

PURIFICATION OF LACTIC ACID

by

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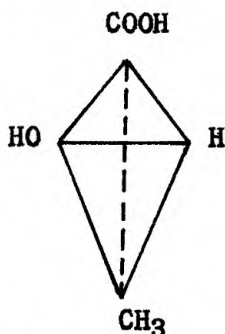
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INTRODUCTION

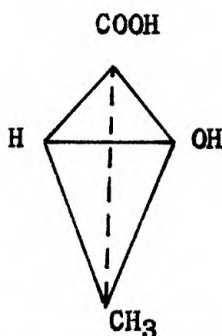
Properties of Lactic Acid

Lactic acid is alphahydroxy propionic acid, $\text{CH}_3 - \underset{\text{H}}{\overset{\text{OH}}{\text{C}}} - \text{COOH}$. Because

of the asymmetry of the alpha carbon atom, this acid exists in two modifications, a dextro acid and a levo acid.



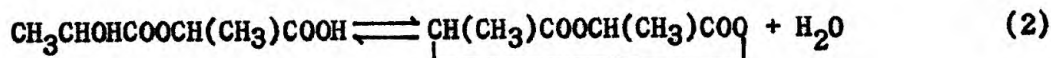
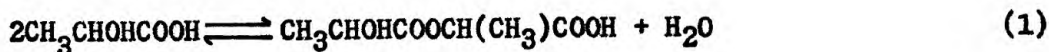
Dextro-Lactic Acid



Levo-Lactic Acid

The commercial acid is a mixture of the two forms, usually in equal proportions; and it is, therefore, inactive with respect to the rotation of the plane of polarized light. Lactic acid is both an alcohol and an acid; and, therefore, its molecules can form esters with one another. In water solutions containing less than 20 percent of lactic acid, the acid is in the simple monomeric form; but solutions of greater concentration contain some esters involving two or more of the simple molecules. According to Watson (23), "when a dilute solution of lactic acid is concentrated, two molecules of lactic acid combine to form one molecule of lactyllactic acid, $\text{CH}_3 \text{CHOH} \cdot \text{COOCH}(\text{CH}_3) \cdot \text{COOH}$ and one molecule of water. When the solution is further concentrated by heating, one molecule of lactyllactic acid loses

a molecule of water to form a lactide.¹¹ Such changes may be represented by the following equations;



It has been determined that lactic acid boils at 260°C under atmospheric pressure, but at this temperature lactic acid undergoes decomposition. It is probable that lactic acid decomposes into ethanol and carbon dioxide as designated by Equation (3).



The high boiling point is due to the formation of an extensive network of hydrogen bonds. Each lactic acid molecule contains at least two active hydrogen atoms and three oxygen donor atoms. Hydrogen bonding with water molecules makes lactic acid difficult to separate from aqueous solutions.

The fermentation process which produced lactic acid also results in the presence of numerous impurities such as dextrans, sugars, proteins, and inorganic salts. These substances are troublesome to remove when purified lactic acid is desired.

Pure 100% lactic acid is a water-white, viscous solution. It is soluble in water and most of the organic solvents. It is very hygroscopic and corrosive. The specific gravity of 85% lactic acid is about 1.206 at 25°C (16), and it is strongly acid to litmus paper.

Lactic acid is one of the oldest known organic acids and perhaps one of the most widely distributed in nature. It is the primary acid constituent of

sour milk (from whence it derives its name), one of the most prevalent organic acids of the soil and a normal constituent in the blood and muscle tissue of animals.

According to the latest price report appearing in Chemical and Engineering News, Oct. 30, 1961, the impure, technical grade lactic acid sells for about 12¢ per pound (equivalent to 23¢ per pound of 85% acid), whereas the U.S.P. 85% pure grade sells for around 85¢ per pound. This clearly indicates that a less expensive procedure for purifying lactic acid is needed to stimulate its production (2).

Preparation of Lactic Acid

Lactic acid may be prepared in a maximum of 50 percent yield from the alkaline degradation of hexose sugars at atmospheric pressure or in greater yields under superimposed pressure, or it may be synthesized from the interaction of acetaldehyde and carbon monoxide at high pressure. However, direct fermentation is the sole commercial method now employed, at least in this country (15).

The culture commonly employed for the conversion of lactose to lactic acid in the fermentation process is a mixed culture of a lactobacillus of the bulgaricus type and a mycoderma. The cultures are incubated at 110°F (43°C) for 24 hours between successive transfers. It is necessary from time to time to neutralize the lactic acid being formed during the fermentation; otherwise, the fermentation would decrease in rate and eventually stop. In most cases, hydrated lime is used as the neutralizing agent. The reasons for this are that: (a) hydrated lime is a comparatively cheap

material to use for the neutralization of the acid during its fermentation; (b) calcium lactate can be purified by recrystallization; and above all, (c) soluble lactate or lactic acid can easily be prepared in a relatively pure condition from calcium lactate by reaction with the proper metal sulfates or sulfuric acid, since calcium sulfate is practically insoluble in water and hence can readily be separated from the product by filtration. The hydrated lime should be added in the form of a slurry and thoroughly mixed with cheese whey by adequate agitation after each addition. Liming should be done every four to six hours, the quantity added being controlled by determinations of acidity, or, better, the pH value. The acidity of whey should be kept between pH 5.0 and 6.0. After about 42 hours the fermentation is completed; i.e. the sugar is practically all fermented. The whey is neutralized with lime to approximately pH 7.0, or 0.1% acid, and heated to the boiling point. The heating is continued until the whey protein is completely coagulated and then discontinued to allow the coagulum to settle (27).

The supernatant liquid is decanted to a storage vat and the sludge filtered in a filter press. For the production of the refined grades of calcium lactate and lactic acid, the solution should be decolorized and further clarified before concentrating. A small percentage of vegetable carbon is added, and the solution is heated to boiling and maintained at this temperature for 15 minutes.

Heating is then discontinued and, with the agitator running, hydrated lime is added slowly until the reaction of the liquid is approximately pH 10.0. Stirring is continued until a sample taken from the vat separates sharply into a grayish precipitate and a clean supernatant liquid. The

batch is then filtered in a filter press, yielding a clean, water-white solution of calcium lactate. Lactic acid, if available, is added to the solution until its reaction is approximately pH 7.0, or its acidity is about 0.05 percent. Sulfuric acid may be used for this purpose; but, if it is used, a filtration is necessary for removal of the calcium sulfate formed in the neutralization.

The neutral calcium lactate solution is evaporated to 15° B \acute{e} . (Sp.gr. 1.115) and pumped to the crystallizers. Cold water is circulated in the jacket of the crystallizer until no further crystallization takes place; this requires 10-12 hours. The wet crystalline mass is shoveled into the basket of the centrifugal filter in which the mother liquor is spun out and the crystals washed several times with small quantities of cold water. The crystals may then be either dried and marketed as technical calcium lactate; or, if a better grade is desired, the crystals can be recrystallized.

According to Seidell (19), 100 grams of H₂O dissolve 3.1 gm. of calcium lactate hydrate $\text{Ca}(\text{CH}_3\text{CHOHCOO})_2 \cdot 5\text{H}_2\text{O}$ at 0°C, 5.4 gms. at 15°C, and 7.9 gms. at 30°C. This is shown in Fig. 1. The solubility data of anhydrous calcium lactate in methanol are shown in Table 1.

In industry, lactic acid is prepared from a solution of calcium lactate as produced in the fermentation or after any of the steps of refining. The solution of calcium lactate used should be of a concentration of approximately 20° B \acute{e} (Sp.gr. 1.160, or approximately 25% lactic acid). If fermentation liquor is used, it should be concentrated to 20° B \acute{e} and kept warm until the sulfuric acid is added. If crystals are used, they should be dissolved in sufficient warm water to give a solution of this density. If solutions of this density are allowed to cool, they are likely to set into

