Marine Protein Concentrate

UNITED STATES DEPARTMENT OF THE INTERIOR
FISH AND WILDLIFE SERVICE
BUREAU OF COMMERCIAL FISHERIES

Fishery Leaflet 584

UNITED STATES DEPARTMENT OF THE INTERIOR
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A SUMMARY REPORT ON THE DEVELOPMENT OF A
PROPOSED COMMERCIAL MANUFACTURING PROCESS
AND ON THE PROPERTIES OF A TEST SAMPLE

By

Bureau of Commercial Fisheries
Technological Laboratory
College Park, Maryland

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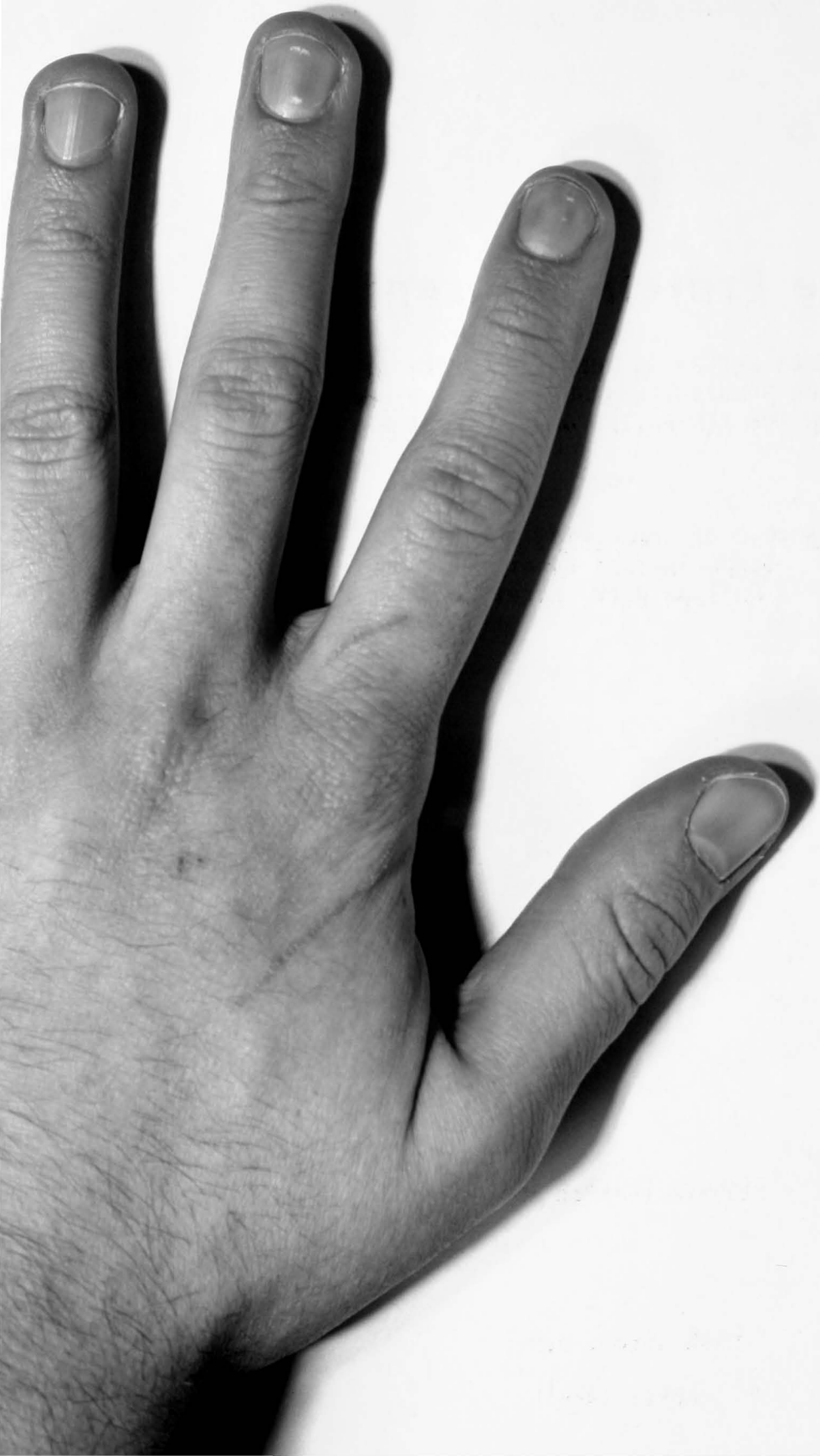

FOREWORD

Laboratory and small-scale production studies by Bureau of Commercial Fisheries scientists have demonstrated that a highly nutritious protein concentrate suitable for human consumption can be prepared from fish.

The development of a commercially feasible method for manufacturing such a product has two-fold significance: (1) Marine Protein Concentrate (MPC) can be a powerful weapon in the war on world-wide malnutrition, and (2) large-scale manufacture of the protein concentrate in the U.S. would mean important economic benefits to the domestic fishing industry from the fisherman through the processor. It has been estimated that billions of pounds of fish suitable for use as a concentrated protein product can be harvested annually off the coasts of the United States.

The research summarized in this report was conducted in response to a directive by Secretary of the Interior Stewart L. Udall that the Bureau develop, as rapidly as possible, a commercially feasible method for making a marine protein concentrate that would be nutritious, healthful, wholesome, and inexpensive. This report is based upon Bureau research as well as upon work carried out by other scientists in this country and abroad.

While the results detailed in this report indicate a significant achievement in the technology of manufacturing a protein concentrate from whole fish, it should not be assumed that research in this field is complete. More work needs to be done on species of schooling fish other than the hake used in the initial experiments and other manufacturing processes besides the chemical method require research and refinement. However, the major obstacles have been surmounted and those problems that lie ahead appear amenable to solution.

Donald L. McKernan

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Director

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ABSTRACT

General information is given on a method to convert red hake into a high-quality marine protein concentrate for human consumption. Plans, as well as estimates of processing and product costs, are shown for a proposed commercial process. Data are presented on the physical, chemical, nutritional, bacteriological, and toxicological studies on marine protein concentrate, produced by a method closely approximating the proposed commercial process.

INTRODUCTION

The world population picture changes rapidly with passing years. In most of the emerging countries during the past 2 decades there have been such significant advances in world health that populations of these countries have increased more rapidly than their economic development. The result is widespread malnutrition, particularly protein malnutrition, for roughly two-thirds of the world population. At the same time, pressures from the population explosion have greatly reduced the proportion of arable land available for crops and more especially for grazing animals that could provide vitally needed animal protein.

Man must, therefore, turn to other sources of animal protein available in enormous quantities and at low cost.

The sea surrounding every continent contains large underutilized or unutilized resources of fish. The present U. S. catch alone, now some 5 billion pounds, could be expanded three to five times. What is needed is to develop a method of using fish as a high-quality protein supplement to upgrade lesser quality vegetable proteins, which are today the world's principal source of this important nutrient. The nature of the product must be such that it can be stored, shipped, and marketed at normal temperatures throughout the world and yet retain the high nutritional value of the fresh fish. The product must also be inexpensive and be available in a form that can be readily introduced into a wide

variety of traditional food products, to ensure that it will find its way into the diets of the peoples in developing countries.

MPC is a high-protein product that meets our requirements. The Bureau of Commercial Fisheries was, therefore, given the task of developing different processes to produce various marine protein concentrates that would meet these specifications. Because of the urgency of the need, Secretary Stewart L. Udall suggested that the research be accelerated to develop as rapidly as possible one simple, practical, and commercially feasible method for the preparation of a wholesome, nutritious, stable, and low-cost MPC.

This report summarizes some of the results of these accelerated MPC investigations at the Bureau of Commercial Fisheries Technological Laboratory, College Park, Md. The laboratory has developed one commercial MPC process and has assessed the properties of a test product, derived from whole fish of one species and prepared by a method that closely approximates the commercial process.

