in solution by cautiously fusing with 5 to 10 g. of KHSO_4 , bringing the temperature up to a dull red heat for about one-half hour and using a cover on the crucible to avoid spattering during the process of fusion. The zirconium may be determined by practically any of the methods mentioned above, using this solution, provided that the quantity of zirconium in the solution is not greater than 0.1 g. calculated as ZrO_2 . Most precipitates of zirconium are more or less gelatinous, bulky, and difficult to filter and wash, and therefore if larger quantities are taken, accurate results cannot be attained.

If the zirconium alone is desired and there is no need of separating the iron, aluminum, etc., from the solution, the phosphate method is the only one which gives accurate results and a clean separation from both the above-mentioned elements.

The solution which has been freed from silica, containing not over and preferably less than 0.05 g. ZrO_2 , is diluted to 200 cc. volume, made 10 per cent acid with H_2SO_4 , 2 cc. hydrogen peroxide added, heated nearly to boiling and a slight excess of disodium phosphate solution added. The zirconium precipitate is a white or greenish white flocculent, gelatinous precipitate. After allowing to stand for about 2 hrs. or longer the precipitate is filtered onto an 11 cm. filter paper and thoroughly washed with hot dilute sulfuric acid. The paper and contents are then transferred to a platinum crucible, ignited, and finally blasted to remove as much of the carbonaceous matter as possible. The proper ignition of this precipitate is one of the most difficult steps of the whole procedure. The weight of zirconium pyrophosphate, ZrP_2O_7 , multiplied by the factor 0.4632 yields the weight of ZrO_2 .

Iron is determined in the solution from the silica filtration by the method of precipitation as the sulfide from ammonium tartrate solution. This method is well known. To 100 cc. portions of the silica-free solution 0.5 g. of tartaric acid is added, the solution heated to boiling, made strongly alkaline with NH_4OH , and H_2S added. The iron precipitates as the sulfide, is filtered out, washed, and weighed as Fe_2O_3 .

The titanium may also be determined on a separated portion of the original solution (if all HF or HNO_3 has been first removed) by Weller's colorimetric method with H_2O_2 or by titration with methylene blue.

Aluminum is usually determined by difference, the aluminum, zirconium and titanium being precipitated with sodium thiosulfate, SO_2 , or phenylhydrazine practically free from iron, if the percentage of iron in the original is not high. In certain cases it is necessary first to remove the iron by the above-mentioned method and destroy the ammonium tartrate by the ordinary methods of wet combustion with nitric and sulfuric acids before the precipitation of the others is attempted.

V—BIBLIOGRAPHY

Before much work on zirconium was attempted, all of the methods for the production of the metal, etc., described in the literature were reviewed and tried. At the start there were many conflicting data regarding the properties and reactions of zirconium and its

compounds. A complete bibliography of the subject contained about five hundred references, from some forty-one different journals.

It has been arranged in alphabetical order with regard to names of authors under the following headings:

- 1—Zirconium Minerals
- 2—Preparation, Properties, and Uses of Zirconium Salts
- 3—Analytical Chemistry of Zirconium
- 4—Zirconium Metal
- 5—Zirconium Alloys

The bibliography also includes references on the use of ZrO_2 as a refractory and as an opaquing agent in enamels, etc. All of the technical uses have been faithfully included. Section 5 has been devoted to the preparation and properties of zirconium alloys.

A CHEMICAL STUDY OF FROZEN FISH IN STORAGE FOR SHORT AND LONG PERIODS

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The present investigations upon fish commercially frozen and stored were undertaken in the hope of furnishing exact information upon which the public could base its opinion concerning the wholesomeness of frozen fish as food. The part of these investigations of practical interest to the trade has already been published in *Departmental Bulletin* 635 of the U. S. Department of Agriculture, and entitled "The Commercial Freezing and Storing of Fish."

The magnitude of the frozen fish industry of this country is indicated by the fact that over 120,000,000 lbs. of food fish,¹ valued at more than \$12,500,000 to the wholesaler, entered cold storage in 1918. Depending upon the locality, the amount of frozen fish in storage during a year represents from 4 to 15 per cent of the annual catch.² Furthermore, there are over 190 fish-freezing and storage plants in the United States, and the number is increasing. These statistics show that the practical advantage and economic necessity of cold-stored fish have long been realized by those familiar with the industry. Until recently, however, no chemical investigation has been made of the effect upon the food value of the freezing and storing of fish.

HISTORICAL

In 1913 several Dutch scientists³ made experiments with ice-chilled and frozen fish to see what changes in microscopical appearance occurred under refrigeration and what part bacteria played in the process of deterioration. This commission found that certain changes take place in the reaction of the tissues to litmus, the flesh of some species being acid, and, of

¹ Estimate based on U. S. Dept. of Agriculture, Bureau of Markets, Report of Stocks of Frozen and Cured Fish, December 15, 1918; issued December 28, 1918.

² Hearings before the Committee on Agriculture, House of Representatives, on Cold Storage Legislation, August 14, 1919, p. 203.

³ J. M. Bottemaine, "Notes on the Investigation of Preserving Fish by Artificial Cold." (a) Preliminary Report, 48 pages; (b) Preprint, 3 pages; 3rd International Congress of Refrigeration, 3rd Section (The Netherlands), Washington-Chicago (1913).

some, alkaline during decomposition. Some differences in the histology of fresh and frozen fish were noted. All of this work was, however, of a preliminary nature and not in any sense an exact chemical investigation.

The quantitative chemical study of changes in fish flesh upon standing or during cold storage is a new one. The researches of Richardson and Scherubel,¹ Emmett and Grindley,² and others upon meat during storage opened the way in that field. Pennington³ and Wiley, *et al.*,⁴ have done fundamental work on the chemistry of ripening processes in the flesh of poultry and game.

The investigations of Smith⁵ and of Perlzweig and Gies⁶ on chemical changes in fish during cold storage are the pioneer contributions to our knowledge of this subject. They determined the chemical composition, ammonia content, etc., of fresh fluke, *Paralithys dentatus*, and flounder, *Pseudopleuronectes americanus*, before freezing, and also made the same determinations at intervals during two years on the same lot of storage fish. These fish were all handled according to the usual trade practices, except that they were probably frozen more promptly after catching than the average commercially frozen fish. Even at the end of two years of storage these investigators were unable to detect any appreciable variations in chemical composition or nutritive value attributable to freezer storage. There were no significant changes in the percentages of the ordinary food constituents, or of ammoniacal nitrogen, acidity of fats or reaction of the aqueous extracts; all of which would tend to show that neither autolytic nor bacterial influences had been active. Furthermore, two years after storage the fish were found palatable and entirely acceptable as food.

