small portions at first. Be sure suction is on before filtering.) Wash beaker with several small portions of distilled water and add them to the crucible.

Transfer the filtrate to a 400 ml. beaker, decolorize with a few drops of bromine water, and boil to expel excess bromine. Determine the metals in the usual way.

B. Blank (soluble barium, calcium, or strontium salts present in the pigment).—Weigh 5.000 grams of the color pigment into a 250 ml. volumetric flask. Add 175 ml. of water containing 5 ml. of glacial acetic acid. Stopper the flask and shake violently for ca. 5 minutes; dilute to volume and filter through a dry filter. Determine metals on a 200 ml. aliquot (equivalent to 4 grams of color pigment).

The difference between the percentage of metals found in A and in B is the percentage of metal, combined organically.

This method gives doubtful results for D&C Red No. 15, D&C Red No. 16, and D&C Red No. 31, because the proposed determination of the blank breaks down the organic combination of metal and dye in these pigments. For other pigments containing certified coal-tar colors it appears to give results corresponding to at least 98 per cent of the theoretical, based on the pure dye content of the pigment, as determined by titration with titanium trichloride.

Since most lakes of calcium, barium, and strontium are made by boiling the sodium salt of the dye with the appropriate metallic chloride this method has been found to be practical for checking the completeness of the conversion.

BACTERIOLOGICAL AND PHYSICAL CHANGES OCCURRING IN FROZEN EGGS

INFLUENCE OF DEFROSTING AND PROLONGED STORAGE ON BACTERIAL COUNT AND ON ODOR

By Roy Schneiter,* M. T. Bartram, and H. A. Lepper (U. S. Food and Drug Administration, Washington, D. C.)

The bacterial changes occurring in frozen eggs during shipment and under conditions of commercial storage have received but scant attention within recent years, and a search of the literature reveals little information on this subject.

Stiles and Bates[1] packed, under laboratory conditions, various grades of eggs in approximately 50 ml. quantities, and stored them at 10°F. for a period of one year. Flasks of each product were removed at intervals for bacteriological examination. The study revealed little variation in the bacterial content of strictly fresh and commercially fresh frozen eggs during this period, while the change in checks, cracks, dirties, spots, and rots was even less pronounced, although a very gradual decline in the average total count of the various grades was apparent.

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[1] U. S. Dept. Agriculture Bull. 158 (1912).

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Nielsen and Garnatz[2] obtained a sharp drop in the bacterial content of frozen eggs containing 14 per cent of salt when held at −18°C. The greatest reduction occurred in 41 days, and thereafter (up to 208 days) little change was evident. Frozen eggs with 10 per cent of sugar failed to show a reduction in bacterial count until after 114 days, and there was a continued decrease through the 203rd day.

The experiments presented here were undertaken to show the bacterial and physical changes occurring in eggs of good and poor quality during the usual conditions of preparation, freezing, shipment, and storage, and during prolonged storage. The influence of improper methods of preparation and of insanitary plant practices on the quality of frozen eggs was ascertained. The effect on these products of thawing and refreezing was also studied to determine the influence of accidental defrosting.

EXPERIMENTAL

Preparation.—All the experimental packs, consisting of 30 pound cans of eggs, were prepared during the latter part of May or early June in egg-breaking establishments in Kansas City under the usual commercial conditions, and they were frozen, shipped, and stored in the regular channels with commercial packs. They were divided into six groups, as shown in Table 1.

