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Oxidized flavors in strawberry ice cream

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Oxidized Flavors in Strawberry Ice Cream

By E. W. Bird, O. E. Ross, C. A. Iverson, O. R. Ause
AND J. J. Willingham

AGRICULTURAL EXPERIMENT STATION
IOWA STATE COLLEGE OF AGRICULTURE
AND MECHANIC ARTS

R. E. Buchanan, Director

DAIRY INDUSTRY SECTION

AMES, IOWA
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Oxidized Flavors in Strawberry Ice Cream

I. An Investigation of Factors That Might Cause Oxidized Flavors

By E. W. Bird, O. E. Ross and C. A. Iverson

At the time this project was initiated only a small amount of material had been published concerning this defect and even that was subject to considerable controversy. That the defect under consideration is important has been emphasized by other workers and needs no reiteration here. It was the opinion of the authors that the study should be made with ice cream manufactured in a commercial manner and from commercial products. This was done wherever possible.

The objectives of the project were to attempt to determine which single factor or group of the following possible factors—oxidases in the fruit, copper and iron in the mix, strawberries and types of added solids—was responsible for the occurrence of the defect. It was likewise considered advisable to see whether or not such changes as occur in the fat, if the defect were a fat oxidation, were sufficient to cause detectable variations in the iodine, acetyl and Reichert-Meissl numbers of the fat of the ice cream.

LITERATURE REVIEW

Palmer and Miller (27) reported that when concentrated peroxidase (from horse radish) was added to cream, the quality of butter made from it was as good after eleven months as was that of the check samples. Palmer and Combs (26) considered that the natural oxidases of butter catalyzed the development of tallowiness in butter made from raw cream or from cream pasteurized at a temperature that does not inactivate the enzymes. Oxidase enzymes exist in plants, milk, fruits, etc. Fang and Cruess (14) found them in apricots, pears, peaches, prunes, figs, lemons, tomatoes, bananas and dates. Reed (28) likewise found the enzyme in citrous fruits. Ezell and Crist (13) reported both catalase and oxidase in lettuce, radishes and spinach, and Haber (16) reported oxidases in tomatoes. Harrison and Thurlow (17) have shown that oxidation of lactic acid is induced when hypoxanthine is oxidized in the presence of xanthine oxidase (in the presence of ferrous iron) and that $\beta$-hy-
droxy-butyric acid is oxidized by cysteine in the absence of peroxidase although the latter increased the oxidation.

A number of articles relative to oxidized flavors in strawberry ice cream have appeared to date; the bulk of them during the time these studies were in progress. They treat of a number of factors which might be of importance in the occurrence of the defect.

The question, whether or not the flavor is one of oxidation of fat, has been considered. Tracy and his collaborators (35, 36, 37, 38, 39, 40, 41, 42) consider that the defect is one of fat oxidation. They base their conclusions on the following facts: 1. There is a greater tendency for its development the higher the fat content of the mix; 2. the lower the score of butter used in ice cream the greater the tendency toward oxidized flavor development and 3. the oxidation-reduction potential seems to be higher or to increase in those samples in which the defect occurs. Ross (31) considered it a fat oxidation involving small amounts of fat but that these changes could not be detected by the iodine, acetyl and Reichert-Meissl numbers. Dahle and Folkers (5, 7, 8, 9) believe that the oxidation of the fat of cream employed in assembling mixes is not involved in the flavor defect occurrence because: 1. It occurred in mixes to which no fat other than that introduced by skimmilk or skimmilk products was added and 2. because of the relationships obtained between the oxidation-induction periods of the fat from some of their experimental ice creams and the development of the flavor defect. It is not clear what these authors may think relative to small amounts of fat introduced by skimmilk, condensed skimmilk, etc.

The effect of strawberries has been discussed by several workers. Mack and Fellers (24) believe that acidity and oxidizing enzymes in fruit cause oxidation of fat in strawberry ice cream and that treating the fruit at 165°F. for 20 minutes inactivates these enzymes and improves the keeping quality of the ice cream. Tracy et al. (41, 42) likewise consider the berries a causative agent and state that treating at 175°F. for 20 minutes retards the defect development. They likewise believe (38, 39, 40) that the fiber portion of the berry retards the development of the off-flavor but that the juice portion accelerates it. This is interesting in view of the fact that addition of all parts of the fruit reduces the oxidation-reduction potential of the mix, although the rise in oxidation-reduction potential, when both berries and copper are added to the same mix, is accelerated. Tracy (37) states that strawberries are of minor importance in causing the defect. Dahle and Folkers (6) state that “Every type
or brand of commercial strawberries used was capable of producing the off-flavor," and that sterilization of the berries failed to check the development. Later (8) they state that heating berries to 180°F for 1 hour did not prevent development of the defect but that reduction of the acidity of the berries to pH 7.0 did. Dahle and Carson (4) encountered the defect in vanilla ice cream. Ross (31) stated that berries were not a causative agent.

Acidity has received some consideration. Dahle and Folkers (7, 8, 9) consider that acidity of the fruit, together with copper, is responsible to some degree for the development of the defect. Sommer (33) believes a low pH is a factor in the oxidized flavor development. Tracy et al. (38, 39) believe that because of negative results obtained when citric acid was used as an acidulant the acidity of the fruit is not important.

Sommer (33) reasoning by analogy from work with cream considered that factors causing the defect should likewise increase the oxidation-reduction potential of the ice cream. Tracy et al. (38, 39, 40) present data which indicate that the oxidation-reduction potential rises as the oxidized flavor develops; copper causes it to rise and strawberries lower it. The rate of rise of the oxidation-reduction potential seems to be accelerated when both strawberries and copper are added. In this connection Webb and Hileman (43) state that they found no particular correlation between absolute oxidation-reduction potential among milks from different cows and the development of oxidized flavor but that oxidized flavor induced by copper contamination was accompanied by an increase in the potential.

There is general agreement among the various workers (4, 5, 7, 29, 38, 39, 40) that high homogenization pressures retard the development of oxidized flavors in strawberry ice cream, in comparison with low homogenization pressures or no homogenization.

Metal contaminations in dairy products and the effects of the resultant metal salts on the flavors of dairy products have received a great deal of attention. Hunziker et al. (20) and Guthrie et al. (15) are in agreement that the chromium-nickel steels corrode least and impart less flavor defects than other metals studied. Both groups state that copper, poorly tinned copper or copper alloys are not satisfactory. Iron is likewise an offender.