Recently, Almy⁷ investigated the chemical changes in mackerel (*Scomber scombrus*) after freezing and cold storage for 9 mo. Unlike the so-called "bottom" fish used in the researches of Smith, which contain less than one per cent of fat, mackerel are tender, oily fish, very prone to rapid deterioration unless handled carefully and kept constantly under refrigeration. This investigation represented the first attempt to study the nitrogen distribution in the flesh of frozen fish, but, as the work was a continuation of that reported in the present paper, no attempt will be made to describe the methods used but only to summarize the conclusions based on the study of a typical fat-containing fish. Almy found that during storage there were in frozen mackerel slight increases in the amount of total nitrogen and coagulable nitrogen in the aqueous extracts, but no change in the content of ammonia and amine nitrogen. There were also slight increases in the acidity of the aqueous extracts of the

flesh and of the fat extracted from the flesh of the cold stored mackerel. In no case, however, was there any significant change in the food value of the fish or any evidence of deterioration at the end of 9 mo. At the end of this period of storage some of the mackerel were cooked and prepared for the table. Unlabeled portions of these cooked storage mackerel and of perfectly fresh mackerel were served to a representative group of 15 people in New York City, and none of them was able to detect any difference between the fresh and frozen fish as judged by appearance and flavor.

METHODS OF THE PRESENT INVESTIGATION

HISTORY OF FISH ANALYZED

Two species were used in the present investigation: weakfish, *Cynoscion regalis*, and bluefish, *Pomatomus saltatrix*, which is carnivorous and a voracious enemy of all fish smaller than itself. The medium sized fish of both species were studied. All of the specimens were caught off the New Jersey coast and promptly landed at the shore without any contact with ice. In all the lots but one the fish were eviscerated before they were landed. As soon as possible the different lots were weighed and carted a short distance to a fish freezer, where the specimens for freezing were washed and frozen in pans on brine pipes in the usual way. The temperature of the freezing room was -5°F. , and of the storage room about 0°F. This is lower than the customary storage temperature, but the isolated nature of the plant necessitated such a practice as a safeguard to allow time for repairs in case of a break in the refrigerating machinery. The fish to be analyzed immediately in the freshly frozen condition were quickly chilled and frozen, and while still at a temperature below 0°F. were taken to the laboratory in Philadelphia where they arrived hard frozen. They were then thawed slowly by standing over night in an ice-box, cleaned, and prepared at once for analysis in the laboratory. The frozen fish for long holding were shipped in cork-insulated shipping cases, and upon reaching Philadelphia were at once transferred to a storage room where a temperature of about 15°F. was maintained during the various periods of storage. Each lot was boxed in the commercial way and reglazed whenever necessary, which was about three times during a year. As indicated in the tables, some of the weakfish were not glazed. One lot was stored without removal of entrails (in the so-called "round" condition), and another lot was wrapped in commercial semi-parchment paper without any glazing. These variations from the commercial practices were made in order to study the effects of such modifications.

PREPARATION OF SAMPLES FOR ANALYSIS

The thawed fish were prepared for sampling by separating as completely as possible the edible portion from the heads, tails, skins, entrails, and bones, which were discarded. The edible part was weighed and the difference between this weight and that of the whole fish represented the refuse or inedible portion. The flesh was ground three successive times in a meat chopper, and the samples of individual fish mixed thoroughly

¹ THIS JOURNAL, 1. (1909), 95.

² *Ibid.*, 1 (1909), 413.

³ Hearings before Committee on Manufactures, U. S. Senate, on Foods Held in Cold Storage, May 25, 1911, p. 57.

⁴ U. S. Dept. of Agriculture, *Bulletin* 115 (1908).

⁵ *Biochem. Bull.*, 3 (1913), 54.

⁶ *Ibid.*, 3 (1913), 69.

⁷ L. H. Almy, unpublished report of a study of the chemical changes occurring in frozen mackerel during storage for a year. Research performed in partial fulfillment of the requirements for the degree of Doctor of Philosophy at Columbia University, 1917.

by hand. In the case of bluefish, which were of considerable size, two specimens were used; but of the weakfish three were analyzed in each lot, the edible portions being united, ground again, and well mixed before final sampling for analysis.

The following section contains a description of the various determinations of chemical changes in the fish during cold storage, and is intended to include every determination that offered any possibility of giving useful information about fish flesh and its changes. Possibly, some of these determinations are unnecessary and some may be of little value, but all should help to give information about the adequacy of the methods themselves as applied to problems of this sort. Most of the determinations have been adapted from the work of early investigators upon meat and poultry. Any new methods or modifications will be apparent to those familiar with chemical work on cold storage problems.

ANALYTICAL PROCEDURE

MOISTURE, ETHER EXTRACT, ASH AND TOTAL NITROGEN—The methods used for the estimation of the percentage of moisture, ether extract, and ash are those described in *Bulletin 107* (revised) of the Bureau of Chemistry, U. S. Department of Agriculture. Total nitrogen was determined by the method of Kjeldahl and Gunning, using 0.1 *N* acid and alkali.

FAT-FREE SOLIDS—It is a well-recognized fact that the flesh of fat fish contains a lower per cent of moisture than that of lean fish. The reciprocal relation of the per cent of moisture and fat in the flesh of fish of the same species is such that the per cent of fat-free solids is more or less constant. An increase of 2 per cent in the fat-free solids therefore shows that the per cent of moisture has decreased by 2 per cent. The per cent of fat-free solids was computed in each case by subtracting the per cent of fat from the per cent of solids.

AMMONIA AND AMINE NITROGEN BY AERATION—Loosely bound basic nitrogen compounds, which in fish flesh include ammonia and amine nitrogen, were estimated by a modification of the Steel-Gies method described by the authors.¹

TOTAL SOLUBLE NITROGEN AND COAGULABLE NITROGEN—The extraction of the flesh and the determinations of soluble nitrogen and coagulable nitrogen were made by the methods previously outlined.¹

SOLIDS—One hundred cc. portions of the water extract of the flesh were evaporated on the steam bath in weighed platinum dishes, the residue being dried at 100° C. to constant weight.

ASH—The organic matter of the soluble solids was burned off in a muffle at low red heat to avoid any loss of chlorides.

ORGANIC EXTRACTIVES—This is the difference between ash and solids already determined.

ACIDITY OF THE EXTRACTS—Owing to the presence of both proteins and phosphates in extracts of fish flesh, it is not believed that direct titrations of acidity can give results of absolute value. For comparative

studies in investigations of this sort, however, such direct titrations would certainly show any significant increases in acidity due to changes occurring during a prolonged period of storage. Correction was made for the very slight acidity of the distilled water used in preparing the extracts. One hundred cc. of the original extract were titrated with 0.1 *N* sodium hydroxide, using phenolphthalein as indicator. The end-point was taken as the point at which the alkali produced a pink color, permanent for one minute. In the tables the results are stated as the number of cc. of 0.1 *N* sodium hydroxide required to neutralize the entire extract from a 50 g. sample.