DEVELOPMENT OF A COMMERCIAL MPC PROCESS

On the basis of knowledge and practical experience available to Bureau scientists at the beginning of this accelerated program, we decided to "freeze" all exploratory research and to concentrate on the study of a single solvent extraction method. Isopropyl alcohol was chosen as a solvent because of its unique qualifications, such as usage in other food processing endeavors, low toxicity, low price, low boiling point, good efficiency in extracting water and lipids, and bactericidal properties.

Red hake was chosen as the raw material because (1) it is abundant off the U. S. coast; (2) it is largely unutilized as a food fish; (3) because we could carefully control the quality of the hake supplied to the laboratory; and (4) because this fish is available for a considerable period during the year.

To devise plans for a sound commercial processing method, we followed a logical sequence of development, starting with laboratory experiments and ending in small pilot plant experiments (model-scale unit studies). Because of favorable results in the preceding studies, both as to the process and the product, we were able to use the data to formulate general plans for an industrial-scale plant (50 tons of raw fish processed per 24 hours) and to estimate processing and product costs involved for this size plant during 250 days of operation per year. (This size plant and this number of working days are considered minimum for a commercial endeavor and maximum for a pilot endeavor.)

Our presentation of the plans and cost estimates for the proposed commercial process, however, is preceded by a general discussion of certain factors that need special emphasis because of the process and the raw material. These factors, we believe, are as important to assure the production of a high-quality MPC as are the suggested processing conditions. This section dealing with the proposed commercial process,

therefore, is divided into three parts--general considerations, plans for large-scale production units, and processing costs.

GENERAL CONSIDERATIONS

The design of a plant for the manufacture of MPC by a solvent-extraction procedure must meet requirements for handling food materials and must provide safeguards against explosions. Superimposed upon the physical requirements and attendant to them is the necessity to define certain specifications for the raw material and the solvent to be used in the process. There are, as well, overall requirements for the sanitation that must be maintained at all stages of the operation.

Physical Features of the Plant

Among the factors important to an initial decision on the design and construction of a plant are the location and size of the plant and the building materials and requirements.

Location and size of plant.--The efficient and economic operation of a 50-ton-a-day plant (raw fish input) requires a supply of fish for a working schedule of at least 250 days per year. If costs of the product are to be kept down and quality of the product is to remain high, the plant should be located as near as possible to a source of the desired species of fish. Land transportation from port of landing to plant adds greatly to raw material costs and may detract from fish quality. Preferably the source of fish should be close enough so that most of the fish can be landed the same day they are caught or at least within 24 to 36 hours. If this is impossible, satisfactory preservatives must be used to ensure that the processing plant will handle raw material of high-grade quality.

If emergencies, such as breakdowns, occur at the plant and make it necessary to store the raw fish for a longer time, the fish can be ground, mixed with isopropyl alcohol and held in tanks. By halting enzymatic or bacterial decomposition, the solvent effectively checks the spoilage of the fish. It would be equally satisfactory, if economic considerations warrant, to grind promptly the fresh catch on fishing vessels and hold the ground fish in tanks with recommended levels of isopropyl alcohol. This technique would maintain quality enroute to the processing plant.

Selecting the location and size of the plant requires very careful and deliberate consideration. Failures of several early efforts at MPC manufacture were due largely to lack of such planning and to failure to maintain high-quality in the raw fish supply.

Buildings and process equipment.--The basic principles that determine plant design are (1) MPC is a human food product; (2) an inflammable solvent is used in the process; and (3) to minimize contamination, the storage area for the finished, dry MPC must be isolated from the raw fish handling and processing sections. Regulations covering the first

two aspects are well defined in various State codes and include such requirements as stainless steel or ceramic-lined processing vessels, pumps, tanks, and other equipment in contact with the product; fully sanitary (dairy-type) piping and valves of similarly corrosion-resistant material easily disassembled for cleaning; and explosion-proof motors, switches, controls, and other electrical equipment.

Details on requirements for flooring, ceiling, lighting, ventilation, plumbing, water, dust control, cleaning and sanitizing, packaging, and storage may be obtained from various local, municipal, and State codes and from the U. S. Public Health Service and Food and Drug Administration regulations relating to the handling of human food products.

Specifications for the Solvent and Fish

Solvent.--Suitable quality isopropyl alcohol is available commercially in drums or tank cars. It is synthetically prepared from propylene and is sold, with only traces of impurities, as the anhydrous alcohol or in the form of a constant-boiling mixture with water (91 volume percent isopropyl alcohol) containing 87.7 percent by weight of the alcohol. The specifications for commercial isopropyl alcohol are given in Table 1. Proper storage conditions are important to avoid formation of rust in the container and the contamination of the alcohol by water, dust, and dirt.

Raw material.--The hakes, species of the genera Urophycis, Merluccius, and Theragra, variously known as red hake, ling, squirrel hake, silver hake, walleye pollack (Alaska pollack), whiting, Pacific hake, white hake, southern hake, and merluzza (South American) are all soft-fleshed fish with a maximum storage life in ice of about 10 days. The flesh is white and normally low in fat content. In the usual commercial catch their size is quite variable, apparently even when taken from the same grounds at any given time. Small fish, 9 to 12 inches long and weighing less than a pound sometimes make up the bulk of the New England ground catch, but larger fish ranging up to 25 to 30 inches and 8 to 10 pounds are common.

Although only red hake (Urophycis chuss) were used in the research reported here, it is probable that all Urophycis, Merluccius and Theragra species are acceptable for the production of MPC by the solvent extraction method.

On the vessel, the fish should be stowed in ice below deck as promptly as is possible. Under no circumstances should a second trawl load be dumped on the deck until the catch of the previous haul has been stowed properly. The flesh of hake is so soft that a chute to the floor of the hold should be used to minimize bruising of the flesh. Icing should be adequate to chill the fish rapidly and to maintain them at 38° F. or less until they are landed. Use of finely crushed ice will avoid undue bruising of the flesh. Fish should be protected from bulkheads and the outer surfaces of the hull by a maintained layer of ice. The fish and ice should not be piled too deeply in the pens because these fish crush easily.

Table 1.--Commercial specifications for 91 percent and anhydrous isopropyl alcohol[1/]

Type of isopropyl alcohol	Minimum concentration of alcohol	Specific gravities 20°/20° C.		Acidity	Maximum color	Maximum non-volatile matter	Distillation		Residual odor	Water dilution transparency
		Min.	Max.				Min. initial	Max. dry point		
	Percent by volume	G./ml.	G./ml.	Weight percent of acetic acid	Hazen units	Mg./100 ml.	°C.	°C.		
91 percent	91.0	0.8175	0.8190	0.002	10	2	79.7	80.7	None	Clear at infinite dilution
Anhydrous	99.0	0.7876	0.7876	0.002	10	2	81.5	83.0	None	Clear at infinite dilution

1/ Either of these commercially available forms of the solvent is considered acceptable for the process herein described. Economic and other considerations would determine choice.

When delivered, the fish should be examined routinely by an experienced inspector to provide satisfactory control of the quality of the raw material. The fish should meet the following general requirements: belly walls should be intact; gills should be still red - not discolored grey or brown; eyes should be normal, with bright color, and not be sunken and clouded; and the skin should be generally intact.

The fish should be unloaded in baskets or boxes or by an elevator conveyor in such a manner that the flesh will not be damaged by cutting, tearing, or crushing. Raw fish in the plant should be stored for as short a time as practical to ensure that the processing equipment operates at an efficient level (average 75 percent capacity during the period on stream). Unless some suitable method of holding the fish is available, as described earlier, vessel landings should not be scheduled so that the raw fish will have to be held at the plant for more than 48 hours--normally, the fish should start through the process within 12 hours after being landed at the plant.

Sanitation

Good food-plant sanitation practices must be followed in the manufacture of MPC to ensure a final product of good quality and one that is safe microbiologically. Sanitation practices should start on the deck of the fishing vessel and be continued through the plant until the finished product has been packaged.

On the vessel.--The film-like coating over the skin of fish often has large numbers of microorganisms, which may contaminate heavily not only the vessel hold but also the deck boards, hold surfaces, and items repeatedly used in handling the fish--such as shovels, wash tanks, baskets, boxes, and penboards. After the fish have been unloaded at the dock, the hold of the vessel and all other surfaces and utensils involved should be washed down thoroughly, preferably with a pressure gun and a detergent solution. After the organic detritus has been removed, a spray of a solution of sodium hypochlorite--300 parts per million of available chlorine--will give effective sterilization.

