a light solution of sodium chloride—is treated hourly with a measured quantity of sodium hypochlorite, so that it constantly contains from 1 to 3 p. p. m. of free chlorine (Table IV); and the fillets emerging from this apparatus are in excellent condition to be packed into the individual one-pound cartons or wrapped in transparent Moistureproof Cellophane and deposited in the 10-pound dealer packages (Table V).

Table IV—Effect of Chlorinating 35° Salinometer Sodium Chloride Brine, in Brining Machine

Test	Count per Cubic Centimeter[a]		Per Cent Reduction
	Before chlorinating[b]	After chlorinating[c]	
1	320,000	1,450	99.54
2	213,500	1,350	99.36
3	245,000	27,000	88.97
4	170,000	Sterile	100.00
Average	237,100	7,450	96.85

[a] Method of counting: In making a count on fish flesh a comparatively large sample—usually 10 grams—is mascerated with knives and forceps and placed in a flask with 100 cc. of sterile water, which is later further diluted sufficiently to assure ease of counting. One cubic centimeter of the diluted sample is plated on Bacto Nutrient Agar, and incubated 48 hours at 20° C. The count is recorded in bacteria per gram of fish. In making a count on fish slime or other liquids or fluids, the method is the same, except that the results are given as bacteria per cubic centimeter of sample.

[b] Freshly-mixed brine, through which fillets had been passed for about 6 hours, before the samples were taken. Average temperature about 13° C.

[c] Chlorination accomplished by adding sodium hypochlorite to bring residual chlorine content to about 5 p. p. m. Counts in this column represent average of samples—the clear liquid was in all cases sterile, but there were many small particles of flesh, mucous, etc., which apparently had not yet been sterilized by the action of the chlorine, which was added 15 to 20 minutes prior to sampling.

Table V—Effect of Passing Haddock Fillets for 20 Seconds through 35° Salinometer Sodium Chloride Brine Chlorinated to 5 p. p. m. of Residual Chlorine by Addition of Sodium Hypochlorite

Test	Count per Gram		Per Cent Reduction
	Before brining	After brining	
1	111,000	77,000	30.63
2	236,000	114,000	51.65
3	1,790,000	413,000	76.92
Average			53.06

Throughout the plant all surfaces with which the product may come in contact are smooth, non-porous, and easily cleansed. Floors are of granolythic concrete, sloped to frequent drains. Walls are enameled white, with a 5-foot dado of gray enamel, and a sanitary coping. Ceilings, also, are white. Inner walls and ceilings of refrigerated rooms are of mastic finish enameled white. All equipment is of monel metal, aluminum, bronze, galvanized iron, nickel-chromium alloy, or chromium plating, each material being used where extended research and much expensive experience has indicated that it renders the best service. Table tops are of monel. Belts on the quick-freezing machines are of monel or stainless steel. Pans for holding partially processed product are of monel.

Monel pans in which fillets are supplied to the packing tables are, during working hours, kept standing in a strong (7 p. p. m.) sodium hypochlorite solution when not in use, and every night are given a thorough scrubbing in the same strength solution.

When operations at the Gloucester plant were first started, the girl packers wore rubber gloves; but it was found a practical impossibility to prevent particles of fish adhering to the gloves, and the packers now wash their hands in chlorinated seawater before starting work and at frequent intervals during the day.

During working hours a janitor constantly flushes floors and machines with chlorinated seawater, and at night everything is scrubbed and washed down with a high-pressure automobile pump employing a strong sodium hypochlorite solution.

A well-equipped chemical and bacteriological laboratory and a testing kitchen are operated in connection with this Gloucester plant, and all product is subject to strict laboratory control. Figure 7 illustrates the "Lot History Sheet," which has been of great value in assuring a uniformly satisfactory finished product. The form consists of four sections, each of which represents one of the four phases of production and testing. The upper left-hand section covers the history of the raw material. In the upper right-hand quarter complete manufacturing data are recorded. The lower left-hand square shows the results of a bacteriological examination of two representative samples, taken at random in the packing room, distinctively labeled, and then replaced with the regular production packages to await their return at the quick-freezing machine. The remaining section of the record shows the result of a careful cooking test of each of the products included in the day's production. No part of the production covered by any Lot History Sheet is released for shipment until the sheet has been examined and approved by a responsible member of the laboratory organization.

An Investigation of Cane-Molasses Distillery Slop[1]

With Special Reference to Certain Organic Acids

E. K. Nelson and C. A. Greenleaf

Food Research Division, Bureau of Chemistry and Soils, Washington, D. C.

