THE DESIGN OF A PILOT PLANT FOR THE PRODUCTION OF MONOSODIUM GLUTAMATE

by

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I. INTRODUCTION

The production of monosodium glutamate has grown from a by-product of the sugar beet industry to an important position in today's food commodities. Monosodium glutamate is made from Steffan's waste, corn gluten, wheat gluten, and other similar proteinaceous materials. In trying to find more and better uses for soybeans, it was discovered that soybean meal was an excellent raw material for making monosodium glutamate because of the high percentage of proteins in the meal. For years in the Far East, soybean meal has been utilized for the production of monosodium glutamate, but American industry had to be educated to make use of the potential of the soybean.

For the various processes required for the isolation and purification of glutamic acid for the conversion into monosodium glutamate, the yields obtained have been low of the order of between 50 and 70 per cent by weight of the total glutamic acid originally contained in the proteinaceous substances processed. The difficulty involved in the manufacture of monosodium glutamate is in getting the glutamic acid in

a refined state. Other amino acids are obtained during the initial hydrolysis step, and they are difficult to remove to obtain the "edible" glutamic acid of commerce.

The proposed production of monosodium glutamate from soybean meal is an answer to higher yields and less expensive equipment and reagents. In using the soybean meal, which is itself a highly valuable cattle food, the glutamic acid is taken out leaving valuable amino acids in the waste after the initial hydrolysis step. Therefore, this waste referred to as humin can be sold as a hydrolyzed cattle food.

It was the purpose of this investigation to design a pilot plant for the production of monosodium glutamate. The problem involved the investigation of synthesis methods and conditions, cost analyses, and actual design of the pilot plant.

II. LITERATURE REVIEW

The following section of this thesis is a comprehensive survey of the literature pertaining to the production of monosodium glutamate. The literature review has been subdivided into trade information for monosodium glutamate, soybean meal, soybean production in Virginia, mcnosodium glutamate, and pilot plant design.

Trade Information for Monosodium Glutamate

Information on the uses of monosodium glutamate, possible substitutes, plant locations, future trends, and packaging are contained in the following paragraphs.

Uses. Monosodium glutamate is primarily used as a food flavoring. It is reported to intensify the flavors of soups, meats, vegetables, and gravies. It is also reported to increase the mental activity of retarded children. The 1952 summary of uses of monosodium glutamate shows a distribution of over two million pounds yearly to the dry and canned soup industry, two million to the canned meat industry,

under a million to restaurants, and under a million for table use[26].

Competition. A hydrolyzed protein product is on the market as a substitute for monosodium glutamate. This product, however, is not uniform in quality and is said to possess a rather strong taste in itself[38].

Location of Present Plants. There are at present six plants in this country producing monosodium glutamate. The location of the plants, the raw material used, the 1948 production, and the estimated capacity are given in Table I, page 5[38].

Future of Industry. The future of the industry will depend on the degree to which the industrial and retail market can be further saturated. If monosodium glutamate is to be used in baby food, a large industrial market will be opened. Also, it is believed that the retail market will be saturated to a much greater extent than it has been. Less than a million pounds per year is used for table use in this country. In Japan, however, nine million pounds are sold yearly to seventy million people[26,27,38,39,45].

Packaging. Bottles, cans, and fiber drums may be used as containers for monosodium glutamate. For retail use, shaker cans and paper bags are used[38].

TABLE I

Plants Producing Monosodium Glutamate [26,27,38,39,95]

Company	Location	Production,[a] million lb	Capacity,[a] million lb	Process
Huron Chemical Company	Harbor Beach, Mich.	2.7	3.0	beet waste
International Minerals and Chemicals	Rossford, Ohio	---	3.0	beet waste
International Minerals and Chemicals	San Jose, Calif.	3.4	4.0	beet waste
A. E. Staley	Decatur, Ill.	0.5	1.5	corn and soybeans
General Mills	Keokuh, Iowa	1.0	1.0	wheat
Great West Sugar	Johnston, Colo.	---	---	beet waste

[a] Production and capacity based on 1948.

Soybean Meal

The basic raw material necessary for the production of monosorium glutamate from soybeans is soybean meal. Soybean meal will be discussed under the headings of analysis, amino acid content, and uses of the amino acids.

