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OFF FLAVOR DEVELOPMENT IN CANNED
APPLE SAUCE

Thesis for the Degree of M. S.

MICHIGAN STATE UNIVERSITY

William Van Hodge

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OFF FLAVOR DEVELOPMENT IN CANNED APPLE SAUCE

By

WILLIAM VAN HODGE

AN ABSTRACT

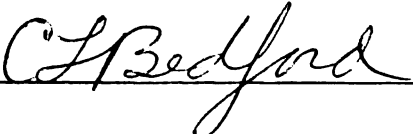
Submitted to the College of Agriculture, Michigan State University of
Agriculture and Applied Science in partial fulfillment of
the requirements for the degree of

MASTER OF SCIENCE

Department of Food Technology

1959

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A study was carried out during the winter and spring of 1959, to investigate the effect of temperature and length of storage on eighteen single variety apple sauces. The apples had been harvested at the proper stage of ripeness and stored at 32 to 34°F. Thirty pounds of fresh, sound apples were selected from each of the varieties used and made into apple sauce under simulated commercial conditions. The sauce was then adjusted to 17 percent soluble solids with sucrose and a representative sample of each sauce was frozen, to serve as control. The remainder of the sauce was canned in No. 303 cans and divided into three lots. These lots were stored at 32°F, 70 to 75°F, and 85°F. The 85°F lot was stored at this higher temperature to accelerate the development of storage effects.

The results of the measurements made on the apple sauce stored at various temperatures indicate that the development of the off flavor component, n-caproic acid, is a function of time and temperature. This development continues to increase quite rapidly for some time, then gradually tends to level off. The tin content and caproic acid content play a role in the development of off flavor and dark color in apple sauce.

These are not the only factors responsible for off flavor since sauces stored at 85°F had comparable amounts of tin and caproic acid as that stored

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at 70° F and both exhibited off flavor, but the sauce stored at 85° F was much more undesirable in this respect, and also showed darkening of the sauce. No explanation for this can be given at this time. There was considerable variation in the amount of caproic acid developed among the various varieties tested.

There was small differences in pH, total acidity, acetaldehyde, amide N, soluble solids, consistency, viscosity and tin content with little or no change in the quantities of the factors during storage, except in the case of tin, which tended to increase with increased storage time. No relationship could be found between these factors and the development of caproic acid.

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INTRODUCTION

The canned apple sauce industry has expanded considerably in recent years, and apple sauce has become one of the important processed foods in the United States. In 1940 the United States pack was 2,364,864 cases (#303 equivalent), and in 1958-59, 16,002,901 cases, which was an increase of nearly a million cases per year. The 1958-59 pack of apple sauce was equal to about 49 percent of the canned peach pack of that year (5). The main areas of production are California, Washington and Oregon in the western part of the United States, and New York, Pennsylvania, Maryland, Virginia and West Virginia in eastern United States. Although apple sauce is produced in large quantities in Michigan, it has not as yet become a major processed item in this State.

Cruess (9) has reported that owing to the change in consumer demand nearly all of the Gravenstein variety in California is now being used in the production of apple sauce. This is being generally reflected in all areas, as there has been a steady decline in the per capita consumption of all varieties of fresh apples (a decrease from 27.6 pounds per person in 1936 to 19.2 pounds in 1957), and an increased consumption of processed apples (an increase from 2.8 pounds per capita to 6.9 pounds) (7).

The varieties of apples used in the production of apple sauce vary

with the area of production. In California, the Gravenstein variety is the one used chiefly for sauce (9), while in New York, Pennsylvania, Maryland and Virginia blends of such varieties as York, Jonathan, Golden Delicious, Rome Beauty and Stayman are used in their apple sauce.

Recently it has been reported (17) that canned apple sauce develops an "off flavor" described as "tinny" or "goaty" during storage. Because of the fact that flavor is one of the important quality factors in selling apple sauce, a study was made to determine the effect of temperature and length of storage on the development of off-flavor in apple sauces produced from eighteen varieties of apples grown commercially in Michigan. This study was undertaken during the winter and spring of 1959.

REVIEW OF LITERATURE

Commercially packed apple sauce is a relatively new processed food, being about forty years old. It is believed that the Barlow Brothers Canning Company was the first commercial processor of apple sauce, using modern methods of processing.

Apple sauce is prepared from sound fresh apples (Pyrus malus) of the proper ripeness. The apples are washed, peeled and cored, trimmed, sorted, chopped or sliced, blanched, and pulped (2, 6, 9, 10, 21). The sauce may or may not contain added sweetening, water, salt and spices, and is sufficiently heat processed to assure preservation (25). Heating of the chopped or sliced apples is generally accomplished by one of two methods-- direct steam injection or with the conventional steam jacketed kettle process. The former method is in much wider commercial usage, and the Allen type processing equipment is most commonly used. It is preferred because it provides a rapid, efficient and easily controlled procedure.