In the plant.--Many of the features of the building layout, equipment design, lighting, ventilation, and materials-handling process proposed for MPC manufacturing plants are common to any well-designed food processing plant because of their important role in maintaining good sanitation.

Even the best equipment and building construction concepts are of little value, however, unless vital cleaning operations are given continuous attention. In general, it is good practice to flush equipment with cold or preferably lukewarm water (38°-46° C. - 100°-115° F.) to remove or loosen adhering particles of fish. Further operations include: (1) cleaning the equipment with a suitable solution at the proper temperature for optimum effect; (2) thoroughly brushing the hard-to remove solids; (3) rinsing with hot water (82° C. - 180° F.); and (4) final sanitizing.

Also important to the maintenance of good sanitation is control of rodent and insect pests. Proper screening and good building construction are effective only if screens are kept in good condition, doors are kept closed, and other safeguards against entrance of these pests are maintained. Insecticides and rodenticides, when needed and where permitted, should be handled with care, because most are toxic to humans and should never be used in such a manner that they might injure plant personnel or contaminate the product. These chemicals should be stored entirely separate from the storage areas holding the product or the packaging material. All such pesticide containers should be clearly and conspicuously labeled as a toxicity and as to proper methods of application.

In the final analysis, the worker is the most important barrier against product contamination, and perhaps more importantly he is responsible for maintaining good sanitation. Each worker should have a regular medical examination. He should be provided with clean protective outergarments and be required to maintain high standards of personal hygiene.

Packaging.--All precautions taken to prepare and package a sterile sanitary product are useful only if the package is effective under all conditions of handling, storage, and distribution. The packaging material itself must be clean and free from substances that might affect product odor, flavor, and appearance, or cause microbial contamination. According to the Federal Food, Drug, and Cosmetic Act, Sec. 402 (2) (6), a "food would be deemed adulterated--if its container is composed, in whole or in part, of any poisonous or deleterious substance which may render the contents injurious to health." Other factors important in a suitable packaging material for MPC are moisture-vapor permeability, gas permeability, heat sealability, stability to sunlight and ultraviolet light, and normal temperature effects. All these factors must be considered.

PLANS FOR COMMERCIAL PRODUCTION UNITS

Having considered general matters, associated with the commercial production of MPC, we must now turn our attention to matters specific to the construction of a plant capable of processing 50 tons of raw fish per day. The rationale for the selection of process and equipment is presented in this section, after which pertinent operating procedures are described. To illustrate the continuous sequence of the projected production procedure, we present an overall materials-handling schedule, which is followed by a general discussion section.

All of the discussions pertain to a plant with a capacity of 50 tons of raw material per day. Such items, however, as the following schematic flow diagram would remain essentially unchanged for plants with processing capacities either somewhat smaller or larger than that of the 50-ton plant. It would primarily be the dimensions or capacity of the equipment and the schedules of operation that would vary for

plants of different capacities. All the equipment discussed is of conventional design and is believed to be readily available.

Selection of Process and Equipment

Process.--Based on the experimental results obtained to date in this program and on the engineering analyses performed, a scheme or proposal for the large-scale manufacture of MPC is presented for the information of the public and for review of commercial concerns interested in MPC. The process uses red hake as the starting material and isopropyl alcohol as the solvent for the extraction of water and lipids. A schematic flow diagram is presented in Figure 1, which shows a manufacturing technique that can be described as a three-stage, countercurrent extraction of suitable quality fish by isopropyl alcohol.

Equipment.--In selecting the equipment, we took great care to ensure that every suggested piece of equipment is commercially available and suitable for the processing of a human food product such as is contemplated for MPC.

The equipment chosen for the production of MPC comprises the following major items.

1. Food grinder of the meat-grinder type, easily disassembled for cleaning and sterilizing.

2. Process tanks, including one covered stainless-steel mix tank with variable-speed agitator capable of thoroughly mixing the viscous ground fish-solvent slurry and two covered, jacketed, stainless-steel extraction tanks, each equipped with a variable-speed agitator.

3. Continuous horizontal centrifuge.

4. Conventional solvent recovery plant.

5. Rotating (double-cone) vacuum dryer.

6. Vacuum system with solvent deodorizing equipment.

7. Grinding equipment.

8. Slurry pumps.

9. Solvent-storage tanks.

Operating Procedures for the Production of MPC

Figure 1 shows an installation capable of processing 50 tons of raw fish per day. The proposed operating procedure for such a plant is based upon the essential operating and equipment features of the model-

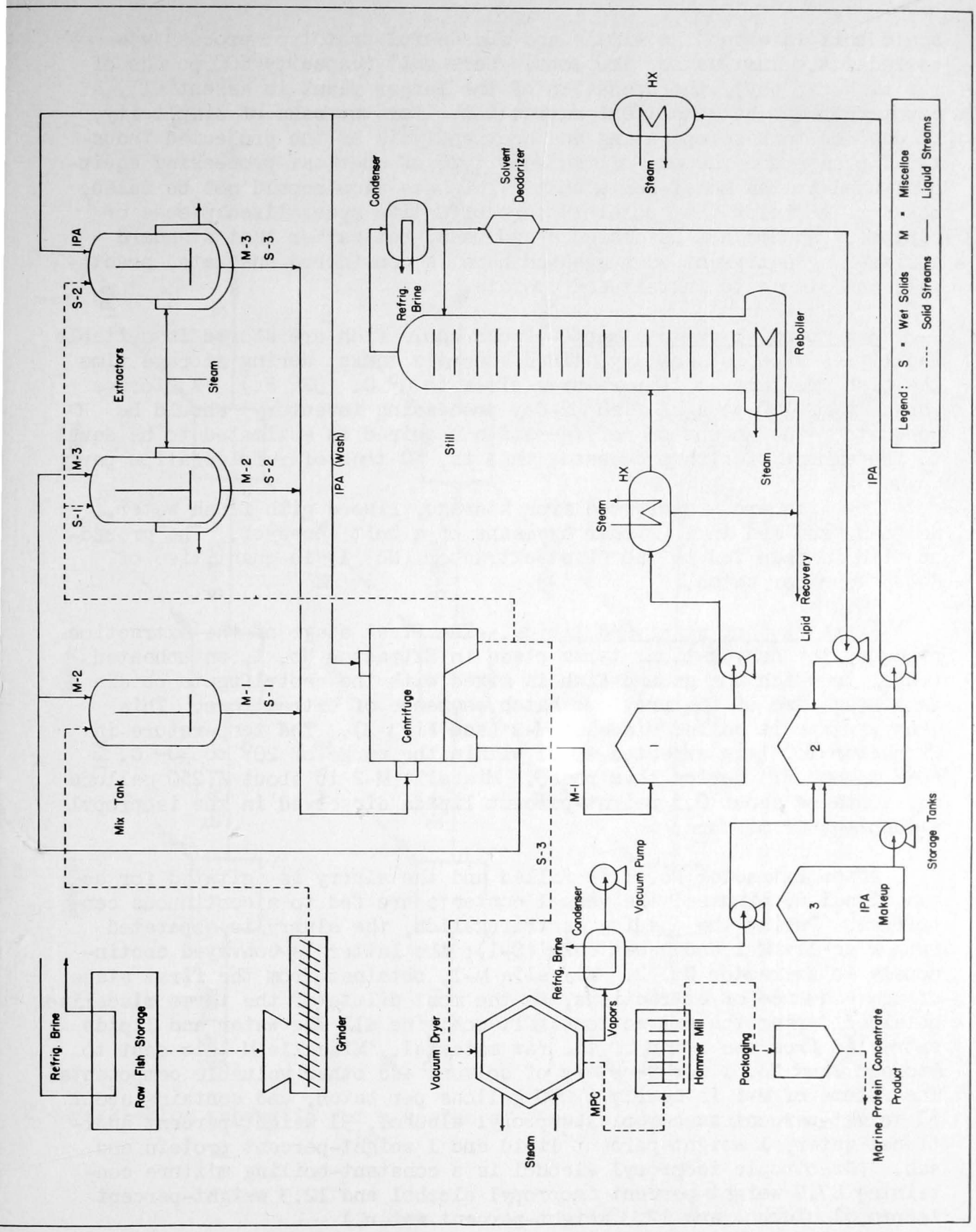

Figure 1--Flow diagram for the manufacture of marine protein concentrate (3-stage countercurrent extraction)

scale unit in which the simple and successful prototype process was worked out. Just as for the model-scale unit (capacity 600 pounds of raw fish per day), the operation of the larger plant is essentially a countercurrent batch solvent extraction. For the sake of simplicity, we decided to base operating and cost analysis of the projected industrial plant upon the use of the same type of chemical processing equipment used in the model-scale unit. This decision should not be taken, however, to infer that possibly more effective specialized pieces of equipment should not be designed and used, but rather that standard engineering equipment as suggested here is considered adequate, practical, and simple to install and operate.

