cessfully by the armed forces. The condition which favored such use was high solution concentration.

No general improvement in detergency was attained by addition of the excellent caking inhibitor, bentonite, to the sulfate-built detergent.

Acknowledgment

The assistance of Earl L. Brown and others in the preparation of the experimental data is gratefully acknowledged.

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Improvement of Keeping Quality of Edible Fats by Some Commercially Available Tannins*†

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AS revealed by the number of publications in recent years, a great deal of effort has been expended in research work toward the development of suitable antioxidants and synergistic combinations to be added to fats and oils. Several years ago the study of tannins for the purpose of improving the keeping quality of fats was begun in this laboratory. Tannins were selected for investigation because of their wide occurrence in foods (1) and because their chemical structures suggested antioxidant properties.

A search of the literature at that time revealed little or no data on tannins as antioxidants. Lea (2) has two notes of some interest in his book "Rancidity in Edible Fats." On page 167 he states "substances insoluble in fat-solvents or decomposed by alkali, e.g., proteins, tannins, glucosides or carbohydrates may, perhaps, also assist in protecting fats in the tissue." On page 171 the statement is made that "Chevreul (1856) was familiar with the fact that oak and certain other woods delayed the drying of linseed oil and that American Indians are said to have used the bark of trees for retarding the development of rancidity of bear grease."

Moureu and Dufraisse (3) included tannic acid in a group of substances which tended to retard oxidation changes of another group of organic substances which were relatively easily oxidized and which included fats.

Olcott and Matill (4) listed tannic acid with the "inactive compounds" when tested on crude esters of hydrogenated cottonseed oil. A footnote explained that some of the substances in this "inactive list," when added at levels of 0.02% and 0.1%, exhibited slight antioxygenic activity. Some of the substances in this table were specifically mentioned as possessing slight antioxidant effect, but tannic acid was not included.

Von H. Schönfeld (5) lists concentrated tannin as an agent for removing mucilage from fats. Later Musher (6) claims the use of gallotannin in treatment of paper so as to retard rancidity. Musher (7) and Verbeek (8) in patents which were pending at the same time presented data showing that tannic acid can be added directly to fats to retard the development of rancidity. Musher showed that 50 P.P.M. of tannic acid, when added to lard, increases the keeping quality from two hours by the active oxygen method to six hours. Verbeek showed that when 50 P.P.M. of tannic acid was suspended in oleo oil, the keeping quality was increased from eight hours to 15. Musher was primarily concerned with the enhanced effect resulting from heating the tannin and fat to a temperature "over 250°F." Verbeek was primarily concerned with the improvement of the keeping quality of fats by a treatment with tannins in which the tannins and a tannin compound were removed, as by filtration.

This paper presents data on the process of improving the quality of fats and consists of bringing into contact with relatively dry fat (containing less than 0.2% moisture) a dry tannin material (usually containing less than 12% moisture) and then removing the tannin and tannin complex by filtration. It also shows the effect of varying the temperature of treatment and how this variable is related to Musher's patent.

Experimental

A—Methods

(1) Standardized Procedure for Treatment of Fats With Tannin: Three hundred grams of fat are weighed into a 500-ml. wide-mouth Erlenmeyer flask and heated on an electric hot plate to 160°F. while being stirred with an electric agitator. 0.300 ± .001 gm. of the tannin is added and the stirring continued for 14 minutes. The temperature is held at 160°F. ± 5°F. throughout the process. 0.5 gram of Dicalite Speedplus (commercial grade of diatomaceous earth) is added and stirring is continued for one more minute. The fat is filtered through a Whatman No. 4 filter paper. The tannin and Speed-

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[*] A paper presented at the A.O.C.S. Fall Convention at Chicago, Nov. 7-9, 1945.

[†] Part of the data were presented at the Conference on Problems Related to Fat Deterioration in Foods under the auspices of Committee of Food Research, Research and Development Branch, Military Training Division, Office of The Quartermaster General in Washington in June 1945.

plus is transferred with the oil to the filter paper. The first 50 ml. which filters through is discarded. The control is heated to a temperature of 160°F. and Speedplus is added in the ratio of 0.5 gm. to 300 gm. of fat. The fat is then filtered through Whatman No. 4 filter paper. The first 50 ml. which filters through is discarded.