Ellenberger and White (11) consider copper very important in developing oxidized flavors in frozen cream. Hunziker (19) considers copper and its salts active agents in causing oxidized flavors. Iron and its salts likewise act as
catalysts, but their action is much less pronounced. Donauer (10) found that copper caused oxidized flavors in butter, while iron was not so serious an offender. Hunziker and Hosman (21) found salts derived from copper and its alloys very important in catalyzing the oxidation of butter fat but state that the action of iron salts is relatively slight. Rice (29) and Rice and Miscall (30) consider that copper, especially in the presence of oxygen, is very important in the development of oxidized flavors in sweetened condensed milk and in milk. Erb and McMasters (12) found that ice cream prepared with condensed milk from copper pans developed oxidized flavors more quickly than that prepared with condensed milk from rubber-lined pans. Roundy and Jackson (32) found that mixes condensed in copper pans generally developed oxidized flavors while those from stainless steel or nickel pans did not when stored for 3 months at 42°F.

It is evident then that there is excellent agreement among the reports on oxidized flavors in strawberry ice cream that copper is an important factor in causing the flavor defect (4, 5, 7, 8, 9, 23, 31, 33, 35, 36, 37, 38, 39, 40, 41, 42).

The type of "added solids" has received attention. The use of condensed milk from copper pans seems to contribute to the defect largely, while a fine quality product is apparently obtained from condensed milk from stainless steel pans (4, 32). Commercial dry skimmilk seems less likely to cause the defect than condensed skimmilk (6, 9, 31). Tracy (35) states that excessive use of serum solids may cause stale flavors. He and his collaborators emphasize the use of fresh high grade dairy products to aid in preventing the defects (35, 40, 41.) Dahle and Folkers' results (5, 6, 7, 8, 9) indicate that the quality of the dairy products may not be so very important. Tracy et al. (41) state that the source of serum solids is not so important but that the fat source is.

EXPERIMENTAL
METHODS AND MATERIALS EMPLOYED
ANALYTICAL METHODS
FAT EXTRACTION

A number of methods, including churning, for the recovery of fat from ice cream were tried. It was possible to recover approximately 50 percent of the fat by churning after the ice cream had been subjected to hardening room temperatures for several days. This no doubt resulted from a
dehydration of the proteins serving as protective-colloids in the fat-serum interfaces. A 50 percent recovery was deemed insufficient for representative checks of the fat of the ice cream. For this reason the following modified Röse-Gottlieb extraction method was resorted to.

Two hundred grams of ice cream, filtered through cheesecloth to remove pulp and seeds, were placed in a 1 l. glass-stoppered separatory funnel. The stopcock plug and glass stopper were lubricated with graphite by rubbing with a soft lead pencil so that no foreign greases would be introduced. Conc. NH₄OH (35.0 ml.), ethyl alcohol (125.0 ml.), ethyl ether (200.0 ml.) and petroleum ether (200.0 ml.) were added in the order named, and the contents of the separatory funnel were shaken for 1 minute after the addition of each reagent. The funnels were allowed to stand approximately 15 minutes for the separation of the fat-ether layer from the aqueous-alcoholic layer. Two such extractions were made with each sample, and the ether extracts from both were filtered into a 2 l. round-bottomed flask. Seventy-five grams of anhydrous calcium chloride were added, and the mixture was refluxed for 1 hour to remove alcohol and water. The heating was discontinued, the calcium chloride was allowed to settle (usually about 30 minutes) and the ether solution was filtered into Mojonnier fat dishes. These extracts were reduced in volume on a steam hot plate until all the solution could be transferred to one dish. The evaporation was continued in order to remove the ethers, after which the dish was placed in a Mojonnier fat oven at 135°C. and 21 inches vacuum for 10 minutes in order to remove any traces of ethers that might remain. The fat was then filtered through a hot water funnel, and samples were weighed for the fat constants as quickly as possible after this filtration.

**FAT CONSTANTS**

Iodine numbers were determined by the Hanus method (Woodman (44)) and Reichert-Meissl numbers by the method outlined by Leach and Winton (22). For the determination of the acetyl number the fat was acetylated as described by Woodman (44). Reichert-Meissl numbers were determined on the acetylated and non-acetylated fat from the same sample. The difference between the two Reichert-Meissl numbers was considered equivalent to the acetyl radical taken up in the acetylation of the fat, and the acetyl number was calculated from this difference. This method, except for the method of saponifying the fat, is essentially that described by Woodman (44). It was employed because
it yielded much better checks between duplicates than other methods which were tried.

**OXIDASE DETERMINATIONS**

These were determined according to Ezell and Crist (13), who employed Harvey's (18) modification of the simplified Bunzell (2) oxidase apparatus. One modification was made in this piece of equipment, viz., a piece of glass tubing was sealed onto the open end of the manometer, and a stopcock (T) was sealed into the closed arm at V (fig. 1). The stopcock was so placed that it would be just above the water level in the thermostat. In this way the zero levels could be adjusted after the materials had come to the temperature of the waterbath (but before mixing) without removing it from the waterbath at all. Two ml. samples were employed when oxidase preparations were investigated, 4 ml. samples when ice cream was the material. In either case 5 ml. of 1 percent pyrogallol and 1 ml. N/1 NaOH (in alkali vial P) were employed.

**COPPER ANALYSES**

Copper was determined colorimetrically as the diethyl-dithiocarbamate (3). The method described by McFarlane (25) was employed, except that the colors were matched in aqueous solution in Nessler tubes, adding standard copper solution to the standard until its color was the same as that of the unknown, rather than extracting the copper salt with amyl alcohol.

**IRON ANALYSES**

Iron was determined colorimetrically as ferric sulfocyanate, according to Stuttgart (34).

**MANUFACTURE OF ICE CREAM**

**COMPOSITION OF MIXES**

Mixes containing 14 percent fat, 10 percent serum solids, 15 percent sugar and 0.3 percent gelatin were made as nearly alike as possible with the exception of the sources of serum solids when these were varied in comparisons.

**SOURCES OF SERUM SOLIDS**

Condensed skimmilk when used was either purchased from a commercial manufacturer or was made in the vacuum pan in the Department of Dairy Industry, Iowa State College. In either case copper pans were employed. The

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3The samples for these determinations were strained through two thicknesses of cheesecloth after melting to remove fruit pulp and seeds; 100-gram samples of the strained material were dried and ashed.
Dry skim milk was either a roller dried skim, pre-condensed in copper pans, or was a commercial grade purchased on the open market.