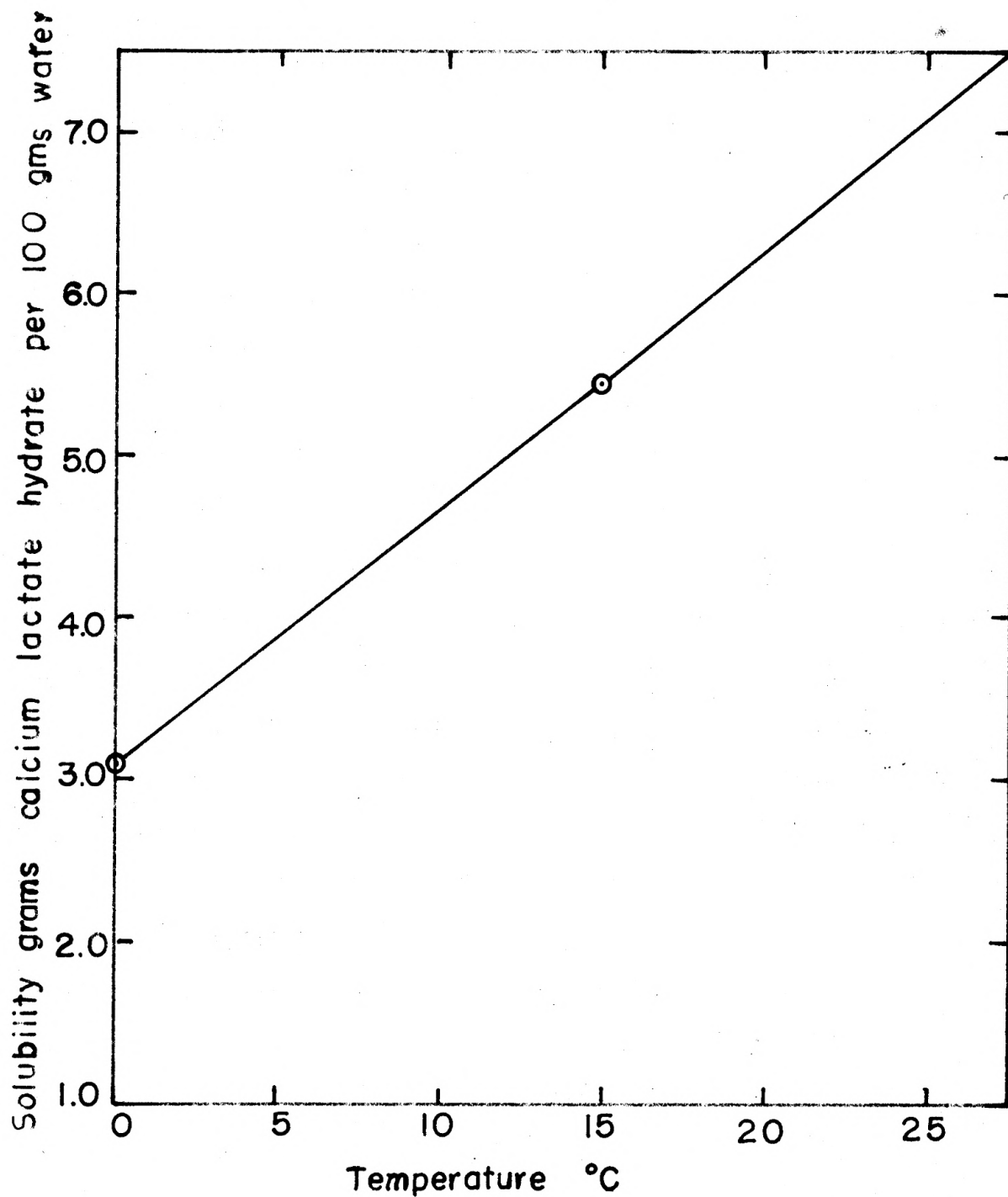


Fig. 1 Solubility of calcium lactate hydrate in water (19).

Table 1. Solubility of calcium lactate in methanol, from Seidell (19).

Temperature °C	:	Grams $\text{Ca}(\text{CH}_3\text{CHOHCOO})_2$ Per 100 gms. CH
15	:	11.0 (13.3)
25	:	20.0
35	:	30.0
45	:	41.0
55	:	52.0
65	:	*56.0
69.8	:	*73.8

*Evidence was determined that above 55°C an alcoholate of the composition $\text{Ca}(\text{CH}_3\text{CHOHCOO})_2 \cdot 5\frac{1}{2} \text{CH}_3\text{OH}$ was formed.

hard masses. Sulfuric acid of 66° Be (Sp.gr. 1.855, concentration 93.2%) is run slowly into the warm, agitated solution of calcium lactate until tests on filtered samples show that equivalence has been reached. When pouring the sulfuric acid into the calcium lactate solution, care must be exercised to keep the temperature of the mixture below 43.5°C (100.3°F) otherwise, anhydrite (CaSO_4) may crystallize and upon cooling it will change to gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) and caking of the whole mass may result. The solubility curves of gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), plaster of paris ($\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$), and anhydrite (CaSO_4) are shown in Fig. 2. The solubility data are listed in Tables 2, 3, and 4. The excess calcium lactate in the mixture can be determined by means of ammonium oxalate solution and excess sulfuric acid by means of barium chloride solution. The calcium sulfate is removed by filtration in a filter press equipped with rubber plates and frames which avoids corrosion difficulties, and washed with water. The washings are kept separate from the filtrate and are used to dissolve calcium lactate in a subsequent operation, or to dilute the lactic acid solution to 22%, if that final concentration is desired. By following the directions given, a solution containing somewhat more than 22% of lactic acid should be obtained. This may either be diluted to 22% or used in making acid of greater concentration, as described below (27).

In making acid of 44% or 50% concentration by the building-up process, crystals of calcium lactate are dissolved in the lactic acid made as described above, sulfuric acid is added as before, and the calcium sulfate is removed by filtration. This should give an acid of 35 to 40% concentration. By one more "building-up" operation, the concentration of the acid can be increased to a percentage greater than 44 or 50 as desired. It may then be reduced to exactly 44 or 50% by addition of water, or preferably the dilute

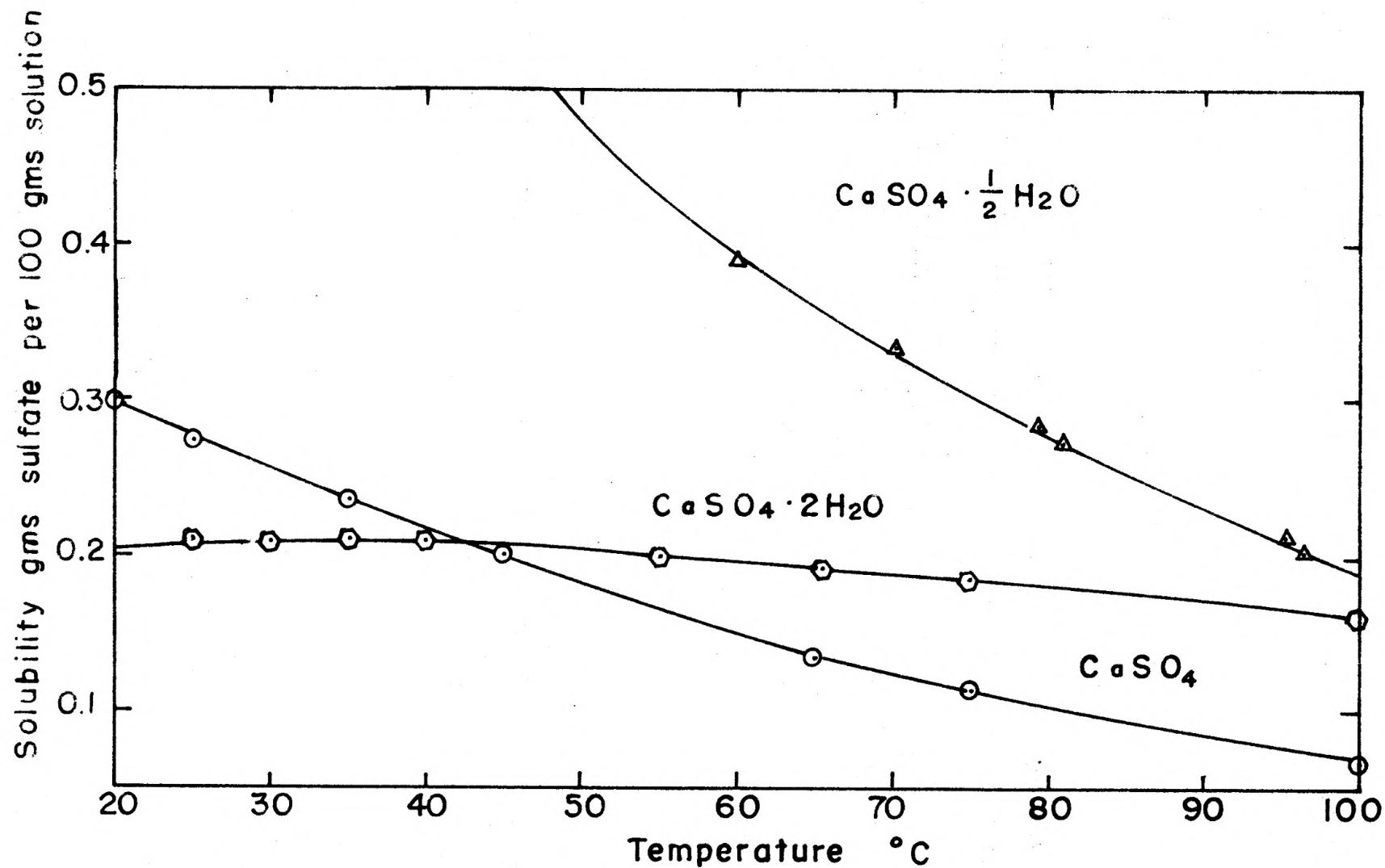


Fig.2 Solubility of calcium sulfates in water(19).

Table 2. Solubility of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ in water, from Seidell(19).

Temperature °C	: Grams CaSO_4 per 100 cc. solution	: Millimols per liter	: Density of solutions
0	0.1759	12.926	1.00197
10	0.1928	14.177	1.00173
18	0.2016	14.817	1.00059
25	0.2080	15.295	0.99911
30	0.2090	15.361	0.99789
35	0.2096	15.405	0.99612
40	0.2097	15.413	0.99439
55	0.2009	14.765	0.98796
65.3	0.1932	14.200	0.98256
75	0.1847	13.575	0.97772
100	0.1619	11.900	-
107	-	11.390	-

Table 3. Solubility of unstable calcium sulfate
hemi-hydrate in water, from Seide11 (19).

Temperature °C	: Grams CaSO ₄ per 100 : gms. sat. sol.	: Solid phase
0.9	0.956	CaSO ₄ $\frac{1}{2}$ H ₂ O
5.2	0.946	"
14.5	0.924	"
21.7	0.811	"
29.3	0.688	"
34.5	0.640	"
42.4	0.555	"
44.8	0.523	"
60.0	0.389	"
70.5	0.336	"
79.5	0.286	"
81.0	0.272	"
95.5	0.213	"
96.5	0.201	"

Table 4. Solubility of calcium sulfate
(anhydrite) in water, from
Seidell (19).