Analysis. The proximate and ultimate analyses of the soybean meal are as follows[36]:

Proximate analysis:	moisture free basis, weight per cent
moisture	12.0 max
protein	44.0 min
carbohydrate	7.0 max
fat	0.5 min
ash	6.0 max
N.F.E.	27.0 min

Ultimate analysis: moisture free basis, weight per cent; hydrogen, carbon, oxygen, and nitrogen are excluded from this analysis

K	1.67
Na	0.37
Ca	0.28
Mg	0.22
P	0.66
S	0.41
Cl	0.024
I	0.00054
Fe	0.0097
Cu	0.0012
Mn	0.0028
Zn	0.0022
Al	0.0007

Amino Acid Content. The following table gives the amino acid content of several different soybean meals. Composition is expressed in weight per cent[36].

Material	Serine	Threonine	Leucine	Isoleucine	Valine	Glutamic Acid
solvent extracted meal	2.4	2.1	4.5	3.5	3.0	9.0
expeller meal	2.2	2.2	4.0	2.9	2.6	7.8
meal	---	1.7	---	---	---	---
meal	---	---	3.23	2.29	2.06	---
meal	---	---	---	---	---	7.64

Uses of the Amino Acids. The following is a listing of the uses of the amino acids found in soybean meal. Glutamic acid has been excluded from this list[36].

SERINE: Biochemical and nutritional studies, culture media, microbiological tests.

THREONINE: Biochemical and nutritional studies.

LEUCINE: Biochemical and nutritional studies.

ISOLEUSINE: Biochemical and nutritional studies.

VALINE: Medicine, food, culture media, biochemical and nutritional studies.

Soybean Production in Virginia

As shown in Table II, page 9[31], the soybean production in Virginia is heavily settled in the Tidewater and Eastern Seaboard sections of the state. The total production of soybeans in Virginia in 1950 was 2,880,000 bushels. The average yield was 19.0 bushels per acre. The price of soybeans in 1950 was $2.49 per bushel in season. The soybean season is from September 1st to August 31st[31].

Monosodium Glutamate

The synthesis of monosodium glutamate is a hydrolysis reaction. In the reaction water and soybean meal are reacted in the presence of hydrochloric acid. Other topics which will be discussed under this heading are hydrolysis of proteins, properties of protein amino acids, properties of glutamic acid hydrochloride, glutamic acid, and monosodium glutamate, and methods of manufacture of glutamate.

Hydrolysis of Proteins. The most characteristic reaction of the proteins is their hydrolysis, which may be accomplished by the use of acid or alkaline solutions, or by means of enzymes.

TABLE II

Soybean Production in Virginia (1)

County[a]	Acres
Princess Anne	5000
Norfolk	5000
Hanover	5000
Accomac	5000
Northumberland	5000
Westmoreland	1000
Essex	1000
Caroline	1000
King William	1000
King George	1000
Lancaster	1000
Gloucester	1000
King and Queen	1000
Nansemond	1000
Southampton	1000
Sussex	1000
Dinwiddie	1000
Henrico	1000
Charles City	1000
Surry	1000
Isle of Wight	1000

[a] Counties listed have a production of 1000 acres or more.

As hydrolysis proceeds there is evidence that the original molecules are being broken down into simpler substances (proteoses). Thus, salts which precipitate the original protein may fail to cause a precipitate after a short period of hydrolysis, or a protein which could be coagulated by heat might lose this property. Further hydrolysis yields still more simple substances (peptones). The complete hydrolysis of a simple protein gives a mixture of amino acids. Of these, 25 have been definitely certified. They are α-amino acids of a number of different chemical types. The relative amounts of the different amino acids vary widely in the different proteins, and no protein is known which yields but one amino acid upon hydrolysis[52].

Properties of Protein Amino Acids. Amino acids formed are colorless crystalline compounds and water-soluble except cystine and tryosine. They are, in general, soluble in dilute alcohol, not soluble in absolute alcohol or in ether or common organic solvents. The amino acids have no true melting points but decompose when heated to 200 to 350 °C. These properties resemble those of inorganic salts.

The amino acids are acid, neutral, or basic in a reaction, as they possess, respectively, an overplus of carboxyl groups, an equality of carboxyl and amino groups, or an overplus of amino groups. All are optically active save glycine, for which activity is impossible. Either the d- or the l-form of a given acid is found in proteins, but not both. A number of acids, for example, glycine, alanine, aerine, and norleucine are sweet in taste.