THE disposal of the large quantities of distillery slop resulting from the manufacture of alcohol from cane molasses is a problem. The best that can be done at present is to concentrate it so that it will support combustion and burn it, recovering ammonia from the combustion gases and potassium salts from the ash.

For the purpose of throwing some light on the composition of this product and also of studying the changes, if any, taking place in the acids of molasses on fermentation, a quantity of the concentrated slop was obtained as drawn from the evaporators at the Baltimore plant of the United States Industrial Alcohol Company.

Analysis of Distillery Slop

Analysis of the slop afforded the following results: total solids (*in vacuo* at 100° C.), 51.22 per cent; ash, 12.65 per cent; CaO, 1.78 per cent; MgO, 0.47 per cent; Fe_2O_3 and Al_2O_3, 0.15 per cent; Na_2O, 0.38 per cent; K_2O, 5 per cent; Mn_3O_4, 0.015 per cent; SiO_2, 0.10 per cent; P_2O_5, 0.21 per cent; SO_3, 0.37 per cent; chlorine, 2.17 per cent; nitrogen, total, 1.43 per cent; nitrogen, ammoniacal, 0.06 per cent;

[1] Presented before the Division of Agricultural and Food Chemistry at the 77th Meeting of the American Chemical Society, Columbus, Ohio, April 29 to May 3, 1929.

nitrogen, amino, 0.30 per cent; pentoses, 1.11 per cent; reducing substances (as invert sugar), 4.63 per cent; alkalinity of ash, 11.4 cc. 1 *N* hydrochloric acid per gram of ash; non-diffusible (collodion membrane), 11.67 per cent.

The volatile acids were found to be acetic, about 0.9 per cent, and formic, about 0.3 per cent. The non-volatile acids identified were succinic, about 0.5 per cent; tricarballylic, about 1 per cent; lactic about 3 per cent; and a small amount of aconitic acid with a possible trace of citric acid.

Glycerol (0.6 per cent as crude glycerol) was extracted by suitable solvents. Attempts to identify definite compounds, such as carbohydrates, by means of phenylhydrazine, acetylation, and precipitation with ammoniacal copper solutions were uniformly unsuccessful.

Experimental

VOLATILE ACIDS—Fifty grams of slop plus 3.85 grams of 96 per cent sulfuric acid (calculated from the alkalinity of the ash) were diluted to 150 cc. and distilled with steam at constant volume according to the method of Dyer (*1*), 500 cc. of distillate being collected. This required 16.70 cc. of 0.5 *N* sodium hydroxide for neutralization. The sodium salts were evaporated to dryness, redissolved, treated with 16.70 cc. 0.5 *N* sulfuric acid, diluted to 150 cc., and redistilled with steam as described above. The distillate was collected in successive, measured portions and titrated with 0.1 *N* sodium hydroxide. The distillation curve constructed from these data lay throughout between the known curves for acetic and formic acids, indicating the presence of: acetic, 75 per cent; formic, 25 per cent; or acetic acid, 0.376 gram; formic acid, 0.096 gram. Since the 500-cc. distillate from the first distillation contains, according to Dyer, 84 per cent of the acetic acid and 62.7 per cent of the formic acid actually present, the calculated figures are: acetic acid, 0.447 gram or 0.89 per cent; and formic acid, 0.153 gram or 0.31 per cent. The presence of formic acid was confirmed by reduction of mercuric chloride, and that of acetic by the characteristic odor of the ethyl ester.

NON-VOLATILE ACIDS—Two kilograms of slop were diluted with water and precipitated with lead subacetate. The acids recovered from the lead precipitate were esterified, affording 43.6 grams of crude esters. By fractionating these esters three times at 10 mm., the following fractions were separated: (1) under 98° C., 4.2 grams; (2) 130° to 145° C., 1.7 grams; (3) 155° to 160° C., 28 grams; (4) 160° to 170° C., 1.2 grams. Hydrazides were prepared from 0.7 gram of each of these fractions.

Fraction 1 afforded a hydrazide melting at 165° to 166° C., showing no depression when mixed with succinic hydrazide, and corresponding to succinic hydrazide in optical crystallographic properties.

Fraction 2 appears to be a mixture. Some succinic hydrazide (m. p. 162° to 166° C.) was obtained and a hydrazide of higher melting point, which gave a sharp depression in melting point when mixed with malic hydrazide. It is probably the same as the hydrazide from Fraction 3.