(2) Ferric Chloride Test: Place 80 grams of melted fat in a 4-oz. oil bottle. Add 1 ml. of 1% ferric chloride solution. Shake well so as to extract water soluble material from fat. The presence of bluish-green or black colored water droplets shows a positive reaction. A blank is run for comparison. About 5 P.P.M. of tannic acid will give a faintly positive test.

(3) The A.O.M. (Active Oxygen Test): The method described by King, Roschen, and Irwin (9) was used.

(4) Color: The color was read, using a Lovibond Tintometer and a 5¼-inch column. The first figure is for yellow and the second for red.

(5) Copper Determination: Extract by refluxing for 30 minutes a suitable sample (approximately 50 grams) with 50 ml. of 1:1 hydrochloric acid. Pour the mixture on a wet, hot filter paper and wash flask and paper several times with hot, distilled water. Evaporate the filtrate to approximately 5 ml. and add 5 ml. of bromine water. Boil off the excess bromine. Dilute to 50 ml. In a 50-ml. Nessler tube place a suitable aliquot, add 5 ml. of 1 to 5 ammonia hydroxide solution and 5 ml. of copper reagent (0.1% sodium diethyl dithiocarbonate. Prepare suitable standards containing .005 to .05 mg. of copper. Compare unknown with nearest standard.

(6) Preparation of Copper and Iron Stearate: In the preparation of fresh cupric hydroxide, ammonia was added to aqueous copper acetate until a deep violet solution was formed and then more cupric acetate was added until a heavy precipitate formed and an excess of ammonia was not present. The precipitate was filtered with suction and dried overnight in a 105°C. oven; 50% excess of the theoretical amount of cupric hydroxide was added to melted stearic acid. The liquid mixture was heated at 140°F. with stirring for five minutes. While the solution was still hot, just enough chloroform was added to bring about solution. The hot solution was filtered through a fast paper and the flask and paper were washed with hot chloroform. The chloroform was driven off the filtrate by heating on a steam bath.

The iron stearate was prepared in a similar manner from ferric chloride. The ferric hydroxide was washed with distilled water to remove the chloride ion. The ferric and cupric stearates were added directly to the lard.

B—Materials

(1) Fats and Oils: Commercially processed fats were used. The cutting, the killing, the scrap cut lards, and the rendered pork fat were prepared by steam rendering. The cutting lard is secured from the fatty tissues which are obtained from cutting of the chilled carcass into commercial cuts, and from trimming of these cuts. Killing lard is obtained from fats, except leaf fat, removed as the carcass is dressed. Scrap cut lard is derived from other miscellaneous sources, such as freezer stock. Rendered pork fats are derived from other edible tissues, such as bacon trimmings, as described in Meat Inspection Regulations (10). Lard samples were secured after the rendered fats had been allowed to settle and were stored in glass at 45°F. until used. Samples were not used after four days.

Oleo stock was secured by open kettle rendering at 155°F. from selected beef fats. The oleo stearin, which has a titer of about 50°C., and the oleo oil, which has a Wiley melting point of about 33-34°C., were secured by conventional seeding and pressing of oleo stock (11).

The peanut and cottonseed oils were refined, bleached, and deodorized. The shortenings were all hydrogenated vegetable products which had been well deodorized. The butterfat was secured from fresh, high-score butter by melting on a steam bath, decanting off the fat layer, and filtering until clear.

(2) Tannins: All samples represented commercially available products. U.S.P. tannic acid (12) is a yellowish-white to light brown powder, containing less than 12% moisture and less than 0.5% ash, very soluble in water, acetone and ethyl alcohol, and is made from gall nuts. The technical tannic acids contained 65-85% tannin and were similar to U.S.P. powder but were not purified to the same extent and may be derived from sources other than gall nuts. The chestnut and spruce powdered extracts were typical commercial tannin preparations.