**Type of Strawberries**

Unless otherwise noted, frozen strawberries were employed. Fresh berries, obtained chiefly from the Department of Horticulture, Iowa State College, were washed, stemmed and placed in 1 gallon brightly tinned, single service containers and were treated with sugar at the rate of 2 parts of berries to 1 part of sugar. These cans of treated berries were frozen and stored in the hardening room until used, at temperatures —10°F. to 0°F.

**Assembly of Mixes**

Fifty pound batches of mix were prepared. These were pasteurized at 158°F. for 30 minutes in 10 gallon cans, with constant agitation. The cans were held in a waterbath during the heating and pasteurization. They were homogenized in a two-stage Manton-Gaulin homogenizer with 2,500 pounds pressure at the first and 1,000 pounds pressure at the second stage. The mixes were then cooled over a surface cooler to approximately 60°F. and were held subsequently at 30°F. for 24 hours.

**Freezing**

These mixes were frozen in a Cherry-Burrell brine freezer. Half-batches (23.5 pounds) were frozen. The berries, at the rate of 10 percent non-sugared fruit, were added at the freezer, and approximately 100 percent overrun was maintained.

**Samples and Judging**

In each freezing, except in the preliminary trials, two samples of ice cream were drawn from the freezer into quart, cardboard, single service containers. This was done after freezing but before the addition of the strawberries. These were control samples—one for copper analysis, when desired, and one for judging. The berries were then added,
the whipping finished, and two fruit samples were then taken as before. The quart single service containers were coat-
ed on the inside with paraffin, melted in a double boiler apparatus made with two Pyrex beakers. This was done to prevent flavor contamination from the package. The double-boiler effect was employed to prevent imparting a smoky or cracked oil flavor to the paraffin.

Two to three members of the dairy industry staff with considerable experience in ice cream scoring judged the samples for flavor. The samples were key-numbered to obscure their identity. They were in no regular order and were judged in classes of six. Each judge placed the sample numbers, his flavor scores and flavor criticisms for the class scored on a sheet of paper before the judges conferred regarding the samples. Averages of the scores and composite criticisms were recorded for the samples.

CHECK OF EXPERIMENTAL METHODS

EFFECT OF THE EXTRACTION PROCEDURE ON THE FAT CONSTANTS

Ten samples of well mixed butter were divided into two portions. One of these was heated to separate the fat and serum phases, and the resulting fat was filtered through a dry filter paper in a hot water funnel. Two 25 gram samples of the other lot were extracted by the procedure described under Analytical Methods. Iodine, Reichert-Meissl and acetyl values were run on these fats. A statistical treatment of the data showed that there was no significant difference between the Reichert-Meissl and acetyl numbers of the two fats but that there was between the iodine numbers. The iodine numbers of the filtered fat were, with one exception, somewhat higher than those of the extracted fat, so that oxidation by peroxides in the ether solution or during solvent removal probably occurred. Although these differences were significant statistically, the maximum, minimum and average variations were but 0.67 unit, 0.11 unit and 0.401 unit, respectively. Although the extraction method described was not as satisfactory as might be desired, it was considered the best available for obtaining fat from ice cream.

CHECK OF TECHNIQUE IN OXIDASE MEASUREMENTS

In order to be certain of the technique of the oxidase measurements it was considered necessary to carry out determinations on materials known to contain considerable quantities of the enzyme. Potato oxidase was chosen as the material. Graph 1 of fig. 2, which is representative of

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4All statistical analyses of the data on which this bulletin is based were made in the statistical laboratories of the Iowa Agricultural Experiment Station. The methods employed were selected by Dr. A. E. Brandt and the calculations were carried out under the direction of Dr. Brandt and Miss Gertrude M. Cox.
a number of determinations made, shows that the method as conducted yielded the type of curves that other investigators have shown are characteristic for oxidases.

RESULTS

PRELIMINARY TRIALS

ENZYME STUDIES

It was considered advisable to check the effect of hardening room temperatures. Potato peelings were ground with calcium carbonate in a food chopper in order to neutralize the acids which otherwise might have inhibited the enzyme action (13). The juice was pressed out by hand through two thicknesses of cheesecloth and was diluted 1:4 with distilled water. Graph 1, fig. 2, is typical of the results obtained, and it indicates that hardening room temperatures would have little effect on the oxidase activity. One trial run for 28 days indicated that the enzyme activity was not appreciably impaired.

STRAWBERRY AND STRAWBERRY ICE CREAM OXIDASE ACTIVITY.

A number of trials were run on strawberries, without the neutralization procedures employed above. Neutralization was not practiced because the berries are not neutralized when used in ice cream. Graph 2, fig. 2 presents representative reaction curves. These are not at all the characteristic oxidase type of curve that was obtained with the potato-peel extract. One oddity encountered was the initial increase in pressure followed by a more or less linear reaction-rate curve. Graph 3, fig. 2 presents typical curves for strawberry ice cream. The rate of oxygen absorption by the ice cream is much slower than that of the strawberries but is again an apparently nearly linear function of time in the time range studied. No equilibrium is obtained.

It is rather contrary to expectation that the oxygen absorption should proceed at a slower rate and be less at any time interval for the mixes containing condensed skim milk than for those containing dry skim milk. These graphs indicate that oxygen absorption under the conditions of the oxidase test is not of a type that indicates oxidases functioning in the oxidative process. They also indicate that under the conditions of the test oxygen absorption is a function of time and proceeds at a more or less constant rate.

EFFECT OF ADDING ENZYMES TO ICE CREAM

Potato oxidase extracts were mixed into one series of ice cream samples drawn from the freezer before any flav-

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5Dry skim milk was used as a source of added solids in these experiments.
oring had been added. A 1-quart sample of the same unflavored mix was held as a control. Potato oxidase extract was added at the rate of 0.2 ml., 1.0 ml., 5.0 ml. and 10.0 ml. per quart and was thoroughly mixed into the ice cream. The samples were scored at weekly intervals for 6 weeks. No oxidized flavors developed in any of the samples in several such trials. In the sample containing 1 ml. potato extract a mild potato flavor was encountered; in those with the higher concentrations pronounced potato flavors existed.

Tomatoes have a milder flavor than potatoes. They are sufficiently acid to lower the pH of the ice cream mix as strawberries do. The pH values of some strawberry ice cream samples investigated were from 5.3 to 5.9. When sufficient tomato juice (from raw tomatoes) was added to yield comparable pH values a distinct tomato flavor was encountered. Ice cream samples were prepared as in the preceding experiment except that 1.0, 2.0, 10.0 and 20.0 ml. quantities of tomato juice were added. No oxidized flavors developed in these samples during 4 weeks.