Temperature °C	: Grams CaSO_4 per 100 : gms. sat. sol.
20	0.298
25	0.274
35	0.242
45	0.201
65	0.136
75	0.114
100	0.067

acid obtained by washing the filter cake of calcium sulfate. It should be obvious that it is not necessary to balance the acid and lactate precisely until the last stage of a "building-up" operation. The limiting concentration of lactic acid obtained by the "building-up" process is 76.8%. At this concentration the removal of water by gypsum is just balanced by its addition as calcium lactate hydrate. More highly concentrated lactic acid, such as 85%, is obtained by evaporation of water from approximately 50% acid in an acid resistant vacuum evaporator. Decolorization and removal of heavy metals should precede this evaporation (27). Alternately, an entraining liquid such as benzene may be employed to steam distill out some of the water (23).

Utilization of Lactic Acid

Lactic acid is marketed in four principle grades: technical, containing 22 or 44% dark colored acid; edible, containing 44% light colored or 50% water-white acid; U.S.P. as 85%; and plastic, as acid in concentrations not less than 50%. The technical grade is used mainly in the production of leather to neutralize the lime used in the processing of hides. Edible grades are mainly for food and beverage acid uses. U.S.P. grades are used as a modifier for cow's milk for infant feeding as well as many medical applications. Plastic grades are used in the manufacture of transparent cast phenolic resins, alkyd and acrylic resins as plasticizers and catalysts. A plasticizer is considered to function by neutralizing a part of the Van der Waal's forces or secondary valence bonds which prevent the resin molecules from slipping past one another when the material is stressed. After these forces or bonds

have been partially neutralized or weakened by the plasticizer, the resin is more flexible. Lactic acid can be used as a satisfactory plasticizer except for the fact that it is rather expensive.

According to Needle and Aries (25), "the potential annual market for lactic acid is about 200,000,000 pounds if the price of the purer grades can be reduced significantly.

Purification of Lactic Acid

The principle impurities in crude lactic acid include unfermented sugars, inorganic salts, (particularly calcium salts), heavy metals, such as iron, acetic acid, and similar substances in addition to water. Direct volatilization of lactic acid is difficult because of its low vapor pressure and its sensitivity to heat. The boiling points of lactic acid at various pressures are given in Table 5 and Fig. 3.

Numerous methods have been devised for the purification of lactic acid, but most of these involve chemical steps such as the formation of insoluble salts or volatile esters. These processes require considerable time and some involve expenditure for chemicals as well.

Recrystallization Processes. When converting calcium lactate into lactic acid, the purity of the latter depends primarily on that of the former. In order to obtain a purer calcium lactate the crystals obtained from the first crystallization are dissolved in hot water, the solution treated with activated carbon, filtered, evaporated, and recrystallized. Some of the sugars originally present in the calcium lactate solution are removed in this way (20).

Table 5. Vapor pressure data of lactic acid.*

Pressure	:	Boiling point
1 atm		260°C (decompose)
40 mm Hg		158°C
10.0 mm Hg		125°C
5.0 "		112°C
1.0 "		84°C
0.5 "		72°C
0.1 "		52°C

*Data supplied by Clinton Corn Processing Company, Clinton, Iowa.

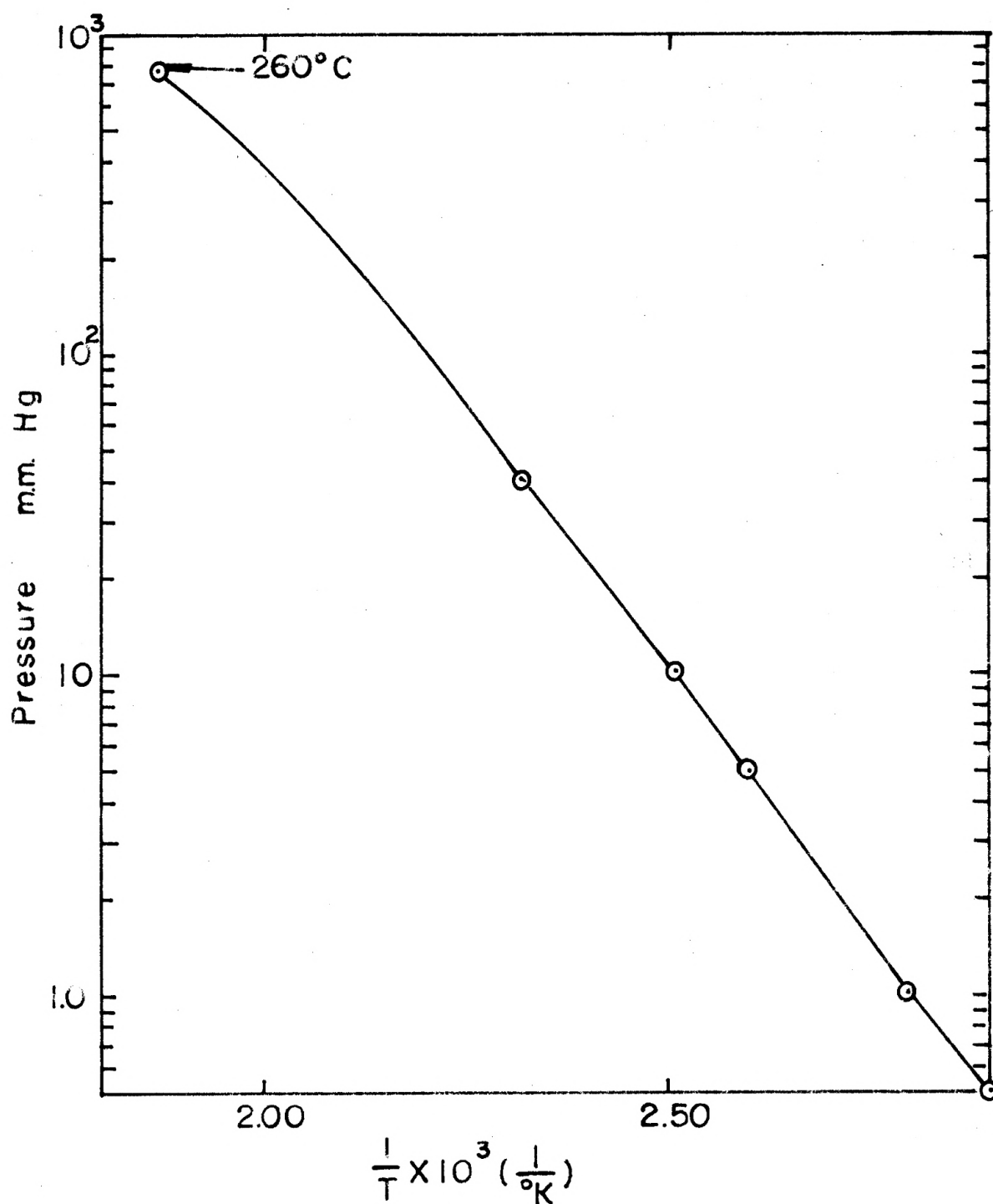


Fig. 3 Vapor pressure of lactic acid

A somewhat purer lactic acid can be made by converting the calcium lactate into the zinc salt by the use of zinc carbonate or sulfate. The zinc lactate is recrystallized and dissolved in water. The zinc is subsequently precipitated with hydrogen sulfide and the zinc sulfide filtered off. The zinc salt is best suited for this operation because it crystallizes better than any other lactate. Both of these recrystallization processes leave traces of impurities in the solutions, however.

Solvent Extraction Processes. Solvent extraction of crude lactic acid obtained by fermentation methods has been extensively studied and used in commercial processes (15). In 1933, Jenemann (10) proposed an extraction method using isopropyl ether to extract lactic acid followed by extraction of the ether solution by distilled water. This method, however, has proved to be hazardous, because the isopropyl ether is inflammable and may form peroxides which are explosive. In 1947, Dietz and Schopmeyer (2) made a study of the recovery of lactic acid as an ester directly from dilute solutions. In the method evolved the dilute lactic acid solution (5 to 6% lactic acid) was esterified with ethanol and the ester extracted with 1, 2-dichloroethane. The esters were recovered from the solvents by distillation. In this process the impurities were not removed completely, particularly those non-volatile impurities which were likely to be extracted with the esters. The most recent study of the liquid-liquid extraction of lactic acid was made by Weiser and Geankoplis (25). They determined the selectivities of various organic solvents and concluded that isoamyl alcohol was most suitable. The solvent was recovered by steam distillation. A steam to solvent ratio of about 7.9 by weight was required. Although the solvent selected was a

superior agent for discriminating against the common impurities, the method does leave traces of them in the product solution.

Precipitation of Heavy Metals. Potassium ferrocyanide is employed to remove the heavy metals present in the lactic acid that result from corrosion of metal tanks. The quantity of potassium ferrocyanide solution to be added is determined by experience or by a laboratory test on a sample of the acid. The quantity should be sufficient to precipitate all the heavy metals present, but an excess should be avoided as far as possible. After the addition of the potassium ferrocyanide solution, a small percentage of activated carbon is added, and the solution heated nearly to boiling. The flocculated mixture of carbon and heavy-metal ferrocyanide is removed by filtration. Organic impurities are not affected, however.