Samples of egg batter for bacteriological examination were removed from each can in the experimental packs immediately after the container was filled. These samples, well-stirred, were taken with aseptic precautions from the containers by means of sterile straight glass tubes (0.25″ ×18″). The odor and appearance of the product in each can were noted, after which all cans, except pack F, were immediately placed in a sharp freezer at from −10 to −14°F. The cans in pack F were slow frozen at 0°F. The cans were resampled approximately 60 hours after being placed in the freezer by drilling, with an electric drill and a one inch bit, three cores equidistant between the side and center of each can and one-third of the periphery apart. The chips from the top layer were discarded, and the others were collected in sterile jars. The sampling procedure used for these and subsequent samples was the same as that recommended by Schneiter.[3] The frozen eggs were maintained at a temperature of from +5 to −13°F. for approximately three months and were then shipped to Washington, D. C. with a commercial car-lot shipment. Two special recording thermometers enclosed in regular 30 pound egg cans were included with the shipment, and records of re-icing were obtained. The temperatures recorded during transit varied from 24° to 32°F., but never exceeded the latter figure. Immediately upon receipt the eggs were examined, samples

[2] First Food Technical Conference, 1940, pp. 289–294.
[3] *This Journal*, 22, 625 (1939).

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were removed, and the cans were placed in commercial storage at from 0° to −5°F.

Approximately one month after shipment one can of each type of product (whites, yolks, or whole eggs) from each of packs A and B, two cans from each of packs C and E, and one can from each of D and F were

TABLE 1.—*Experimental packs*

PACKS	NO. OF CANS	TYPE OF PRODUCT	QUALITY OF SHELL EGGS	PLANT SANITATION
A	6 6 6	Whites Yolks Whole	Selected eggs from good grade of current receipts consisting of fresh, sound, clean, uniform shell eggs.	Good
B	6 6 6	Whites Yolks Whole	Average run of good grade current receipts; included selected eggs as well as small, weak, and slightly cracked eggs.	Good
			Good egg batter % plus rots (%)	Good
C	1	Whole	Good eggs 97 Rots 3	
	1	Whole	Good eggs 95 Rots 5	
	1	Whole	Good eggs 90 Rots 10	
	1	Whole	Good eggs 75 Rots 25	
	1	Whole	Good eggs 52 Rots 48	
	1	Whole	Good eggs 0 Rots 100	
D	2	Whole	Included checks, cracks, leakers, and washed dirties.	Good
E	1	Whole	Average run of good current receipts plus washed dirties	Fair
	1	Whole	Second-grade egg batter plus checks, cracks, and leakers.	Fair
F	2	Whole	Low-grade breaking stock and candling rejects.	Poor

removed from storage and placed on the roof of the storage plant in the sun, where they remained for six hours at temperatures ranging from 80° to 100°F. The cans were then removed to the interior of the building and left overnight (18 hours) at 64°F., at which time the contents were thawed, except for a mushy central core. The temperatures of the well-stirred product ranged from 37° to 46°F. The eggs were sampled and examined, and the cans were replaced in the freezer for refreezing, after which they were again examined.

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After storage for one year in Washington the cans were sampled and examined. All of them, with the exception of one of each type of product from packs A and B, four cans from C, and one can from each of D and F, were removed from storage, thawed, and refrozen in the same manner as that outlined above except that the temperature in this instance did not exceed 95°F. during the thawing period. The temperatures of the product after exposure on the roof ranged from 36° to 44°F., and they had risen to 44°–60°F. by morning. Samples were removed from the liquid eggs and again after refreezing.

The cans noted previously, which were not removed for defrosting, were maintained in storage for a total period of six years, with examination after five and six years of storage.

Bacteriological and Organoleptic Examination.—All samples of egg batter and frozen egg drillings collected for bacteriological studies were examined according to the procedures outlined by Schneiter.[3] Total plate counts of viable microorganisms were determined by plating appropriate dilutions on dextrose agar with incubation at room temperature (25°–32°C.) and at 37°C. for 72 hours. The incidence of the coliform group of bacteria was determined by lactose broth presumptive test with partial confirmation on Levine's eosin-methylene blue agar. Hemolytic staphylococci and streptococci and anaerobic microorganisms were determined on veal blood agar and alkaline cooked-meat medium, respectively; however, the results were of no apparent significance and are not included in this study.

The results of the bacteriological and physical examination are given in Table 2. The individual cans of packs A and B are not listed, but the results are summarized. The counts obtained at 37°C. are also not given, but in general they were consistently lower than those obtained at the lower incubation temperatures.