Data and Discussion of Results

Before discussing the results, it may be of value to point out that a recent review of 133 papers published since 1921 in the field of autoxidation of fats, and oxidation inhibitors for fats, showed 75 were concerned with inhibitors, and in 32% of these papers the effectiveness of the substance was expressed as "protection factor," "anti-oxygenic index," or "antioxidant value," which were used interchangeably. This value is secured by dividing the keeping quality of the control containing the antioxidant by the keeping quality of the control. It is obvious that a value calculated in this manner is not fixed for any particular type of fat or concentration of inhibitor. It is greatly dependent upon the keeping quality of the control or substrate. Unfortunately, in too many cases the keeping quality of the control has not been given with the result that the value of the data is greatly reduced and might actually be misleading.

TABLE 1

Effect of Tannic Acid Treatment on Lard
0.1% U.S.P. Tannic Acid No. 18720

Material	A.O.M. Keeping Quality		I_V	I_F
	Control	Treated		
	hours	*hours*		
No. 1 Cutting Lard	6.0	37.3	31.3	6.2
No. 2 Cutting Lard	8.8	34.5	25.7	3.9
No. 3 Cutting Lard	9.0	38.0	29.0	4.2
No. 4 Cutting Lard	10.0	38.0	28.0	3.8
No. 5 Cutting Lard	11.5	40.5	29.0	3.5
No. 6 Cutting Lard	15.0	47.8	32.8	3.2
Neutral Lard	10.5	36.5	26.0	3.5
Killing Lard	19.0	48.0	29.0	2.5
Scrap Cut Lard	11.8	36.5	24.7	3.1
Rendered Pork Fat	4.5	23.0	18.5	5.1

For the past 10 years in this laboratory it has been helpful to use the increase in induction period of the substrate as a measure of the effectiveness of the antioxidant added. We use the symbol "I_A" to indicate this value. Lips and McFarlane (13), and Riemenschneider, Turer and Ault (14) use this method. The keeping quality of the control should be given.

Keeping quality of fats can be increased by treatments or processes, such as hydrogenation, deodorization, refining, or removal by adsorption of metallic ions, etc. To distinguish improvements from such processes from those secured by the addition of antioxidants, such improvements have been expressed in our laboratory in a manner analogous to that used for antioxidants. Thus I_F (improvement factor) corresponds to "protection factor" and I_V (improvement value due to process) corresponds to I_A (improvement due to antioxidant). The I_V and I_A values have proved the more useful and therefore are recommended. In any case, the life of control should be given.

The data are presented in Tables 1 to 6.

Table 1 shows that the variation in I_V is less than in I_F when the keeping quality of the substrate varied

TABLE 2

Effect of Tannic Acid Treatment on Keeping Quality
of Various Fats

Material	A.O.M. Keeping Quality		I_V	I_F
	Control	Treated		
	hours	*hours*		
Peanut Oil	25.0	34.0	9.0	1.4
Cottonseed Oil	20.0	25.5	5.5	1.3
Type I Shortening	133.5	159.0	25.5	1.2
High K. Q. Hydrogenated Vegetable Oil Shortening	116.0	150.0	34.0	1.3
Oleo Oil	7.0	77.8	70.8	11.1
Oleo Stearin	35.0	133.0	98.0	3.8
Oleo Stock	17.8	110.5	92.7	6.2
Butterfat	38.0	88.0	50.0	2.3

TABLE 3

Removal and Inactivation of Iron and Copper as Iron and Copper Tannates—Cutting Lard Was Used

	P.P.M. Added	Treatment	A.O.M. Keeping Quality	D_V[1]	I_V	Note
			hours			
A	0	0	9.5	....	....	Control for A series
A	10 Fe	0	2.3	7.2	....	....
A	10 Fe	T.A.(0.1%)	13.0	....	10.7	Some Fe Tannate left in
B	0	0	12.0	....	....	Control for B series
B	5 Fe	0	4.5	7.5	....	....
B	5 Fe	T.A.(0.1%)	22.0	....	17.5	Some Fe Tannate left in
C	0	0	12.0	....	....	Control for C series
C	1 Cu	0	1.0	11.0	....	....
C	1 Cu	T.A.(0.1%)	**4.5**	....	3.5	....