Oxidation of Organic Impurities. A partial purification of lactic acid may be made by subjecting the crude solution of the lactic salts or free acid to a mild oxidation (6)(7). Some of the oxidizable impurities are destroyed by the addition of oxidizing agents. The oxidizing agent is added to the lactic acid in aqueous solution; time is allowed for the agent to react; and the solution is heated to remove the gases formed. Solutions of calcium or sodium hypochlorite, potassium chromate or permanganate, hydrogen peroxide, nitric acid, and chlorine or ozone gas have been used as oxidizing agents (6)(7)(13)(21)(22). Excess reactants and products must be removed or destroyed and the final solution is not sufficiently pure for use in the plastics industry.

Steam Distillation. One of the oldest procedures for the purification of lactic acid is steam distillation under high vacuum (11)(12). This is very expensive, chiefly because of the large steam consumption. The purity of the finished product depends upon the purity of the starting solution and

upon the efficiency of the vapor system in separating entrainment. In such a system the equipment is usually arranged with a partial condenser or dephlegmator which strips the bulk of the distilled acid from the vapors at 50% or higher concentration. Acid not recovered in this fraction is recycled. Most of the non-volatile impurities are left behind, but volatile acids are carried over into the product solution.

Azeotropic Distillation Processes. Watson (23) showed that a highly concentrated lactic acid could be produced by distillation in the presence of liquids such as benzene, toluene, isopropyl ether and carbon tetrachloride, which form minimum constant boiling mixtures with water. The water is readily removed and leaves a residue of more or less anhydrous acid with a total acidity of 100% or more. It is essential that the withdrawing liquid be immiscible with water. It is also desirable that it be immiscible with concentrated lactic acid so that it may be readily recovered. However, the latter requirement is not so important in the case of low boiling liquids which may be easily vaporized from the acid. The total acidity of the concentrated acid can become approximately 105%, calculated as lactic acid. The figure exceeds 100% because of the presence of lactyllactic acid and a small amount of lactide. The newly concentrated acid contains about 50 or 60% of free lactic acid, the remainder consisting chiefly of lactyllactic acid. The main object of this process is to obtain a highly concentrated lactic acid. In the process some of the volatile impurities are removed. However, it does not remove non-volatile impurities.

Esterification Processes. The method of purification involving ester hydrolysis was first proposed by Smith and Claborn (20) and later modified by Weisberg and Stimpson (24), Wenker (26), and Schopmeyer and Arnold (18).

The process consists of converting impure lactic acid into lactic ester by esterification with a primary alcohol such as methyl, ethyl, propyl, or isopropyl, distilling the ester and some water as an azeotrope, hydrolyzing the ester to lactic acid and alcohol with distilled water, and removing the alcohol by fractional distillation. This procedure avoids crystallization and filtration procedures and yields an acid of plastic grade.

In the method proposed by Smith and Claborn (20) in 1939, calcium lactate crystals were dissolved in a sufficient amount of methanol (see Fig. 4) and insoluble materials were removed by filtration. The filtrate was acidified with sufficient sulfuric acid to liberate the lactic acid, forming calcium sulfate, and to catalyze the esterification. The mass was heated at refluxing temperature for 4 to 8 hours to establish the esterification equilibrium and then filtered to remove the precipitated materials. The excess methanol was removed by fractional distillation at atmospheric pressure, and the water and most of the ester were distilled under vacuum at a low temperature. This process was later found to be commercially impractical because the yields were far less than theoretical, due chiefly to the fact that the ester could not be completely separated from the calcium sulfate sludge. Moreover, the process required such an extended time that the cost of the recovered lactic acid was excessive.

In 1942, Weisberg and Stimpson (24) proposed a method which showed some improvement over the one proposed by Smith and Clayborn. It consisted of acidifying a salt of lactic acid with sulfuric acid, reacting the acid with a lower alcohol to form an ester, which was distilled, and a residue containing a portion of the ester. The ester was hydrolyzed in a column under superatmospheric pressure. They found that as the pressure in the column increased

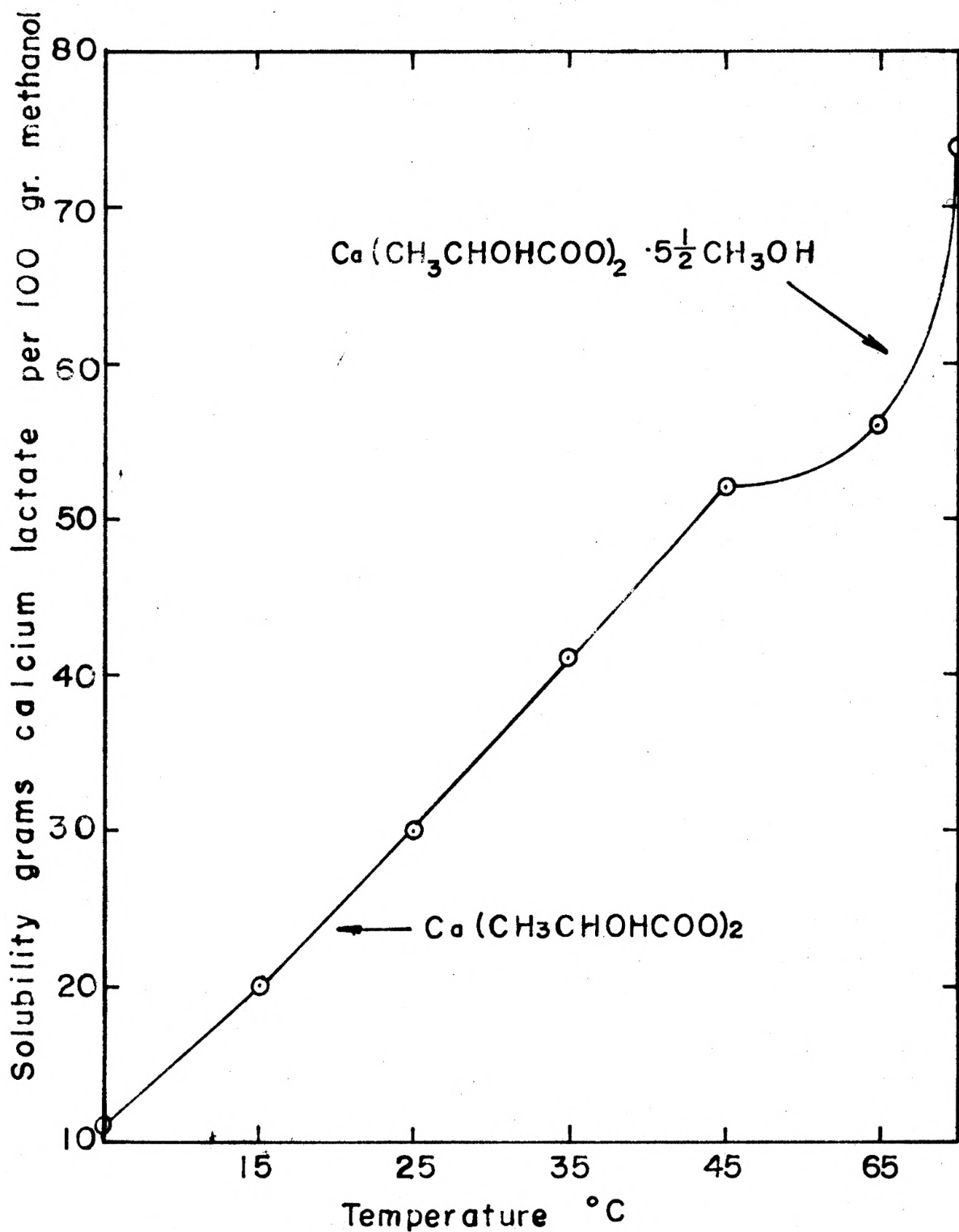


Fig. 4 Solubility of calcium lactate in methanol. (19).

the time required to complete the hydrolysis decreased. Water was added to the residue which combined with the ester to form a constant boiling mixture. This was separated from the residue by distillation.

Later, Wenker (26) proposed a method which used concentrated crude lactic acid (approximately 80 to 85% lactic acid) as a starting material instead of lactic acid salts. The crude lactic acid was esterified with methanol in a tank. A trace of sulfuric acid was added as a catalyst. The vapor containing methyl lactate, methanol and water was run to the bottom of a fractional distilling column where the methanol was separated from the methyl lactate and water. The liquid from the bottom of the column was introduced into a hydrolysis tank containing pure water and a trace of sulfuric acid catalyst. Both reboiler tanks were heated with steam jackets. Hydrolysis of methyl lactate took place in the second tank and the vapor went back to the bottom of the distillation column where the methanol was separated. Part of the methanol vapor was condensed in a partial condenser and returned to the top of the column as reflux. The remainder was condensed in a total condenser for reuse in the esterification tank. A run took 12 to 16 hours after which the sulfuric acid in the hydrolysis tank was treated with calcium lactate and the calcium sulfate precipitate was filtered out.

Another esterification process was proposed by Schopmeyer and Arnold (18). It makes possible the continuous reuse of the methanol. It is continuous, convenient, and easy to control. At the beginning of the process, methanol and 85% lactic acid, together with a small amount of sulfuric acid as catalyst were charged into the esterification kettle. The ingredients of the esterification mixture were approximately in the proportion of 1.5 moles of methanol, 1.0 mole of lactic acid and 0.005 mole of sulfuric acid. This

mixture was refluxed for 30 minutes to 1 hour to permit a considerable amount of the lactic acid ester to form in the esterification kettle. Then the aqueous solution of the lactic acid to be purified, in concentrations of 44% or 22%, was fed continuously in sufficient amount to balance the rate of distillation. The water present in the lactic acid used, together with the water formed by the esterification reaction, was partly vaporized along with the methyl lactate ester and the excess unreacted methyl alcohol. The vapors were introduced into the fractionating column at a point near the middle of the column. The methanol rose in the column and was condensed in the condenser. Part of the condensed methanol returned to the esterification kettle and the rest went back to the column as reflux. At the temperature of the column, the lactic acid ester underwent hydrolysis. The hydrolysis was supposed to be practically complete near the bottom of the column and a purified, aqueous lactic acid was collected in the reboiler.