Raw material pretreatment.--Fresh whole fish are stored in suitable facilities such as sanitary, lined concrete tanks; during storage time the fish are kept at temperatures close to 0° C. (32° F.). A storage capacity of 100 tons of fish (2-day processing inventory) should be adequate. The amount of refrigeration required is estimated to be equal to the amount of fish processed; that is, 50 tons of refrigeration per day.

The fish are transferred from storage, rinsed with fresh water, weighed, and fed to a grinder by means of a belt conveyor. The ground-up fish is then fed to the first extractor (No. 1) in quantities of 8-1/3 tons per batch.

Extraction of water and lipids.--The first stage of the extraction process, the dehydration, takes place in Extractor No. 1, an unheated vessel in which the ground fish is mixed with the centrifugate obtained from stage two of the previous batch sequence of extractions. This centrifugate is called Miscella M-2 (see Fig. 2). The temperature in Extractor No. 1 is expected to be within the range of 20° to 30° C. (68° to 86° F.) during this phase. Miscella M-2 is about 4,250 gallons and contains about 0.5 weight-percent lipids dissolved in the isopropyl alcohol-water mixture.

After Extractor No. 1 is filled and the slurry is agitated for an additional 50 minutes, the vessel contents are fed to a continuous centrifuge. During the ensuing centrifugation, the slurry is separated into Miscella M-1 and a wet cake (S-1); the latter is conveyed continuously to Extractor No. 2. Miscella M-1, obtained from the first stage of the sequence of extractions, is the most dilute of the three miscellae obtained during the extractions. It contains all the water and lipids extracted from one batch of the raw material. Miscella M-1 is sent to Storage Tank No. 1 for recovery of solvent and other valuable components. The volume of M-1 is nearly 6,000 gallons per batch, and contains about 67 weight-percent azeotropic isopropyl alcohol, 31 weight-percent additional water, 1 weight-percent lipid and 1 weight-percent protein and ash. (Azeotropic isopropyl alcohol is a constant-boiling mixture containing 87.7 weight-percent isopropyl alcohol and 12.3 weight-percent isopropyl alcohol and 12.3 weight-percent water.)

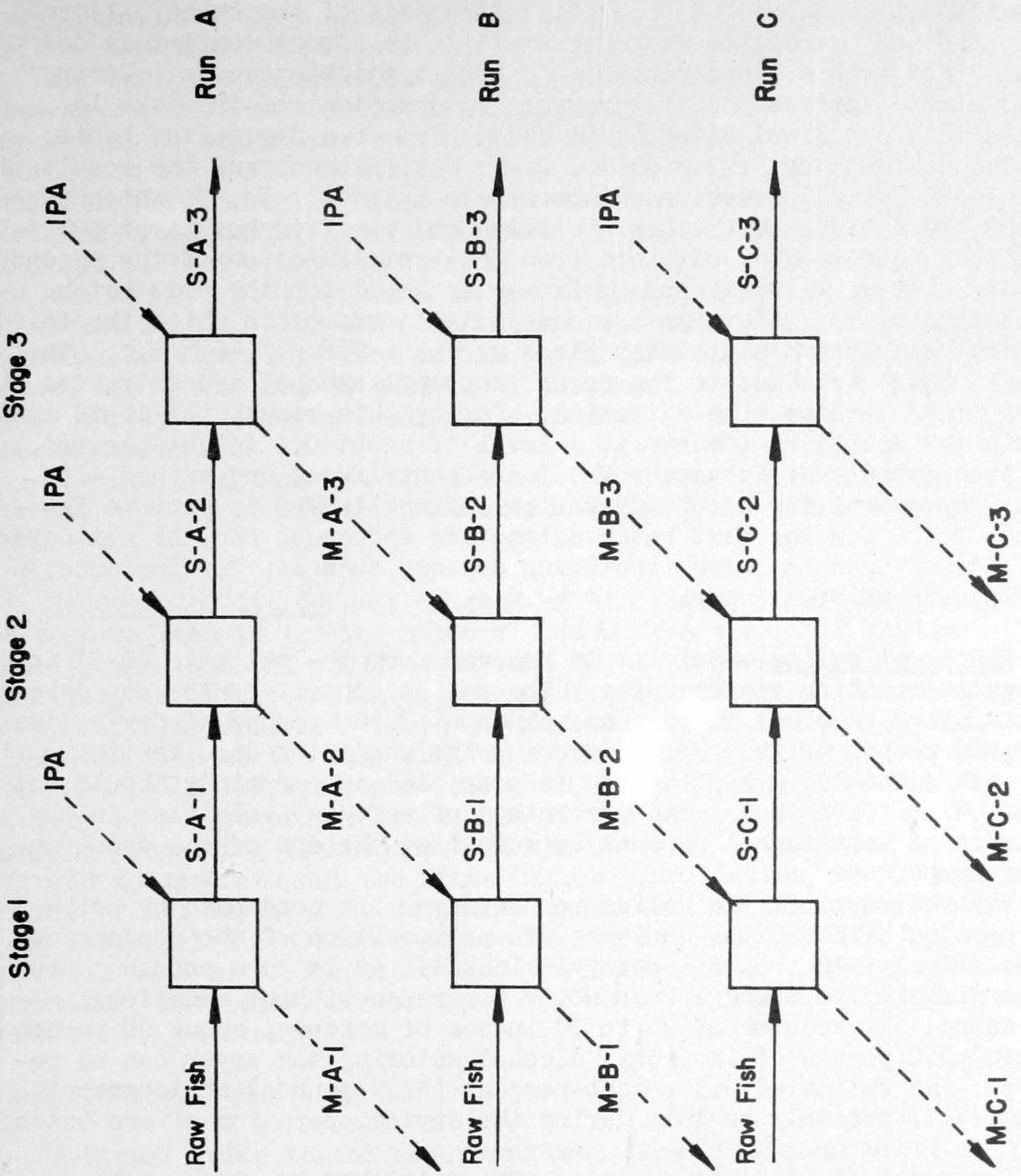

Figure 2--Flow schematic for a series of 3-stage countercurrent extractions

The second extraction stage takes place in the jacketed Extractor No. 2. The wet cake (S-1) from the first stage is added continuously with agitation to the extractor, which contains the centrifugate, or Miscella M-3, obtained from the third stage of the previous batch sequence of extractions. Extraction temperature during this stage is maintained at about 75° C. (167° F.). Analysis of the cake (S-1) entering the hot extraction stage shows it to be almost completely dehydrated but with a lipid content of about 5 weight-percent. During the 90-minute extraction, the average lipid content of the cake is reduced to about 1 weight-percent, calculated on a dry solids basis.

The contents of Extractor No. 2 are centrifuged, and the resultant wet cake (S-2) is conveyed continuously to Extractor No. 3, which contains 3,400 gallons of freshly distilled and purified hot isopropyl alcohol. Miscella M-2, obtained from the centrifugation of the second extraction stage is delivered to Extractor No. 1 for the next batch.

Extractor No. 3 is also a steam-jacketed vessel in which the third and final extraction step takes place at about 75° C. (167° F.). The wet cake (S-2) is added to the fresh isopropyl alcohol and extracted for about 70 minutes with agitation. During this stage, the lipid content in the solids is lowered to a level of about 0.3 weight-percent.