It has been reported by several researchers (2, 6, 21, 26) that the quality of apple sauce is determined by four factors--flavor, color, consistency, and absence of defects. Flavor has been considered the most important of these factors, although color and consistency play a very important role. Pfund (21) reported that in addition to sweetness and a moderate degree

of acidity, odor was also important in determining the flavor of apple sauce.

Power (22) working with fresh apple parings, isolated amyl alcohol, formic, acetic, caproic and caprylic acids, and concluded that the odorous constituents of apples consisted of amyl esters of the above named acids. More recent work (15, 16, 28) has shown the presence of primary alcohols from one to six carbon atoms, aldehydes, ketones and esters of formic, acetic, propionic, butyric and caproic acids in stored apples, which contribute to the volatile fraction of the apple flavor. It is recognized that during the manufacture of apple sauce some of these volatile compounds are lost in the cooking and filling processes. The result is a lack of typical apple flavor in the sauce (17). It has been suggested (4) that apple essence be added to the sauce in order to replenish the lost flavor.

In addition to flavor lost during processing, it has been reported that apple sauce during storage develops a flavor and aroma which is disliked by the consumer (1), and described as "rank or tinny". Mattick, Moyer and Shallenberg (17) have isolated n-caproic acid from stored canned apple sauce. They tested sauces made from Baldwin and McIntosh apples, and from blends of Rhode Island Greening, Baldwin, Ben Davis, Northern Spy and Wealthy, and of Baldwin, Cortland, Rome Beauty, and found n-caproic acid in concentrations up to 120 ppm, depending on storage time. They concluded that it was largely responsible for the rank flavor of apple sauce. The source of

caproic acid is not known. The amount of caproic acid isolated from esters in apple essence by White (29) appears to be too low to account for the amount found in apple sauce.

Flavor changes in canned apple sauce have also been reported as due to changes in the non-volatile constituents (acids, sugars and salts) (21, 26). Pfund (21) and others reported that during storage the total acid content tends to decrease and result in flatness. This was particularly noticeable in some varieties, such as the Golden Delicious.

Sugar content also plays an important part in the flavor of the apple sauce (6, 21, 26, 28). Dryden and Hills (10) found that a sugar content of about 22 percent with an acid content of 0.45 percent was preferred. If the acid content was increased or decreased, a corresponding change in sugar content was necessary.

Although consistency is recognized as an important factor in apple sauce quality, no objective method of measurement is recognized in the present standard (2). Consistency measurements have been reported, by using the line spread procedure (6, 9, 21, 26, 27, 28) with the Stormer viscosimeter (13, 15, 28) and with the Brookfield viscosimeter (15). It is indicated that a measurement of consistency would be useful to detect undesirable changes (28).

METHODS AND MATERIALS

The eighteen apple varieties used in this study are as follows:

Baldwin, Ben Davis, Caville Black, Cortland, Fameuse, Golden Delicious, Grimes Golden, Jonathan, McIntosh, Northern Spy, R. I. Greening, Stark, Stayman, Tolman Sweet, Wagener, Wealthy, Winter Banana and Wolf River.

They were obtained from Michigan State University orchard during the fall of 1958, and were stored at $32^{\circ}\text{F} \pm 1^{\circ}\text{F}$ with a relative humidity of 85 ± 5 percent. All varieties were stored about four months before processing. The apples were removed from storage and held at room temperature for three hours prior to sorting and processing. Thirty pounds of sound apples of each variety were used in the preparation of sauce. The apples were washed, peeled, cored and cut into twelfths. The prepared slices were covered by cold tap water (57°F) until processed, to minimize browning. The sliced fruit was steam blanched for three minutes to soften the flesh and to stop enzymatic activity. They were immediately pulped in a laboratory size Langsenkamp pulper, using a $3/32$ inch screen. The sauce was adjusted to a soluble solids content of 17 percent, and the sweetened sauce was filled hot into #303 (apple sauce) cans, given a 7-minute exhaust, closed and hot-water processed five minutes. Representative samples of sauce of each variety was taken prior to processing and put into plastic containers and frozen, to serve as checks.

After cooling the cans in running cold water, each lot of apple sauce was divided into three lots; one lot was stored at 32° F, one lot at 70 to 75° F, and the third lot at 85° F. The higher temperatures were used to accelerate the effect of storage conditions.

MEASUREMENTS AND DESCRIPTIONS

pH and Total Acidity

A ten-gram representative sample of apple sauce was thoroughly mixed with 200 ml of freshly boiled, cooled, distilled water, and the pH determined, using a Beckman Zeromatic glass electrode pH meter. The sample was then titrated to a pH of 8.2 and the results expressed as percent malic acid. In the pH determination it was found necessary to add at least a small quantity of freshly boiled distilled water to assure even distribution of the hydrogen ions. The optimum quantity of distilled water was found to be 200 ml for both pH and total acidity determinations. Initial studies showed the pH values obtained on the dilute sauce were the same as those obtained on the undiluted sauce.