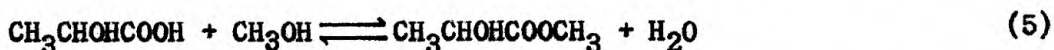
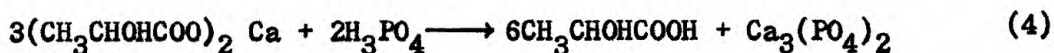
This process required fairly concentrated lactic acid as a raw material, and the esterification reaction was rather slow at the reflux temperature.

Purpose of this Research

In this investigation a method was sought by which it would be possible to produce a pure lactic acid from the dilute solution of calcium lactate produced by fermentation. It was thought desirable to attempt this in a single step to reduce handling problems. It was also hoped that time could be saved by speeding up the esterification and hydrolysis reactions.

DESCRIPTION OF THE PROCESS

This process differs from the conventional ester hydrolysis process in that dilute calcium lactate solution obtained from the fermentation is used as a starting material instead of fairly concentrated crude lactic acid (usually of the strength of about 50%) as required by most of the ester hydrolysis processes. The process involves first converting the calcium lactate to lactic acid, esterifying the acid with methanol, distilling the ester away from nonvolatile impurities, and hydrolyzing the ester to give pure lactic acid. The main reactions involved are:



Orthophosphoric acid instead of sulfuric acid is used to acidify the calcium lactate and an excess is provided to serve as a catalyst in the esterification reaction (5). The substitution of orthophosphoric acid for sulfuric acid has the advantage that the calcium phosphate produced in equation (4) forms soft sludges. If sulfuric acid were used the calcium sulfate formed might set into hard masses of gypsum wherever there was insufficient water for the hydration reaction.

Materials

The materials used in this study were distilled water, methanol, calcium hydroxide, 98% sulfuric acid, 85% orthophosphoric acid, 85% lactic acid, and

44% light technical grade lactic acid. The methanol was reagent grade produced by E. I. duPont and Co. The calcium hydroxide was reagent grade produced by The Matheson Company, 98% sulfuric acid, 85% orthophosphoric acid, and 85% lactic acid were all reagent grades and were purchased from Baker & Adamson. Light technical grade lactic acid (44%) was supplied by The Clinton Corn Processing Company. Because of the hygroscopic and corrosive nature of lactic acid it was handled in glassware and acid-resistant plastic tubing.

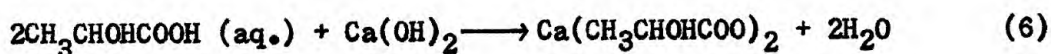
Equipment

Plate I, Appendix, shows the fractional distillation column and the associated equipment used. The column was made of borosilicate glass 3 ft. long and $2\frac{1}{2}$ in. inside diameter. The column was insulated with asbestos pipe covering and packed with $\frac{1}{2}$ in. stoneware Raschig rings. Esterification was carried out in the 3-neck glass flask shown on the right. Heat was supplied by a Bunsen burner directly underneath the esterification flask. Another 3-neck glass flask was placed below the column as a reboiler to receive the product. A specially designed glass distillation head shown in Plate II, Appendix, was placed between the glass condenser and the column. The head regulated the reflux ratio at 3/1, 1/1, or 1/3 as desired. Three thermometers were used, one in the esterification flask, one in the reboiler flask, and one at the reflux head. Connections were made with standard taper ground glass joints. All the tubing used in this study, except those for the condenser water, were made of Teflon.

Experimental Procedure

In this investigation, two methods were tried using the same equipment. The first was the Schopmeyer and Arnold (18) process.

In the second method a dilute solution of calcium lactate was prepared by reacting dilute light technical grade (44%) lactic acid with a slurry of calcium hydroxide. This step was added because insufficient dilute calcium lactate was available from fermentation of whey. The reaction follows.



Orthophosphoric acid (85%) was charged into the reaction flask in slight excess over the amount needed to react with all the calcium lactate. Some distilled water was added to dilute the acid to about 25%. Dilute calcium lactate prepared as described above was introduced into the reaction flask, stirred, and heated to boiling. The acidification reaction follows.



The completion of the acidification reaction (Equation 4) was indicated by the settlement of the calcium phosphate particles. This required about 15 minutes. Methanol was then fed slowly to the esterification flask. The excess of orthophosphoric acid served as an esterification catalyst. Vapors containing methanol, water, and methyl lactate went into the column and were fractionated in the same manner as before. The methanol vapor and a little water vapor rose in the column and passed through a distillation head (Plate II) to a condenser where the vapors were condensed. Part of the condensate went back to the column as reflux - ratios used were 3/1 and 1/1 - and the

rest went back to the esterification flask continuously. As the operation proceeded the concentration of methanol increased and the concentration of lactic acid decreased due to the formation and vaporization of methyl lactate ester. This was indicated by the lowering of the boiling point of the esterification mixture. The methyl lactate vapor which entered the column underwent hydrolysis at the temperature of the column. The hydrolysis was believed to be substantially complete near the bottom of the column and a purified, aqueous lactic acid solution was collected in the reboiler. The contents of the reboiler were boiled throughout the operation and any unhydrolyzed methyl lactate present in the reboiler was probably hydrolyzed at the temperature of the reboiler under the influence of the large amount of water present there. Calcium phosphate, unreacted lactic acid, together with the non-volatile impurities originally present in the crude calcium lactate solution remained in the esterification flask and were removed after the process was completed.

Analytical Methods

Three standard tests were made on the product. These were free acidity, total acidity, and ash content. The free and total acidity determination of the residue remaining in the esterification flask was also made in order to give a lactic acid balance.

For the free and total acidity determination, the titrimetric method proposed by Fetzer and Jones (6) was employed. For each determination, an appropriate quantity of acid was placed in a tared weighing bottle and reweighed to obtain the weight of the sample. The acid was then transferred

to a 500 ml. Erlenmeyer flask using 75 to 100 ml. of distilled water. To this 100 ml. more of distilled water and 9 drops of phenolphthalein indicator were added and the whole thing was mixed thoroughly. The sample was titrated to a faint pink color with approximately 0.5 N sodium hydroxide. The alkali was added slowly and the flask was shaken vigorously to avoid overneutralization. The reading was recorded as free acid. Then the standard alkali was added rapidly until the 50.00 ml. reading was reached. The flask was shaken vigorously and then set aside for 20 to 30 minutes. Next it was back-titrated with approximately 0.5 N Standard acid to a faint pink color. The reading was recorded.

Since the sodium hydroxide and sulfuric acid used in this investigation were 0.4809 N and 0.5049 N respectively, the calculations were;

$$\frac{(\text{ml. of } 0.4809 \text{ N alkali})(0.04332)}{(\text{Sample weight})}(100) = \% \text{ free lactic acid}$$

$$\frac{(47.62 - \text{ml. of } 0.5049 \text{ N standard acid})(0.04548)}{(\text{Sample weight})}(100) = \% \text{ total lactic acid}$$

For the ash content determination 1 to 2 gms. of sample were accurately weighed into a porcelain crucible. The crucible was then heated in a muffle furnace for 4 to 5 hours. The temperature of the furnace should not exceed 550°F. The crucible was weighed again when it was cooled.

$$\frac{(\text{weight of crucible} + \text{ash} - \text{tare weight of crucible})}{(\text{weight of crucible} + \text{sample} - \text{tare weight of crucible})}(100) = \% \text{ ash content}$$

EXPERIMENTAL RESULTS

A total of 23 runs was made in this investigation - 11 runs by the method of Schopmeyer and Arnold (18), 12 runs by the new method. These are tabulated in Tables 6 and 7. The Schopmeyer and Arnold method was regarded as a basis for comparison.

Material Losses

The over-all material balances of the experimental runs were quite satisfactory for this scale of operations. With one exception the losses ranged from 0.57% to 17.10%. The loss in run No. 6 was 27.58% because vapors escaped through the stirrer seal. In view of the fact that methanol has the highest vapor pressure of any of the constituents methanol probably accounts for most of the lost material. The material hold-up in the packed distillation column was difficult to determine. Over a number of runs such errors probably balanced out, but the first run in each series had high losses which were probably due to incomplete recovery of the hold-up material.

Yields

The material balances showed that considerable lactic acid remained in the reaction flask. Conversions of purified lactic acid ranged from 15.6% to 73.1%. Reasons for the low yields are discussed below.

Table 6. Runs with sulfuric acid.