[1] Denotes decrease in keeping time or "damage value."

from six to 15 hours. If one- or two-hour lard had been used, the difference would have been still more pronounced. The keeping quality of good lard can be increased 20 hours with an effective sample of tannic acid. This sample of tannic acid was quite effective as can be seen from Table 6.

TABLE 4

Removal of Copper by Use of Tannic Acid

	Description of Sample	P.P.M. of Copper in Fat
A	Refined Lard	Less than 0.5
B	A to which 8.9 P.P.M. of Copper had been added	8.9 to 9.4
C	B treated with 0.5% Tannic Acid	Less than 0.5

Table 2 shows the large variation in improvement of keeping quality dependent upon the type of fat used as the substrate. Animal fats are improved to a greater extent than vegetable fats. This is interpreted as being due to the effect that more pro-oxidants are removed from them and that, in general, the fatty acid constituents of the beef fats, at least, are, in themselves, less prone to be oxidized than those of the peanut oil or cottonseed oils. A quantitative interpretation also requires consideration of antioxidants present in the various fats.

Table 3 shows the detrimental effect of small quantities of metals. It is of interest to compare the effect of 1 P.P.M. of copper, which reduced the A.O.M. from 12 hours to 1, with the data published in the National Provisioner (15). It was reported in this reference that 0.2 P.P.M. of copper reduced six-hour lard to two hours. Under the conditions of the tests colloidal soap was formed and was not completely removed in the case of iron, as evidenced by the color of iron tannate in the filtrate. Even with some iron tannate left in suspension the keeping quality excelled that of the control. It is thought that this occurred, to some extent, with the samples containing the cupric stearate. It was thought that with a more efficient filter cake, the iron and copper could be removed more completely. It should also be recognized that such relatively high concentrations of metals do some damage before they are removed or inactivated.

TABLE 5

Effect of Temperature of Treatment on Cutting Lard Using 0.1% U.S.P. Tannic Acid No. 18720

Temp. of Treat.	A.O.M. Keeping Quality	I_V	Peroxide Value[1]	Color	$FeCl_3$ Test	Note
°F.	*hours*					
......	8.8	......	0.2	5/0.4	Neg.	1st Control
160	34.5	25.7	......	5/0.4	Neg.	....
200	35.5	26.7	......	5/0.6	Neg.	....
250	16.0	7.2	......	6/1.3	Neg.	....
300	3.5	—5.3	......	7/1.6	Pos.	T. A. Froths
350	3.5	—5.3	......	8/1.2	Pos.	T. A. Froths
400	11.8	3.0	......	9/2.1	Pos.	T. A. Chars
......	11.5	......	0.2	5/0.4	Neg.	2nd Control
450[2]	47.5	36.0	......	30/4.6	Pos.	T. A. Chars
160	11.5	......	0.2	5/0.4	......[4]	....
300	4.3	—7.2	4.0	6/1.6	......[4]	....
400	2.5	—9.0	5.0	7/1.6	......[4]	....
450	1.7	—9.8	5.0	9/1.8	......[4]	....
160[3]	11.0	......	0.2	5/0.4	......[4]	....
300[3]	0.0	—11.0	26.0	7/1.2	......[4]	....
400[3]	1.0	—10.0	6.0	7/1.6	......[4]	....

[1] Peroxide value is expressed as millimoles of oxygen per 1,000 grams of fat.

[2] This and following were run on a second control.

[3] Sixty minutes instead of the usual 15 minutes were used in the heating period.

[4] No tannin added.

TABLE 6

Effectiveness of Various Samples of Gallotanin Type Tannins Substrate Cutting Lard,* 0.1% Tannin