A third comparison was made as follows: Three mixes were prepared. To the first was added an amount of Streptococcus lactis culture sufficient in amount to reduce the pH to 5.9. To the second, butter culture was added in quantity equal to the S. lactis culture to lower the pH; to the third an equivalent amount of skimmilk was added. Four 1-quart samples of each of these mixes were frozen in a hand freezer after having been treated as follows: (a) No further treatment; (b) 10 ml. potato oxidase extract added; (c) 5 ml. diacetyl solution6 (1 drop diacetyl (Eastman) to 100 ml. H2O) added and (d) 10 ml. potato oxidase extract plus 5 ml. diacetyl solution added. No sallowy flavors developed in any of these samples.

EFFECT OF ADDITION OF DIACETYL

Diacetyl determinations on strawberries, run in Doctor Hammer's Dairy Bacteriology laboratories for the authors of this bulletin, showed that the berries contained traces of this chemical. For this reason additional trials with diacetyl were made. Several comparisons of samples containing diacetyl with control samples failed to show an oxidized flavor development as a result of the addition of diacetyl.

6Diacetyl was added at the suggestion of Dr. B. W. Hammer who had had evidence that when added to butter diacetyl seemed to cause oxidative effects when restricted amounts of water (less than 0.5 percent) were present.
RESULTS WITH SAMPLES PREPARED TO APPROXIMATE COMMERCIAL CONDITIONS

After the preliminary results had been obtained it was considered that either oxidative enzymes, which had been considered a probable cause of the defect, were not of particular importance or else that conditions resulting from ingredients introduced into ice cream through the normal channels of manufacture reacted in such a way as to yield results appreciably different from those got when one or another ingredient was added to an experimental mix.

It was decided, therefore, to prepare pairs of mixes identical in all respects except that the source of serum solids in one sample of each pair would be dry skimmilk while that in the other would be condensed skimmilk. The condensed skim was prepared in copper condensing equipment. The dry skimmilk was prepared chiefly by the atmospheric roll process in the Dairy Industry Department, Iowa State College. The mixes were prepared as previously described. These samples were scored at 1, 7, 14, 20 and 28 days.

Oxidase activity, copper and iron analyses were run on each of the ice creams prepared. Fat constants were determined on the extracted fat from each ice cream prepared. Check samples were taken for every sample of ice cream manufactured, as has been stated under Methods. This gave opportunity to determine whether strawberries contributed to the cause of the oxidized flavor. Oxidase activity as employed here was expressed as ml. oxygen absorbed in 8 hours, because of the fact that no equilibrium was attained with ice cream samples up to 8 hours. The pressures were read at hourly intervals for all oxidase tests, but no difference in type of curve was obtained in any case.

OXIDASE ACTIVITY

The normal curve for oxidase activity indicates that as the oxidation proceeds the enzyme loses its ability to oxidize, and the curve finally becomes asymptotic to the abscissa. It was considered that if oxidation resulting from enzymes occurred the volume of oxygen absorbed under the conditions of the test should be less at the end than at the beginning of the storage period. The volume of oxygen absorbed during 8 hours at the end of the storage period was subtracted from the volume absorbed at the beginning of the storage period, and the rate of development of oxidized flavor was compared with this difference. It was considered that the greater the positive difference so obtained, the more rapid the rate of oxidized flavor development should be if oxidases were a factor.
Statistical treatment of the data shows that with neither the dry skimmilk samples nor the condensed skimmilk samples did such a parallelism exist when rate was expressed as: (a) Developed 1 to 20 days, (b) developed between 21 and 28 days and (c) not developed. It appears that these data check those of the preliminary trials in that oxidase enzymes seem not to be causative agents of the defect.

IRON CONTENT

With the exception of one sample the iron contents of the dry skimmilk samples ranged from 1.57 to 3.54 ppm.; the other sample contained 0.11 ppm. The mean value was 2.35 ppm. The condensed skimmilk samples contained from 1.45 to 2.45 ppm., with the mean 1.97. A statistical treatment of these data shows that the higher the iron content of the sample the less the tendency to develop oxidized flavors. This correlation is statistically significant. The result is contrary to what would have been expected, and it indicates that iron in the form in which it occurred in the mixes serves rather to retard than to accelerate the development of the flavor defect.

COPPER CONTENT

The copper content of the dry skimmilk samples ranged from 0.20 to 1.08 ppm. with a mean of 0.54 ppm., while for the condensed skimmilk samples the range was 0.24 to 2.12 ppm. and the mean 0.97 ppm. Statistical treatment of these data revealed that among them there is no definite correlation between the rate of oxidized flavor development and the copper content of the samples.

FAT CONSTANTS

There was no correlation shown by statistical treatment of the data insofar as change in Reichert-Meissl or acetyl numbers and rate of development of the flavor defect were concerned. The data do, however, show a tendency for the decrease of the iodine number to be greater for those samples in which the rate of development was most rapid and vice versa, although this correlation is not significant statistically. The changes in fat constants were taken as the differences between the constants at the beginning and at the end of the storage period. For this portion of the problem the average length of the storage period was approximately 60 days. Rates of development were figured in three classes: (a) 1 to 20 days, (b) over 20 days and (c) no development.

The tendency found for correlation between iodine number decrease and rate of flavor development would indicate
that possibly oxidation of the fat is involved in the development of the flavor defect.

STRAWBERRIES

When the fruit and control samples were compared for development or non-development of oxidized flavor and for rate of development of oxidized flavor, it was found that no significant difference existed between the two classes. These data indicate, therefore, that fruit is neither a causative nor a catalyzing agent in the development of oxidized flavors in strawberry ice cream. In order to illustrate this more plainly the actual number of samples which developed the defect is compared with the expected number (which latter was calculated by pooling the 22 fruit and the 22 check samples) in table 1.

TABLE 1. A COMPARISON OF THE EXPECTED AND ACTUAL NUMBERS OF FRUIT AND CONTROL SAMPLES THAT DEVELOPED AND DID NOT DEVELOP OXIDIZED FLAVORS.

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<th>Did not develop oxidized flavor</th>
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<td></td>
<td>Expected no.</td>
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<td>Fruit</td>
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<td>Control</td>
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</tbody>
</table>

A statistical comparison of the dry and condensed skim-milk samples shows that there is a significant difference between the two types of ice cream. The dry skim-milk samples in this study had the better keeping quality. When these data were pooled and the expected values were calculated and compared to the actual numbers of samples developing the defect, the following results were obtained.