Feed									Product												
Run No.	time hours	temp. °C	Reflux ratio	Ash %	H ₂ SO ₄ gms.	85% lactic acid gms.	44% lactic acid gms.	Total pure lactic acid gms.	Total pure water gms.	Methanol gms.	Total gms.	Ash %	Pure lactic acid gms.	Lactic acid distilled %	Residue gms.	Reboiler gms.	Holdup gms.	Total gms.	Loss gms.	% Loss	Remarks
1	4.75	100.0-86	3/1	0.78	2.0	424	510.0	584.7	349.3	192.0	1128.0	1.78	234.0	40.0	526.5	465.0	31.1	1022.6	100.4	8.9	
2	6.08	97.0-89	3/1	0.78	1.0	212	377.5	346.2	243.3	101.2	691.7	3.52	151.5	43.7	220.8	396.2	30.0	647.0	44.7	6.46	
3	5.0	98.0-88	3/1	0.78	1.0	212	431.0	369.5	273.5	101.2	745.2	2.53	147.5	40.0	225.2	483.2	27.1	735.5	9.7	1.3	
4	5.5	96.0-87	3/1	0.78	1.0	212	546.9	421.2	337.7	101.2	861.1	2.12	178.2	42.3	245.9	558.3	-	804.2	56.9	6.6	Feed line tubing broke.
5	6.25	99.0-83	3/1	0.78	1.0	212	522.0	410.0	324.0	101.2	836.2	0.68	172.6	42.2	211.5	566.5	37.4	815.4	21.2	2.53	
6	6.88	95.5-90	3/1	0.78	1.0	212	544.3	419.7	336.6	101.2	858.5	0.34	306.6	73.1	216.0	380.7	25.0	621.7	236.8	27.6	Stirrer seal leak.
7	4.75	100.0-87	1/1	0.78	1.0	212	540.0	417.7	334.3	202.4	955.4	0.38	105.8	25.2	313.5	464.5	28.0	806.0	149.4	15.6	
8	6.5	90.5-81	1/1	0.78	1.0	212	529.0	412.7	328.3	202.4	944.4	0.26	223.7	54.2	365.0	500.0	30.0	895.0	49.4	5.23	
22% lactic																					
9	6.5	91.0-84	1/1	0.78	1.0	212	500.0	290.2	421.8	202.4	915.4	0.23	133.6	46.0	279.0	481.0	35.0	795.0	120.4	13.15	
10	7.0	98.0-86	3/1	0.78	1.0	212	700.0	334.2	577.8	128.0	1041.0	0.265	177.2	53.2	418.0	547.0	10.0	975.0	56.0	6.34	
11	5.5	99.0-82	3/1	0.78	-	212	700.0	334.2	577.8	128.0	1040.0	0.24	98.5	29.5	315.0	615.5	30.0	960.5	77.5	7.46	

Table 7. Runs with phosphoric acid.

Feed									Product														
Run No.	Run time : hours	Reaction flash : temp. : °C	Reflux : ratio	Ash : %	Excess : H_3PO_4 : gms.	85% H_3PO_4 : gms.	$Ca(OH)_2$: gms.	Methanol : gms.	44% lactic acid : gms.	Pure lactic acid : gms.	Pure HOH : gms.	Water : added : gms.	Total : gms.	Ash : %	Pure lactic acid : gms.	Lactic acid : distilled : %	Residue : gms.	Reboiler : gms.	Holdup : gms.	Methanol : and water : gms.	Total : gms.	Loss : gms.	Loss : %
12	1.42	110.0-90.0	3/1	0.78	1.0	78.0	74.1	128.0	410.0	180.4	229.6	-	690.1	0.405	35.8	19.9	217.0	314.5	40.0	-	571.5	118.6	17.10
13	5.0	95.0-92.0	3/1	0.78	1.3	40.0	37.0	100.0	205.0	90.2	114.8	400.0	782.0	0.25	30.5	33.75	307.5	303.0	35.0	105.0	750.5	31.5	4.02
14	4.0	94.5-86.0	3/1	0.78	1.3	40.0	37.0	100.0	205.0	90.2	114.8	400.0	782.0	0.28	22.7	25.15	226.5	405.0	30.0	87.0	748.5	33.5	4.29
15	4.0	87.5-84.5	3/1	0.78	1.3	40.0	37.0	200.0	205.0	90.2	114.8	400.0	882.0	0.21	20.0	22.20	365.5	337.0	30.0	117.5	850.0	32.0	3.62
16	4.0	83.0-80.0	3/1	0.78	1.3	40.0	37.0	300.0	205.0	90.2	114.8	300.0	882.0	0.12	17.1	18.92	410.0	285.0	5.0	168.0	868.0	14.0	1.64
17	4.0	82.0-80.0	1/1	0.78	5.58	45.0	37.0	300.0	205.0	90.2	114.8	300.0	887.0	0.11	16.9	18.72	440.0	259.0	25.0	141.5	865.5	21.5	2.42
18	4.0	84.0-78.0	1/1	0.78	9.8	50.0	37.0	300.0	205.0	90.2	114.8	300.0	892.0	0.092	18.9	20.90	424.0	255.0	45.0	102.0	826.0	66.0	7.42
19	4.0	83.0-78.5	3/1	0.78	9.8	50.0	37.0	300.0	205.0	90.2	114.8	200.0	792.0	0.11	19.0	21.10	371.5	280.0	35.0	101.0	787.5	4.5	0.57
20	7.0	83.0-73.0	1/1	0.78	9.8	50.0	37.0	300.0	205.0	90.2	114.8	300.0	892.0	0.092	22.6	25.10	327.0	301.0	38.0	166.0	832.0	60.0	6.75
21	4.0	78.0-70.0	1/1	0.78	9.8	50.0	37.0	300.0	205.0	90.2	114.8	-	592.0	0.082	14.1	15.60	210.0	175.0	35.0	132.0	552.0	40.0	6.76
22	4.0	95.0-82.0	1/1	0.78	9.8	50.0	37.0	100.0	205.0	90.2	114.8	200.0	592.0	0.11	21.1	23.30	151.0	324.0	25.0	62.0	562.5	29.5	5.00
23	7.0	94.0-82.5	3/1	0.78	9.8	50.0	37.0	100.0	205.0	90.2	114.8	400.0	792.0	0.197	36.8	40.75	295.0	389.5	35.0	65.0	784.5	7.5	0.94

Effect of Feed Composition

The feed for the improved method consisted of crude calcium lactate solution, methanol, water, and orthophosphoric acid. The amount of orthophosphoric acid used was in excess of that theoretically needed to acidify the calcium lactate. The excess orthophosphoric acid served as an esterification catalyst. It was found in this investigation that the esterification rate increased as the amount of water present in the esterification flask decreased in accordance with the law of mass action. In runs 9, 10, and 11 crude lactic acid of 22% strength was used instead of 44%. When comparing run 8 with run 9, other conditions being equal, the yield dropped from 54.2% in run 8 to 46.0% in run 9. This indicates that the yield increases as the concentration of the lactic acid in the feed increases. It is clear that the amount of methanol used to esterify the acid should be in excess of that theoretically required. Unfortunately, it has been found that too much methanol reduced the boiling point of the esterification mixture considerably and this in turn slowed down the reaction rate. The esterification reaction was far from complete in all of the experimental runs.

Effect of Time of Run

Probably the effect of the length of time of run was one of the most significant variables in this operation. It was found that the yield of lactic acid increased as the length of time of run increased. One run of 1 hour and 25 minutes duration showed 19.9% yield. Most of the runs ran between 4 hours

and 7 hours and showed higher yields. Obviously, under those conditions, the esterification of lactic acid is a slow process.

Effect of Concentration of Catalyst

Filachione and Fisher (4) reported that volatilization of lactic acid was facilitated by the use of considerable excess of catalyst. However, it was found in this investigation that the concentration of catalyst was not significant. Evidently enough sulfuric or phosphoric acid catalyst was present in all runs except run 11. In run 11, no catalyst was used and the yield (29.50%) was low compared with other runs. This showed that while the presence of the catalyst promotes the esterification rate, the esterification reaction will proceed at a slower rate without a catalyst.

Effect of Temperature

It appeared that the rate of esterification increased at a higher esterification temperature. In runs 14, 15, and 16 the esterification temperature ranges were 94.5°C-86°C, 87.5°C-84.5°C, and 83°C-80°C, and the yields were 25.15%, 22.20% and 18.92% respectively. In run 21 the yield (15.60%) was the lowest and the esterification temperature range (78°C-70°C) was also the lowest of all the experimental runs.

Purity of the Products

The ash content of the lactic acid products was taken as an indication of its purity. The ash content of the products obtained from the first four

runs as listed in Table 6 was higher than the ash content of the crude lactic acid, i.e. 0.78%. This resulted from washing out the impurities originally present in the packed column. As the column became cleaner after each run the ash content of the products became closer to their real values. According to Table 6 and Table 7, the ash content of products obtained from run 5 on was much less than the ash content of the crude lactic acid. This indicates that the products obtained were purer than the feed. Another fact which showed that the products were of higher purity than the feeds was that the color of the products was much lighter than the color of the crude light technical grade lactic acid used as feed.

DISCUSSION OF RESULTS

The principal difficulty with the experimental runs centered around the unsatisfactory yields of converted lactic acid despite runs up to 7 hours in length. Had the lactic acid been converted completely to the ester it would have been distilled rapidly from the reaction flask. A substantial fraction of the lactic acid remained unreacted.

It is clear that the reaction rate was low in the temperature range used (78° to 100°C). Raising the temperature would improve the rate. The rough rule for chemical reactions is that raising the temperature 10°C approximately doubles the rate.

Another factor accounts in part for the incomplete reaction. The reaction equilibrium is probably incomplete. The second series of runs reported in Table 7 contained relatively more water and the yields of converted lactic acid were generally lower than the first series reported in Table 6. Since

water is a product of the esterification reaction, its removal would shift the equilibrium toward more complete esterification of the lactic acid. As a matter of fact, water removal was attempted in some early runs by using a high reflux ratio at the top of the distillation column thus returning less water with the distilled methanol. The trouble was that the reaction flask became pasty as all liquid except the returning methanol was distilled off. Methanol has a low boiling point (64.7°C); so it vaporized rapidly from the hot reaction flask.