The contents of Extractor No. 3 are centrifuged and washed with fresh isopropanol for about 50 minutes. Miscella M-3 is sent to Extractor No. 2 for the next batch extraction sequence, and the wet cake (S-3) is conveyed to a desolventizing device, such as, for instance, a rotating vacuum drum dryer.

Desolventization.--Solvent is removed from the wet cake (S-3) in the heated rotating vacuum dryer. The solids content of the wet cake at this stage is about 50 percent, or about 2,500 pounds of dry solids and 2,500 pounds of isopropyl alcohol. The suggested desolventizing temperature should be kept as low as possible and certainly should not exceed 90° C. (194° F.). The percentage of residual volatiles in the MPC is to be less than 1 percent by weight at the end of the desolventizing stage.

Vapors drawn off the solids in the dryer are condensed by using refrigerated brine as the coolant. The composition of the condensate is essentially azeotropic isopropyl alcohol. It is pure enough to be pumped directly to Storage Tank No. 2 for reuse without additional processing. At vacuums of 26 to 29 inches of mercury, about 99 percent of the 2,500 pounds of isopropyl alcohol entering the dryer can be recovered. An estimated 0.5 weight-percent (12.5 pounds) of isopropyl alcohol will probably be lost during the drying operation of one batch.

Treatment of finished product.--The dried MPC is conveyed to a mill, where the MPC is ground to a fine powder. The powder is packaged in 50-pound lined fiberboard containers and sent to storage, ready for shipment.

The three-stage countercurrent extraction process, briefly described above, produces 15,000 pounds of MPC per day from 100,000 pounds of raw, lean fish.

Recovery of solvent.--The bulk of the isopropyl alcohol used in this extraction is recovered from Miscella M-1. Each batch of 8-1/3 tons of raw fish yields about 6,000 gallons of Miscella M-1, which is held in Storage Tank No. 1. The miscella is pumped continuously from this tank to a distillation column. The feed material to the column is preheated in a steam-coiled heat exchanger, which also serves as a settling tank for the removal of the relatively fast settling fines.

The azeotropic isopropyl alcohol is recovered by condensing the overhead vapors from the distillation column. Part of the condensate, composed of 87 weight-percent alcohol and 13 weight-percent water, is returned to the still as reflux. The recovered solvent is deodorized with activated carbon or other device to remove trace quantities of odoriferous compounds, such as amines. Storage Tank No. 2 contains the isopropyl alcohol recovered from the vacuum dryer and the distillate from the column; it also contains the isopropyl alcohol required to make up solvent losses. For each batch, the solvent losses are anticipated to amount to about 1 percent of the 3,400 gallons of freshly distilled solvent used--that is, 34 gallons of isopropyl alcohol per batch. A 30-day supply of isopropyl alcohol is stored in Storage Tank No. 3 for process makeup.

The still bottoms leaving the reboiler contain water, isopropyl alcohol, as well as lipids, fine solids (fines) and other water-soluble components.

Recovery of lipids.--One aspect of the plant design that has not yet been defined is the recovery of lipids from the still bottoms. Presumably, the fish oil would exist as a separate phase because of its low solubility in water; therefore, separation should be relatively simple. In experiments performed on the distillation of Miscella M-1, however, a large portion of the oils adhered to the protein fines. This situation must be anticipated when lean fish are processed--that is, the recovery of a high proportion of the fish oils may require fairly complicated processing techniques. If the miscella is filtered or recentrifuged to separate out the fines before distillation, considerable amounts of oil may be lost with the fines. If the fines are washed with fresh isopropyl alcohol to recover the lipids, the distillation equipment will need to be larger so that the added costs of these additional operations, materials, centrifuges, and other equipment could offset the value of the recovered oil.

Overall Material-Handling Schedule

A visual guide to the countercurrent flow of solvent and fish solids is presented in Figure 2.

The material-handling schedule for a proposed 50-ton-per-day plant is shown for a 12-hour period in Figure 3, which illustrates the contents and activities of the major equipment units. It can be seen that fresh isopropyl alcohol fed from the storage tank to Extractor No. 3 at Hour-3 finally leaves the system as part of the Miscella M-1 at Hour-11. Similarly, the batch of ground fish entering Extractor No. 1 at Hour-0 yields the solid product from the dryer at Hour-9. It is important to

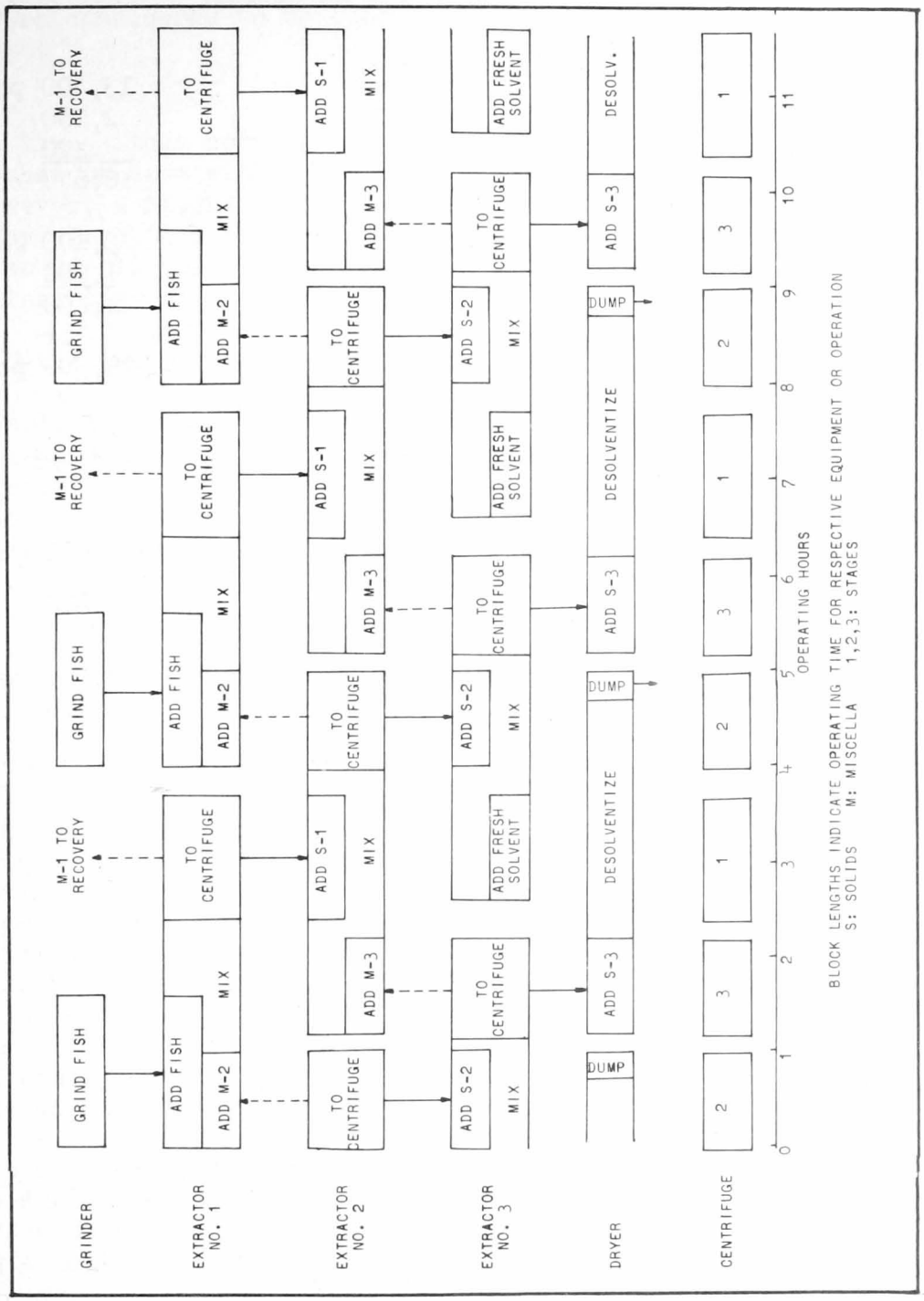

Figure 3--Time schedule of operations for a countercurrent solvent extraction of marine protein concentrate

note, however, that every 4 hours, a fresh batch of fish (8-1/3 tons) enters the first stage of the extraction and that every 4 hours, 2,500 pounds of MPC leave the dryer.