The results of the first analysis made on the unfrozen batter are typical for the various types of eggs used. All the eggs in pack A were normal, and the bacteriological counts ranged from less than 10,000 to 30,000 per gram, with members of the coliform group never present in dilutions greater than 1:1000. In pack B the counts were somewhat higher, but not in excess of 300,000, except in the case of the whole eggs, where counts up to 2,000,000 per gram were obtained. A portion of the batter used in filling the six cans in this lot had stood in the churn over the lunch period (30 minutes) under hot summer conditions, and the same conditions prevailed in the first can of pack F. It presented a noticeably strong odor although the bacterial content is not high. The eggs in pack C were definitely putrid, and the counts correspondingly high, while the results on packs D and E indicate normal eggs with the variable counts encountered when dirty eggs or cracked leaky eggs are used.

Influence of Freezing and Shipment.—The results of the second analysis show decreases in bacterial count typical of those obtained on freezing

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TABLE 2.—*Influence of shipment and defrosting on odor and bacterial content of frozen eggs under short-time storage*

PACK	TYPE	FIRST ANALYSIS BEFORE FREEZING			SECOND ANALYSIS 2½ DAYS AFTER FREEZING			THIRD ANALYSIS AFTER SHIPPING 2½–3 MONTHS AFTER PACKING			AFTER ONE YEAR IN STORAGE								
											FOURTH ANALYSIS BEFORE DEFROSTING			FIFTH ANALYSIS DEFROSTED			SIXTH ANALYSIS REFROZEN		
		ODOR	COUNT/GM.†	COLIFORM*	ODOR	COUNT/GM.	COLIFORM	ODOR	COUNT/GM.	COLIFORM	ODOR	COUNT/GM.	COLIFORM	ODOR	COUNT/GM.	COLIFORM	ODOR	COUNT/GM.	COLIFORM
A	Whites	(1) Normal	17,000	+2	(1) Normal	7,000	+1	(1) Normal	3,950	−(1 gm.)	(3) Normal	200	−	(5) Normal	1,000	−	(5) Normal	152	−
	Yolks	(1) Normal	28,000	+1	(1) Normal	19,000	+1	(1) Normal	7,300	+1	(3) Normal	9,000	+1	(5) Normal	13,200	−	(5) Normal	5,740	+1
	Whole	(1) Normal	10,000	+1	(1) Normal	1,500	−(0.1 gm.)	(1) Normal	1,600	−	(3) Normal	700	+1	(5) Normal	2,860	−	(5) Normal	1,460	+1
B	Whites	(1) Normal	150,000	+4	(1) Normal	77,000	+3	(1) Normal	64,300	+2	(3) Normal	47,300	+1	(5) Normal	122,000	+1	(5) Normal	115,000	+1
	Yolks	(1) Normal	128,000	+4	(1) Normal	111,500	+4	(1) Normal	215,000	+3	(3) Normal	1,329,000	+3	(5) Normal	2,082,000	+3	(5) Sl. Putrid	19,969,000	+3
	Whole	(2) Normal	1,800,000	+4	(1) Strong	1,730,000	+5	(1) Strong	3,200,000	+5	(3) Strong	32,210,000	+6	(5) Putrid	18,400,000	+3	(5) Putrid	22,520,000	+5
C	*per cents* rots 3	Stale	16,000,000	+6	Stale	6,600,000	+6	Stale	42,000,000	+7	(4) Putrid	256,000,000	+7	Putrid	220,000,000	+6	Putrid	590,000,000	+6
	rots 5	Putrid	29,000,000	+6	Putrid	8,900,000	+6	Putrid	60,000,000	+7	(4) Putrid	128,000,000	+7	Putrid	340,000,000	+7	Putrid	420,000,000	+7
	rots 10	Putrid	22,000,000	+6	Putrid	8,000,000	+6	Putrid	65,000,000	+7	Putrid	158,000,000	+7						
	rots 25	Putrid	150,000,000	+6	Putrid	8,000,000	+6	Putrid	23,000,000	+7	Putrid	80,000,000	+7						
	rots 48	Putrid	350,000,000	+6	Putrid	53,000,000	+7	Putrid	120,000,000	+8	Putrid	151,000,000	+7						
	sour 100	Sour	270,000,000	+6	Sour	240,000,000	+7	Sour	52,000,000	+8	Putrid	91,000,000	+7						
D	Whole	Normal	10,000	+2	Normal	62,000	+2	Normal	10,000,000	+4	Strong	29,600,000	+4						
	Whole	Normal	4,900,000	+6	Normal	1,600,000	+6	Normal	5,300,000	+6	(4) Putrid	60,000	−	Putrid	47,000,000	+6	Putrid	1,100,000	−
E	Whole	Normal	190,000	+4	Normal	140,000	+4	Normal	560,000	+5	(4) Normal	7,000	−	Normal	8,000,000	+5	Sl. Taint	200,000	−
	Whole	Strong	18,000,000	+6	Strong	1,900,000	+5	Strong	21,000,000	+6	(4) Putrid	35,000,000	+6	Putrid	13,000,000	+6	Putrid	52,000,000	+7
F	Whole	(2) Strong	680,000	+4	Strong	470,000	+4	Strong	5,100,000	+5	(4) Stale	10,000,000	+5	Stale	16,000,000	+4	Strong	53,000,000	+3
	Whole	Normal	2,800,000	+5	Normal	270,000	+5	Normal	8,600,000	+5	Sl. Strong	2,000,000	+4						