The amino acids are amphoteric which means that they can act either as acids by virtue of the carboxyl group or as bases by virtue of the amino group. In solution, the molecules may have positive and negative charges, due to the chemical behavior of the opposed groups. This type of ion is known as a zwitterion (dipolar ion). At some definite pH the acid and basic ionization would be of equal extent. This is known as the isoelectric point. Taking glycine for an example, the reaction of the zwitterion with acid and base is as follows:

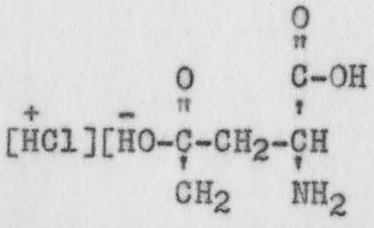
(1) $+H_3N-CH_2-C(=O)-O^- + HCl = H_3N-CH_2-C(=O)-OH \; Cl^-$

(2) $+H_3N-CH_2-C(=O)-O^- + NaOH = H_2N-CH_2-C(=O)-ONa^{-+}$

Inspection of these equations shows that an amino acid by combining with proton or hydroxyl is able to prevent sudden changes of pH when acid or base is added to its water solution. Any substance which acts in this way is called a buffer(54).

Properties of Glutamic Acid Hydrochloride, Glutamic Acid, and Monosodium Glutamate. The physical and chemical properties of glutamic acid hydrochloride, glutamic acid, and monosodium glutamate are discussed in the following sections:

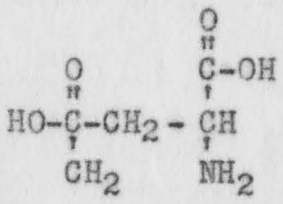
1-Glutamic Acid Hydrochloride. 1-Glutamic acid hydrochloride is formed by treating 1-glutamic acid with hydrochloric acid. The molecular structure of 1-glutamic acid hydrochloride is as follows:



The molecular weight and crystal form of the material are 183.60 and triclinic, respectively. The indices of refraction of the hydrochloride are α equals 1.546 and β equals 1.559. The 1-glutamic hydrochloride decomposes at 201 °C.

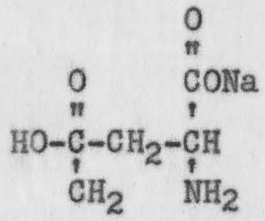
It is very soluble in water, soluble in alcohol, and slightly soluble in concentrated hydrochloric acid[34].

<u>l-Glutamic Acid</u>. l-Glutamic acid is a colorless amino acid and it is sometimes known as l-α-aminoglutstic acid. The molecular structure of l-glutamic acid is as follows:



The molecular weight and crystal form of the material are 147.13 and rhombic, which plate from water, respectively. The indices of refraction of the acid are α equals 1.490, β equals 1.605, and γ equals 1.620. The density and melting point of the material are 1.538 and 202 °C, respectively, and it will decompose at 213 °C. It is very soluble in water, soluble in alcohol, and insoluble in ether. Some of the physiological properties of l-glutamic acid are hastening of cell differentiation, stimulation of the learning process of rats, aiding transamination reactions, and beneficial in treatment of petit mal and psychomotor seizures[34].

l-Monosodium Glutamate. l-Monosodium glutamate is the monosodium salt of l-glutamic acid. The molecular structure of l-monosodium glutamate is as follows:



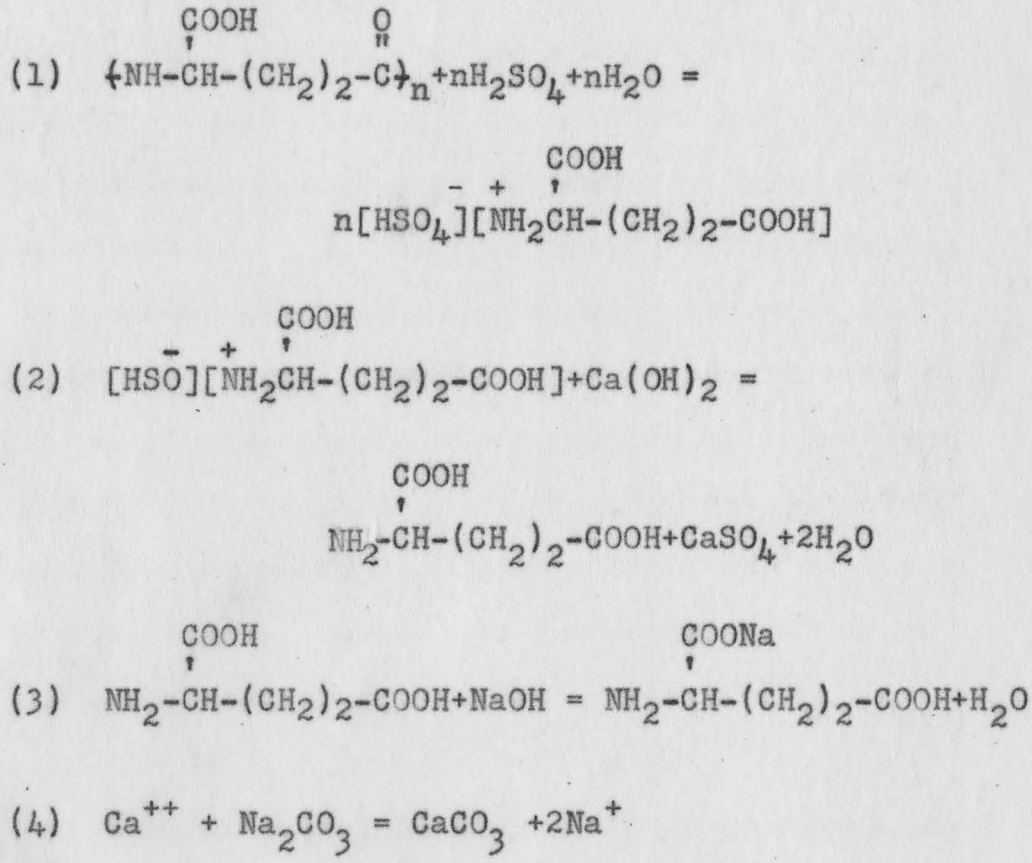
The molecular weight and color of the material is 169.12, and white or almost white, respectively. Its melting point is 230 °C. The l-monosodium glutamate has a peptone-like color and a meat-like taste[34].

Methods of Manufacture of Glutamate. The following discussions are presented: the manufacture of monosodium glutamate (1) by using sulfuric acid and calcium hydroxide, (2) by removing the soluble carbohydrates with hydrochloric acid, and (3) by using hydrochloric acid and caustic soda.

Manufacture of Monosodium Glutamate Using Sulfuric Acid and Calcium Hydroxide. l-Glutamic acid is made by hydrolyzing gluten or soybean protein by means of sulfuric acid, and purified by removing sulfate ions by means of calcium hydroxide, the remaining calcium ions as sulfite,

carbonate, or phosphate, and other amino acids by selective crystallization. One part gluten is refluxed with two parts of 50 per cent sulfuric acid for seven hours or until hydrolysis is complete. The hydrolyzate is diluted with water, cooled, and sufficient slurry of calcium hydroxide is added to precipitate all the sulfate ions and to bring the reaction to a pH of 11.0. Humin and calcium sulfate are removed by filtration and the cake washed. The filtrate is treated with sufficient sulfur dioxide, carbon dioxide, and sodium carbonate to precipitate the remaining calcium ions, the pH value being maintained at neutrality by addition of sodium hydroxide solution. The insoluble calcium salt is removed by filtration and the cake washed. The filtrate is acidified with hydrochloric acid to a pH value between 5.5 to 6.0, and concentrated in a vacuum to 65 °C to a specific gravity of about 1.22. It is held for 24 hours at room temperature to permit leucine, tyrosine, and other unwanted amino acids to crystallize. These amino acids are removed by filtration and the filtrate adjusted to a pH of 3.2 by means of hydrochloric acid. It is allowed

to stand at room temperature from one to six days to permit the glutamic acid to crystallize. The crystals are removed by filtration and washed. The glutamic acid is white and sufficiently pure to be used without further treatment for making monosodium glutamate. The chemical equations for the above method are as follows[31]:



Manufacture of Monosodium Glutamate by Removing the Soluble Carbohydrates with Hydrochloric Acid. Rokusho obtained a yield of 11.0 to 12.5 per cent of glutamic acid based on the protein present in Manchurian soybean oil cake. They stated that the best yield of glutamic acid was obtained by first removing the soluble carbohydrates and ash from alcohol extracted soybean cake with 0.5 to 2.0 per cent hydrochloric acid leaching at the isoelectric point of the protein (pH=4.7) and hydrolyzing with 18 per cent hydrochloric acid[36]. Also, glycinin contains about 20 per cent of glutamic acid; the soybean meal is suitable material for the manufacture of monosodium glutamate. The test results were obtained by hydrochloric acid hydrolysis of meal defatted with a benzene-methyl alcohol mixture which removes a large portion of the carbohydrates, as these prevent the crystallizing out of the glutamic acid hydrochloride[32].