The requirements for rapid and complete esterification of lactic acid are to raise the temperature in the reaction vessel, to distill out the water as completely as possible, to provide for thorough contact of the reaction mixture with recycled methanol before it is distilled out at the high reaction temperature, and to arrange that the reaction mixture be kept fluid.

These requirements suggest that a high boiling liquid immiscible with water be added to the reaction. Toluene, which boils at 110.7°C should be satisfactory. It will also be necessary to introduce recycled methanol into the reaction system in a manner designed to insure good contact.

RECOMMENDATIONS

In view of the problems encountered in this investigation, some changes should be made in order to make the process feasible. The following is a new process resulting from the experience gained.

Plate III, Appendix, shows diagrammatically the arrangement of the suggested process. It consists of an esterification reaction system - flasks A and B, one fractional distillation column C for methanol recovery, and a

solvent recovery system consisting of decanter F and distillation columns I and G. Condensers are provided at D and K, and reboilers are included at E, H, and J for the distillation columns.

At the start of a run orthophosphoric acid, methanol, and toluene will be charged into the reaction flasks A and B. The mixture will be stirred and heated until methanol starts to distill into the column C. Then crude dilute calcium lactate solution will be fed continuously to the reaction system. Since toluene and water form a binary constant boiling mixture (B.P. 84°C) they will distill into column C and appear in reboiler E. Next this reboiler can be brought to a boil, and the methanol fraction will appear at the condenser D where part of this stream will be refluxed to column C and part of it returned to reaction flask A. Before methanol can escape the reaction system it must pass into flask B allowing sufficient contact time for esterification to occur. Calcium phosphate will precipitate, and the mixing should be vigorous enough to keep the solid in suspension. The excess phosphoric acid will serve as the esterification catalyst. Enough toluene should be present and enough heat provided at flask B to distill water rapidly into the column C. The boiling temperature in flask B should be only slightly below the boiling point of the solvent-water constant-boiling-mixture, the amount of the depression being due to the quantity of methanol present.

As the two liquid layers accumulate in reboiler E, they will overflow into the decanter F; and when liquid appears in reboilers J and K these will be heated to drive solvent-water constant-boiling-mixture to condenser K. Toluene solvent in flask H will be returned to the reaction flask B to provide for the continuous removal of water.

Enough methanol should be returned to flask A to provide for rapid esterification of the liberated lactic acid. Enough toluene solvent should be present in A and B to provide for rapid removal of water, and an excess of phosphoric acid must be maintained until the last of the dilute calcium lactate has been fed in.

The methyl lactate distilled into column C should be hydrolyzed by the time it reaches flask J even though the operation is expected to proceed much more rapidly than in the past. The contents of flask J should contain the pure lactic acid and the water charged in the form of dilute solution. If desired, its water content may be reduced by introducing some solvent toluene and permitting the solvent-water constant-boiling-mixture to distill out of column I to condenser K. During this last operation the line from the decanter F to the column I should be shut off.

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LITERATURE CITED

- (1) Burton, L. V.
Byproducts from milk. Food Industries, 9, 571, (1937).
- (2) Dietz, A. A., Ed. F. Degering, and H. H. Schopmeyer.
Recovery of lactic acid from dilute solutions. Ind. Eng. Chem.,
39, 82, (1947).
- (3) Fetzer, W. R., and R. C. Jones.
Determination of free and total acidity in commercial lactic acid.
Analytical Chemistry, 24, 835, (1952).
- (4) Filachione, E. M., and C. H. Fisher.
Purification of lactic acid. Ind. Eng. Chem., 38, 228, (1946).
- (5) Groggins, P. H.
"Unit Processes in Organic Synthesis," 5th. ed., p. 701, McGraw-Hill
Book Company, Inc., New York, (1958).
- (6) Haag, I. L.
Purification of lactic acid. U. S. Patent, 2,013,096, (1935).
- (7) Haag, I. L., and C. A. Vana.
Purification of lactic acid. U. S. Patent, 2,101,998, (1937).
- (8) Hamburger, Toni.
Process of purifying lactic acid. U. S. Patent, 1,459,395, (1923).
- (9) Horsley, L. H.
"Azeotropic Data," p. 252, American Chemical Society, Washington,
D. C., (1952).
- (10) Jenemann, J. A.
Lactic acid. U. S. Patent, 1,906,068, (1933).
- (11) Lawrie, J. W.
Purification of lactic acid. U. S. Patent, 1,594,843, (1926).
- (12) Macallum, A. D.
Purification of lactic acid. U. S. Patent, 2,013,104 (1935).
- (13) Macallum, A. D.
Purification of lactic acid. U. S. Patent, 2,125,383 (1938).
- (14) Montgomery, Rex., and R. A. Ronca.
Chemical production of lactic acid and other acids from molasses.
Ind. Eng. Chem., 45, 1136, (1953).

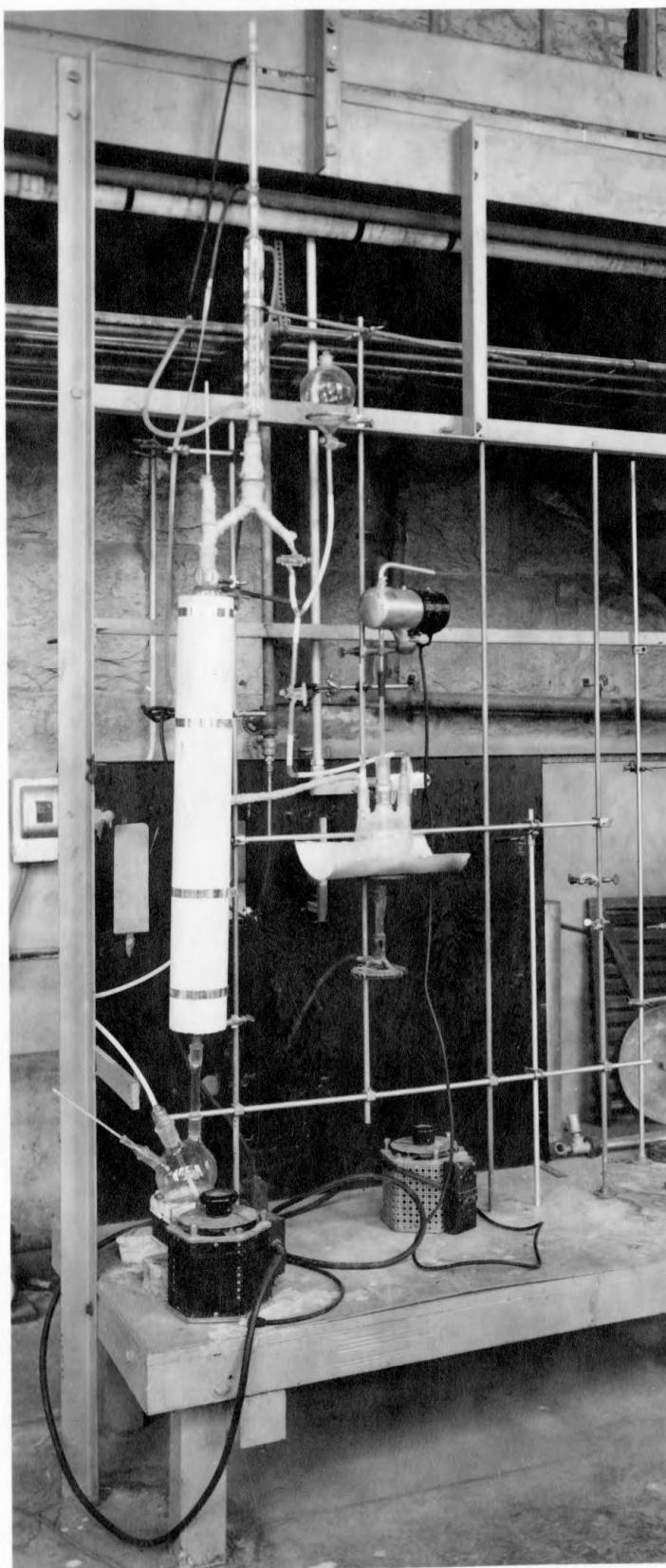
- (15) Peckham, G. T., Jr.
The commercial manufacture of lactic acid. Chem. Eng. News, 22,
440, (1944).
- (16) "Pharmacopaeia of the United States of America," 12th revision, p. 23,
(1943).
- (17) Schatzkes, J.
Purification of lactic acid. Brit. Pat., 173,479, (1921).
- (18) Schopmeyer, H. H., et.al.
Lactic acid purification. U. S. Patent, 2,350,370, (1944).
- (19) Seidell, Atherton.
"Solubilities of Inorganic and metal organic compounds," 3rd Ed.,
Vol. I, 248, D. Van Nostrand Co., Inc., New York, (1940).
- (20) Smith, L. T. and H. V. Claborn.
The production of pure lactic acid. Ind. Eng. Chem. News Ed., 17,
641, (1939).
- (21) Urbain, O. M.
Process for producing lactic acid. U. S. Patent, 2,009,753, (1935).
- (22) Vana, C. A.
Deodorization of lactates. U. S. Patent, 2,013,082, (1935).
- (23) Watson, P. D.
Composition of lactic acid. Ind. Eng. Chem., 32, 399, (1940).
- (24) Heisberg, S. M. and E. G. Stimpson.
Preparation of lactic acid. U. S. Patent, 2,290,926, (1942).
- (25) Weiser, R. B. and C. J. Geankoplis.
Lactic acid purification by extraction. Ind. Eng. Chem., 47, 858,
(1955).
- (26) Wenker, H.
Purifying hydroxy aliphatic acids. U. S. Patent, 2,334, 524, (1943).
- (27) Whittier and Webb.
"By products from milk," p. 39-41, Reinhold Publishing Company,
New York, (1950).