For the 50-ton-per-day plant, the following quantitative relations should be applicable when a lean fish is processed. These values have been used in the assembly of the three-stage countercurrent extraction equipment as well as in the calculation of the materials schedule for each of the six batches to be processed per 24 hours.

1. Ground fish composition

Water	13,300 pounds
Protein and ash	2,950 "
Lipids	420 "
	16,670 "

2. Fresh solvent added

In Mix Tank No. 3	3,400 gallons
In centrifuge hot wash	1,000 "
	4,400 "
	or
	29,300 pounds

3. Solvent to fish ratio

Isopropyl alcohol/wet fish - 29,300/16,670 = 1.75/1

Isopropyl alcohol/dry fish solids - 29,300/3,370 = 8.7/1

4. Solvent recovered

From distillation column	4,000 gallons
From vacuum drum dryer	360 "
	4,360 "

5. MPC produced

MPC	2,500 pounds
Solids lost	450 "

Discussion

The capability of this type of process to extract lipids and water from fish was demonstrated in laboratory- and model-scale unit experiments. Probably, we cannot assume that the same efficiency of extraction can be realized in a 50-ton-per-day plant as in laboratory-scale equipment. Certain factors of safety were therefore incorporated in the design of the plant: The ratio of solvent to raw fish was fixed at 1.75 pounds solvent per pound of raw material, corresponding to 25.7 gallons per 100 pounds of fish. This represents an increase in the ratio of solvent used over and above the ratio that small-scale experiments have shown to be effective; the increase is considered desirable in order to present a conservative economic evaluation of the process.

In addition to the increased solvent-to-fish ratio, the extraction temperature in the final two mix tanks in the 50-ton-per-day plant will be 75° to 80° C. (167° to 176° F.), because laboratory tests show that this extraction temperature guarantees a satisfactory removal of lipid

from fish solids. Since steam-heated tanks are used and the liquid feed is preheated, there should be no problem in maintaining this temperature.

Another factor of safety (for lipid removal) in the design of the 50-ton-per-day plant is the use of 70° C. (158° F.) solvent for washing the centrifuge cake at each stage of the extraction. This technique is common industrial practice and will lower the amount of lipids carried to the next stage of the extraction in the liquid associated with the wet cake. The design, and the economic evaluation of the design, are therefore considered to be conservative.

PROCESSING COSTS

A three-stage countercurrent batch extraction process should yield a finished product with a residual lipid content of less than 0.5 percent. If, however, a slightly higher content of lipid were permissible, for instance close to 1 percent, a two-stage extraction process might be used, which would slightly reduce processing costs. The costs for both these operations therefore are examined below.

Three-Stage Countercurrent Extraction

Table 2 presents costs for items of equipment for a three-stage, 50-ton-per-day batch commercial plant. Total equipment costs are $231,500.

Table 3 presents the total capital investment, which we estimate to be less than $900,000.

Table 4 presents total costs of operation per day for the three-stage, 50-ton plant. With fish priced at 1.0¢ per pound, the MPC would cost 13.9¢ per pound.

Two-Stage Countercurrent Extraction

Table 5 presents costs for items of equipment for a two-stage, 50-ton-per-day batch commercial plant. Total equipment costs are $194,500.

Table 6 presents costs of the total capital investment, which we estimate to be less than $760,000.

Table 7 presents total costs of operation per day for the two-stage 50-ton plant. With fish priced at 1.0¢ per pound, MPC would cost 13.1¢ per pound. It is 0.8¢ cheaper than the cost of the product prepared by a three-step operation.

Table 2.--Estimated equipment costs for a three-stage 50-ton-per-day (raw material input) batch plant

(s/s is stainless steel)

Item	Description	Size	Material of construction	Estimated cost
				Dollars
1	Grinder	8,500 lb./hr.	316 s/s	11,000
2	Agitated tank T-1	7,500 gal.	316 s/s	17,000
3	Agitated tank T-2 (heated)	6,500 gal.	316 s/s	24,000
4	Agitated tank T-3 (heated)	6,000 gal.	316 s/s	22,500
5	Centrifuge	100 gal./min.	316 s/s	38,000
6	Storage tank ST-1	7,000 gal.	304 s/s	10,500
7	Storage tank ST-2	7,000 gal.	Steel	3,500
8	Storage tank ST-3	10,000 gal.	Steel	4,500
9	Complete still	30 $gal._3$/min.	Copper	30,000
10	Solvent deodorizer	8 $ft.^3$	Steel	1,000
11	Vacuum dryer	150 $ft.^3$	316 s/s	38,000
12	Mill	625 lb./hr.	316 s/s	5,000
13	Packaging equipment	625 lb./hr.	316 s/s	10,000
14	Conveyor-fish	8,500 lb./hr.		5,500
15	Conveyor-wet solids	5,600 lb./hr.		4,500
16	Conveyor-dry solids	625 lb./hr.		2,500
17	Pumps	to 100 gal./min.		4,000
	Purchased equipment costs			231,500

Table 3.--Total capital investment estimates for a three-stage 50-ton-per-day (raw material input) batch plant

	Item	Estimated cost
		Dollar
1	Purchased equipment costs (PE)	231,500
2	Equipment installation (43% PE)	99,500
3	Piping (36% PE)	83,300
4	Instrumentation (10% PE)	23,200
5	Building (40% PE)	92,600
6	Fish storage installed (200 tons of fish)	7,000
7	Physical plant cost (PPC)	537,100
8	Engineering and construction (20% PPC)	107,400
9	Direct plant cost (DPC)	644,500
10	Contractors' fee (7% DPC)	45,100
11	Contingency (10% DPC)	64,500
12	Fixed capital (IF)	754,100
13	Working capital (total operating costs for 60 days)	125,300
	Total capital	879,400

Table 4.--Total operating costs per day of a three-stage 50-ton-per-day (raw material input) batch plant

	Item	Estimated Cost
		Dollars
1	Electric power, 1¢ per kw. hr. (200 kw./hr.)	48.00
2	Steam, 55¢ per 1,000 lb.	224.40
3	Refrigeration, 90¢ per ton (50-ton)	45.00
4	Fish, 1¢ per pound (100,000 lb.)	1,000.00
5	Isopropyl alcohol, 50¢ per gal. (227 gal.)	113.50
6	Supplies and maintenance materials, 0.0015% IF	11.31
7	Operating labor, 96 hr. @ $2.50, 8 hr. @ $3.50	268.00
8	Maintenance labor, 0.0015% IF	11.31
9	Payroll extras, 15% of items 7 & 8	41.90
10	General overhead, 30% of items 7 & 8 & 9	96.36
11	Amortization, 0.0224% IF	168.92
12	Taxes and insurance, 0.006% IF	45.25
13	Interest on working capital, 0.00725 times sum of above twelve items	15.03
	Total operating costs	2,088.98
	For a production rate of 15,000 lb. MPC per day: Total operating cost = 13.9¢ per pound of MPC On a fish-free basis: Total operating cost = 7.3¢ per pound of MPC	

Table 5.--Estimated equipment costs for a two-stage 50-ton-per-day (raw material input) batch plant

Item	Description	Size	Estimated cost
			Dollars
1	Grinder	9,375 lb./hr.	12,000
2	Agitated tank T-1	5,500 gal.	15,000
3	Agitated tank T-2 (heated)	5,000 gal.	21,000
4	Centrifuge	85 gal./min.	36,000
5	Storage tank ST-1	5,000 gal.	9,000
6	Storage tank ST-2	7,000 gal.	3,500
7	Storage tank ST-3	10,000 gal.	4,500
8	Complete still	30 gal./min.	30,000
9	Solvent deodorizer	8 $ft.^3$	1,000
10	Vacuum dryer	100 $ft.^3$	31,000
11	Mill	625 lb./hr.	5,000
12	Packaging	625 lb./hr.	10,000
13	Conveyors	14,725 lb./hr.	12,500
14	Pumps	200 gal./min.	4,000
	Purchases equipment costs		194,500

