composition of fresh corn oil with thermally oxidized oil (Table I) indicated that the amount of linoleic acid was greatly reduced during thermal oxidation. This decrease probably resulted from the oxidation, scission, and polymerization of the double bonds present in linoleic acid. Treatment with urea resulted in the separation of the unchanged fatty acids from the oxidized material. From several such urea fractionations it was found that 64% of the fatty acids formed urea adducts; the remaining 36% failed to form complexes with urea. The composition of these fractions is summarized under Section II, Table I.

The nonurea adduct-forming fraction contained some linoleic acid, probably also small amounts of oleic and saturated fatty acids which were not removed by urea fractionation. The urea adduct-forming fatty acids appeared to be free of polymers and seemed to be composed of a mixture of normal straight-chain fatty acids. Their hydrogenation at room temperature and at atmospheric pressure in ethyl acetate with platinum oxide as catalyst yielded a mixture of solid fatty acids. A mixed melting point with stearic

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² Portion of a thesis presented by E. G. Perkins as partial fulfillment of the requirements for the degree of Doctor of Philosophy in Food Technology. Present address: Armour and Company, Research Division, Chicago, Ill.

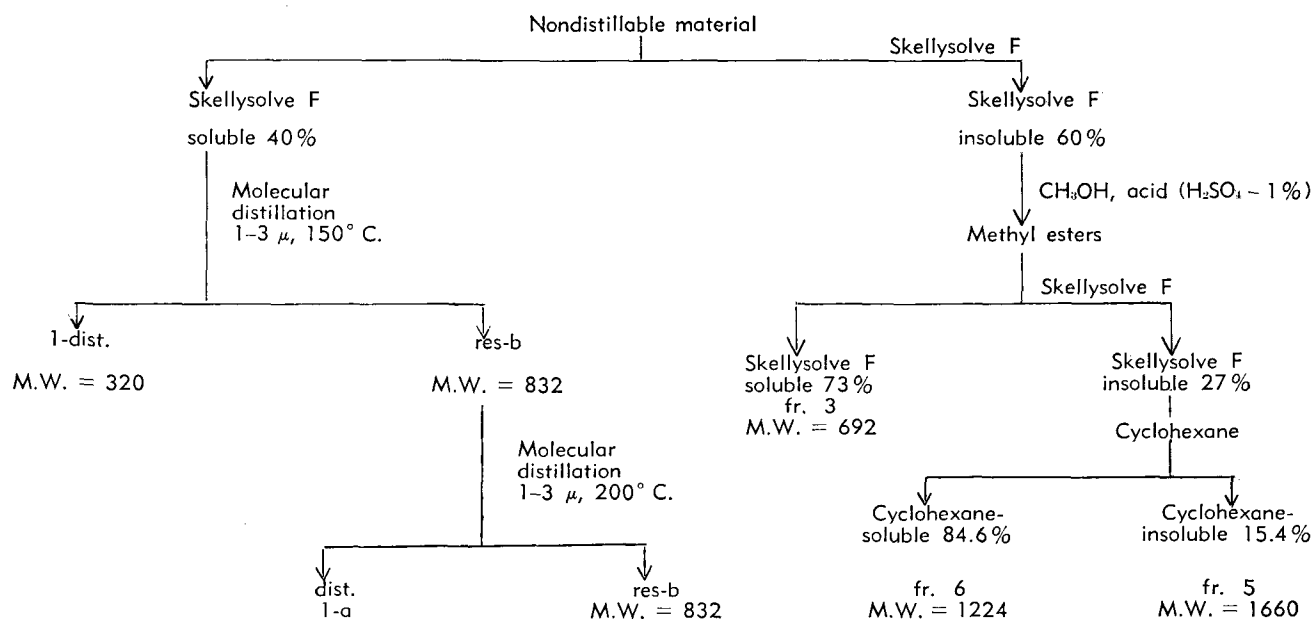


FIG. 1. Fractionation of nondistillable material from NAFM

acid of 68–69°C. indicated that this mixture was largely composed of stearic acid.

The distillate obtained on high-vacuum distillation of the nonurea adduct-forming fraction was composed of linoleic, oleic, and saturated fatty acids (Section III, Table I). The composition of the distillate was confirmed by paper chromatographic identification of the fatty acids as their mercuric acetate adducts, according to the method of Inouye *et al.* (18). A spectrophotometric analysis of the nondistillable material indicated that a small amount of diene had not been removed by high-vacuum distillation.