TABLE 2. A COMPARISON OF THE EXPECTED AND ACTUAL NUMBERS OF SAMPLES, CONTAINING: 1. DRY SKIMMILK AND 2. CONDENSED SKIMMILK THAT DEVELOPED AND DID NOT DEVELOP OXIDIZED FLAVORS.

<table>
<thead>
<tr>
<th>Developed oxidized flavor</th>
<th>Did not develop oxidized flavor</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Expected no.</td>
</tr>
<tr>
<td>Dry skim-milk samp.</td>
<td>9.5</td>
</tr>
<tr>
<td>Cond. skim-milk samp.</td>
<td>9.5</td>
</tr>
</tbody>
</table>

SUMMARY AND CONCLUSIONS

1. The data obtained indicate that oxidases from the fruit are not responsible for causing oxidized flavors in strawberry ice cream.
2. Contrary to expectation, the samples showed less tendency to develop oxidized flavors the higher the iron content of the sample. Statistically this correlation is significant. This fact would indicate that the iron very probably is in combination with some milk constituent in the ferrous form and serves in this combination as an anti-oxidative catalyst.

One fact that has come to the attention of one of the authors recently may be interesting in this connection. Accretions of hemispherical shape and greenish color which had formed on the sides of 10-gallon cans in which superheated condensed skimmilk was sold, were brought to these laboratories for analysis and for a diagnosis of the causes of their formation. When ashed, the bulk of the ash seemed to be ferric oxide. Because of the color of the accretions they were macerated in distilled water, filtered and the filtrate tested for ferrous iron. A strong test for ferrous iron was obtained.

3. Insofar as these results show, the total copper content of the ice cream sample is not an index which will show whether ice cream samples will develop oxidized flavors. They do indicate, however, that the form in which the copper may exist in the samples may be of vital importance.

4. In regard to fat constants, such variations as existed between the initial and final Reichert-Meissl numbers and initial and final acetyl values determined on fat extracted from ice cream at the beginning and end of an approximately 60-day storage period do not show any correlation with the rate of development of oxidized flavors. There is a tendency, however, for the drop in iodine numbers of these fats to be greatest with those samples which develop the defect most rapidly. This is not, however, a significant correlation statistically. The trend in the iodine number changes points toward fat oxidation during development of the oxidized flavors.

5. These data indicate that strawberries do not contribute in causing the oxidized flavor in strawberry ice cream.

6. The ice creams made with dry skimmilk as a source of serum solids developed oxidized flavors to a significantly lesser extent than did those in which condensed skimmilk was employed.
II. A Comparison of Flavor Defects and Copper Contents of Strawberry Ice Cream in Which Commercial Dry Milk and Condensed Whole and Skimmilks (Prepared in Copper Pans) Were Employed as Sources of Serum Solids.\(^7\)

By E. W. Bird, O. R. Ausse, J. J. Willingham and C. A. Iverson\(^8\)

When the data of part I of this bulletin were obtained they indicated that certain factors involved in the cause of oxidized flavors in strawberry ice cream warranted further study. The number of samples investigated in part I were small (11 pairs); the data secured with regard to the role of strawberries in causing the flavor defect were not in agreement with others that had been reported; the role of copper seemed less important than had been stated by other workers, and it was considered that additional information was desirable concerning sources of serum solids.

For the reasons stated this problem was undertaken. It was hoped that if companion mixes, differing only to the extent that was necessary in order to have the additional serum solids furnished by dry skim, condensed skim and condensed whole milks, were frozen, it would be possible to obtain information relative to: (a) The effect of strawberries, (b) the role of added solids, (c) the effect of heat treatment of varying amounts of fat during the condensing process, (d) the effect of age of condensed milks and (e) the effect of copper content of the ice cream in the development of oxidized flavors in strawberry ice cream.

**EXPERIMENTAL**

**METHODS AND MATERIALS EMPLOYED**

**ANALYTICAL METHODS**

Copper analyses were the only ones determined on these samples. These were run by the same procedure as in part I except that instead of matching the copper diethylidithiocarbamate colors in aqueous solution in Nessler tubes the copper salt was dissolved in amyl alcohol (25) and the alcoholic solutions so obtained were compared with those from the standards in a Duboscq-Spencer colorimeter.

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\(^7\)Project 340 of the Iowa Agricultural Experiment Station.

\(^8\)The authors of this bulletin are indebted to Mr. Glenn Woods of the Des Moines Cooperative Milk Producers Association at Des Moines, Iowa; to Mr. Thomas Wright, of the Blue Valley Creamery at Cedar Rapids, Iowa and to their respective companies for the excellent cooperation they accorded in furnishing condensed milk for use in the experimental work presented in parts II and III of this bulletin.
MANUFACTURE OF ICE CREAM

The composition of the mixes, the method of assembling and processing the mixes, the type and amounts of fruit used, the types of samples (including controls) and the methods of judging were identical with those of part I. Likewise, these ice creams were made during the winter (October to April), the period during which the flavor defect is said to be most likely to occur.

FREEZING

This procedure differed from that employed in part I. Two quarts of plain mix were frozen in a 4-quart motor driven “hand” freezer for the control samples. A second 2-quart lot of the same mix was frozen with strawberries for the fruit samples. This practice was resorted to because with three comparisons each week considerably more strawberry ice cream would have been obtained than could have been disposed of commercially during the winter months.

SOURCES OF SERUM SOLIDS

Commercial dry skimmilk purchased through the usual outlets, condensed whole milk prepared in copper pans in one Iowa plant and condensed skimmilk prepared in copper pans by a second Iowa plant were used as sources of serum solids. It was not possible to obtain both types of condensed milk from any plant close enough to Ames to enable satisfactory shipping connections. For this reason skim and whole condensed milks were got from different companies.

RESULTS

The procedure in this study was to prepare three mixes as nearly alike as was possible, using the three sources of serum solids mentioned. During the first, third, fifth, etc., weeks fresh condensed milk was employed in the preparation of the mixes. The condensed milks used during the first, third, fifth, etc., weeks were held for 1 week in the 5-gallon cans (in which they were received) at a temperature of approximately 30°F., and these were used as “1-week old” condensed milks when the three samples were prepared during the second, fourth, sixth, etc., weeks. Fifteen such comparisons were made with “fresh” and 15 with “1-week old” condensed milks. A total of 30 weeks, therefore, was required to complete these freezing experiments. Inasmuch as these samples were frozen during the winter season, 2 years were needed to obtain the data presented.