NITROGEN PRECIPITATED BY TANNIN AND SALT—Proteose and peptone nitrogen were determined by the method of Bigelow and Cook.¹ Fifty cc. portions of the extract, after removal of coagulable protein, were treated in a 100 cc. volumetric flask with 15 g. of sodium chloride, and, when the latter was thoroughly dissolved, chilled to ice-box temperature (12° C.), treated with 30 cc. of a 24 per cent solution of tannic acid (cold), made up to volume with cold water, shaken, and allowed to stand at the temperature of the ice-box for 24 hrs., after which the precipitate was filtered off. The solution was kept cold, and the nitrogen in 50 cc. of the filtrate was determined according to the usual Kjeldahl procedure, except that the addition of potassium sulfate was unnecessary because of the large amount of sodium chloride present. Care was taken during the early part of the oxidation that the escaping hydrochloric acid did not cause the material to foam out of the flask. Since tannic acid invariably contains a certain amount of nitrogen, a blank determination must be made with every experiment. The per cent nitrogen in the tannin salt precipitate was obtained by subtracting the per cent of nitrogen in the tannin salt filtrate from the per cent of non-coagulable nitrogen. The results obtained by this method were sometimes erratic but are included in the table as an indication of changes in peptone and proteose nitrogen.

PROTEOSE NITROGEN BY ZINC SULFATE PRECIPITATION—For this determination 50 cc. of the filtrate from the coagulated protein were concentrated to 30 cc. in a 100 cc. beaker and the resultant solution was saturated with zinc sulfate, according to the method of Bömer.² When cold, the solution was filtered through a thin asbestos layer in a Gooch crucible and the precipitate washed with a saturated solution of zinc sulfate. The precipitate, together with the asbestos mat, was transferred to a Kjeldahl flask, and the nitrogen determined as usual.

NITROGEN IN PEPTONE BASES, ETC.—The figures under this heading in the tables were obtained by subtracting the sum of the amine and ammonia nitrogen, amino-acid nitrogen, proteose nitrogen, and coagulable nitrogen from the total soluble nitrogen.

AMINO-ACID NITROGEN—This determination was made according to the general method of Sørensen³ on 50 cc. of the filtrate after removal of coagulable nitrogen

¹ *J. Am. Chem. Soc.*, **28** (1906), 1485.

² *Z. anal. Chem.*, **34** (1895), 563.

³ *Biochem. Z.*, **7** (1907), 64.

¹ *J. Biol. Chem.*, **33** (1918), 486.

in the extract. The presence of ammonia and the amines tends to interfere with the Sørensen titrations for amino acid but the results have comparative value in detecting changes during cold storage.

AMMONIA AND AMINE NITROGEN BY AERATION—Portions of 150 cc. of the original extract were analyzed for ammonia in the same manner as the fish flesh.

DETERMINATION OF FAT CONSTANTS—The methods employed for the gross extraction of the oil from the flesh and for the determinations of specific gravity, index of refraction, iodine number, saponification number and acid value of the oil are the same as those described by the writers.¹

In the tables, the average of two or more concordant determinations is given in every case.

OBSERVATIONS AND RESULTS OF CHEMICAL ANALYSES

(1) *Unglazed Eviscerated Weakfish Stored without Wrapping (Series 1001)*

APPEARANCE AFTER STORAGE—These weakfish, stored with neither a glaze nor parchment wrapping, were unmarketable at the end of 4 mo. The whole fish were dried and wrinkled in appearance, while the noses and eyes were whitish and dried out.

At the end of 8 mo. this lot of fish was very uninviting in appearance and unmarketable. As they were unfit for food, no further samples were taken after 8 mo. of storage. The whole fish were badly wrinkled, and white. The eyes were also white and sunken. When cooked the flesh seemed tasteless and dry.

GROSS ANALYSIS—The quantitative variations in the composition of certain fish due to season, age, and individual differences are often very great, as shown by previous investigators and confirmed by the present writers,² and the differences attributable to the two latter causes must be borne in mind before giving undue emphasis to variations in the gross analysis of cold stored fish in the accompanying tables. The tendency to lose moisture during storage is common to all flesh products. The loss of moisture from these unglazed fish was greater than that from any of the other fish as shown by the chemical analysis. The percentage of fat varied greatly but this is a characteristic of weakfish and was not due to changes in cold storage. The acidity of the aqueous extract showed no definite change.

NITROGENOUS CONSTITUENTS—Any of the minor but significant changes due to bacterial or enzyme action on the nitrogenous constituents of the stored fish would be indicated by the amounts of such constituents, but more especially by their relation to the total nitrogen, commonly called the "distribution of nitrogen." In general, these unglazed weakfish at the end of 8 mo. showed no greater changes in their nitrogen distribution than the glazed eviscerated weakfish (Series 1002) at the same period. Apparently, 8 mo. of storage temperatures did not produce significant alteration in the nitrogenous constituents of the frozen weakfish even without the protection of this glaze. Ammonia and amine nitrogen increased in amount, but only at about the same rate as in the glazed weakfish. The changes in the soluble and coagulable nitrogen were irregular.

CONSTANTS OF FAT EXTRACTED FROM THE FLESH—Since there is known to be considerable variation in the chemical properties of oils from individual fish, depending on their food, the relation to time of spawning, etc., it does not seem wise to consider that any of the changes in the composition of the fat in these fish, as recorded in Table IV, were due primarily to storage except in the case of the acid value.³ This value at the end of the 8 mo. storage period showed a considerable increase as compared with that of the oil of the fish when first frozen. However, this change in the acid value was of the same magnitude as that of glazed weakfish (Series 1002) at the same period. Although this lot of weakfish did not have the protection of either glaze or wrapping, it showed no greater signs of aging than similar glazed fish; hence, we may conclude that the slight changes in

oils of these frozen fish during the 8 mo. of storage are probably not due to their oxidation by oxygen of the air.

(2) *Glazed Eviscerated Weakfish Stored without Wrapping (Series 1002)*

APPEARANCE AFTER STORAGE—Up to the end of 13 mo. the fish of this lot looked bright under their glaze and were neither white nor wrinkled, except to a slight extent on the nose. The eyes were bright but slightly sunken. After cooking, the flesh had a normal appearance and flavor.

After 2 yrs. in storage all of these weakfish were unmarketable owing to the whitish and wrinkled appearance, especially of the heads. The eyes were opaque and much sunken, and the gills yellow and dry. Some of the fins were yellowish and dry. When cooked, they were not unpalatable, but tasteless and flat.

GROSS ANALYSIS—There were no noteworthy changes in the amounts of the various constituents during storage, aside from the usual variations found in the composition of fish of the same species even when of nearly uniform size and taken from the same school. Loss of moisture based on percentage of fat-free solids continued to occur until the twelfth month of storage, after which the results do not seem to indicate further loss of moisture. There was no definite increase in the acidity of the aqueous extract of the flesh.

NITROGENOUS CONSTITUENTS—The percentage of ammonia and amine nitrogen made slight but definite increases during storage. However, these increases were not as marked or as uniform as in the case of frozen bluefish (Series 1006). The per cent of total soluble nitrogen increased up to the eighth month, then decreased. The per cent of coagulable nitrogen showed a similar rise and fall. The percentages of amino-acid nitrogen, as determined by the Sørensen titration method, showed a slight tendency to increase in the storage fish.