Fraction 3 afforded a good yield of a hydrazide melting at 195° to 196° C. Ten grams of this fraction were saponified, and the acid separated melted at 155° to 157° C. Mixture with tricarballylic acid gave no depression in melting point. A mixture of the hydrazide with tricarballylic hydrazide also gave no melting point depression. Optical crystallographic examination confirmed the identity of the acid as tricarballylic acid.

The hydrazide from Fraction 4 appeared to be a mixture. When it was seeded with citric trihydrazide, some crystals were obtained which had the appearance of the citric acid derivative, but they were insufficient in amount for a melting point or an optical crystallographic examination. It is probable that a trace of citric acid is present.

Direct ether extraction of the acidified slop gave a higher yield of succinic acid (2.5 grams from 500 grams of slop) and a far lower yield of tricarballylic acid than was obtained by the esterification method, indicating that the esterification method left some lead succinate unprecipitated and that the extraction of tricarballylic acid from aqueous solution by ether was far from complete.

A special examination was made for lactic acid. Five hundred grams of slop were acidified, diluted with 200 cc. of water, and extracted for several days in a large extractor designed for extracting liquids with ether. The ether was evaporated and the extracted acids were neutralized with barium hydroxide and made up to 300 cc. volume. Seven hundred cubic centimeters of alcohol were added, and the mixture was well shaken and allowed to stand overnight. The insoluble barium salts were filtered and washed with a little 70 per cent alcohol. The acids from the precipitate were recovered by removing the barium with sulfuric acid, and fractionally crystallized. The acids yielded were succinic, tricarballylic, and a small quantity of aconitic acid, all being identified by optical crystallographic methods.

The barium salts remaining in solution in 70 per cent alcohol were decomposed with the required quantity of sulfuric acid, and barium sulfate was removed by filtration. The filtrate, concentrated to 100 cc., was boiled with zinc carbonate, the excess was removed by filtration, and the solution, concentrated to 75 cc., was treated with 225 cc. of alcohol. On cooling and standing overnight, a copious crystalline precipitate of zinc salt was filtered off and dried over sulfuric acid. Optical examination showed it to be zinc lactate. The zinc salt weighed 24.6 grams and after drying *in vacuo* at 100° C. it weighed 19.0 grams, corresponding to 14 grams of lactic acid, or 2.8 per cent of the original slop.

The acid recovered from the zinc salt gave acetaldehyde on oxidation. The hydroxy acid test with ferric chloride, and the guaiacol and codeine reactions for lactic acid established its identity as lactic acid.

SEPARATION OF GLYCEROL—The glycerol was separated from 500 grams of slop in the following manner: The slop was diluted with an equal volume of alcohol, and the resulting heavy precipitate was separated and washed with 50 per cent alcohol. The alcoholic solution was evaporated, 200 cc. of absolute alcohol were added, and when the residue was thoroughly disintegrated and the soluble part in solution 300 cc. of absolute ether were gradually added while the solution was stirred. The precipitate was separated and the alcohol-ether solution evaporated. The residue was treated with dry acetone, an equal volume of absolute ether was added and, after being separated from the precipitated material, the solvents were evaporated. The residue was dissolved in a little water and filtered from a small quantity of fatty matter, and the water was carefully evaporated in a tared beaker. The drying was finished in a vacuum desiccator. The residue, crude glycerol, weighed 2.89 grams, 5.9 grams per kilogram or 0.59 per cent. The presence of glycerol was confirmed by the preparation of the benzoate; melting point, 72° C.

Summary

An analysis was made of concentrated molasses slop as it comes from the evaporating pans of a large alcohol distillery, and the organic acids were identified. Considerable quantities of formic, acetic, succinic, tricarballylic, and lactic acids, a small quantity of aconitic acid, and a possible trace of citric acid were found. Examination of a sample of the molasses from which this slop was derived revealed

no succinic and tricarballylic acids and only a little lactic acid.

The succinic acid and most of the lactic acid are evidently products of the fermentation. The disappearance of the greater part of the aconitic acid and its replacement by tricarballylic acid leads to the conclusion that, in the process of fermentation, the aconitic acid has been reduced to tricarballylic acid.

Glycerol was identified, but the quantity was not large enough to make its extraction appear profitable.

Acknowledgment

The writers' appreciation is hereby expressed to the officials of the United States Industrial Alcohol Company for their coöperation in furnishing material for this investigation.