pH and Volatile Acid Content

The procedure for the distillation of volatiles was similar to that described by Mattick et al. (17). A 100-gram representative sample of sauce was weighed into a 500 ml round bottom boiling flask with 50 ml of freshly boiled distilled water and steam distilled at the rate of 5 ml per minute until 200 ml were collected. The steam generator consisted of a 3 liter flask with a 3 necked top, fitted with an internal coil of nichrome wire immersed in boiled distilled water and regulated with a variable voltage

transformer. The distillate was condensed with a Allihn condenser, and collected in a beaker containing 50 ml of freshly distilled water. The discharge tip of the condenser was placed below the surface of the water in the collection beaker, and the beaker was placed in an ice water bath to minimize the loss due to volatilization. The pH of the distillate was determined, using a Beckman Zeromatic glass electrode pH meter, and then titrated to a pH of 8.2 with (0.01 N NaOH) to determine the volatile acid content of the sauce. The results were expressed as mg of n-caproic acid per 100 grams of sauce.

Identification of the Volatile Acid Component

The procedure used for the quantitative identification of the volatile component was similar to that described by Block et al. (3). The titrated samples were concentrated about 1,000 times to obtain a sample of the sodium salt of the acid sufficiently large for the chromatographic identification procedure. The sodium salt of the acid was converted to an ammonium salt to eliminate the possibility of the acetic spots being masked or obliterated by the sodium ion. The ammonium salt of the acid was spotted on Whatman's No. 4 chromatographic paper, and developed with the solvent (1 part concentrated NH_4OH and 100 parts 95% $\text{C}_2\text{H}_5\text{OH}$). The chromatogram was dried and sprayed with bromophenol blue indicator (50 mg of indicator in 100 ml of water plus 200 mg of citric acid). The presence of

ammonium salts was indicated by the appearance of blue spots on a yellow background.

Acetaldehyde

The procedure and apparatus for the separation of acetaldehyde from apple sauce was similar to the steam distillation procedure described by Mattick et al. (17). The quantitative determination of acetaldehyde was similar to that described by Joslyn et al. (14). A 200 ml sample of the steam distillate was placed into a 500 ml Erlenmeyer flask containing 50 ml of a neutral buffer solution (3.35 grams KH_2PO_4 plus 15 grams $\text{Na}_2\text{PO}_4 \cdot 12\text{H}_2\text{O}$ per liter) and 10 ml of bisulfite solution (18.9 grams anhydrous Na_2SO_3 and 150 ml of $\text{N. H}_2\text{SO}_4$ per liter). The flask was stoppered and shaken. After standing for 20 minutes, 1 ml of freshly prepared 1% starch solution and 10 ml of acid solution (250 ml concentrated HCl per liter) were added. The excess bisulfite was then titrated with 0.1 N iodine solution until a blue end point was reached.

The solution was then made alkaline by adding 100 ml of an alkaline buffer (8.75 grams H_3BO_3 and 400 ml of 1 N, NaOH per liter). This liberated the bound bisulfite and the blue color disappeared. The solution was then titrated with 0.01 N iodine solution until the blue end point returned. A blank was run on the reagents using 100 ml of distilled water. (1 ml of 0.01 N iodine = .22 mg acetaldehyde).

Ammonia N and Amide N

The procedure used for the determination of ammonia and amide nitrogen content of apple sauce was similar to that described by Pucker et al. (23) except the quantitative determination was made titrametrically. A representative 100 gram sample of apple sauce was blended with 100 ml of distilled water for two minutes, then filtered through E and D No. 619 folded 20 cm filter paper.

For ammonia N, a 50 ml representative sample of the water extract was transferred to a 500 ml Kjeldahl flask in a 55°C water bath and 5 ml of a saturated sodium borate solution (pH 10) was added. The sample was aerated for 15 minutes at the rate 2-3 bubbles per second with an air inlet tube and the distillate was condensed and collected in a beaker containing 5 ml 0.1 N. HCl and 20 ml of distilled water. The residual acid was titrated with 0.1 N NaOH using methyl red as an indicator.

For amide N, a 20 ml representative sample of the water extract was transferred to a 500 ml Kjeldahl flask and 2 ml 6N. H_2SO_4 added. The mixture was refluxed on a steam bath for three hours, cooled and then the acid was partially neutralized by the addition of 10 ml of 0.1 N. NaOH. The remainder of the procedure used for amide N determination was the same as that used for ammonia N determination.

Soluble Solids

The soluble solids content was determined with an Abbe refractometer on the liquid obtained by expressing a representative sample of apple sauce in a double layer of cheese cloth.

Consistency and Viscosity

Consistency and viscosity measurements were made at 70°F with a Bostwick consistometer and Brookfield Synchro Lectric viscosimeter. The Bostwick consistometer measurement results were expressed as cm of flow in 15 seconds. The Brookfield viscosimeter was operated at 12 rpms using a No. 4 spindle with the sauce filled to a depth of 3 1/2 inches in tall 200 ml beakers, the results expressed as relative viscosity measurements (centipoise).

Tin Content

The procedure used for the quantitative determination of tin in canned apple sauce was similar to that described by Morris et al. (20).