Manufacture of Monosodium Glutamate Using Hydrochloric Acid and Caustic Soda. The process involved is the hydrolysis of gluten with

hydrochloric acid and then treating the hydrolyzate with caustic soda to obtain a pH of between 6.0 and 6.4. The humin is separated from the partially neutralized hydrolyzate by filtering, and the filtrate is concentrated to the point of incipient crystallization of sodium salts. The salts are filtered from the concentrate, readjusting to a pH of between 6.0 and 6.4, and cooled. On cooling, a precipitate containing substantial amounts of amino acids other than glutamic acid is formed. The filtrate from the above is treated with hydrochloric acid to give a pH between about 3.0 and 3.2. The solution is adjusted to normal room temperature with the crude glutamic acid crystallizing out. The crude glutamic acid is separated out with hot water at about 60 °C with the mother liquor left being recycled to the concentrating step. The glutamic acid is then treated with caustic soda to obtain monosodium glutamate[30].

Industrial Pilot Plant Design

The pilot plant is used frequently in industry to investigate process variables on a semi-plant scale. The pilot plant[41] is usually built along with the commercial plant, and additions or deletions are made to the commercial plant as they are determined in the pilot plant. Pilot plant design by Esso Laboratories is a typical example of industrial pilot plant design. This topic will be discussed under the headings of preliminary investigations, market survey by development division, development laboratory data, pilot plant design, construction of pilot plant, and operation of pilot plant.

Preliminary Investigations. Before a process can be developed, a need for the products produced by this process must exist. Chemical engineers are assigned to the compilation of market survey. All competitive products, existing processes, prices, and all other related subjects are examined from the market viewpoint. This information is presented to the technical management for their consideration. The next step is the investigation of fundamental problems involved in the process. This information is also presented to the technical

management for their consideration. The next step is the investigation and analysis of the problem through technical management discussions. Here the ideas are discussed according to feasibility and economic return on the investment.

Market Survey by Development Division. The initial step in the market survey, which is much more extensive than the one in the preliminary investigations, is the preliminary design of the plant. The plant is designed from existing data and from this design of the plant, the additional data necessary for a complete design of the plant are determined. The request for these additional data is sent to the development laboratory. A second cost analysis is also determined at this stage of the pilot plant design. In this analysis the cost of equipment, cost of materials, labor costs, and all other costs involved in building and operating a plant are considered.

Development Laboratory Data. In this stage of the plant design, all data and information that is not available in the literature is determined in the laboratory. This involves the use of bench scale equipment to determine process specifications for any new

process that will be used, obtaining engineering data such as densities and vapor pressures, and the development of any new materials to be used in the process. During this time, notes on safety are compiled and special data for safety considerations are determined.

Pilot Plant Design. All of the previous information is initially sent to a correlations group. Here the data are correlated so that they can be used in the design of the pilot plant. All possible correlations and facts that can be gleaned from these data are obtained and sent to the pilot group. The chemical engineers in this group have the job of completely designing an integrated pilot plant, which can be used for extrapolation to commercial size.

Construction of Pilot Plant. Pilot plant construction usually costs in the neighborhood of several hundred thousand dollars and the size of the pilot plant will vary. The time required to build the pilot plant is from eight months to a year. In the pilot plant the engineer is not solely worried about economics, but he is concerned with flexibility. Flexibility is necessary so that many variables may be studied. During the construction of the pilot plant

the groups are reorganized. One group has the specific purpose of programming the pilot plant operations. All of the groups give their data and findings to the construction group. This group correlates all information and translates it into the construction of the pilot plant.

Operation of Pilot Plant. The operation of the pilot plant is under the supervision of engineers. They are responsible for the design features and for the actual operation of the pilot plant. All of the variables of operation are studied. The pilot plant operations group is assisted by two other groups. One group is responsible for the collection of engineering data and the other group is responsible for the collection of data obtained from the study of the different variables. Both groups correlate their data and present the correlations to the operations group.

III. EXPERIMENTAL

This section is concerned with the purpose and plan of investigation, materials, apparatus, the method of procedure used, sample calculations, and the data and results obtained in this investigation.

Purpose of Investigation

The purpose of this investigation was to design a pilot plant for the production of monosodium glutamate. The problem involved the investigation of optimum synthesis methods and conditions, cost analysis, and actual design of the pilot plant.