The empirical formulas of the fractions obtained on solvent fractionation of the nondistillable material were calculated from their carbon hydrogen content, from their molecular weights as determined by the Rast method, and from their neutralization equivalents (Table II). Since these methods are subject to limitations in accuracy, the results obtained are only to be considered as approximations. The fraction (1-dist.) obtained on distillation of the nondistillable material (mol. wt. 320) corresponded to linoleic acid. The residue *b* and its distillate 1-a contained highly oxygenated, high-molecular-weight materials.

A comparison of the observed iodine values of the various fractions with their calculated iodine values gave only an estimate of the amount of unsaturation present in the fractions since iodine does not add quan-

titatively to double bonds of polymers (Table III). Examination of the data presented in Table III indicated that the number of double bonds present in the fractions varied from two to four, depending upon the size of the molecule.

A sample of thermally oxidized oil was hydrogenated³ to an iodine value of 33 with 3% Rainey nickel catalyst under 500 lbs. of pressure at 150°C. A sample of the nonurea adduct-forming fatty acids, when hydrogenated over 1% platinum dioxide in ethyl acetate at atmospheric pressure and room temperature, could not be hydrogenated to an iodine value lower than 55. This type of behavior may indicate that the double bonds were in some way hidden or otherwise blocked so that either the hydrogen or catalyst or both could not reach them. Examination of the infrared spectra of fractions 1 res-b, 1-a, 3, 5, and 6 at 6.0 μ and 10.3 μ indicated the presence of unsaturation, but the bands found were of very weak intensity.

Information concerning the distribution of oxygen in the isolated fractions was obtained from the determination of neutralization equivalents and of hydroxyl groups (Table IV). Attempts to determine *alpha*-dihydroxy groups quantitatively by the method of Pasehke and Wheeler (19) did not prove satis-

³ The hydrogenation was done through the courtesy of R. J. Vander Wal, Armour and Company, Chicago, Ill.

TABLE I
Comparison of the Properties and Fatty Acid Composition of (I) Fresh Corn Oil with Thermally Oxidized Corn Oil, (II) the Urea and NAFM,^d and (III) the Distillable and Nondistillable Material of the NAFM

	I		II		III	
	Fresh oil	Thermally ^b oxidized oil	Urea adduct-forming acids	Nonurea adduct-forming material	Distillable ^c material	Nondistillable material
Iodine value.....	126.0	70.1	62.0	83.0	113.0	70.0
Molecular wt. (Rast).....	294	454	300	512	320	630
n_D^{25} / ^d	1.4724	1.4812	1.4528	1.4828	1.4676	1.4981
Oleic.....	28.2%	44.4%	61.8%	57.9%
Linoleic.....	55.4	10.0	3.5	9.5	33.6	9.5
Triene.....	0	0	0	0	0	0
Saturated.....	16.4	23.1	34.7	8.5	0
Polymeric material ^a	0	22.5	0	0	90.5

^a Polymeric material calculated from results of distillation. ^b Corn oil thermally oxidized for 48 hours at 200°C. \pm 5°C. ^c Molecular distillation of NAFM at 150°C. under 1–3 μ pressure. ^d NAFM = Nonurea adduct-forming material.

TABLE II
Analysis of the Fractions Isolated from the Fatty Acids of Thermally Oxidized Corn Oil^a

Fraction ^a	1-dist.	res.-b	dist. 1-a	3	5	6
Type.....	RCO ₂ H	RCO ₂ H	RCO ₂ H	RCO ₂ CH ₃	RCO ₂ CH ₃	RCO ₂ CH ₃
Iodine value.....	107.0	78.7	81.0	64.5	56.6	66.6
M. wt. (Rast).....	320	852	607	692	1660	1224
Sap. equiv.....	400	380	330	277	598
% C.....	75.22	73.98	73.41	73.58	70.50	72.23
% H.....	11.60	10.99	10.98	10.58	9.61	10.22
% O.....	13.18	16.13	15.69	15.84	19.81	17.65
Empirical formula (approx.).....	C ₁₉ H ₃₇ O ₂	C ₄₀ H ₈₁ O ₂	C ₃₇ H ₆₆ O ₂	C ₄₁ H ₈₇ O ₂	C ₃₈ H ₁₅₉ O ₂₀	C ₇₄ H ₁₂₄ O ₁₃

^a The origin of the fractions is outlined in Figure 1.

factory as reproducible results could not be obtained. Examination of the distribution of oxygen in the isolated fractions indicated that the oxygen present in the molecules was in the form of carboxyl and hydroxyl groups

In an attempt to determine whether or not any Diels Alder types of cyclization had taken place during thermal oxidation, the fraction corresponding to a dimer (molecular weight = 692) was treated with palladium on carbon catalyst for 7 hrs. at 350°C., both in a sealed tube and in an open tube. After the reaction was discontinued, the product was dissolved in ethyl ether, the catalyst was removed by filtration, and the product was examined in the ultraviolet region of the spectrum for the presence of aromatic structures. The sample thus obtained showed no adsorption peaks in the ultraviolet region. A model compound prepared from the reaction of linoleic acid with maleic anhydride, when treated under these same conditions, gave a band at 256 millimicrons in the ultraviolet region of the spectrum characteristic of aromatic structures.