A representative 25 gm sample was weighed into a 800 ml Kjeldahl flask and 10 to 15 ml concentrated H_2SO_4 and 50 ml concentrated HNO_3 were added. The flask was then heated until all brown fumes had dissipated and the volume of the water white solution was 10 to 15 ml. It was necessary to add additional 5 to 10 ml of concentrated HNO_3 in order to obtain a water

white solution. The Kjeldahl flask was cooled and a small quantity of KClO_3 , 15 ml concentrated HCl , and 10 ml H_2O were added, after which the flask was heated until white fumes appeared and the solution was clear. The contents were then transferred to a 300 ml Erlenmeyer flask and 25 ml of concentrated HCl and 0.5 grams of aluminum foil was added. The flask was connected with a rubber stopper and glass tube to a beaker containing 10% KHCO_3 , and the hydrogen gas allowed to escape through the KHCO_3 solution. The flask was then cooled in an ice water bath which drew in a small quantity of the KHCO_3 . The partially neutralized contents of the flask were immediately titrated with 0.01N I_2 and the results expressed as mg Sn. per 100 grams of sauce.

RESULTS AND DISCUSSION

Off Flavor Component

The main, rank, volatile component found in the steam distillate was identified as n-caproic acid by the use of paper chromatography procedure. This is in agreement with the results reported by Mattick et al. (17). No other volatile acids were found.

Since the volatile acidity of apple sauce was found to be caproic acid, steam distillation of this component and titration offered an efficient routine procedure for its measurement. However, to obtain comparable results between replicate samples, it was necessary to carefully standardize the method as to the rate of distillation (5 ml per minute). Distilling at this rate and collecting 200 ml of distillate, the recovery of added n-caproic acid was from 98 to 101 percent. Faster rates of distillation resulted in variations in the amount of caproic acid recovered and lower rates gave incomplete recovery even when the amount of distillate collected was increased to 500 ml. No explanation can be given for the erratic recovery at the faster rates of distillation, but condensation of the steam in the boiling flask was the major problem at the lower distilling rates.

As shown in Table I, the amount of n-caproic acid formed during storage differed considerably between apple varieties. The largest amount

was formed in the sauce made from Wealthy apples. Mattick et al. (17) reported that the concentration of caproic acid ranged from 0 to 12 mg per 100 grams of apple sauce. No explanation can be given as to the differences found between the varieties, since the source of the caproic acid is not known. The rate of development of caproic acid varied with the time and temperature storage (Table I). The average caproic acid content of the apple sauces stored at 32°F for 84 days was similar to those stored at 70°F for 42 days, or at 85°F for 35 days. The results indicate that the amount of caproic acid formed tended to increase linearly with time up to 70 and 56 days at 70 to 85°F respectively. Further storage of 21 days at 85°F showed only slight increase in the caproic acid content indicating a leveling off in its formation. Mattick et al. (17) found a linear relationship between storage time and caproic acid formation for sauces stored at 20°C for 22 weeks.

The odor and taste of apple sauce stored at 70°F was not considered unpleasant after 70 days of storage, but the taste of sauce stored at 85°F became objectionable after 56 days, and both odor and taste were very objectionable after 77 days. An odor similar to that found in apple sauce stored for 77 days at 85°F was obtained when 11 mg SnCl_2 and 11 mg n-caproic acid were mixed with a 25 gm sample of thawed frozen apple sauce. This indicates that tin and caproic acid play a role in the development of the objectionable odor and flavor of canned apple sauce.

TABLE I

Caproic Acid Formation in Canned Apple Sauce

Variety	Storage Temperature								Varietal Average
	-5°F	32°F	70°F			85°F			
	49 days	84 days	42 days	63 days	70 days	35 days	56 days	77 days	
	mg/100 grams ^{1/}								
Baldwin	2.9	6.6	7.5	11.7	12.0	8.8	15.6	15.7	11.1
Ben Davis	3.0	9.0	5.8	12.5	13.0	8.7	18.0	18.0	12.1
Caville Black	2.7	6.5	6.4	7.8	8.5	8.6	13.8	14.4	9.4
Cortland	3.5	7.8	6.7	12.8	12.5	7.6	17.1	17.5	11.7
Fameuse	3.8	11.2	6.8	9.0	9.7	8.4	11.9	12.5	9.9
Gold. Delicious	3.2	10.7	6.7	9.3	9.7	6.3	14.7	14.5	10.3
Grimes Golden	3.9	8.9	5.4	11.4	11.6	6.3	15.2	15.7	10.7
Jonathan	3.9	8.2	5.9	10.4	10.7	6.3	6.8	17.1	9.3
McIntosh	3.5	7.6	6.5	10.5	12.4	8.8	15.5	15.6	10.9
Northern Spy	3.5	7.6	9.4	11.4	12.1	9.1	14.0	15.3	11.3
R. L. Greening	3.1	6.0	8.7	13.1	13.7	11.3	13.4	17.0	11.9
Stark	2.0	6.1	6.8	11.7	12.4	8.2	17.0	17.0	11.3
Stayman	3.1	6.4	7.3	11.6	12.2	6.3	14.7	15.1	10.5
Tolman Sweet	2.7	7.9	2.9	6.2	7.6	2.9	9.6	10.2	6.8
Wagener	2.6	7.4	4.2	8.4	9.0	6.7	10.7	12.2	8.4
Wealthy	3.0	10.0	8.4	11.6	12.1	11.5	16.6	16.0	12.3
Winter Banana	2.5	7.2	5.6	5.8	6.9	6.7	11.3	13.6	8.1
Wolf River	2.7	6.4	8.5	12.1	12.5	9.3	16.5	16.8	11.7
Average	3.0	7.8	6.6	9.7	11.0	7.9	14.6	15.3	

^{1/} Each datum represents the average value of 3 determinations. Individual values given in the appendix table.