CONSTANTS OF FAT EXTRACTED FROM THE FLESH—The only fat constant which changed during storage was the acid value, the latter increasing at an irregular rate during the entire storage period of 2 yrs. and 1 mo. As this was interstitial fat, the increase in the acid value was probably due to enzyme action rather than to bacterial or oxidation changes which might affect external fat. From unpublished work, it is known that the flesh of properly frozen fish is practically sterile.

(3) *Unglazed Eviscerated Weakfish Stored with Paper Wrapping (Series 1003)*

APPEARANCE AFTER STORAGE—After 8 mo. these wrapped fish appeared somewhat dried out and wrinkled on the heads. The eyes were sunken and dull but not opaque. After cooking, the flesh tasted normal in every way. At the end of 13 mo. the fish were entirely unmarketable, being much wrinkled and white with opaque and deeply sunken eyes. As their condition was so bad, it was not deemed necessary to carry further the studies on these unglazed wrapped fish. When cooked, they tasted flat and stringy, with a slight bitter after-taste.

GROSS ANALYSIS—The most striking fact about the composition of the fish in this lot is that they were badly dried out at the end of 13 mo. storage. Judging by chemical analysis and by appearance, the wrapping around these unglazed fish slightly retarded their desiccation but did not keep them from becoming unmarketable and unfit for food after 13 mo. The ordinary food constituents showed no significant changes.

NITROGENOUS CONSTITUENTS—As usual, there was a gradual increase in the percentage of ammonia and amine nitrogen during storage for 13 mo. However, this increase was no more marked than that found in glazed weakfish stored for about the same period. Like the glazed fish (Series 1002) the total soluble nitrogen and the non-coagulable nitrogen increased to the eighth month, then fell.

CONSTANTS OF FAT EXTRACTED FROM THE FLESH—There was the usual increase in the acid value of the oils from the fish after 13 mo. of storage. The magnitude of this change was about the same in the case of the glazed weakfish (Series 1002) after storage for the same period.

(4) *Glazed Weakfish Stored with Entrails Intact and without Wrapping (Series 1004)*

APPEARANCE AFTER STORAGE—After storage for 13 mo., the appearance of these fish was still good, only the slightly sunken condition of the eyes indicating aging. One of the weakfish in this lot had in its stomach 4 small fish about 6 in. long. None of these ingested fish showed any signs of enzyme action on the part of digestive juices or bacteria. However, at the end of storage for 2 yrs., the fish were entirely unmarketable owing to their general wrinkled and whitish appearance and sunken dull white eyes. Upon cooking, the flesh was tasteless and poor in quality, though not inedible.

GROSS ANALYSIS—There were no noteworthy changes in the percentages of the ordinary food constituents during storage.

¹ *J. Biol. Chem.*, **33** (1918), 487, 493.

² *Ibid.*, **33** (1918), 483.

³ Analyses of a large number of fish have shown that the acid value of the fat varies with the content of fat. In general the lean fish have a higher acid value than the fat fish of the same species. This relationship between the acid value of the fat and the state of nutrition of the fish is being studied further. Consideration has been given to this factor in arriving at conclusions in regard to changes due to storage.

TABLE I—GROSS FLESH ANALYSIS

No.	Description of Fish	Storage Period	Weight of Fish Analyzed G.	Edible Portion Per cent	Per cent on Moist Basis						Per cent on Dry Basis				
					Moisture	Fat-free Solids	Ash	Ether Ex-tract	Total Nitro-gen	Am-mon-ia and Amine Nitro-gen	Ash	Ether Ex-tract	Total Nitro-gen	Am-mon-ia and Amine Nitro-gen	
1002/1	Weakfish	Fresh	1273	55.1	75.59	19.26	1.18	5.15	3.00	0.016	4.83	21.10	12.29	0.065	
1002/2		Fresh	1104	50.0	78.70	19.54	1.24	1.76	2.96	0.018	5.82	8.26	13.89	0.084	
1001/1		Eviscerated, not glazed, not wrapped in paper	1 mo.	1585	51.9	75.86	20.77	1.14	3.37	3.06	0.014	4.72	13.96	12.67	0.059
1001/2			2 mo.	1840	50.3	75.98	20.67	1.08	3.35	3.12	0.018	4.50	13.94	12.98	0.073
1001/3			4 mo.	1597	57.6	71.78	20.19	1.12	8.03	3.19	0.016	3.97	28.45	11.34	0.058
1001/4		8 mo.	991	56.5	74.72	22.93	1.31	2.35	3.42	0.024	5.18	9.27	13.53	0.093	
1002/1	Eviscerated, glazed, not wrapped in paper	2 mo.	2027	50.6	77.61	20.48	0.96	1.91	2.99	0.017	4.29	8.53	13.35	0.076	
1002/2			4 mo.	1655	53.1	76.65	20.88	1.10	2.47	3.13	0.015	4.71	10.59	13.40	0.066
1002/3			8 mo.	1657	45.5	76.92	21.09	1.18	1.99	3.12	0.023	5.11	8.62	13.52	0.101
1002/4			1 yr. 1 mo.	1543	52.8	75.14	22.04	1.16	2.82	3.20	0.020	4.67	11.34	12.87	0.081
1002/5			2 yrs. 1 mo.	2123	51.5	75.16	21.97	1.10	2.87	3.17	0.023	4.42	11.54	12.75	0.091
1003/1	Eviscerated, not glazed, wrapped in paper	4 mo.	1660	51.7	77.12	21.53	1.16	1.35	3.13	0.019	5.07	5.90	13.67	0.083	
1003/2			8 mo.	1721	56.3	72.87	20.96	1.16	6.17	3.16	0.021	4.28	22.74	11.64	0.076
1003/3			1 yr. 1 mo.	1505	52.3	75.57	22.03	1.28	2.40	3.30	0.023	5.24	9.82	13.50	0.093
1004/1		Not eviscerated, glazed, not wrapped in paper	4 mo.	1528	47.2	74.49	20.63	1.16	4.88	3.09	0.019	4.55	19.13	12.11	0.075
1004/2				10 mo.	1518	48.9	75.95	20.98	1.14	3.07	3.12	0.030	4.74	12.76	12.97
1004/3			1 yr. 1 mo.	1858	43.9	75.41	21.40	1.13	3.19	3.18	0.028	4.60	12.97	12.93	0.112
1004/4			2 yrs. 1 mo.	1863	42.8	75.77	20.52	1.13	3.71	3.12	0.032	4.66	15.31	12.88	0.132
Bluefish															
1006/1	Eviscerated	Fresh	5432	50.0	76.02	22.85	1.34	1.13	3.44	0.019	5.58	4.71	14.34	0.081	
1006/2		Fresh	2982	51.4	75.64	22.54	1.32	1.82	3.31	0.024	5.41	7.40	13.58	0.097	
1006/1	Eviscerated, glazed, not wrapped in paper	4 mo.	4354	52.3	74.20	22.47	0.99	3.33	3.34	0.016	3.84	12.90	12.94	0.060	
1006/2			5 mo.	5030	49.5	73.98	22.83	1.14	3.19	3.41	0.022	4.38	12.26	13.10	0.082
1006/3			8 mo.	4250	48.4	74.32	23.66	1.27	2.02	3.56	0.025	4.95	7.87	13.82	0.097
1006/4			1 yr.	4801	47.7	74.74	23.41	1.22	1.85	3.37	0.029	4.83	7.32	13.34	0.114
1006/5			1 yr. 4 mo.	4210	53.9	74.29	23.90	1.23	1.81	3.37	0.026	4.78	7.04	13.10	0.100
1006/1a			1 yr. 4 mo.	3930	51.4	75.67	23.70	1.26	0.63	3.56	0.021	5.18	2.59	14.63	0.067
1006/6			2 yrs. 3 1/2 mo.	3796	51.0	73.78	24.89	1.23	1.33	3.63	0.028	4.69	5.07	13.85	0.107