Samples of the NAFM were treated according to the aromatization method of Paschke and Wheeler (19), and the final products were examined in the ultraviolet for the presence of aromatic structures. The samples showed no absorption in the regions specific for aromatic compounds.

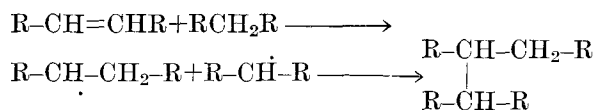
The infrared spectra of fractions 1 res.-b, 1-a, 3, 5, and 6 indicated the presence of hydroxyl groups and were essentially identical. The infrared spectra of these fractions were also identical with methyl linoleate, with the exception of the bands for the hydroxyl group at 2.7-2.8 μ, 9.0, and 7.5-7.9 μ.

Discussion

Several hypotheses have been proposed to account for polymer formation when unsaturated fatty acids are heated in the absence of air, but few of them have taken into consideration the role of oxygen in such systems. The attack of oxygen at 200°C. can be assumed initially to take place at the methylene group adjacent to a double bond with the formation of a conjugated hydroperoxide. The reaction mechanisms proposed by Farmer and other researchers support

this view (2, 3, 4). Evidence (5, 6) has been obtained which indicates that oxygen attacks predominately at carbons 9 and 13. This type of reaction can be assumed to take place at high temperatures, if even instantaneously, forming conjugated hydroperoxides which could then decompose, cause polymerization, or be oxidized.

When unsaturated fatty acids are heated in the absence of air, it has been shown that polymers are formed through Diels Alder type reactions by the addition of a 1,4-diene to any other double bond to form a tetrasubstituted cyclohexene ring structure (20-26). Sunderland (27) has proposed a mechanism for this type of polymerization for unsaturated fatty acids. He proposed a mechanism in which a carbon-carbon double bond abstracted a hydrogen atom from a methylene group in another molecule, then joined the two molecules through a single carbon-carbon bond:



However only circumstantial evidence exists for the presence of these compounds in thermally oxidized oils. Such evidence depends primarily upon the increased viscosity, increased index of refraction, and

TABLE III
Comparison of Calculated Iodine Values and Observed Iodine Values in the Isolated Polymer Fractions

Fraction	Calcd. for one double bond	Observed iodine value	Calcd. from molecular weight	No. of double bonds
1 res.-b.....	31.2	78.7	62.4	2.6
1-a.....	41.2	81.0	82.4	1.9
3.....	36.8	64.0	73.6	1.8
5.....	15.3	56.0	61.2	3.7
6.....	20.8	66.0	62.4	3.2

decreased unsaturation observed in thermally oxidized oils.

The suggestion by Paschke *et al.* (19) that linoleate polymerized in the presence of oxygen could form cyclic monomers which contained hydroxyl groups took into account the influence of oxygen in poly-

TABLE IV
Distribution of Oxygen in Isolated Polymer Fractions

Fraction	Oxygen	Molecular weight (Rast)	Atoms oxygen per molecule	Hydroxyl	Oxygen as hydroxyl	Atoms oxygen as hydroxyl	Oxygen as carboxyl	Atoms oxygen as carboxyl	Oxygen unaccounted for
	%			%	%		%		
1 res.-b.....	16.13	832	8	8.90	8.28	4	8.06	4	0.31
1-a.....	15.64	607	6	5.20	4.92	2	10.46	4	0.39
3.....	15.84	692	6	7.25	6.77	2	11.56	4	0.03
5.....	19.81	1,662	20	8.83	8.31	8	11.88	12	0.38
6.....	17.65	1,224	13	11.86	11.11	9	5.43	4	0.36

TABLE V
Hypothetical Structures of Three Possible Dimers Isolated from Thermally Oxidized Corn Oil