Consistency

There was considerable difference between the consistencies of the sauces made from the various varieties (Table II). This indicates that to obtain uniform consistencies for the sauces made from various varieties of apples, it would be necessary to adjust the amount of water added during the processing operation.

pH and Total Acidity

There was no appreciable changes in pH or total acidity during storage of individual varieties and their controls (Tables III and IV). The greatest variation was between the total acidities of the varieties and ranged from 0.1 to 0.55 percent malic acid. No significant relationship was found between acidity and caproic acid production. ($r = .420$).

Soluble Solids

There was no change in soluble solids content during the storage of the sauce. The variations in soluble solids contents of various varieties were due to experimental error in the soluble solids content adjustment prior to processing (Table V).

Tin

There was considerable variation in the tin content from various varieties. Fameuse, Caville Black, Wagener, Wealthy and Winter Banana

TABLE II

Consistency of Apple Sauce

Variety	Viscosity												Consistency											
	Storage Temperature												Storage Temperature											
	-5°F				32°F				70°F				-5°F				32°F				70°F			
	49	days	84	days	42	days	63	days	70	days	35	days	49	days	84	days	42	days	63	days	70	days	35	days
	Centipoise (absent 000) ^{1/}												cm per 15 seconds ^{1/}											
Baldwin	35	29			32	35	35				26	30	26	0.5	0.5		0.6	0.5	0.6			0.7	0.5	0.7
Ben Davis	48	49			48	47	48				42	42	42	0.7	0.7		0.7	0.7	0.7			0.7	1.0	1.0
Caville Black	24	20			22	22	24				19	23	16	1.0	1.0		1.0	1.0	1.0			1.0	1.0	1.0
Cortland	38	29			27	28	27				26	21	21	1.0	1.0		1.2	1.2	1.2			1.2	1.2	1.2
Fameuse	29	28			32	33	37				24	23	23	0.7	0.7		0.7	0.7	0.7			0.7	0.7	0.7
Golden Delicious	24	21			16	18	17				19	22	19	0.5	0.6		0.6	0.5	0.5			0.7	0.5	0.7
Grimes Golden	19	19			16	19	18				16	24	16	0.7	0.7		0.7	0.7	0.7			0.7	0.7	0.7
Jonathan	46	48			47	45	44				38	29	29	0.4	0.5		0.4	0.4	0.5			0.5	0.5	0.7
McIntosh	29	28			24	23	24				29	38	29	1.0	1.0		1.2	1.0	1.0			1.0	1.0	1.0
Northern Spy	25	22			19	18	18				18	19	18	0.7	0.7		0.7	0.7	0.7			0.7	0.7	0.7
R. I. Greening	35	30			32	30	31				32	32	32	0.5	0.5		0.5	0.5	0.5			0.5	0.5	0.7
Stark	49	49			49	49	49				49	45	48	0.2	0.2		0.2	0.2	0.2			0.2	0.2	0.2
Stayman	35	35			36	34	34				33	36	33	0.5	0.5		0.5	0.5	0.5			0.5	0.5	0.5
Tolman Sweet	37	28			35	34	36				27	28	28	0.5	0.5		0.5	0.5	0.5			0.5	0.5	0.5
Wagener	25	22			22	23	25				17	28	25	0.5	0.5		0.5	0.5	0.5			0.5	0.5	0.5
Wealthy	25	25			21	25	24				26	23	22	1.0	1.0		1.2	1.0	1.0			1.0	1.0	1.0
Winter Banana	23	18			17	18	18				18	18	17	0.4	0.5		0.5	0.5	0.5			0.7	0.5	0.5
Wolf River	33	31			28	28	25				26	30	26	1.4	1.2		1.2	1.2	1.2			1.2	1.0	1.0

^{1/} Each datum represents the average value of two determinations.