NITROGENOUS CONSTITUENTS—With the exception of the pronounced tendency of ammonia and amine nitrogen to increase in these uneviscerated weakfish, there were no other changes different from these discussed in the case of the glazed eviscerated weakfish (Series 1002). No other lot of fish exhibited such a tendency to form ammonia and amine nitrogen as did the weakfish stored "in the round." As all the lots of weakfish came from the same school, and had a normal uniform content of nitrogen in this form at the start, it would appear that the presence of the entrails in the fish during storage was concerned in this matter. The importance of this question, however, is such that until further study has been made no definite conclusions should be drawn.

CONSTANTS OF FAT EXTRACTED FROM THE FLESH—Except for the usual increase in the acid value there were no significant changes in the composition of the fat of these fish as revealed by chemical analyses.

(5) *Glazed Eviscerated Bluefish Stored without Wrapping (Series 1006)*

APPEARANCE AFTER STORAGE—There were no noticeable changes in the appearance of these bluefish until the end of 16 mo. of freezer storage. When removed at the end of the sixteenth month, the lot showed a certain amount of drying out on the noses of the fish, which were wrinkled and yellowish; the eyes were somewhat sunken, but eyes and skin were fairly bright. Upon cooking, the flesh was palatable and the flavor did not noticeably differ from that of fresh bluefish. These fish were in marketable condition and only a person familiar with the appearance of frozen fish would have classed them as "old stock."

The bluefish which were in storage for 2 yrs. and 3.5 mo. were in unmarketable condition when taken out for analysis. The noses and skin were dried, wrinkled, and yellowish. The gills were dry and brown. Some of the fins were swollen and soft. The eyes were sunken and opaque. The flesh seemed soggy after thawing but there was no odor of decomposition, only a peculiarly strong fishy odor. Upon cooking, the parts of the flesh near the skin were bitter and unpalatable, while the interior portions had a good flavor but were tough. As the fish had been kept glazed during the whole period of storage, there had been no opportunity for molds to attack their surface. From every point of view these fish were unmarketable and hardly fit for food.

GROSS ANALYSIS—There were no variations in the proportions of the gross food constituents of bluefish which could not be explained as being due to a loss of moisture during the periods of storage or to natural variations in the composition of this species of fish. The difference between the average initial per cent of fat-free solids and the per cent of this constituent at the end of the several periods of storage indicates a gradual loss of moisture amounting to about 0.7 per cent at the end of one year and to about 2.2 per cent at the end of 2 yrs. and 3.5 mo., in spite of the protection afforded by the ice glaze. At the close of the latter period the physical appearance of the fish confirmed

the analytical findings in this regard. The acidity of the aqueous extracts showed no change.

NITROGENOUS CONSTITUENTS—There is a slight but definite tendency for the percentage of ammonia and amine nitrogen to increase during storage, both as determined on the flesh itself and on its aqueous extract. There was a considerable decrease in the amount of coagulable nitrogen after one year of storage. The proportion of soluble nitrogen showed a less marked decrease. Proteose nitrogen, as determined by zinc sulfate precipitation, was another constituent that decreased perceptibly during storage.

CONSTANTS OF FAT EXTRACTED FROM THE FLESH—The acid value of the oil of the bluefish showed a definite increase during storage and a relatively high value at the end of over 2 yrs. In addition there appeared to be a decrease in the iodine number. This may have been due to a slight oxidation of the oil, a change which probably was wrought by the action of the air on the oil near the surface of the fish after this part of the fish had become somewhat dried out.

DISCUSSION

One of the inevitable effects of cold storage upon frozen fish is loss of moisture. This evaporation of water at temperatures from -5° to 15° F. is indeed slow, and within certain limits decreases in amount with decreasing temperatures. At the beginning of the storage period the proportion of fat-free solids in the freshly frozen weakfish averaged 19.40 per cent. After 8 mo. the fat-free solids (Table I) of the frozen weakfish, which had been stored in the eviscerated condition without a protective coating of ice-glaze or paper, were 22.93 per cent, indicating a decrease of 3.53 in the per cent of moisture. In contrast to this, the fat-free solids of the frozen weakfish, which had been stored for 25 mo. in the eviscerated condition and "sealed" with a thin layer of ice, were 21.97 per cent, corresponding to a decrease of 2.5 in the per cent of moisture. It is plain that the ice-glaze greatly retarded evaporation of water from the flesh. This slight desiccation of the fish is shown, not only by chemical analysis, but by the appearance as well. The unglazed fish stored without wrapping were unmarketable at the end of 4 mo. owing to their dried-out appearance, whereas the glazed fish were in marketable condition even at the end of 13 mo. Loss of

TABLE II—COMPOSITION OF THE COLD WATER EXTRACT OF THE FLESH
Per cent on Moist Basis