Constants of dimers	Theoretical values for dimers			Found Fraction 3
	A	B	C	
Iodine value.....	77.9	81.7	79.7	64.5
Percentage of OH.....	10.4	5.46	8.00	7.25
Saponification equivalent.....	327	311	319	330
Molecular weight.....	654	622	638	692
Empirical formula.....	C ₃₈ H ₇₀ O ₈	C ₃₈ H ₇₀ O ₆	C ₃₈ H ₇₀ O ₇	C ₄₁ H ₆₇ O ₆
Presence of <i>alpha</i> -dihydroxyl group.....	+	—	+	+
Proposed structural formula ^a	$\begin{array}{c} R_1\text{CH}-\text{CH}-\text{CH}-\text{CH}=\text{CHR}_2 \\ \quad \\ \text{OH} \quad \text{OH} \\ R_3\text{CH}-\text{CH}-\text{CH}-\text{CH}=\text{CHR}_2 \\ \quad \\ \text{OH} \quad \text{OH} \end{array}$	$\begin{array}{c} R_3\text{CH}-\text{CH}-\text{CH}=\text{CHR}_2 \\ \\ \text{OH} \\ R_3\text{CH}-\text{CH}-\text{CH}=\text{CHR}_2 \\ \\ \text{OH} \end{array}$	$\begin{array}{c} R_1\text{CH}-\text{CH}-\text{CH}-\text{CH}=\text{CHR}_2 \\ \quad \\ \text{OH} \quad \text{OH} \\ R_3\text{CH}-\text{CH}-\text{CH}=\text{CHR}_2 \\ \\ \text{OH} \end{array}$	Mixture of A, B, C & higher polymers?

^a Position of the double bonds is unknown. R₁ = CH₃(CH₂)₄; R₂ = (CH₂)₇CO₂CH₃; R₃ = CH₃(CH₂)₅.

merization. The products formed according to this mechanism could further polymerize to give high-molecular-weight compounds of high oxygen content. This type of reaction would also cause an apparent loss in unsaturation. However no chemical evidence has yet been published to demonstrate the presence of these compounds in thermally oxidized oils. Furthermore the present study indicated that this type of compound probably did not occur in thermally oxidized oil as no evidence for it could be obtained. Such cyclic monomers should be distillable under high vacuum, but the distillates obtained in the present study all formed straight-chain, urea adduct-forming fatty acids. It is possible that these fatty acids were polymerized into polymer systems containing six-membered rings. Polymers containing six-membered rings could also be formed by the addition of a 1,4-diene system to any dienophile present in the material undergoing polymerization. Dehydrogenation with palladium of the various polymer fractions obtained in the present study and attempted aromatization of them by the bromination-debromination method of Paschke and Wheeler (19) gave no indications of any cyclic material present in thermally oxidized corn oil although a similar dehydrogenation of a model compound gave indications of aromatic structures. These results could also be interpreted to mean that dehydrogenation with palladium and/or bromination-debromination failed because of steric interferences even though such structures may be present in thermally oxidized corn oil.

In the present study qualitative evidence for the presence of *alpha*-dihydroxy groups was obtained, indicating that the polymer fractions contained the *alpha*-dihydroxy group in some part of the molecule. Since several compounds were isolated upon periodate cleavage, they probably contained a mixture of isomeric structures containing the *alpha*-dihydroxy group.

The infrared spectra of the polymeric fractions were almost identical with methyl linoleate, with the exception of the hydroxyl group stretching absorption. This similarity indicated that they may have the same monomeric units.

The mechanism proposed by Sunderland could conceivably account for the formation of these polymers if oxygen were not present (27). Such reactions could take place between linoleic or oleic acid, depending upon the concentrations of these fatty acids in the oils being polymerized. A modification of this hypothesis could possibly explain how the polymers were formed in the thermally oxidized oil if one takes into consideration the distribution of oxygen found

in the isolated polymers, the probable unsaturation of the materials, the similarity of the infrared spectra, and the lack of evidence for the six-membered ring structure.

As Farmer has demonstrated (2), the initial attack of oxygen on an unsaturated system results in the formation of conjugated hydroperoxides. If hydroperoxides were formed at 200°C., they would have decomposed at the high temperatures used in this study to generate free radicals. Some free hydroxyl radicals may also have formed during the oxidation. Any or all of these free radicals may have been present at any one instant in any one of their possible resonance structures. These various free radicals may then have reacted to form increasingly complex polymeric materials. Chain transfer reactions and chain termination reactions could result in an isomeric mixture of varying molecular weight polymers (Table V). This could have been accomplished by combination with another large free radical, by abstraction of a hydrogen atom from another molecule, or by reaction with a free hydroxyl radical resulting from the decomposition of another hydroperoxide molecule.