Table 6.--Total capital investment estimate for a two-stage 50-ton-per-day (raw material input) batch plant

	Item	Estimated cost
		Dollars
1	Purchased equipment costs (PE)	194,500
2	Equipment installation (43% PE)	86,600
3	Piping (36% PE)	70,000
4	Instrumentation (10% PE)	19,500
5	Building (40% PE)	77,800
6	Fish storage, installed (200 tons of fish)	7,000
7	Physical plant cost (PPC)	455,400
8	Engineering & construction (20% PPC)	91,100
9	Direct plant cost (DPC)	546,500
10	Contractors fee (7% DPC)	38,300
11	Contingency (10% DPC)	54,600
12	Fixed capital (IF)	639,400
13	Working capital (total operating costs for 60 days)	117,700
	Total capital	757,100

Table 7.-- Total operating costs per day for a two-stage 50-ton-per-day (raw material input) batch plant

	Item	Estimated cost
		Dollars
1	Electric power, 1¢ per kw. (200 kw./hr.)	48.00
2	Steam, 55¢ per 1,000 lb.	224.40
3	Refrigeration, 90¢ per ton (50-ton)	45.00
4	Fish, 1¢ per pound (100,000 lb)	1,000.00
5	Isopropyl alcohol, 50¢ per gal. (227 gal.)	113.50
6	Supplies and maintenance materials, 0.0015% IF	9.59
7	Operating labor, 72 hr. @ $2.50; 8 hr. @ $3.50	208.00
8	Maintenance labor, 0.0015% IF	9.59
9	Payroll extras, 15% of items 7 & 8	32.64
10	General overhead, 30% of items 7 & 8 & 9	75.07
11	Amortization, 0.0224% IF	143.23
12	Taxes and insurance, 0.006% IF	38.36
13	Interest on working capital, 0.00725 times sum of above twelve items	14.12
	Total operating costs	1,961.50
	For a production rate of 15,000 lb. MPC per day: Total operating cost = 13.1¢ per pound of MPC On a fish-free basis: Total operating cost = 6.4¢ per pound of MPC	

PREPARATION AND PROPERTIES OF MPC TEST SAMPLE

One of the objects of the accelerated program was to demonstrate that a safe, wholesome, nutritious MPC can be made from whole fish. To meet this aim, we prepared an MPC test sample in model-scale unit equipment by an isopropyl alcohol extraction method approximating the conditions of the proposed commercial method. The MPC was then defined according to its various properties.

PREPARATION OF THE MPC TEST SAMPLE

About 350 pounds of MPC were produced by a batch-extraction process in the model-scale unit, using red hake (Urophycis chuss) and isopropyl alcohol. Care and caution were observed at all stages of the procedure, from the handling of the raw material through the processing operation, the packing step, and the storage of the end product.

Procurement of Raw Material

The red hake used in the production of the test sample of MPC was purchased from the Salt Water Fisheries Co., Provincetown, Mass.

About 3,000 pounds of red hake were caught by otter trawl in an area near No Man's Land, (about five miles south of Martha's Vineyard). On board the vessel, the fish were immediately packed in ice.

After the fish were unloaded they were packed into waxed cartons and frozen. The fish were transported in a refrigerated truck to Beltsville, Md., where they were immediately transferred to the freezer in our plant and kept at 29° C. (-20° F.) until used.

Processing Procedure

Sixteen batches of MPC were prepared in the manner described below and ultimately combined as the test sample.

Raw material pretreatment.--For each batch of MPC about 150 pounds of fish were removed from freezer storage and ground. The frozen fish were first reduced to about 1-inch pieces by one pass through a Rietz extructor (prebreaker). 1/ The chunks were collected in a stainless steel pot and were then transferred, still in the frozen state, to a Hobart grinder. The fish were passed once through a plant having 5/8 inch holes, then twice through a plant having 1/8 inch holes. The resulting ground product had the textural appearance of ground beef. The temperature of the fish never exceeded 0° C. (32° F.) during this process.

1/ Trade names referred to in this publication does not imply endorsement of commercial products.

Extraction.--The ground fish were transferred into two 30-gallon stainless steel cans and weighed, and each lot of about 75 pounds of fish was dispersed in 7 gallons of fresh 91 volume-percent isopropyl alcohol. A portable stainless steel agitator was used to slurry the fish and alcohol, the mixing time being 5 minutes. The two slurries were then combined in the mixing tank, and fresh isopropyl alcohol was added to bring the volume of alcohol to 45 gallons. The slurry was agitated for 1 hour. During this time, the temperature of the slurry rose from 0° C. (32° F.) to a final temperature of about 4° C. (40° F.).

The fish slurry was then pumped to a Bird horizontal centrifuge for the separation of the solids and the miscella. The liquid phase was pumped to the storage tanks; and, after they were collected, the solids were weighed and transferred to the extractor. Centrifugation for this stage usually took $1\frac{1}{4}$ hours.

The solids from the dehydration stage were dispersed in the extractor in 45 gallons of fresh isopropyl alcohol preheated to 79° C. (175° F.). The temperature of the mixture was next brought to about 79° C. (175° F.) and held at that level for 1 hour with constant agitation. The slurry was then pumped to the centrifuge to recover the extracted solids. Part of the miscella from the centrifuge was recycled back to the extractor in order to maintain a pumpable slurry. All of the miscella was finally pumped to storage for future recovery of solvent. This extraction procedure was repeated twice more, using 45 gallons of fresh solvent each time. Centrifugation usually took about an hour for each stage.

The solids from the final-stage centrifugation were weighed and transferred to the tumbling dryer for desolventization.

Desolventization.--Since the dryer was of sufficient capacity to hold about 80 pounds of material, the solids from two runs were combined. The dryer was operated at a vacuum of 27 to 28 inches of mercury and a jacket temperature of 43° C. (110° F.). To avoid balling of the product, the dryer was rotated only intermittently during the first hour. Thereafter, the dryer was rotated continuously throughout the remainder of the run. In general, no more solvent condensed after 4 hours. The jacket temperature was then increased to 54° C. (130° F.) for 1 hour and finally to 66° C. (150° F.) for 1 hour. The jacket heat was then turned off, and the temperature slowly fell to about 27° C. (80° F.). The product was allowed to cool in the rotating dryer under vacuum for 1 to 4 hours.

Mixing of final product.--The eight batches of MPC resulting from the 16 runs were combined into three lots by proportioning from each batch. The three lots were next mixed separately in the tumbling dryer for 1 hour. The product was then subdivided into 20-pound lots, placed in Mylar bags, and stored in tightly closed metal cans at room temperature.

PROPERTIES OF MPC TEST SAMPLE

Criteria for the evaluation of the MPC test sample included a determination of its physical, chemical, and sensory properties, a

determination of its protein quality and its microbiological safety, and an examination of its wholesomeness.

These criteria, we believe, provide sufficient data to permit a sound judgment of the quality of MPC from the points of view of acceptability, nutritive value, and safety for use.

Physical Properties

The physical definition of the MPC test sample included observations on the color, particle size, distribution, particle identification, and hygroscopicity.

The color of the sample as determined by reflectance measurements was off-white, with a yellowish cast.

The ground sample was classified according to particle size distribution. A particle size distribution of the MPC test sample that passed a 100-mesh screen was derived.

The test sample was also submitted to different conditions of temperature and humidity to determine the rate at which the sample absorbed moisture. Samples stored at 25° C. (77° F.) and 31.0 percent relative humidity equilibrated rapidly, with only a 1-percent increase in weight. At higher humidities, regardless of temperature, the amount of water absorbed was greater and the time for equilibration increased.

Chemical Properties

The proximate analyses of the individual batches of MPC test sample and of the composite are given in table 8.

The amino acid composition of the composition of the composite test sample is indicated in table 9.

The nonprotein nitrogen content of the product compared favorably with that in other foods and consisted mainly of amino acids.