Per cent on Moist Basis														Per cent on Water-, Ash-, and Fat-free Basis													
No.	Description of Fish	Storage Period	Solids	Ash	Organic Matter	Acidity of Extract Cc.	Nitrogen								Nitrogen												
							Total	Coagu- lable	In Pep- tone Bases, etc. (by Differ- ence)	In Tan- nin- Salt Pre- cipi- tate	Prote- ose	Am- ino Acid	Am- monia and Amine	Total	Co- agu- lable	In Pep- tone Bases, etc. (by Differ- ence)	In Tan- nin- Salt Pre- cipi- tate	Prote- ose	Am- ino Acid	Am- monia and Amine							
1002/1	Weakfish	Fresh	7.49	1.04	6.45	15.4	0.858	0.547	0.233	0.040	0.010	0.054	0.014	4.75	3.03	1.29	0.221	0.055	0.298	0.082							
1002/2	Eviscerated	Fresh	7.28	1.11	6.18	15.0	0.771	0.499	0.228	0.033	0.017	0.054	0.015	4.21	2.45	1.29	0.180	0.092	0.295	0.081							
1001/1	Eviscerated, not	1 mo.	5.83	1.07	4.76	15.1	0.906	0.554	0.232	0.029	0.012	0.090	0.013	4.62	2.82	1.25	0.250	0.058	0.458	0.068							
1001/2	glazed, not	2 mo.	6.04	1.08	4.96	17.8	0.821	0.489	0.232	0.031	0.012	0.074	0.014	4.16	2.45	1.16	0.158	0.060	0.377	0.072							
1001/3	wrapped in	4 mo.	7.28	1.05	6.23	13.6	0.793	0.482	0.202	0.027	0.020	0.074	0.015	4.15	2.52	1.06	0.144	0.103	0.389	0.077							
1001/4	paper	8 mo.	8.88	1.21	7.67	16.4	0.998	0.628	0.266	0.030	0.011	0.076	0.017	4.62	2.90	1.24	0.139	0.052	0.352	0.078							
1002/1	Eviscerated,	2 mo.	6.23	0.97	5.26	18.5	0.834	0.494	0.235	0.035	0.010	0.082	0.013	4.27	2.53	1.18	0.179	0.050	0.421	0.086							
1002/2	glazed, not	4 mo.	4.10	1.05	3.05	15.1	0.843	0.515	0.220	0.026	0.015	0.078	0.015	4.26	2.60	1.13	0.132	0.075	0.393	0.076							
1002/3	wrapped in	8 mo.	4.22	1.14	3.08	16.0	0.949	0.631	0.216	0.045	0.010	0.073	0.019	4.77	3.17	1.09	0.234	0.047	0.365	0.095							
1002/4	paper	1 yr. 1 mo.	7.28	0.97	6.31	16.0	0.807	0.493	0.224	0.034	0.013	0.060	0.017	3.86	2.36	1.07	0.163	0.063	0.286	0.080							
1002/5		2 yrs. 1 mo.	7.36	0.97	6.39	18.0	0.813	0.456	0.254	0.029	0.008	0.077	0.018	3.88	2.18	1.21	0.139	0.040	0.367	0.085							
1003/1	Eviscerated, not	4 mo.	4.77	1.07	3.70	16.6	0.866	0.511	0.262	0.039	0.015	0.064	0.014	4.25	2.51	1.29	0.192	0.073	0.312	0.068							
1003/2	glazed, wrapped	8 mo.	7.85	1.08	6.73	17.0	0.897	0.546	0.256	0.018	0.011	0.071	0.013	4.53	2.76	1.29	0.191	0.057	0.357	0.066							
1003/3	paper	1 yr. 1 mo.	7.56	1.02	6.54	18.6	0.835	0.499	0.235	0.021	0.011	0.075	0.015	4.02	2.40	1.14	0.101	0.052	0.360	0.072							
1004/1	Not eviscerated,	4 mo.	4.48	1.07	3.41	16.0	0.855	0.520	0.233	0.024	0.014	0.071	0.017	4.39	2.67	1.20	0.125	0.074	0.363	0.086							
1004/2	glazed, not	10 mo.	7.23	1.05	6.18	25.5	0.992	0.658	0.205	0.026	0.021	0.087	0.021	5.00	3.32	1.03	0.131	0.104	0.438	0.104							
1004/3	wrapped in	1 yr. 1 mo.	7.72	0.97	6.75	15.6	0.849	0.515	0.221	0.026	0.015	0.073	0.025	4.19	2.05	1.58	0.128	0.073	0.361	0.124							
1004/4	paper	2 yrs. 1 mo.	7.67	0.96	6.71	15.5	0.805	0.452	0.228	0.045	0.015	0.084	0.026	4.15	2.38	1.14	0.232	0.077	0.421	0.132							
Bluefish																											
1006/1	Eviscerated	Fresh	7.16	1.24	5.92	35.4	0.933	0.479	0.281	0.045	0.054	0.102	0.017	4.34	2.23	1.31	0.200	0.251	0.474	0.078							
1006/2		Fresh	8.50	1.30	7.20	36.0	1.064	0.580	0.310	0.054	0.050	0.107	0.017	5.01	2.73	1.46	0.254	0.233	0.504	0.079							
1006/1	Eviscerated,	4 mo.	4.54	1.14	3.40	30.0	0.769	0.421	0.175	0.055	0.044	0.113	0.016	3.58	1.96	0.82	0.254	0.203	0.526	0.074							
1006/2	glazed, not	5 mo.	5.65	1.24	4.41	27.1	0.760	0.329	0.266	0.012	0.035	0.112	0.018	3.50	1.52	1.22	0.055	0.162	0.514	0.081							
1006/3	wrapped in	8 mo.	5.49	1.17	4.32	28.0	0.818	0.440	0.220	0.056	0.034	0.103	0.021	3.65	1.96	0.99	0.250	0.149	0.459	0.093							
1006/4	paper	1 yr.	6.80	1.19	5.61	38.0	0.849	0.311	0.329	0.050	0.044	0.143	0.022	3.82	1.40	1.48	0.227	0.197	0.645	0.097							
1006/5		1 yr. 4 mo.	6.27	1.15	5.12	36.1	0.785	0.302	0.319	0.041	0.028	0.112	0.024	3.45	1.33	1.40	0.180	0.124	0.492	0.106							
1006/1a		1 yr. 4 mo.	6.98	1.18	5.80	31.4	0.863	0.351	0.350	0.071	0.021	0.120	0.021	3.84	1.56	1.46	0.316	0.094	0.636	0.091							
1006/6		2 yrs. 3 1/2 mo.	7.22	1.23	5.99	37.4	0.863	0.332	0.334	0.058	0.032	0.137	0.028	3.65	1.40	1.42	0.245	0.134	0.579	0.118							

moisture is also slightly retarded by wrapping the fish in semi-parchment paper, although it is plain that the paper alone gives insufficient protection from the drying effects of the air. Glazing seals the fish in an almost air-tight manner, and is evidently a necessary precaution to preserve the bright and fresh appearance of fish when removed from storage. It is known that ordinary glazing processes add from 3 to 5 per cent to the weight of the fish, but, on the other hand, during storage both the fish and the glaze lose weight as a result of the evaporation of water.

The rate of evaporation of water from the flesh of the glazed weakfish appeared to decrease markedly after the fish had been in storage for a year (Fat-free Solids, Table I). The losses of moisture from the flesh of all the glazed fish during the first 8 or 10 mo. of storage were small and not such as to produce a material change in the appearance of the fish. Except for an occasional broken or dried fin these fish after thawing resembled closely those often found in markets and sold as fresh fish. This good showing was due to the fact that the fish were frozen in cakes, conforming to the size of the usual commercial pan used for the initial freezing, thus reducing the amount of surface exposed to the air; to the protection afforded by the ice-glaze, which was renewed as required; and to the uniformly low temperature (15° F.) at which the fish were kept. In most commercial fish freezers in this country even better results might be expected, as the average temperature of their storage rooms is 5° or 10° (F.) lower than that of the rooms in which the experimental fish were held.

There is no evidence to show that the proportions

of the food constituents of fish, namely, fat, protein, and ash, underwent appreciable changes in cold storage. At first glance it would seem that the variations in the amounts of these constituents in individual fish of the same school are surprisingly great. However, this lack of uniformity in composition is due almost entirely to the variations in the fat content, which is for the most part compensated for by changes in the moisture content. When due allowance is made for this natural variation, the proportions of protein, as represented by nitrogen in the tables, and ash do not change during storage except by amounts which can be accounted for by the slight loss of moisture.