Plan of Investigation

The plan of investigation for the design of a pilot plant for the production of monosodium glutamate involved a review of the literature pertaining to the production of monosodium glutamate, assembly of laboratory equipment, laboratory experimentation, analysis of data obtained, design of pilot plant, and cost estimation.

Literature Review. The first step in the literature review was a comprehensive survey of "Chemical Abstracts." A list of books, articles from periodicals, list of patents, and other sources containing information concerning the production of monosodium glutamate was obtained from this survey. All of these sources were obtained from the library facilities of Virginia Polytechnic Institute. Particular attention was given to the methods of synthesis, costs, materials of construction, and equipment design.

Assembly of Laboratory Equipment. The synthesis of monosodium glutamate was investigated from the laboratory viewpoint. This involved glassware experimentation. The equipment necessary for this experimentation was standard laboratory glassware and was obtained from the stockroom of the Chemical Engineering Department of the Virginia Polytechnic Institute.

Laboratory Experimentation. The reaction that was studied was the hydrolysis of soybean meal with hydrochloric acid. The method of reacting the soybean meal with water in an acid solution to determine when hydrolysis was complete was the first object of the investigation. The variables that were studied were concentration of acid and time of hydrolysis.

Variables that should be studied in the pilot plant are the effect of using phosphoric or sulfuric acid in the hydrolysis reaction. Also, pilot plant investigation should involve the study of the effect of calcium hydroxide and calcium carbonate as neutralizing agents. The effect of charcoal purification should be studied further and the determination of the purity of the final product monosodium glutamate determined.

Development laboratory data were obtained for the design of the hydrolysis tank, filtering equipment, and evaporators.

Analysis of Data. The data from the hydrolysis of soybean meal in an acid solution were analyzed to determine the optimum synthesis method and conditions. Data were obtained on the production of humin as a by-product and an analysis to determine the economic feasibility of the inclusion of this by-product in the design, proved that this inclusion was an economical necessity.

Pilot Plant Design. An integrated pilot plant for the production of monosodium glutamate was designed.

Estimation of Costs. A preliminary cost analysis was made for the pilot plant production of monosodium glutamate. The capital investment and the research

subsidy necessary to build and operate this pilot plant was determined.

Materials

The materials presented in the following paragraphs were used in the study of the synthesis of monosodium glutamate. The materials are presented with their specifications, place of obtainment, and use.

Alcohol, Ethyl. Scientific grade, 95 per cent. Obtained from Fisher Scientific Co., Silver Spring, Md. Used as a drying agent.

Carbon. Activated, drum 2-375A-14R. Obtained from Carbide and Carbon Chemicals Company, New York, N. Y. Used to study effect of carbon purification.

Carbon. Decolorizing, code 1551. Obtained from Allied Chemical and Dye Corp., New York, N. Y. Used as a decolorizing agent.

Hydrochloric Acid. CP grade, code 1090. Obtained from Allied Chemical and Dye Corp., New York, N. Y. Used as one of the reactants.

Sodium Hydroxide. Pellets. Obtained from Allied Chemical and Dye Corp., New York, N. Y. Used as one of the reactants and to adjust pH.

Soybean Meal. Forty-four per cent protein solvent extracted. Obtained from A. E. Staley Manufacturing Co., Decatur, Ill. Used as one of the reactants.

Water, Distilled. Obtained from laboratory still in the Department of Chemical Engineering, Virginia Polytechnic Institute, Blacksburg, Va. Used as wash water in the filtration processes.

Apparatus

The equipment presented in the following paragraphs were used in the study of the synthesis of monosodium glutamate. The equipment are presented with their specifications, place of obtainment, and use.

Balance. Analytical, Becker chain-o-matic. Manufactured by the Seederer-Kohlbusch Co., Jersey City, N. J. Used to weigh the reactants and yields of monosodium glutamate.

Glassware, Assorted Laboratory. Erlenmeyer flasks, beakers, graduated cylinders, filter flasks, funnels, pipets, and round-bottom flasks. Obtained from Fisher Scientific Co., Silver Spring, Md. Used to contain chemicals and process product.

Heater, Jacket. Serial No 5663, 110 v, 350 w. Manufactured by Glas-Col Apparatus Co., Terre Haute, Ind. Used to heat the reaction and to provide heat during concentration.

Hot Plate. No ROPH 7100, 120 v, 1000 w. Manufactured by Edwin L. Wiegand Co., Pittsburgh, Pa. Used to provide heat in concentration process.