The optical crystallographic examinations were made by G. L. Keenan, of the Food, Drug, and Insecticide Administration.

Literature Cited

(1) Dyer, *J. Biol. Chem.*, **28**, 445 (1917).

Vitamins in Canned Foods

VIII—Home Canning and Commerical Canning Contrasted in Their Effect on Vitamin Values of Pears[1]

Martha M. Kramer, Walter H. Eddy, and E. F. Kohman

COÖPERATIVE RESEARCH FROM THE LABORATORIES OF THE DEPARTMENT OF FOODS AND NUTRITION OF THE KANSAS STATE AGRICULTURAL COLLEGE, TEACHERS COLLEGE, COLUMBIA UNIVERSITY, AND THE NATIONAL CANNERS ASSOCIATION

CRAVEN and Kramer (*1*) reported in 1927 a study of the vitamin C values of raw Kieffer pears and the effect of open-kettle and cold-pack home canning. They reported that 15 grams of raw pears fed daily gave protection against scurvy to the animals and some gain in body weight. The average gain for 4 pigs was 42 grams over a 90-day period. The antiscorbutic factor in pears was destroyed in the open-kettle method of home canning, as animals consuming as high as 19 grams of pears daily lived on the average no longer than the expected survival period when the basal diet alone was given. The antiscorbutic factor is not completely destroyed, however, by the cold-pack method, although animals consuming as high as 20 grams daily survived only about 50 days.

As the destruction of vitamin C in these pears was so marked in comparison with the results obtained with a number of commercially canned foods (*2* to *11*), it seemed of interest to repeat the studies with a commercial pack. In addition to the regular commercial pack of pears, several other packs were included in which variations were introduced suggested by studies on canned apples (*9*). At the same time a similar series of Bartlett pears was canned in a commercial cannery and the vitamin A, B, and C content determined at Teachers College, Columbia University. Kieffer pears are canned commercially only in relatively small quantities. The Bartlett pears comprise the great bulk of commercially canned pears. This is a combined report of the vitamin studies pertaining to the two varieties of pears.

[1] Presented under the subtitle before the Division of Agricultural and Food Chemistry at the 77th Meeting of the American Chemical Society, Columbus, Ohio, April 29 to May 3, 1929.

Vitamin C Content of Kieffer Pears

The vitamin C content of the following lots of Kieffer pears was determined by Kramer:

Table I—Gain or Loss in Weight, Survival Period, and Severity of Scurvy of Guinea Pigs Receiving Varying Amounts of Kieffer Pears as a Source of Vitamin C

Daily Feeding	Control			Raw Pears			Canned Pears											
							Lot 1			Lot 2			Lot 3			Lot 4		
Grams	*Grams*	*Days*	*	*Grams*	*Days*	*	*Grams*	*Days*	*	*Grams*	*Days*	*	*Grams*	*Days*	*	*Grams*	*Days*	*
	−130	34	8															
	−119	39	7															
	−146	32	6															
	−141	33	8															
	−128	28	9															
	−110	51	7															
5				77	90	5												
				58	61	4												
				−121	54	6												
8				101	90	4												
				− 78	55	5												
				− 67	30	5												
10				29	90	4	132	90	6	68	90	6	43	90	3	− 57	88	8
				− 62	90	5	67	90	1	− 87	90	6	137	90	7	− 84	75	7
				− 49	90	2	− 94	32	5	−116	90	6	−103	50	5			
							−125	41	3	− 88	28	7						
12				62	90	5												
				56	70	3												
				− 45	37	2												
				− 73	67	2												
15				285	90	1	54	90	1	93	90	1	141	90	2	89	90	6
				177	90	.	92	90	1	19	90	6	114	90	4	20	90	7
				− 51	90	3	118	90	4	137	90	3	− 54	36	2	−112	28	7
				−154	44	2	− 83	36	6							−120	48	6
20				259	90	.	253	90	.	114	90	5	253	90	.	142	90	2
				268	90	.	232	90	.	96	90	1	182	90	.	167	90	1
							222	90	.	− 27	90	3	212	90	1	− 86	40	3
							100	90	.							−103	86	6
							52	90	.									
30							191	90	.	225	90	.	157	90	.	55	90	1
							184	90	.	261	90	.	239	90	.	− 39	68	2
													233	90	.	−107	33	4
																−129	39	4

* Figures represent relative severity of scurvy symptoms as disclosed by autopsy.