COMPARISON OF COPPER CONTENTS OF FRUIT AND CHECK SAMPLES

The colorimetric determination of copper is a long and
tedious procedure. It was considered that if it could be shown that no appreciable difference existed between the copper analyses of fruit and check samples, it would be necessary to determine the copper in the fruit samples only. Eighteen pairs of samples fairly equally distributed among the dry skim, condensed skim and condensed whole milk samples were analyzed. A statistical treatment of the data showed that there was no significant difference between the copper analyses of the fruit and check samples. These data show, therefore, that the berries neither increase the copper content by incorporating additional copper nor decrease it appreciably by dilution. This lends assurance that in comparing fruit and control samples for oxidized flavor development, any difference that exists cannot be attributed to the variation of copper content between them.

**COMPARISON OF SAMPLES MADE WITH DRY SKIM, CONDENSED SKIM AND CONDENSED WHOLE MILK**

**OXIDIZED FLAVORS**

For convenience in determining rates of oxidation the time to develop oxidized flavor was divided into (a) 1 to 14 days, (b) between 15 and 28 days and (c) not developed. The dry skimmilk samples possessed better keeping quality in these trials, only one fruit sample having developed the defect and that in the (b) rate class. The condensed skim-milk samples were divided among the rate classes as follows9: (a) 14 samples, (b) 6 samples and (c) 9 samples, while those of the condensed milk were: (a) 1 sample, (b) 14 samples and (c) 15 samples.

The condensed whole milk samples were less susceptible toward the development of oxidized flavors, therefore, than were the condensed skimmilk samples. In general the condensed skimmilk samples, as received, were of poorer quality than were the condensed whole milk samples. Despite this fact, it appears that heat treatment of larger amounts of fat in copper pans in the manufacture of condensed whole milk than in the manufacture of condensed skimmilk does not render the ice creams prepared with the former more readily susceptible to oxidized flavor development than that prepared with the latter.

**FLAVOR SCORES**

It was considered that an analysis of the data from the condensed milk ice creams on flavor score alone would be of

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9There were but 29 samples here because the last “1-week old” sample was not frozen.
some interest in connection with the heat treatment of larger amounts of fat in the condensed whole than in the condensed skimmilk samples. This analysis showed that there was no significant difference between the initial scores of the two types of ice cream, that the rate of deterioration of the ice cream (based on initial and final scores, 28 days storage) made with condensed skimmilk was more rapid than that with condensed whole milk and that as regards the deterioration of the samples, there was a significant difference between the two types of ice cream in favor of that prepared with condensed whole milk.

These data would indicate either that heat treatment of larger amounts of fat during the condensing process does not render the ice cream made from it more susceptible to deterioration or that larger quantities of copper in the condensed skimmilk cause more rapid deterioration in the ice cream made from it and overshadow the effect of the heat treatment of the fat.

AGE OF CONDENSED MILK

There was no significant difference between the ice creams made with “fresh” or “1-week-old” condensed milk under the conditions of the experiment, concerning either development or non-development of oxidized flavors or the rate of development of oxidized flavors. When flavor score was considered there was a tendency for the ice cream made with the condensed milk held 1 week in the 5-gallon cans to exhibit better keeping quality than that made with the fresh condensed milk. This tendency was somewhat more pronounced with the samples containing condensed skimmilk than with those containing condensed whole milk. This may result either from a deposition of copper from the condensed milk onto the walls of the can (30) or to solution of iron from the cans (see part I).

COMPARISONS OF FRUIT AND CHECK SAMPLES

With all three types of ice cream samples there was no significant difference between fruit and control samples with respect to development or non-development of oxidized flavors. These data are in agreement with those of part I in that strawberries did not contribute toward development of oxidized flavors.

COMPARISON OF COPPER CONTENT OF SAMPLES VS. RATE OF DEVELOPMENT OF OXIDIZED FLAVORS

DRY SKIMMILK SAMPLES

Two control samples and only one fruit sample developed
oxidized flavors in this group, so that no correlation between copper content and rate of development existed. The maximum, minimum and mean copper analyses of this group were 2.5, 0.63 and 1.15 ppm., respectively. The fruit sample which developed the defect contained 1.33 ppm. copper.

**CONDENSED WHOLE MILK SAMPLES**

There was no significant difference statistically between the copper contents of the three rate classes, viz., (a) developed 1 to 14 days, (b) developed 15 to 28 days and (c) not developed. The means of the copper analyses of the three groups which are 1.29, 1.00 and 0.956 tend to show this, because of the little variation among them.

**CONDENSED SKIM MILK SAMPLES**

There was a significant difference between the copper contents of the (a) and (c) rate classes but not between the (a) and (b) rate classes. The means will again illustrate this: (a) 3.15 ppm., (b) 3.23 ppm. and (c) 1.93 ppm. When the probable upper limit for the non-appearance of oxidized flavors was calculated (from the condensed skim-milk sample data) the mean value 2.38 ppm., was obtained. A similar probable lower limit of the mean calculated for the developed group is 2.70 ppm. A range of 0.32 ppm. in these means might be considered to exist in which the development and non-development are problematical.

These figures are not presented with the idea of implying that such rigid fixed limits exist above or below which oxidized flavors will or will not develop but rather to show that these data, when compared with the condensed whole milk and dry skimmilk data in the two preceding paragraphs, definitely indicate that limits in copper content that would be set for development or non-development of oxidized flavors in strawberry ice cream will no doubt vary with the type of ingredients used and will be considerably different if based on the actual copper content of the ice cream rather than on the basis of copper added in ionic form (8, 9).

The three lots of data presented above show correlation between the appearance or non-appearance of the defect and the copper content of the samples in lots of ice cream containing condensed skimmilk but not in those containing dry skim or condensed whole milk.

**CONCLUSIONS**

1. The effect of heat treatment of different amounts of fat during condensation of skim or whole milks was appar-
ently of no influence on the keeping quality of ice creams made with condensed whole or condensed skimmilk.

2. Aging condensed milk (made in copper pans) for 1 week in 5-gallon cans at 30°F. did not affect the keeping quality of ice cream made from it as compared with ice cream made with the same milk when fresh. This was true with both condensed skim and condensed whole milks.

3. The keeping quality of ice cream made in these experiments was best for samples made with dry skimmilk, intermediate for those containing condensed whole milk from copper pans and poorest for those in which condensed skimmilk from copper pans was employed.