* Reciprocal of dilution; i.e., 2 =positive in 1:100 dilution, etc.; − =negative in 1:10 dilution.
† All plates incubated at room temperature (25°–32°C.).
(1) Average of 6 cans.
(2) Stood 30 minutes before filling (summer temperature).
(3) One can from each group defrosted and refrozen, after third analysis.
(4) Can defrosted and refrozen, after third analysis.
(5) Average of 5 cans.

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the egg batter. The odor of the eggs in the various packs remained unchanged except for the whole eggs of pack B, which had become noticeably strong. The bacterial counts on these had increased slightly over those of the batter.

The results of the third analysis, made after a storage period of approximately three months and after shipment to Washington, D. C., indicate that except for a slightly decreased count no significant change had occurred in the good eggs of pack A. However, in the remaining packs the bacterial counts had undergone slight to marked increases, although in no instance could any change in the odor of the product be noted. This condition indicates that the increase in bacteria occurred during the shipping and that although the temperatures, as shown by the recording thermometers, never exceeded 32°F., they were sufficiently high for bacterial increases to occur in those packs that contained abnormal eggs and had high original bacterial content. This should be contrasted with the eggs of good quality.

Influence of Defrosting.—The 12 cans defrosted after the third analysis were sampled while in the thawed state and again after refreezing. These results are given in Table 3. In general, all of the eggs so treated showed slight increases in bacterial count while in the liquid state, with the most marked increase occurring in packs A and B. The number of organisms observed in the mixed defrosted product in no instance exceeded 24 times those in the product prior to thawing. This is in sharp contrast to the results obtained by Brownlee and James,[4] who observed average increases of 180, 360, and 660 per cent in samples taken from the center, midway, and outside positions of cans during a 24-hour defrosting period in water at 11.5°C. On refreezing, the total counts, except for the can of whole eggs in pack B, returned to the levels observed on the third analysis. The odors of the thawed eggs in pack A and the whites and yolks of pack B were normal, while the defrosted can of whole eggs in pack B was definitely putrid, as were the eggs in pack D and in one can of pack E.

The results of the examination after the eggs had been in storage one year are recorded under the fourth analysis (Table 2). Continued decreases in the bacterial counts in pack A were obtained, particularly in the whites and whole eggs, and all were normal in odor and appearance. The cans that had been defrosted one year previous to this analysis were entirely similar in all respects to the ones not so treated. Pack B showed some decrease in the average counts on the whites, while the yolks and whole eggs had undergone increases in bacterial content. All of the eggs in this pack remained normal except that the whole eggs had the same "strong" odor noted on freezing and the previously defrosted can was putrid.