Hydrometers. Range 1.180-1.251, subdivision 0.001. Obtained from Fisher Scientific Co., Silver Spring, Md. Used to determine specific gravity of mother liquor.

pH Meter. Model H, 115 v, 50-60 cy. Manufactured by Fisher Scientific Co., Pittsburgh, Pa. Used to determine pH for each step in the production of monosodium glutamate.

Powerstat. Type 110, 115 v, 50-60 cy, 7.5 amp, 0-135 v regulation, maximum KVA = 1. Manufactured by Superior Electric Co., Bristol, Conn. Used to control the temperature during hydrolysis and concentration.

Reactor, Three Neck. Pyrex, 1000 ml. Obtained from Fisher Scientific Co., Silver Spring, Md. Used as the reaction vessel.

Refrigerator. No 17-180, 115 v, 50-60 cy, ac. Obtained from Fisher Scientific Co., Silver Spring, Md. Used to cool the mother liquor allowing the glutamate hydrochloride crystals to form at 0-4 °C.

Stirrer, Magnetic. One hundred and fifteen volts, 50-60 cy. Obtained from Fisher Scientific Co., Silver Spring, Md. Used to stir the reaction solution.

Thermometers. Range 0-300 °F, two degree divisions. Obtained from Fisher Scientific Co., Pittsburgh, Pa. Used to measure reaction temperature.

Method of Procedure

The following paragraphs are a detailed description of the experimental procedure employed in the preparation of monosodium glutamate.

Preparatory Operation. The desired quantities of soybean meal and hydrochloric acid were weighed on an analytical balance and added to the hydrolysis flask.

Reaction Procedure. The reaction procedure was carried out by a number of different processes which were hydrolysis, filtration, concentration, cooling, and drying. These are discussed in the following paragraphs.

Hydrolysis. The hydrolysis reaction of soybean meal in the presence of hydrochloric acid was agitated. The temperature of the reaction was controlled by a heating jacket. The time and temperature of the hydrolysis reaction was 20 hours and 120 °C, respectively.

Filtration. The mother liquor from the hydrolysis flask was filtered in a buchner funnel to remove the humin.

Removing Impurities. The filtrate was treated with 0.6 gram of decolorizing charcoal to remove

impurities. The mixture was stirred for one hour and then allowed to stand for 12 hours at ambient temperature and pressure.

Filtrating Impurities. The mother liquor from the purifying flask was filtered in a buchner funnel to remove the impurities.

Concentration. The filtrate was then concentrated to a specific gravity of 1.22 by evaporating the water from the filtrate.

Cooling. The mother liquor after concentration to a specific gravity of 1.22 was cooled to 0 to 4 °C and allowed to stand for 12 hours at this temperature.

Filtrating Glutamic Acid Hydrochloride. The mother liquor after cooling was filtered in a buchner funnel to filter out the glutamic acid hydrochloride.

Redissolving. The glutamic acid hydrochloride was redissolved with water and stirring for one hour. The pH was changed from 2.0 to 3.0 to 3.2 by adding sodium hydroxide and thus forming glutamic acid. A pH of 3.0 to 3.2 was the isoelectric point of glutamic acid.

Filtrating Glutamic Acid. The mother liquor from the redissolving flask was filtered in a buchner funnel to filter out the glutamic acid.

Neutralizing. The glutamic acid was treated with water and sodium hydroxide to form monosodium glutamate at a pH of 7.

Concentration. The solution of monosodium glutamate formed during neutralization was then concentrated to a specific gravity of 1.22.

Cooling Monosodium Glutamate. The monosodium glutamate after concentration to a specific gravity of 1.22 was cooled to 4 °C to allow the monosodium crystals to form.

Filtrating Monosodium Glutamate. The monosodium glutamate after cooling was filtered in a buchner funnel.

Drying. The monosodium glutamate was treated with ethyl alcohol to absorb the moisture retained on the crystals of monosodium glutamate. The crystals were then dried by heat.

Analysis. The only analysis performed on the monosodium glutamate was the determination of the melting point of the material by the standard laboratory capillary tube method.

Data and Results

The experimental data and results obtained during this investigation are presented in the following paragraphs. Included are the data from the investigation of the optimum synthesis method and conditions, as well as the results of the pilot plant design and cost estimation.