Upon autolysis or bacterial attack the flesh of fish yields both ammonia and amines. In comparative investigations, such as here presented, the sum of the ammonia and amine nitrogen, or nitrogen determined by aëration, may be used as one index of changes in the nitrogenous constituents of fish flesh during storage. It is impossible to say whether such observed changes are due primarily to enzyme action or to the growth of bacteria. In either case, the nitrogen determined by aëration represents end-products which collect and indicate aging processes in fish. During storage there seemed to be a slight tendency for ammonia and amine nitrogen to increase, but this was usually not marked until after one year of storage. Bluefish had a more pronounced tendency to form ammonia and amine nitrogen than most of the lots of weakfish. In the latter, the effects of glazing and wrapping on the production of this form of nitrogen were not apparent. However, the weakfish which were stored with entrails intact exhibited a relatively

TABLE III—NITROGEN DISTRIBUTION CALCULATED IN PER CENT OF TOTAL NITROGEN

No.	Description of Fish	Storage Period	Total	Per cent Nitrogen					Amino Acid	Ammonia and Amine	
				Soluble	Coagulable	In Peptone Bases, etc. (by Difference)	In Tannin-Salt Precipitate	Proteose			
<i>Weakfish</i>											
1002/1	Eviscerated	Fresh	100	28.60	18.26	7.71	1.33	0.33	1.80	0.50	
1002/2		Fresh	100	26.13	15.21	8.01	1.12	0.57	1.83	0.51	
1001/1	Eviscerated, not glazed, not wrapped in paper	1 mo.	100	29.61	18.07	7.40	1.60	0.38	2.93	0.44	
1001/2		2 mo.	100	26.25	15.64	7.40	1.00	0.38	2.38	0.45	
1001/3		4 mo.	100	24.82	15.07	6.36	0.86	0.61	2.32	0.46	
1001/4		8 mo.	100	29.19	18.33	7.81	0.88	0.33	2.23	0.49	
1002/1	Eviscerated, glazed, not wrapped in paper	2 mo.	100	28.07	16.63	7.78	1.18	0.32	2.77	0.57	
1002/2		4 mo.	100	26.93	16.43	7.06	0.83	0.48	2.48	0.48	
1002/3		8 mo.	100	30.44	20.23	6.98	1.49	0.30	2.33	0.60	
1002/4		1 yr. 1 mo.	100	25.19	15.40	6.89	1.06	0.40	1.87	0.63	
1002/5		2 yrs. 1 mo.	100	25.66	14.37	8.05	0.92	0.26	2.42	0.56	
1003/1	Eviscerated, not glazed, wrapped in paper	4 mo.	100	27.67	16.34	8.38	1.26	0.48	2.03	0.44	
1003/2		8 mo.	100	28.38	17.29	8.08	0.57	0.35	2.24	0.42	
1003/3		1 yr. 1 mo.	100	25.28	13.09	9.16	0.64	0.32	2.26	0.45	
1004/1	Not eviscerated, glazed, not wrapped in paper	4 mo.	100	27.66	16.82	7.54	0.79	0.47	2.29	0.54	
1004/2		10 mo.	100	31.80	21.12	6.57	0.83	0.66	2.79	0.66	
1004/3		1 yr. 1 mo.	100	26.72	13.07	10.10	0.82	0.46	2.30	0.79	
1004/4		2 yrs. 1 mo.	100	25.82	14.81	7.09	1.44	0.48	2.62	0.82	
<i>Bluefish</i>											
1006/1	Eviscerated	Fresh	100	27.14	13.94	8.18	1.31	1.57	2.96	0.49	
1006/2		Fresh	100	32.13	17.51	9.55	1.63	1.33	3.23	0.51	
1006/1	Eviscerated, glazed, not wrapped in paper	4 mo.	100	23.00	12.60	5.24	1.63	1.30	3.38	0.48	
1006/2		5 mo.	100	22.26	9.67	7.78	0.35	1.03	3.27	0.51	
1006/3		8 mo.	100	23.04	12.37	6.25	1.58	0.94	2.89	0.59	
1006/4		1 yr.	100	25.16	9.22	9.77	1.49	1.29	4.25	0.63	
1006/5		1 yr. 4 mo.	100	23.31	8.98	9.45	1.21	0.84	3.32	0.72	
1006/1a		1 yr. 4 mo.	100	24.21	9.84	9.82	1.99	0.59	3.38	0.58	
1006/6		2 yrs. 3 1/2 mo.	100	23.79	9.12	9.26	1.60	0.87	3.77	0.77	

strong tendency for this constituent to increase. In fact, the highest percentages of ammonia and amine nitrogen found in any case were in those weakfish frozen "in the round." Since these fish were hard frozen it is difficult to conceive that this increase was due to the slow diffusion of decomposition products into the flesh from the entrails or to the penetration of bacteria from the same source. Whatever the explanation may be, it does not seem impossible that these changes in the flesh are traceable to the presence of the entrails during storage. Further experimentation is necessary before a definite conclusion can be reached.

During storage the bluefish showed a decrease in the amounts of water-soluble and coagulable nitrogen. These constituents increased at first and then decreased in all of the weakfish, except those stored in the eviscerated condition without glazing or wrapping, in which the changes were irregular. The nitrogen in the form of peptone, bases, etc., did not seem to be a variable constituent in the case of any of the fish in storage. The proteose and peptone nitrogen, as precipitated by tannin and salt, did not vary appreciably in amount. Proteose nitrogen, as precipitated by the zinc sulfate method, decreased perceptibly in the bluefish during storage, but not in the weakfish. Bluefish seem to contain, naturally, about three times as much proteose nitrogen as weakfish. Bluefish also contain more amino-acid nitrogen than weakfish, and both species show a slight tendency towards the accumulation of this constituent while in storage.

The interpretation of the meaning of the slight changes in the nitrogenous constituents just described is difficult owing to the small amounts involved, the apparently conflicting tendencies, and to the fact that the role of bacteria and enzymes in such changes is unknown for fish flesh in the hard frozen condition. However, the general conclusion to be drawn from the slight changes observed is that they indicate the beginning of the process of the breaking down of the larger protein molecules into intermediate products, and the

simple substances like amino acids, and ammonia. Such changes are typical of the decomposition of proteins by enzymes or bacteria in ripening processes. The flesh of fish, unlike that of beef or poultry, is known to yield considerable amounts of amines when attacked by bacteria or enzymes. The changes occurring were very slight and of such a nature as would warrant the belief that they were brought about by autolysis rather than by bacterial action.

The iodine number of the fat of the fish which were analyzed after 2 yrs. of storage seemed to be somewhat lower than that of fish which had been stored for shorter periods. This was most pronounced in the case of the bluefish and is probably due to the oxidizing action of the air on the fats near the surface of the fish which was more or less dried out at this stage.