4. These experiments definitely indicate that strawberries do not contribute toward the oxidized flavor development in strawberry ice cream.

5. A significant correlation between development and non-development of oxidized flavor and copper content was found for those ice cream samples containing condensed skimmilk but not for those containing condensed whole or dry skimmilk. The mean copper analyses for the three types of ice cream were: That containing dry skimmilk 1.15 ppm., that containing condensed whole milk 2.54 ppm. and that containing condensed skimmilk 2.69 ppm. The data indicate that setting limits for copper contents above and below which the flavor will and will not develop depends on the type of ingredients employed, their method of manufacture and the state in which the copper exists. These data also indicate that these limits would be greatly different if based on actual copper content of the ice cream than if based on quantities of copper salts added to ice cream.

III. A Comparison of Flavor Defects and Copper Contents of Strawberry Ice Cream in Which Commercial Dry Milk and Condensed Whole and Condensed Skimmilks (from Stainless Steel Pans) Were Employed as Sources of Serum Solids.10

By E. W. Bird, J. J. Willingham and C. A. Iverson

Shortly after the completion of the experimental work presented in part II, one of the companies from which condensed milk had been purchased replaced its copper vacuum pan with one manufactured of stainless steel. The manager

10Project 480 of the Iowa Agricultural Experiment Station.
of this company was as interested in having a study similar to that made in part II conducted with condensed milks from stainless steel pans as were the authors of this paper. It was agreed that if such a study were conducted, the company would manufacture both condensed skim and condensed whole milks so that these would be available as sources of serum solids at the times needed.

The objectives of this project were the same for comparisons of dry skimmilk and condensed skim and whole milks from stainless steel pans as sources of serum solids as were those for the types of solids employed in part II.

EXPERIMENTAL

METHODS AND MATERIALS

The methods and materials here were like those of part II with three exceptions, viz., that condensed milks from stainless steel pans were used instead of condensed milks from copper pans, that both condensed milk types were purchased from one company rather than from two companies, and that the method of freezing was changed somewhat. The dry skimmilk was purchased on the open market as in the previous experiment.

The freezing method was as follows: The mixes were frozen in a direct expansion, triple dasher freezer. No flavoring was added. When approximately 100 percent over-run was obtained, half the ice cream was run from the freezer into a can in which vanilla was subsequently incorporated with it so that this portion could be sold as vanilla ice cream. Immediately after withdrawing half of the batch, the control samples were taken, and then the correct amount of berries was added to the freezer, and the fruit samples were drawn. This more nearly approximated commercial freezing conditions than did the procedure employed in part II.

RESULTS

In this part of the study the same procedures of manufacturing ice cream from "fresh" and "1-week-old" condensed milks were followed. Twenty comparisons were made with each so that a total of 40 weeks was required to complete the freezings. Since these data were again obtained during the "winter" period, 2 years elapsed before all the experimental work was completed.

COMPARISON OF COPPER IN FRUIT AND CHECK SAMPLES

In this section of the study 15 comparisons were made of
the copper contents of fruit and check samples. These were selected in equal numbers from the dry skim, condensed skim and condensed whole milk samples. Again no significant difference was found between the copper contents of the two types of samples. This strengthens the contention made in part II that berries neither raise nor lower the copper content and that such differences as may exist between fruit and control samples cannot be attributed to variations in the copper contents of the two types of samples.

COMPARISONS OF SAMPLES MADE WITH CONDENSED SKIM, CONDENSED WHOLE AND DRY SKIM MILKS

OXIDIZED FLAVORS

None of the samples prepared with either fresh or 1-week-old condensed milk of either type developed oxidized flavors. The 40 dry skim milk samples were divided into the following rate classes: (a) Developed 1 to 14 days, (b) developed 15 to 28 days, and (c) not developed. The number of samples in each class were: (a) 3, (b) 9 and (c) 28.

SCORES OF CONDENSED MILK SAMPLES

It was again considered of interest to compare the scores of the ice creams in which the two types of condensed milk were incorporated in order to check the effect of heat treatment of the fat during the condensing process. When fresh there was no significant difference between condensed whole and condensed skim milk ice creams. When the drop in score is considered, there is a significant difference between the ice cream samples containing condensed whole and condensed skim milk, with the latter deteriorating less rapidly. The ice creams made with 1-week-old condensed milks showed a tendency toward poorer keeping quality than those made with fresh condensed milks; this tendency was slightly more pronounced with condensed whole than with condensed skim milk.

These data indicate that the heat treatment (during the condensing process) of larger amounts of fat or of materials associated with the fat has a tendency to cause ice creams into which these larger quantities of heated fat are incorporated to show a tendency toward poorer keeping quality.

COMPARISON OF FRUIT AND CHECK SAMPLES

Regardless of the source of serum solids no significant difference exists between development or non-development of oxidized flavors in the fruit and the control samples. This
evidence, added to that presented in parts I and II, shows that strawberries do not cause the oxidized flavor in strawberry ice cream.

COMPARISON OF COPPER CONTENT AND RATE OF DEVELOPMENT OF OXIDIZED FLAVORS IN FRUIT SAMPLES

CONDENSED SKIM AND CONDENSED WHOLE MILK SAMPLES

No oxidized flavors developed in either class of samples. There was, therefore, no correlation between copper content and rate of development of these flavor defects. The mean copper contents of the two groups of ice cream samples were: Those containing condensed skim milk 0.68 ppm.; those containing condensed whole milk 0.67 ppm.

DRY SKIM MILK SAMPLES

No significant difference existed between the copper contents of the samples which did and those which did not develop the flavor defect. There were likewise no significant differences among the rate development classes divided (a) 1 to 14 days, (b) 15 to 28 days and (c) not developed. The mean copper values of the three classes again serve to illustrate this, viz., (a) 1.47 ppm., (b) 1.05 ppm. and (c) 1.27 ppm. The differences among these means are very small.

CONCLUSIONS

1. These data indicate that there is a significant difference between the keeping quality of ice cream (as indicated by drop in flavor score) containing condensed whole milk and condensed skim milk, the latter deteriorating less rapidly. This would indicate that heat treatments of larger amounts of fat during the condensing process may cause poorer keeping quality in those samples containing the larger amounts of heated fat.

2. There was a tendency toward poorer keeping quality of those ice creams made with condensed milks stored 1 week, at 30°F. in 5-gallon cans. This tendency was slightly more pronounced with those samples containing condensed whole milk than with those containing condensed skim milk. This again tends to indicate that heat treatment of larger amounts of fats (in the condensing process) may be important from a standpoint of quality in those condensed milks in which the copper content is not more important than the heat treatment.