The eggs in packs C, D, E, and F showed definite increases in bacterial counts with the exception that one of the defrosted cans in each of D and

[4] Proc. Seventh World's Poultry Congress, 1939, pp. 488–492.

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TABLE 3.—*Results of preliminary defrosting on odor and bacterial content of frozen eggs*

PACK	TYPE	BEFORE DEFROSTING			THAWED			REFROZEN		
		ODOR	COUNT*/GRAM	COLIFORM†	ODOR	COUNT/GRAM	COLIFORM	ODOR	COUNT/GRAM	COLIFORM
A	Whites	Normal	1,900	Neg.	Normal	4,900	Neg.	Normal	6,700	Neg.
	Yolks	Normal	7,000	Neg.	Normal	110,000	0.5	Normal	27,000	0.5
	Whole	Normal	2,000	Neg.	Normal	8,000	2	Normal	4,000	Neg.
B	Whites	Normal	6,000	2	Normal	110,000	2	Normal	76,000	2
	Yolks	Normal	120,000	2	Normal	2,900,000	2	Normal	250,000	2
	Whole	Normal	4,100,000	6	Sl. putrid	27,000,000	6	Putrid	13,000,000	5
C	3% rots	Stale	42,000,000	7	Putrid	18,000,000	7	Putrid	14,000,000	7
	5% rots	Putrid	60,000,000	7	Putrid	20,000,000	5	Putrid	17,000,000	6
D	Whole	Normal	5,300,000	6	Stale	7,000,000	6	Putrid	6,800,000	6
E	Whole	Normal	560,000	5	Normal	310,000	6	Normal	100,000	4
	Whole	Strong	21,000,000	6	Sl. putrid	9,600,000	5	Strong	7,700,000	6
F	Whole	Strong	5,100,000	5	Stale	1,500,000	4	Putrid	2,800,000	4

* Incubated at room temperature (25°–30°C.).
† Reciprocal of dilution.

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E gave a much lower count. No changes in odor from the last analysis were noted in the cans not previously defrosted.

Immediately following the fourth analysis all of the cans, with the exception of 12 cans that were to be continued in storage, were thawed and examined, then refrozen and again examined. These results are recorded under the fifth and sixth analyses, respectively. Increased counts of 5-, 1.5-, and 4-fold were observed in the whites, yolks, and whole eggs, respectively, of pack A while in liquid state, but the count returned to the prethawed level, or below, on refreezing. All of the eggs in this pack remained normal, although this was the second defrosting for one can in each of the product types. In pack B the average counts of the yolks underwent a 10-fold increase during thawing and refreezing, and the contents were observed to be either quite strong or slightly putrid. The egg whites in this pack remained normal, and the bacterial counts were not significantly changed by the defrosting.

All the eggs in the remaining packs showed increases in bacterial count, and all were abnormal in odor. In all the analyses it will be observed that the coliform content roughly parallels the total bacterial count and therefore serves as a valuable adjunct in measuring the quality of the product.

Effect of Prolonged Storage on Frozen Eggs.—The 12 cans of eggs from the previous experiment that were not subjected to defrosting after one year in storage, consisting of one can from each type of product in packs A and B, four cans from pack C, and one can from each of packs D and F, were continued in storage for a total period of six years, with examination at the end of five years and again after six years in storage. At this latter date the cans themselves were quite deteriorated, and it seemed advisable to terminate the experiment.

The results of the organoleptic and bacteriological examination of these samples are given in Table 4. The first three analyses are the same as those previously recorded in Table 2 and will not be discussed again.