TABLE III
pH of Canned Apple Sauce

Variety	Storage Temperature							
	-5° F	32° F	70° F			85° F		
	49 days	84 days	42 days	63 days	70 days	35 days	56 days	77 days
Baldwin	3.7 ^{1/}	3.7	3.7	3.7	3.7	3.7	3.7	3.7
Ben Davis	3.7	3.7	3.7	3.7	3.7	3.7	3.7	3.7
Caville Black	3.8	3.8	3.8	3.8	3.8	3.8	3.7	3.7
Cortland	3.7	3.7	3.7	3.7	3.7	3.7	3.7	3.7
Fameuse	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5
Golden Delicious	4.1	4.0	4.0	4.0	4.0	4.0	4.0	4.0
Grimes Golden	3.7	3.7	3.7	3.7	3.7	3.7	3.7	3.7
Jonathan	3.7	3.7	3.7	3.7	3.7	3.7	3.7	3.7
McIntosh	3.8	3.7	3.7	3.7	3.7	3.7	3.8	3.8
Northern Spy	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5
R. I. Greening	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5
Stark	3.7	3.7	3.7	3.7	3.7	3.7	3.6	3.7
Stayman	3.6	3.5	3.5	3.5	3.6	3.5	3.5	3.6
Tolman Sweet	4.6	4.6	4.6	4.6	4.6	4.6	4.6	4.6
Wagener	3.8	3.8	3.8	3.8	3.8	3.8	3.8	3.8
Wells	3.7	3.6	3.6	3.6	3.7	3.6	3.6	3.6
Winter Banana	3.8	3.7	3.7	3.7	3.7	3.7	3.7	3.6
Wolf River	3.5	3.4	3.4	3.4	3.4	3.4	3.4	3.4

^{1/} Each datum represents the average of two determinations.

TABLE IV

Total Acid of Canned Apple Sauce

Variety	Storage Temperature								Varietal Average
	-5° F	32° F	70° F			85° F			
	49	84	42	63	70	35	56	77	
	days	days	days	days	days	days	days	days	
gm malic acid/100 grams									
Baldwin	.34 ^{1/}	.35	.35	.35	.37	.34	.37	.37	.35
Ben Davis	.35	.36	.39	.37	.38	.36	.36	.37	.36
Caville Black	.38	.37	.37	.36	.38	.37	.42	.41	.38
Cortland	.31	.33	.36	.34	.35	.34	.34	.34	.34
Fameuse	.33	.36	.34	.38	.35	.34	.38	.35	.34
Golden Delicious	.25	.26	.27	.27	.28	.27	.27	.26	.26
Grimes Golden	.26	.27	.27	.29	.28	.28	.27	.28	.27
Jonathan	.37	.36	.42	.43	.41	.43	.41	.40	.40
McIntosh	.25	.27	.38	.37	.35	.28	.28	.28	.31
Northern Spy	.44	.41	.41	.40	.41	.43	.40	.42	.41
R. L Greening	.51	.51	.51	.47	.47	.54	.54	.54	.52
Stark	.33	.37	.35	.35	.42	.39	.37	.34	.36
Stayman	.56	.53	.55	.53	.55	.54	.53	.51	.54
Tolman Sweet	.09	.10	.11	.12	.11	.10	.10	.10	.10
Wagener	.45	.42	.37	.37	.38	.44	.38	.37	.40
Wealthy	.35	.38	.48	.47	.39	.45	.39	.40	.41
Winter Banana	.18	.22	.21	.21	.22	.22	.22	.22	.21
Wolf River	.45	.46	.42	.49	.48	.48	.48	.48	.45
Average	.34	.35	.36	.36	.36	.36	.36	.36	

^{1/} Each datum represents the average of two determinations.

TABLE V
Soluble Solids Percent of Apple Sauce

Variety	Storage Temperature							
	-5° F	32° F	70° F			85° F		
	49	84	42	63	70	35	56	77
	days	days	days	days	days	days	days	days
(Degree Brix)								
Baldwin	17	18	18	18	18	18	18	18
Ben Davis	18	18	18	18	17	17	17	17
Caville Black	17	19	19	18	19	18	18	18
Cortland	17	17	17	17	17	17	17	17
Fameuse	17	18	18	18	18	18	18	18
Golden Delicious	17	17	17	17	17	17	17	17
Grimes Golden	17	16	16	16	16	16	16	16
Jonathan	17	17	17	17	17	16	16	16
McIntosh	17	18	18	18	18	18	18	18
Northern Spy	17	18	18	18	18	17	17	17
R. I. Greening	17	18	19	19	19	18	18	18
Stark	18	18	18	18	18	18	18	18
Stayman	17	17	18	17	18	18	17	17
Tolman Sweet	17	17	17	17	17	17	17	17
Wagener	18	18	18	18	18	18	18	18
Wealthy	17	18	18	18	18	18	18	18
Winter Banana	18	18	18	18	18	18	18	18
Wolf River	17	17	17	18	18	18	18	18

showed the least amounts of tin residue, while R. I. Greening, Stark and Wolf River contained the greatest amounts. No tin was found in the frozen samples. The amount of tin found in the canned sauce was dependent upon the time and temperature of storage. Sauces stored at 32°F for 125 days contained 3.3 to 10.2 mg tin per 100 g sauce, at 70°F for 125 days, 5.0 to 12.9 mg per 100 g sauce, and at 85°F for 89 days, 4.8 to 13.7 mg tin per 100 g sauce (Table VI). The tin present in the apple sauce was probably due to detinning of the can. Kohn et al. (16) reported that detinning was due to excessive amount of air in the head space. This could be controlled by either reducing the head space to a minimum or by closing under conditions which will remove most of the head space air. Since, in this study, no precautions were taken to decrease the amount of head space air, the above results could be expected. It was also noted that there was a detinning line at the apple sauce level with pronounced darkening of the sauce in those lots stored at 85°F. No significant relationship was found between the amount of tin in the apple sauce and the amount of caproic acid formed. The correlation coefficient was .360.