Polymers formed in this way would give identical spectra. The polymers would have a high molecular weight and a high hydroxyl content, with some *alpha*-dihydroxyl groups present in the molecules. Such compounds would have no six-membered ring structures if formed through a mechanism similar to that proposed by Sunderland.

Summary

It has been shown that thermal oxidation of corn oil at 200°C. caused the formation of polymeric material. A combination of urea fractionation and molecular distillation was employed to concentrate the polymeric material. Further fractionation of the polymer concentrates was performed with the aid of solvent-extraction procedures. These procedures resulted in the isolation of several polymer fractions with molecular weights ranging from 692 to 1,600. Analyses of the fractions indicated that they were of high oxygen content and that they contained unsaturation, difficult to remove by hydrogenation. The oxygen present in the fractions was shown to be in the form of hydroxyl and carboxyl groups. The polymeric materials could be linked together in a non-cyclic structure.

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A Commercial Process for the Recovery of Deodorizer Distillates^{1,2}

RICHARD J. FIALA, A. E. Staley Manufacturing Company, Decatur, Illinois

A BASIC PROBLEM facing the fat and oil industry is the recovery of the organic distillates from the deodorization process. Currently these distillates are drawn from the deodorizer into the vacuum system where they are condensed with water. This method of operation dilutes the distillates to a point where conventional recovery techniques are neither economical nor efficient. This problem is two-fold since the organic distillates which are lost represent commercially valuable materials and stream contaminants which complicate disposal techniques. Because of increasing community pressures and advancing costs, much concern over the recovery of these materials has been shown.

To correct this situation the Croll-Reynolds Company has developed a novel vapor-scrubbing unit. This unit, the Convactor, effects the removal of essentially all of the organic distillates from the deodorizer effluent vapors, thereby reducing stream pollution. Furthermore the organic distillates are concentrated by the Convactor in a closed-circuit, condensing-water system, making economic recovery of the distillates possible.

At Staley process water from the deodorizer barometric condensers is discharged to a hotwell. Here 50-75% of the organic material is recovered. The process water from the hotwell is then discharged to city sewers. The city Sanitary District treats this water on a toll basis, depending on its BOD content, dissolved solids, and volume. In order to reduce the organic load to the sewer system a method for recovering all of the deodorizer distillates was desired. To do this a Croll-Reynolds Convactor was installed in August, 1957, in the Staley oil refinery in conjunction with a conventional Girdler Semi-Continuous Deodorizer. This paper therefore explains a system for recovering the organic distillates, which were formerly lost in the deodorization process. This system may be divided into two separate operations: the vapor-scrubbing operation, which utilizes a Croll-Reynolds Convactor to remove the organic distillates

from the deodorizer vapors, and the recovery operation, which utilizes a unique process developed by Staley for separating the organic materials from the Convactor condensing-water. For the purpose of simplicity these two operations will be considered separately.

The Vapor-Scrubbing Operation

The vapor scrubbing operation is performed in a Croll-Reynolds Convactor (Figure 1). This consists of a black iron tower, which is divided into four specific sections: a barometric-condenser section, a scrubber-condenser section, a flash chamber, and a water reservoir. The barometric condenser and the flash chamber are joined by a vapor-balance line. This allows flashed vapors to rise from the flash chamber to the barometric condenser for condensation and to discharge from the system *via* the barometric downleg.

The auxiliary equipment required to complete the vapor-scrubber system are a scrubber-condensing-water recirculation pump and a scrubber-condensing-water-level controller. The deodorizer effluent vapors

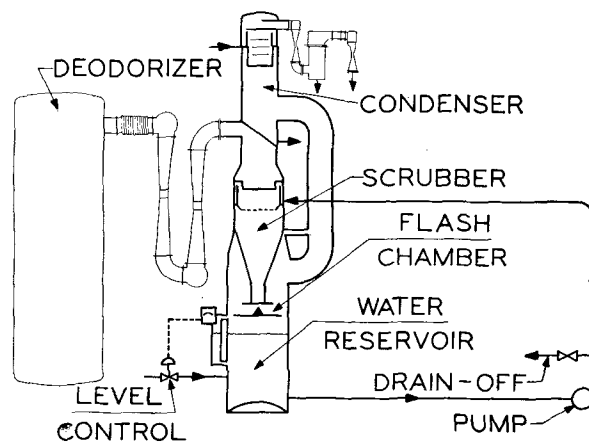


FIG. 1. Croll-Reynolds Convactor.