The physical condition of the eggs after storage for five and six years is quite interesting. Except for the presence of large ice crystals and a leathery texture, which did not affect the edibility of the product after defrosting, all the eggs in packs A, B, D, and F were quite normal in appearance and in odor. The whole eggs in pack B and the eggs in pack C (containing rots) presented a disintegrated appearance and a strong or putrid odor. The odor was much less intense than was the case at the end of one year's storage and became progressively less pronounced after six years' storage.

The bacterial count of the eggs of good quality (pack A) underwent but little change in the five-year period between the fourth and sixth examinations. The egg whites in packs A and B were practically free from bacteria, containing less than 10 per gram, which, in the case of pack B, was a definite decrease. This was undoubtedly due to the presence of bacteri-

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TABLE 4.—*Effect of prolonged storage on odor and bacterial content of frozen eggs*

PACK	TYPE	IMMEDIATELY AFTER FREEZING			THREE MONTHS' STORAGE			ONE YEAR'S STORAGE			FIVE YEARS' STORAGE			SIX YEARS' STORAGE		
		ODOR	COUNT/GRAM†	COLI-FORM*	ODOR	COUNT/GRAM	COLI-FORM	ODOR	COUNT/GRAM	COLI-FORM	ODOR	COUNT/GRAM	COLI-FORM	ODOR	COUNT/GRAM	COLI-FORM
A	Whites	Normal	7,000	+2	Normal	5,700	−	Normal	<10	−	Normal	<10	−	Normal	<10	−
	Yolks	Normal	17,000	+2	Normal	5,000	+2	Normal	7,900	+1	Normal	4,000	+2	Normal	2,700	−
	Whole	Normal	3,000	+2	Normal	3,000	−	Normal	600	+1	Normal	200	−	Normal	400	−
B	Whites	Normal	42,000	+3	Normal	58,000	+2	Normal	186,000	+2	Normal	<1,000	−	Normal	<10	−
	Yolks	Normal	100,000	+5	Normal	270,000	+3	Normal	84,000	−	Normal	33,000	+2	Normal	6,500	+4
	Whole	Strong	1,100,000	+5	Strong	6,200,000	+7	Strong	1,700,000	+5	Strong	1,400,000	+5	Strong	106,000	+4
C	10% rots	Putrid	8,000,000	+6	Putrid	65,000,000	+7	Putrid	158,000,000	+7	Putrid	2,500,000	+4	Putrid	660,000	+5
	25% rots	Putrid	8,000,000	+6	Putrid	23,000,000	+7	Putrid	80,000,000	+7	Putrid	14,000,000	+7	Putrid	3,300,000	+6
	48% rots	Putrid	53,000,000	+7	Putrid	120,000,000	+8	Putrid	151,000,000	+7	Putrid	14,000,000	+7	Putrid	6,200,000	+6
	100% sour	Sour	240,000,000	+7	Sour	52,000,000	+8	Putrid	91,000,000	+7	Putrid	3,000,000	+6	Putrid	700,000	+5
D	Checks, etc.	Normal	62,000	+2	Strong	10,000,000	+4	Strong	29,600,000	+4	Normal	220,000	+3	Normal	<1,000	−
F	Rejects	Normal	270,000	+5	Normal	8,600,000	+5	Strong	2,100,000	+4	Normal	130,000	+2	Normal	11,000	−

† Plates incubated at room temperature (25°–30°C.).
* Reciprocal of dilution; i.e., +3 =positive in 1:1000 dilution; − =negative in 1:10 dilution.

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cidal lysozyme in the egg white, and it may also account for the low counts encountered in the whole eggs as compared to the yolk containing no egg white. The whole eggs in pack B yielded considerably fewer organisms after six years' than after one year's storage; however, the count was significantly higher than that on the other eggs in either pack A or B and still reflected the abuse to which those eggs were subjected when they were held in the churn prior to being placed in the cans.

The bacterial counts of the eggs containing varying percentages of rots likewise underwent a decrease during the five-year period, but they were still of such magnitude that the eggs could probably not be considered an acceptable product. The counts in packs D and F had decreased to a level similar to those of the eggs of good quality of pack A, and except for the high count and strong odor encountered immediately after shipment and after one year's storage, these eggs were never an unsatisfactory product.