Acetaldehyde

There was no correlation between acetaldehyde and off flavor development of canned apple sauce ($r = .190$) (Table VII). The small differences in the amounts found were probably due to varietal differences.

TABLE VI

Tin Content of Canned Apple Sauce

Variety	Storage Temperature			Varietal Average
	32° F 126 days	70° F 125 days	85° F 89 days	
	mg/100 grams ^{1/}			
Baldwin	6.0	8.7	9.0	7.9
Ben Davis	7.2	8.0	9.1	8.1
Caville Black	5.6	6.0	5.0	5.5
Cortland	7.0	6.5	7.2	6.9
Fameuse	4.5	5.0	4.8	4.8
Golden Delicious	9.0	7.3	8.6	8.3
Grimes Golden	9.5	9.6	9.6	9.6
Jonathan	6.0	6.9	6.8	6.6
McIntosh	6.0	9.0	8.5	7.8
Northern Spy	7.5	7.8	7.7	7.6
Red Greening	9.0	10.0	10.3	9.8
York	8.6	11.7	11.5	10.6
Wendland	5.0	7.9	7.4	6.8
Winesap	5.0	6.5	6.8	6.1
Winesap	4.8	5.0	4.8	4.8
Winesap	4.9	5.2	5.0	5.0
Winesap	3.3	5.0	4.5	4.3
Winesap	10.2	12.9	13.7	12.3
Winesap	6.2	7.7	7.8	

^{1/} Each datum represents average of two determinations.

Ammonia N and Amide N

Little or no ammonia nitrogen was found in the apple sauce. Small quantities of amide nitrogen were found present in apple sauce, as shown in Table VII. Also, it can be seen that only small variation exists between variety samples.

TABLE VII

Acetaldehyde and Amide N Content of Canned Apple Sauce

Variety	Acetaldehyde		Amide N
	Stored at 85° F		Stored at 85° F
	34 days	67 days	40 days
	mg/100 grams ^{1/}		mg/100 grams
Baldwin	0.09	0.07	1.5
Ben Davis	0.04	0.07	1.3
Caville Black	0.04	0.09	1.5
Cortland	0.07	0.07	1.4
Fameuse	0.09	0.09	0.9
Golden Delicious	0.04	0.04	1.7
Grimes Golden	0.04	0.07	0.6
Jonathan	0.09	0.09	1.6
McIntosh	0.10	0.07	1.3
Northern Spy	0.07	0.04	1.4
R. L. Greening	0.04	0.04	1.4
Stark	0.04	0.07	1.3
Stayman	0.08	0.04	1.0
Tolman Sweet	0.10	0.09	0.5
Wagener	0.09	0.10	1.7
Wealthy	0.09	0.10	1.1
Winter Banana	0.10	0.10	1.5
Wolf River	0.09	0.09	1.6
Average	0.072	0.072	1.3

^{1/} Each datum represents the average of duplicate determinations.

SUMMARY AND CONCLUSIONS

The purpose of this study was to evaluate the effects of time and temperature on the off flavor development in canned apple sauce produced from eighteen Michigan grown commercial apple varieties. The off flavor component, which was isolated by a carefully standardized steam distillation procedure was identified as n-caproic acid with paper chromatography. N-caproic acid was found to develop as a function of time and temperature. Sauces stored at 32°F contained similar amounts of caproic acid after 84 days of storage as those stored at 70°F for 42 days, or those stored at 85°F for 35 days. The caproic acid formation increased up to 63 and 56 days of storage at 70 and 85°F, respectively, with continued storage showing very little increase in the formation of this component. Considerable variation in caproic acid content was found between sauces from various varieties of apples used, with Ben Davis having the greatest amount, and Tolman Sweet the least amount.

It was concluded that tin and caproic acid content play a part in the off flavor and odor, but this is not the only factor, since sauces stored at 32° and 70°F show comparable amounts of tin and caproic acid, yet do not have the pronounced undesirable off flavor characteristics of the sauce stored at 85°F. At the present time no explanation can be given for this

development. The increase in tin content was probably due to corrosion of the can promoted by the relatively large head space present in canned apple sauce (13) which induced corrosion. There was considerable variation between the varieties as far as consistency, viscosity, soluble solids, total acidity, pH, tin content, and amide nitrogen, but in general there was very little change within the same variety during storage, and no odorous association was apparent between these factors and the development of caproic acid.