Residual lipids in MPC were studied in particular as to their possible role in flavor reversion or in the development of rancidity. Of the total lipids extractable from MPF, 40 percent were identified as neutral lipids (mainly triglycerides and sterols); 10 percent neutral lipids (mainly mono- and diglycerides); 33 percent phospholipids (predominantly sphingomyelin); 8 percent remained uncharacterized. No lipid oxidation had occurred in the sample. Further tests showed that the lipids underwent no oxidative changes even after 3 months' storage of the extracts.

Inorganic analyses of the ash of the MPC sample are presented in tables 10 and 11.

No sand was detected in the sample.

The MPC test product had no fish odor, probably owing to the very small levels of volatile amines observed. Amine hydrochloride mixtures were isolated in quantities of 0.0117 to 0.0134 percent based on the original weight of the MPC. Gas chromatographic separation of the mixtures indicated that trimethylamine, methyl-, and/or dimethylamine, and either ethyl-, diethyl-, or triethyl amines (or a mixture of all three) were present. Hydrogen sulfide, mercaptans, thio-ethers, and disulfides were not present in detectable quantities.

Table 8. Proximate composition of the raw red hake and of individual batches and composite MPC test sample

Component	Proximate composition									
	Raw sample	Batch 1	Batch 2	Batch 3	Batch 4	Batch 5	Batch 6	Batch 7	Batch 8	Composite sample
	Percent									
Protein	15.20	81.78	81.26	78.04	80.74	80.63	79.30	81.85	82.28	81.35
Volatiles	80.21	7.55	7.38	10.78	7.58	7.53	8.91	6.25	7.67	6.73
Ash	3.04	14.35	13.72	13.06	13.80	13.22	13.42	13.48	12.92	13.52
Lipids	5.82	0.17	0.15	0.17	0.13	0.19	0.15	0.21	0.19	0.20

Table 9.--Amino acid composition of raw material and composite MPC test sample[1/]

Amino acid	Concentration in:	
	Raw sample	MPC test sample
	Percent of protein (N x 6.25)	Percent of protein (N x 6.25)
Lysine [2/]	7.40	8.41
Histidine	1.79	2.08
Arginine	6.08	7.13
Aspartic acid	9.51	10.35
Threonine	4.15	4.47
Serine	4.11	4.65
Glutamic acid	14.28	15.39
Proline	4.86	5.21
Glycine	7.82	8.09
Alanine	6.31	6.81
Valine	4.80	5.26
Methionine	3.05	3.30
Isoleucine	4.25	4.56
Leucine	7.06	7.78
Tyrosine	1.78	3.35
Phenylalanine	3.88	4.24
Tryptophan	0.68	1.03
Cystine*	0.84	0.77

* Microbiological determination.

1/ Values not strictly comparable; loss of alcohol - soluble proteins in MPC sample enhances amino acid percentages slightly.

2/ Of the lysine contained in the product, 93 percent was digestible (available) as determined by chemical analysis.

Table 10. Spectrographic analysis of MPC

Element	Maximum concentration	
	Based on ash	Equivalent to original material *
	Percent	
Sodium	ca. 5	ca. 0.5
Potassium	ca. 5	ca. 0.5
Magnesium	2	0.2
Silicon	2	0.2
Aluminum	1	0.1
Strontium	0.9	0.09
Iron	0.9	0.09
Tin	0.09	0.009
Titanium	0.09	0.009
Copper	0.009	0.0009
Zirconium	0.009	0.0009
Manganese	0.009	0.0009
Barium	0.005	0.0005
Nickel	0.005	0.0005
Lead	0.0009	0.00009

Elements checked but not found: zinc, cadmium, indium, bismuth, antimony, arsenic, thallium, gallium, germanium, chromium, cobalt, molybdenum, vanadium, tungsten, and silver.

* Based on an ash content of 12.0 percent.

Table 11.--Mineral content of MPC

Determination	Result
	mg./100 g.
Calcium[1/]	3,780
Phosphorus[2/]	2,930
Sodium[3/]	353
Potassium[3/]	593
Chloride [4/]	1,000
Bromide[5/]	1.5
Iodide[6/]	0.1
Fluoride[7/]	13
Iron[8/]	18.9
Iodine	1 mcg/g
Selenium	2 ppm

1/ Official Methods of Analysis of the Association of Official Agricultural Chemists (1960), section 6.011
2/ Ibid., section 2.023
3/ Ibid., section 6.016
4/ Ibid., section 18.008
5/ Shrader, et al., 1942, Ind. Eng. Chem. Anal. Ed. 14, 1
6/ Houston, 1950, Anal. Chem. 22: 493
7/ Official Methods of Analysis of the Association of Official Agricultural Chemists (1960), section 24.027
8/ Ibid., section 6.007

Sensory Properties

The test sample was analyzed by the Flavor Profile Method (a scientific approach to organoleptic evaluation) immediately after production and after 9 months' storage at room temperature. Regardless of the age of the sample, the panel described the MPC as typical of a processed dry protein product with no evidence at either time of fishy taste or odor.

Nutritive Quality

Rat-feeding tests were made with the MPC test sample immediately after production and after 9 months' storage for comparison against a casein control (table 12).

Microbiological Analyses

Total counts of microorganisms were determined for the raw fish used in the preparation of the test sample and for the finished product. The highest count found in the raw fish was 1,500 organisms per gram. The finished MPC sample had counts of about 40 microorganisms per gram. No coliform types of Salmonella organisms were found in either the raw fish or the finished product.

Wholesomeness.--An approach that has become standard in the isolation and characterization of physiologically potent materials such as vitamins and hormones was applied to determine the presence of absence of toxic substances in the MPC test sample. A lot from the test sample was first exhaustively extracted with hexane. Solids residue from that extraction was next extracted with chloroform. That solids residue was extracted with ethanol, and finally the residue was extracted with water. Each of the liquid extracts of these four solvents was concentrated to dryness.

The test sample, the solids residue remaining after the sequential extractions, and each of the four concentrated extracts were fed to groups of growing rats at very high levels in the diet. The gain in weight, weekly food intake, and food efficiency for each animal in each group were determined. Tests were made for urine concentration, pH and creatine, creatinine, and uric acid contents. A glucose tolerance test and hematology studies were also made. The animals were killed after 4 weeks, and a complete necropsy was carried out. All unusual gross effects were noted. Enzyme tests on the liver and the serum were also included. The blood serum was examined for lipid and nitrogen content.

The results of all experiments on the MPC sample indicate that there is no marked difference from the control group in any of the factors studied, with the exception of the high level of blood urea nitrogen and nitrogen noted for the high-protein-consuming groups, and a marked increase in urinary creatine and in creatinine in the groups receiving the alcohol and water extracts and the MPC as such. The explanation suggested for the increased creatine and creatinine excretion was that the MPC as such probably had a high creatine content which is

Table 12.--Comparison of the protein quality of MPC prepared by the IPA extraction of hake at zero time and after 9 months' storage at room temperature

Diets	Average weight gained by rats	Average weight of food consumed by rats	Protein efficiency ratio[1/]
	Grams	Grams	
Casein control	108.2 ± 3.65	338 ± 7.7	3.00
MPC zero time	142.2 ± 2.24	413 ± 5.02	3.24
Casein control	116.2 ± 10.44	332 ± 18.8	3.00
MPC 9 months' storage	137.3 ± 10.5	373 ± 14.5	3.19

1/ Weight gained per weight of protein consumed = Protein Efficiency Ratio (PER). Data have been adjusted based on a PER value of 3.00 for casein.

accentuated in the water extract and alcohol extract. This possibility is now being examined. Although these results cannot be considered as conclusive, it appears that there is only a small possibility that a biologically active substance is present in the MPC test sample in significant amounts. So far, the results of this study indicate that substances that are biologically active for the rat either are not present in the test sample or are not extractable in the solvents used.

CONCLUSIONS

The accelerated program has shown how MPC can be produced at a relatively low cost by a commercial solvent-extraction method using isopropyl alcohol as the solvent and red hake as the raw material. It also has shown that a test MPC prepared from whole fish by a method approximating the proposed commercial process is highly nutritious, safe, and wholesome and, therefore, should be entirely satisfactory as a dietary supplement for human consumption.

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