APPENDIX

EXPLANATION OF PLATE I

Photograph of the Equipment

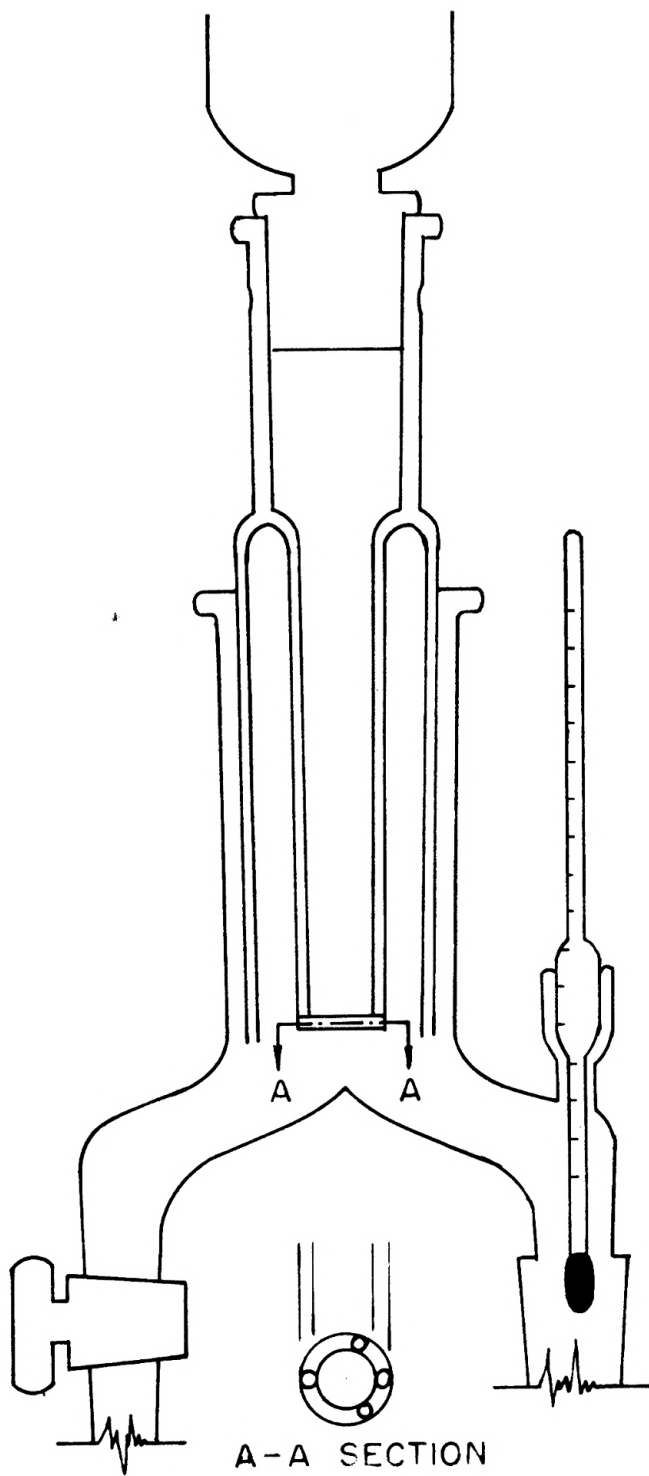
PLATE I



EXPLANATION OF PLATE II

Detail of the Distillation Head

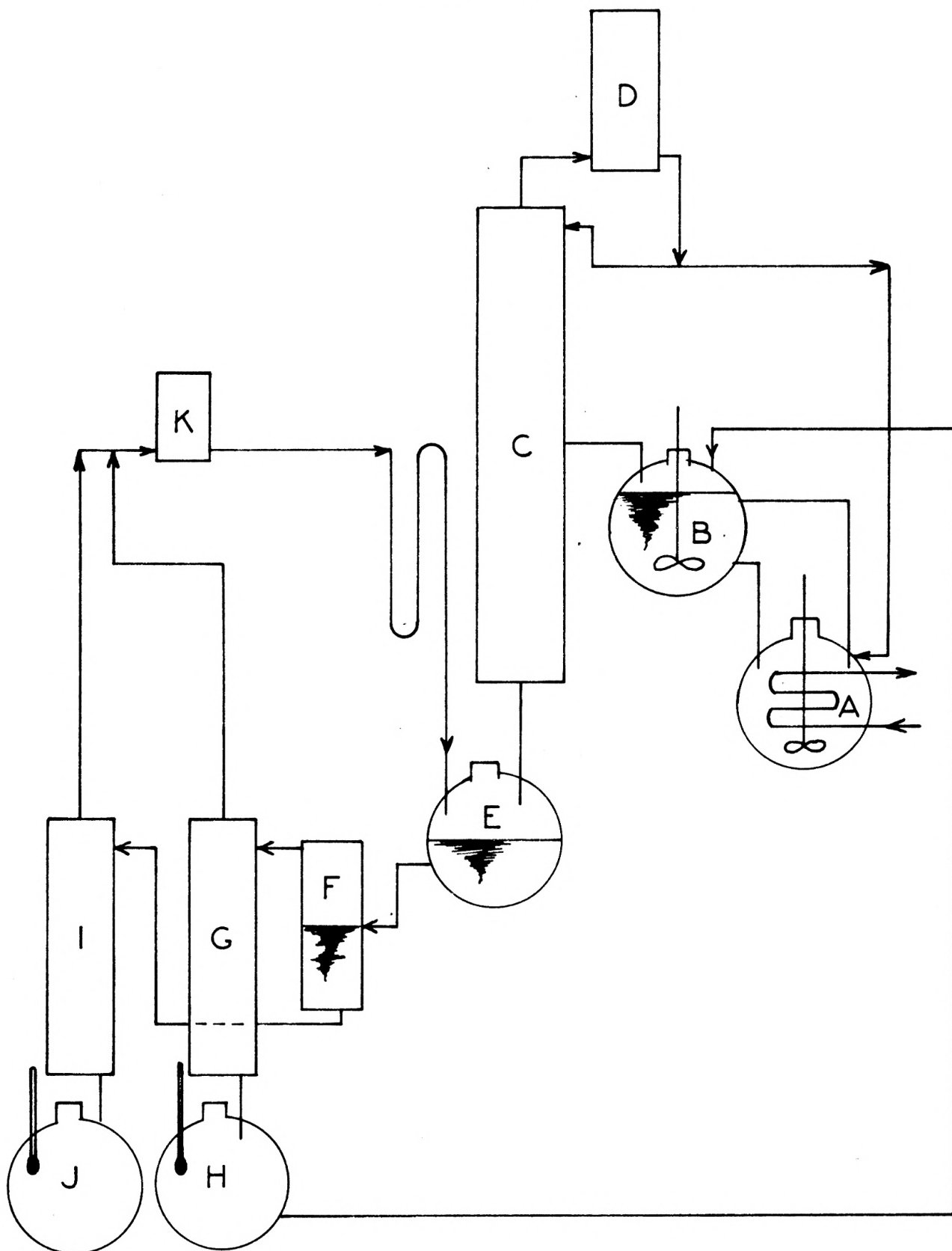
PLATE II



EXPLANATION OF PLATE III

Recommended Changes in the Equipment

PLATE III



PURIFICATION OF LACTIC ACID

by

SIDNEY HSIN-HUAI CHOW

B. S., Taiwan Provincial Cheng Kung University, 1957

AN ABSTRACT OF
A MASTER'S THESIS

submitted in partial fulfillment of the
requirements for the degree

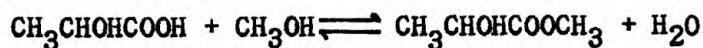
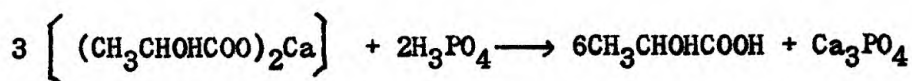
MASTER OF SCIENCE

Department of Chemical Engineering

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1962

Lactic acid has long been known for its complex characteristics. The difficulties involved in the purification of the acid have been studied extensively, and many purification processes have been proposed. However, the price of highly purified lactic acid is still too high to meet the growing demands of the market, particularly for use in the plastic field. It was, therefore, the purpose of this investigation to find a new method whereby purified lactic acid could be produced directly from the crude calcium lactate solution obtained by fermentation of milk. The method developed comprises first acidifying the calcium lactate solution with orthophosphoric acid and esterifying the lactic acid thus obtained with excess methanol using orthophosphoric acid as the catalyst. The methyl lactate in vapor form along with methanol and water vapor are carried into a packed distillation column where the vapors are fractionated and methyl lactate is hydrolyzed. The main reactions involved are:



This process can be made continuous, and treatments such as filtration, crystallization, and addition of oxidizing agents required by older purification methods can be avoided. It is hoped that the simplicity of this process will reduce the cost and stimulate production of the plastic grade lactic acid.