Tannin	A.O.M. Keeping Quality		I_V	$FeCl_3$
	Before	After		
	hours	*hours*		
U.S.P. Tannic Acid No. 1	7.0	35.0	28.0	Neg.
U.S.P. Tannic Acid No. 2	15.5	48.0	32.5	Neg.
U.S.P. Tannic Acid No. 3	9.0	11.0	2.0	Neg.
Tech. Tannic Acid No. 1	9.0	11.0	2.0	Neg.
Tech. Tannic Acid No. 2	11.5	18.0	6.5	Neg.
Tech. Tannic Acid No. 3	9.0	35.0	26.0	Neg.
Tech. Tannic Acid No. 4	9.0	41.0	32.0	Neg.
Chestnut Extract No. 1	9.5	23.0	13.5	Neg.
Chestnut Extract No. 2	6.0	25.0	19.0	Neg.
Chestnut Extract No. 3	12.5	24.5	12.0	Neg.
Chestnut Extract No. 4	11.5	28.3	16.8	Neg.
Spruce Extract No. 1	6.0	8.0	2.0	Neg.
Spruce Extract No. 2	9.5	14.5	5.0	Neg.

* Different samples of cutting lard were used.

An experiment in which relatively high concentrations of copper and tannic acid were used shows that within the limits of experimental error of the method used, all the added copper was removed. See Table 4.

Table 5 shows the interesting relation which exists between the claims of Musher's patents and Verbeck's patent. It should be noted that the I_V is based on the life of the unheated sample. Unfortunately, sufficient quantity of the same sample of lard was not available for making control runs with heating and without adding tannic acid. It was necessary to make such tests on another sample of cutting lard. The harmful effect of the heat treatment at elevated temperatures is shown by the peroxide value, particularly when the time was increased to 60 minutes.

The effects observed are interpreted as follows: At 160 to 200°F. the effect of tannic acid in removing pro-oxidants predominated. At 300°F. the damaging effect of heat and oxygen of the air predominated. At 400-450°F. the antioxidant effect of the thermal decomposition products, particularly pyrogallol, predominated. At 400 and 450°F. the thermal decomposition of the peroxides resulted in a lower peroxide value and also resulted in an induction period before the peroxide reaches 10 which is used in the A.O.M. method as the end point for the induction period.

Table 6 shows the variation in effectiveness of various samples of commercial tannins. Neither the color, the flavor, nor the odor of the lard was adversely affected by treatment with any of these tannins.

Summary

The literature on the use of tannins as antioxidants for fats is reviewed. A process is described for the improvement of the keeping quality of edible fats by treatment with tannin in which the tannin and/or tannin compound is removed by filtration. Data are presented for various types of animal and vegetable fats and oils which were treated with U.S.P. tannic acid by the process described. The process is particularly effective on lard and beef fats. Data are presented which show the effects of varying the temperature at which the treatment is made. A method for testing the effectiveness of removal of the tannin is described. The improvement value I_V, is introduced and is defined as the increase in keeping quality of a fat attributable to a process. It is analogous to the I_A value which is defined as the increase in induction period of a fat due to the addition of an antioxidant.

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Review of Literature on Fats, Oils and Soaps

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Report of the Literature Review Committee

Part 1

Introduction

Domestic production of fats and oils during 1945 amounted to about 9.5 billion pounds compared with 10.8 billion pounds in 1943 and in 1944. Exports to the Allied countries were decreased about 53% as compared to the previous year, and imports tapered off to the smallest volume in many years. Stocks on hand toward the end of the year were being depleted to the lowest since 1936.

The effect of the fat situation on civilian consumption was such that the food fat supplies in 1945 were 41 to 42 pounds per capita (as compared to an average of 48 pounds in 1936-39) in the face of a potential demand of at least 50 pounds per capita. Technical fat users also felt a more or less corresponding deficiency of raw material. Soapmakers were additionally affected by short rosin supplies. Early in the war period, soapmakers were required to increase the percentage of rosin in soap to conserve fats. However, use of rosin was simultaneously expanded in the manufacture of ester gum, synthetic rubber, and miscellaneous chemicals to such an extent that the demand exceeded supplies. Beginning with the second quarter of 1945, the use of rosin in civilian soap was limited by WPB Order M-387 to 25% of the quantity used in civilian soap in the corresponding quarter of 1944.

All predictions suggest that no improvement is expected until mid-1946; for the year as a whole there should be about a 4-pound per capita increase in food fats available for civilian consumption. Demands from our armed forces and allies should decrease. Imports should begin to alleviate the deficiencies in the paint and soap industries, for in normal times these industries were the consumers of those spe-

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