3. There was a significant difference in oxidized flavor development between the ice creams containing dry skim-
milk and those containing the condensed skimmilks. The latter ice creams developed no flavor defects, whereas 12 of the 40 samples of ice cream containing dry skimmilk did.

4. These data again show that strawberries do not cause oxidized flavors in strawberry ice cream.

5. No significant difference was obtained among the copper contents of samples of ice cream containing dry skimmilk when these were considered from the standpoint of developing or not developing the flavor defect or of the rate of development of oxidized flavors. None of the samples containing the condensed milks prepared in stainless steel pans developed the flavor defect.

GENERAL CONSIDERATION OF THE DATA FROM PARTS I, II, AND III

It was considered that a treatment of all the data obtained in the three sections of this bulletin might throw some light on the general aspect of oxidized flavor developments in ice cream. For this reason the following discussion is presented.

FREQUENCY OF OCCURRENCE OF OXIDIZED FLAVORS IN STRAWBERRY ICE CREAM WHEN DIFFERENT TYPES OF SOURCES OF SERUM SOLIDS ARE EMPLOYED

It is appreciated that inasmuch as the five sources of serum solids employed in these studies were not used simultaneously, the data presented in table 3 are not all directly comparable. It is considered, however, that they may serve as a provisional guide when comparisons of serum solids sources are desired.

<table>
<thead>
<tr>
<th>Table 3. A comparison of the percentages of strawberry ice cream samples, containing different kinds of serum solids, that develop and do not develop oxidized flavors.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Serum solids sources</td>
</tr>
<tr>
<td>----------------------</td>
</tr>
<tr>
<td>Commercial dry skimmilk</td>
</tr>
<tr>
<td>Condensed skimmilk from copper pans</td>
</tr>
<tr>
<td>Condensed whole milk from copper pans</td>
</tr>
<tr>
<td>Condensed skimmilk from stainless steel pans</td>
</tr>
<tr>
<td>Condensed whole milk from stainless steel pans</td>
</tr>
</tbody>
</table>

COMPARISONS OF OXIDIZED FLAVORS IN FRUIT AND CONTROL SAMPLES

When all the data were treated statistically, as a single
class, no significant difference was shown between fruit and control samples with regard to development or non-development of oxidized flavors. When rate of development of flavor defect (in rate classes designated in parts II and III) was considered there was a highly significant retardation of rate of development of oxidized flavors in the fruit samples.

It follows then that in addition to the fact that fruit does not cause the defect, these data show that it definitely retards the rate of development of these flavors in those samples in which they do develop. This fact is definitely at variance with the statements of other investigators (see literature review).

COMPARISON OF ICE CREAM SAMPLES CONTAINING DRY SKIMMILK PREPARED IN THE THREE STUDIES PRESENTED

When this statistical treatment was made it was found that there was a highly significant difference in the development of oxidized flavors in ice creams containing dry skim milk purchased during different years. This is illustrated by the percentage of samples (containing dry skimmilk) which developed oxidized flavors, viz., part I, 18.2 percent, part II, 6.3 percent and part III, 30.8 percent.

COMPARISON OF COPPER ANALYSES AND OXIDIZED FLAVOR DEVELOPMENT

When the entire group of fruit samples was pooled it was found that a significant difference existed between the copper content of the samples which did not develop the flavor defect and those which did but that there was no significant difference between the copper contents of those samples in which the defect developed in 1 to 14 days and those in which it developed in 15 to 28 days. The means of these rate classes serve to indicate this: 1 to 14 days, 2.41 ppm., 15 to 28 days, 2.05 ppm. and no development, 1.05 ppm. The probable upper limit of the mean calculated for those samples which may not be expected to develop the flavor is 1.18 ppm. copper, and the probable lower limit calculated for those which may be expected to develop is 1.88 ppm. It would appear then that in the region between 1.18 and 1.80 ppm. copper these experiments furnish no information as to whether the defect will develop or not. In this region causes other than copper content are free to operate—copper content is not critical.

The authors of this paper would like to emphasize again
that it is not their intention to postulate that the above limits are at all rigid or that they may be indiscriminately applied to all cases. It is appreciated that the data pooled are very heterogeneous and that because of this fact they approximate a group of samples, picked up in the open market, concerning which the history is not known. It is believed, however, that because of their number and because the history of the various groups of data is known they permit a more nearly precise forecast than any others that have as yet been presented.

STATE IN WHICH COPPER EXISTS IN THE ICE CREAM SAMPLES

It was noted that in a number of groups of data presented the copper contents of the samples and the rate of development of off flavors show no correlation. It will likewise be noted that the mean copper values for certain groups of samples which do not develop the flavor defect is higher than those in other groups in which the flavor defect occurred between 1 and 14 days. This can be illustrated by comparing the means of the ice creams made with (a) condensed whole milk from copper pans, 1.30 ppm., and (b) condensed skim milk from copper pans, 3.15 ppm., which did not develop the defect, with the means of the dry skimmilk samples (part III), 1.47 ppm., which developed the defect in 1 to 14 days. These data indicate that the gross quantity of copper is not the sole criterion by which it may be judged whether or not the flavor defect will occur but that the state in which the copper exists in the ice cream is of importance.

This is in agreement with the recent observation of Brown et al. (1) that if equivalent amounts of copper (as salt solutions) are added to milk susceptible to oxidation, before and after pasteurization, that added after pasteurization causes the more pronounced oxidized flavor development. The reason for this may be explained in part at least by the following observation: "Ash free" casein precipitated with acetic or hydrochloric acid as many as 10 and 12 times, with solution in ammonium hydroxide between precipitations, was offered for trial when a copper-free source of protein was desired for anemia inducing rations. These samples could not be used in the rations because they contained too great quantities of copper. It would seem that much of the copper might be united with proteins and thus be in a form which either did not catalyze or but feebly catalyzed oxidations.

The possibility that only small amounts of fat need be
oxidized for the production of oxidized flavors was suggested by Ross. The fact that Tracy et al. showed that the susceptibility to the development of the flavor increased with fat content of ice cream and that the tendency (in part I) was for a greater drop in iodine numbers of the fat extracted from those samples in which the defect developed at the more rapid rate, indicates that fat oxidation may be involved. The slow reaction of copper salts available in condensed milks made in copper pans may, as was suggested by Ross, carry these small quantities of fat sufficiently through the oxidation-induction period that little further oxidation is necessary.

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