SUMMARY AND CONCLUSIONS

Studies were undertaken in order to ascertain the bacterial and physical changes occurring in eggs of good and poor quality during the usual conditions of preparation, freezing, commercial storage, and prolonged storage, and the influence on the quality of frozen eggs of insanitary plant practices and improper methods of preparation. Commercial 30 pound cans of frozen eggs were prepared from eggs of good quality, selected from average fresh shell stock, and from several types of eggs of poor quality, e.g., checks, cracks, leakers, washed dirties, and those containing varying percentages of rots. These experimental packs were subjected to the usual conditions of shipment and storage and to intentional defrosting.

Bacterial and physical changes observed lead to the following conclusions:

(1) Frozen eggs of good quality are able to withstand at least two complete thawings and refreezings without significant change in bacterial content or without acquiring abnormal appearance or odor.

(2) Eggs of poor quality, including cracks, leakers, and dirty eggs, usually have high bacterial counts. This condition leads to progressive decomposition of the product unless it is rapidly frozen and maintained in the frozen condition. Insanitary plant practices and improper methods of preparation of the egg batter are conducive to rapid decomposition, especially when freezing is delayed or prolonged.

(3) Prolonged storage of frozen eggs over a period of six years resulted in a considerable reduction of the bacterial content, but the total count still served as a reliable index of the original quality of the product. The counts after six years' storage ranged from 300 per gram in whole eggs of good quality to over 6,000,000 in second grade eggs containing 48 per cent rots. The physical condition of the frozen egg products did not change

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during prolonged storage, except for the formation of ice crystals and small leather-like lumps of separated egg solids in the whole eggs and egg yolks. The odor remained unchanged from that recorded immediately after freezing although that of the putrid eggs became less intense.

(4) Three per cent of rots, which was the lowest amount used in these studies, could be readily detected by an experienced egg examiner.

(5) The total plate count and the coliform index are roughly parallel, and each may serve as a reliable index of the original quality of the product.

(6) There is a rapid reduction in the numbers of viable microorganisms in frozen egg whites, which may be attributed to the presence of the bactericidal lysozyme.

SEMIMICRO METHOD FOR DETERMINATION OF SULFUR IN ORGANIC SUBSTANCES

By J. H. JONES (Cosmetic Division, U. S. Food and Drug Administration, Washington, D. C.)

The oxidation of organic sulfur compounds by a modification of Kahane's method, followed by titration of the sulfate produced with barium chloride, using tetrahydroxyquinone as an indicator, has been found to be a simple, rapid method for the determination of semimicro amounts of organic sulfur.

Kahane and Kahane[1] used a mixture of nitric, perchloric, and iodic acids to oxidize organic sulfur compounds. They determined the sulfate formed by precipitation as barium sulfate, after reducing the iodate remaining in the digestion mixture to iodide. Since either iodate or iodide interferes in the proposed titration of sulfate, iodic acid was not used in the proposed method. However, a number of organic sulfur compounds can be quantitatively oxidized by a mixture of nitric and perchloric acids alone. Other compounds that do not give quantitative results with the nitric-perchloric acid mixture are quantitatively oxidized if treated with aqua regia before the digestion with the nitric-perchloric acid mixture. The proposed procedure, therefore, specifies the use of aqua regia.

Many organic compounds react vigorously if digested with a mixture of concentrated nitric and perchloric acids. It is necessary, therefore, to begin the digestion in dilute solution and to concentrate the oxidation mixture gradually by boiling. With this procedure, the oxidation proceeds smoothly and the mechanical losses due to rapid reaction, shown by Wolesensky[2] to be the chief cause of low results in the determination of sulfur in rubber by Kahane's method, are avoided.

Nitrate ion in considerable concentration interferes with the proposed

[1] *Bull. Soc. Chim.*, (5), 1, 280 (1934).
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