The increase in the acid value of the oils from frozen bluefish and weakfish was the most striking effect noticed in the storage investigations. The weakfish contained a large and also more variable amount of fat when compared with bluefish. The latter did not show such large increases in acid value as the weakfish. This change began to be most noticeable after 8 mo. or a year in storage. Up to this time neither glazing nor wrapping the fish to protect them from the air seemed to have any effect on this aging process in the fat. For this reason, during this time it is probably not influenced as much by atmospheric oxidation as by the bacterial or tissue enzymes in the flesh of the fish itself. The action of such enzymes at the low temperature of storage is necessarily slow and the acids produced in the fats of these fish did not impart a "rancid" flavor to the flesh of the fish which had been stored for a year in the glazed condition.

During the first 13 or 16 mo. of storage the changes in the chemical composition of the fish which had been frozen and stored under the best conditions were not reflected in changes in the flavor of the product after cooking. During the longer periods of storage, however, an undesirable change in the flavor of the cooked flesh could be detected. However, it should be noted,

TABLE IV—ANALYSIS OF FAT

No.	Description of Fish	Storage Period	Specific Gravity 25/25° C.	Index of Refraction 30° C.	Iodine No.	Saponification No.	Acid Value
<i>Weakfish</i>							
1002/1	Eviscerated	Fresh	0.9183	1.4745	142.3	185.8	5.5
1002/2		Fresh	0.9315	1.4749	138.9	182.6	12.4
1001/1	Eviscerated, not glazed, not wrapped in paper	1 mo.	0.9188	1.4709	...	192.6	11.7
1001/2		2 mo.	0.9195	1.4726	132.6	194.8	11.4
1001/3		4 mo.	0.9216	1.4727	129.6	187.1	23.4
1001/4		8 mo.	0.9240	1.4726	...	191.6	17.9
1002/1	Eviscerated, glazed, not wrapped in paper	2 mo.	0.9207	1.4743	132.9	194.1	16.2
1002/2		4 mo.	0.9254	1.4724	124.9	187.3	25.3
1002/3		8 mo.	0.9266	1.4725	128.3	188.7	20.2
1002/4		1 yr. 1 mo.	0.9333	1.4740	136.5	184.1	26.1
1002/5		2 yrs. 1 mo.	0.9267	1.4740	...	180.5	21.3
1003/1	Eviscerated, not glazed, wrapped in paper	4 mo.	0.9221	1.4728	136.7	190.9	15.9
1003/2		8 mo.	0.9141	1.4704	133.6	177.9	20.1
1003/3		1 yr. 1 mo.	0.9187	1.4724	124.1	194.2	12.8
1004/1	Not eviscerated, glazed, not wrapped in paper	4 mo.	0.9261	1.4731	140.3	191.0	22.4
1004/2		10 mo.	0.9147	1.4703	128.6	182.3	20.2
1004/3		1 yr. 1 mo.	0.9214	1.4733	128.8	184.1	25.2
1004/4		2 yrs. 1 mo.	...	...	...	...	...
<i>Bluefish</i>							
1006/1	Eviscerated	Fresh	0.9310	1.4786	126.0	178.9	38.9
1006/2		Fresh	...	1.4745	136.4	172.8	16.3
1006/1	Eviscerated, glazed, not wrapped in paper	4 mo.	0.9325	1.4723	137.9	187.9	21.8
1006/2		5 mo.	0.9148	1.4735	126.5	...	27.4
1006/3		8 mo.	0.9286	1.4745	133.4	188.9	34.1
1006/4		1 yr.	0.9078	1.4694	127.5	172.6	32.0
1006/5		1 yr. 4 mo.	0.9269	1.4745	118.8	176.1	40.4
1006/1a		1 yr. 4 mo.	0.9204	1.4710	118.5	181.3	41.6
1006/6		2 yrs. 3 1/2 mo.	...	...	...	...	...

in connection with the longer storage periods, that the holding temperature (15° F.) is higher than is ordinarily used commercially.

SUMMARY

Two species of salt water fish, *i. e.*, bluefish, *Pomatomus saltatrix*, and weakfish, *Cynoscion regalis*, were frozen by the commonly used air process, stored in a holding room whose temperature remained near 15° F. and examined chemically and organoleptically at the conclusion of holding periods varying in duration from 1 to 27.5 mo. The bluefish before freezing were eviscerated and were kept "glazed" with an ice covering during storage. The weakfish were divided in four lots, each of which was prepared for storage in a different manner in order to determine the effects of glazing, paper wrapping, and the presence of entrails on the keeping quality of these fish.

I—Bluefish and the weakfish stored in the glazed condition remained palatable and wholesome during storage for 16 and 13 mo., respectively.

II—Weakfish, which had not been glazed or otherwise protected against loss of moisture, dried out so rapidly that at the end of 4 mo. they were considered unmarketable.

III—The loss of moisture in weakfish was checked but slightly by wrapping the fish in semi-parchment paper. Paper wrapping is by no means an adequate substitute for the ice-glaze.

IV—The keeping quality of glazed uneviscerated weakfish in storage up to 13 mo. was practically the same as that of the glazed eviscerated weakfish.

V—Both bluefish and weakfish became unpalatable after 2 yrs. of storage. The desiccation and consequent ruining of the appearance of the fish after long periods of holding were more marked than they would have been if the temperature of the storage rooms had been nearer the average commercial holding temperature.

VI—The following changes in the composition of the flesh of the fish during storage were noted:

(a) Water-soluble and coagulable nitrogen decreased in the bluefish, and increased at first, then decreased in all weakfish except those stored in the eviscerated

condition without glazing or wrapping. In the latter the changes were irregular.

(b) Proteose nitrogen decreased in the bluefish but underwent no distinctive change in the weakfish.

(c) Amino-acid nitrogen increased slightly in both bluefish and weakfish.

(d) Ammonia and amine nitrogen increased markedly in the glazed uneviscerated weakfish and only slightly in the other fish.

(e) The acid value of the extracted fat increased in all samples.

(f) The iodine number of the extracted fat decreased in all samples, the change being greatest in the bluefish.

SOLID PHASES OBTAINED BY THE EVAPORATION OF CERTAIN SOIL EXTRACTS¹

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In recent years a very considerable amount of investigation in the Bureau of Soils has been devoted to the nature and functions of soil solution and to water extracts of soils. The results are published in the various bulletins of the Bureau.² For the most part these investigations have concerned themselves with the total material extracted and its relation to plant growth and to the so-called plant food constituents. Similarly in various other publications are found more or less extensive accounts of the relation of water to soil dealing with concentration,^{1-8,*} movement,^{9,10} methods of extraction and materials extracted by water.^{11,15} There can also be found in the literature an enormous amount of material dealing with the mineral composition of sea water, saline lakes and deposits, well and drainage waters, etc. These publications are sum-

¹ Published by permission of the Secretary of Agriculture.² See various publications of the Bureau of Soils, U. S. Department of Agriculture, Washington, D. C.

* Numbers refer to Bibliography, p. 668.