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APPENDIX TABLE

Caproic Acid Production in Canned Apple Sauce

Variety	Storage Temperature							
	-5°F	32°F	70°F			85°F		
	49 days	84 days	42 days	63 days	70 days	35 days	56 days	77 days
	mg caproic acid/100 gr.							
Baldwin	2.8	6.6	7.2	11.4	11.6	8.1	16.0	15.8
	3.0	6.8	7.7	11.9	12.2	9.0	15.8	15.9
	2.8	6.5	7.5	11.7	12.1	9.2	15.1	15.5
Ben Davis	3.0	8.7	5.7	12.4	12.7	8.9	17.6	18.1
	3.0	9.0	5.7	12.6	13.5	9.0	18.0	17.8
	3.0	9.0	6.1	12.4	12.9	8.6	18.1	18.0
Caville Black	2.8	6.6	6.3	7.5	8.5	7.8	13.9	13.8
	2.7	6.8	6.5	8.0	8.5	7.3	13.7	14.6
	2.6	6.1	6.5	7.9	8.7	7.7	13.7	19.2
Cortland	3.4	7.7	6.6	11.9	12.4	8.8	16.9	17.4
	3.6	7.9	6.5	12.2	12.3	8.4	17.3	16.6
	3.5	7.7	6.8	11.5	12.6	8.6	17.3	17.3
Fameuse	3.9	11.4	6.8	9.0	9.6	8.0	11.6	12.1
	3.6	11.1	6.7	8.8	9.5	9.0	12.3	12.8
	3.8	11.1	6.8	8.9	9.9	8.5	11.8	12.5
Golden Delicious	3.2	10.6	6.6	9.0	9.5	6.5	14.7	14.3
	3.2	11.1	6.5	9.3	9.7	5.8	14.6	14.7
	3.2	10.3	6.8	9.5	9.7	6.6	14.9	14.4
Grimes Golden	3.9	8.9	6.1	11.6	11.6	5.8	15.0	15.5
	4.1	8.9	5.0	11.3	11.7	6.7	15.5	15.8
	3.8	8.7	5.1	11.5	11.6	6.3	15.2	15.8
Jonathan	3.9	8.2	5.9	10.1	10.6	5.9	16.4	18.4
	4.1	8.1	5.8	10.9	10.7	6.7	16.4	16.2
	3.8	8.7	5.9	10.3	10.7	6.1	16.9	16.5
McIntosh	3.5	7.8	6.5	10.3	12.3	8.6	15.3	15.3
	3.6	7.6	6.5	10.8	12.4	8.9	15.8	16.3
	3.6	7.8	6.5	10.4	12.4	8.8	15.3	15.8

APPENDIX TABLE CONT'D

Variety	Storage Temperature							
	-5°F	32°F	70°F			85°F		
	49	84	42	63	70	35	56	77
	days	days	days	days	days	days	days	days
mg caproic acid/100 gr.								
Northern Spy	3.6	7.7	9.4	11.4	11.9	9.5	13.7	15.3
	3.5	7.4	9.6	11.6	12.2	8.7	14.4	15.2
	3.6	7.8	9.0	11.4	12.3	9.0	14.9	15.4
R. I. Greening	3.0	5.9	8.5	13.0	13.5	11.2	13.6	14.3
	3.2	6.0	8.5	13.6	13.7	11.3	13.2	14.7
	3.1	5.7	9.2	12.8	14.0	11.7	13.3	16.7
Stark	2.4	6.4	6.5	11.7	12.5	9.2	15.3	17.0
	2.9	6.1	6.8	12.1	12.4	9.3	14.7	17.0
	2.5	6.0	7.1	11.4	12.5	9.2	15.0	16.8
Stayman	3.1	6.3	7.8	11.8	12.0	8.5	17.2	15.2
	3.2	6.5	7.3	11.6	12.2	7.9	16.8	15.0
	3.1	6.4	6.7	11.5	12.4	8.2	17.1	14.7
Tolman Sweet	2.7	7.7	2.9	6.0	7.0	2.8	9.9	9.6
	2.7	8.0	3.1	6.0	7.8	2.8	9.6	10.7
	2.7	7.8	2.7	6.6	7.8	3.0	9.2	10.2
Wagener	2.7	7.5	4.5	8.4	8.1	6.6	11.8	12.3
	2.7	7.5	4.1	8.3	9.4	7.0	11.7	12.4
	2.6	7.2	4.2	8.4	9.4	6.5	11.6	12.1
Wealthy	3.0	9.7	7.5	11.7	12.2	11.4	17.7	16.3
	3.0	10.1	8.7	11.3	11.9	11.7	16.5	15.9
	3.1	9.6	8.8	11.9	12.3	11.6	15.6	15.9
Winter Banana	2.7	7.2	5.8	6.0	6.5	6.6	10.8	13.6
	2.4	7.3	5.4	5.6	7.1	6.8	11.8	13.7
	2.4	7.1	5.6	5.8	7.1	6.6	11.3	13.6
Wolf River	2.7	6.4	8.1	12.3	12.2	9.2	16.4	16.2
	2.8	6.5	8.2	11.7	12.8	9.2	16.8	17.0
	2.5	6.4	8.8	12.2	12.4	9.5	16.4	17.2

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