Variables Studied During Hydrolysis. The variables studied to determine when hydrolysis of the soybean meal was complete were pH, specific gravity, and per cent solids. The pH using 26 per cent hydrochloric acid was 0.0 for 20 hours hydrolysis, and using 5 per cent hydrochloric acid varied from 1.0 to 0.18 for 20 hours hydrolysis with a noticeable break in the pH between 10 and 14 hours hydrolysis. The specific gravity for 26 per cent hydrochloric acid was 1.150 for 20 hours hydrolysis and for 5 per cent hydrochloric acid varied from 1.110 to 1.100 for 20 hours hydrolysis. The per cent solids for both 26 and 5 per cent hydrochloric acid varied from 0.128 to 0.162 per cent for 20 hours hydrolysis.

Monosodium Glutamate Experimental Data. The data obtained during the synthesis of monosodium glutamate are presented in Table III, page 36, to determine hydrolysis time, temperature, concentration, and purification information.

Process Engineering Research Data. The data obtained for the process specifications, material balance, heat balance, and equipment specifications are presented in Table IV, page 37.

Time Data. Figure 1, page 38, shows the effect of time on yield of glutamic acid hydrochloride.

Hydrochloric Acid Concentration Data. Figure 2, page 39, shows the effect of hydrochloric acid concentration on yield of glutamic acid hydrochloride.

Displacement and Batch Washing Data. Figure 3, page 40, shows the effect of batch or displacement washing on removing the soluble solids from the humin.

Material Balance. The material balance for the production of monosodium glutamate in the pilot plant is presented on pages 41 through 56.

Equipment Specifications. The equipment specifications for the equipment required in the pilot plant for

the production of 100 pounds of monosodium glutamate per day are presented on pages 57 through 77.

Equipment Flow Sheet. Figure 4, page 78, is a pictorial presentation of the flow of material through the equipment of 100 pounds of monosodium glutamate per day pilot plant.

Cost Estimation of Pilot Plant. The cost estimation to determine the capital investment and research subsidy necessary to build and operate the pilot plant is presented on pages 79 through 87.

TABLE III

Synthesis Conditions and Yield for the Preparation of Monosodium Glutamate[a]

Test No	Soybean Meal	Hydrochloric Acid		Hydrolysis Temperature,	Hydrolysis Time,	Amount of Wash Water,	Type of Carbon,[e]	Specific Gravity after Concentration,[c]	Cooling Temperature,	Yield[d]	
	gm	gm	%[b]	°C	hr	ml			°C	gm	%
1	60	240	26	120	20	100	f	1.22	0	0.8626	1.43
2	60	240	26	120	20	240	f	1.22	0	0.4520	0.75
3	60	240	26	120	20	100	f	1.22	0	0.5549	0.92
4	60	240	5	120	20	100	f	1.22	0	0.3265	0.55
5	60	240	5	120	20	100	f	1.22	0	0.4921	0.82
6	60	240	2	120	20	100	f	1.22	0	1.1916	1.99
7	60	240	2	120	20	100	f	1.22	0	0.8906	1.49
8	60	240	26	120	2	100	f	1.22	0	0.5630	0.94
9	60	240	26	120	5	100	f	1.22	0	0.4845	0.81
10	60	240	26	120	12	100	f	1.22	0	0.4640	0.77
11	60	240	26	120	20	50	f	1.22	0	0.4277	0.71
12	60	240	26	120	20	200	f	1.22	0	0.5040	0.84
13	60	240	26	20	20	100	f	1.22	0	0.2902	0.48
14	60	240	26	20	30	100	f	1.22	0	0.4773	0.79
15	60	240	2	120	5	100	f	1.22	0	0.5472	0.91
16	60	240	2	120	10	100	f	1.22	0	0.4528	0.76
17	60	240	26	120	20	100	g	1.22	0	3.4356	5.72
18	60	240	26	120	20	100	f	1.22	0	1.5858	2.64
19	60	240	26	120	20	100	h	1.22	0	2.9727	4.95

[a] Based on the yield of glutamic acid hydrochloride
[b] Weight per cent
[c] Specific gravity at 20 °C
[d] Per cent yield based on 60 grams of original soybean meal
[e] Amount of carbon added, 0.6 gram
[f] No carbon treatment
[g] Carbon decolorizing treatment
[h] Activated carbon treatment

TABLE IV

Process Conditions for Equipment Specifications

for Pilot Plant

Specific gravity of main streams, at 20 °C	1.00 min 1.28 max
Thickness of filter cake (12 gm dried humin), inches	0.315
Time of filtration of humin, minutes	2.0
Moisture content of the humin, per cent	65.0
pH of main streams	0.0 min 7.0 max
Temperatures of processes, °C	0.0 min 120.0 max

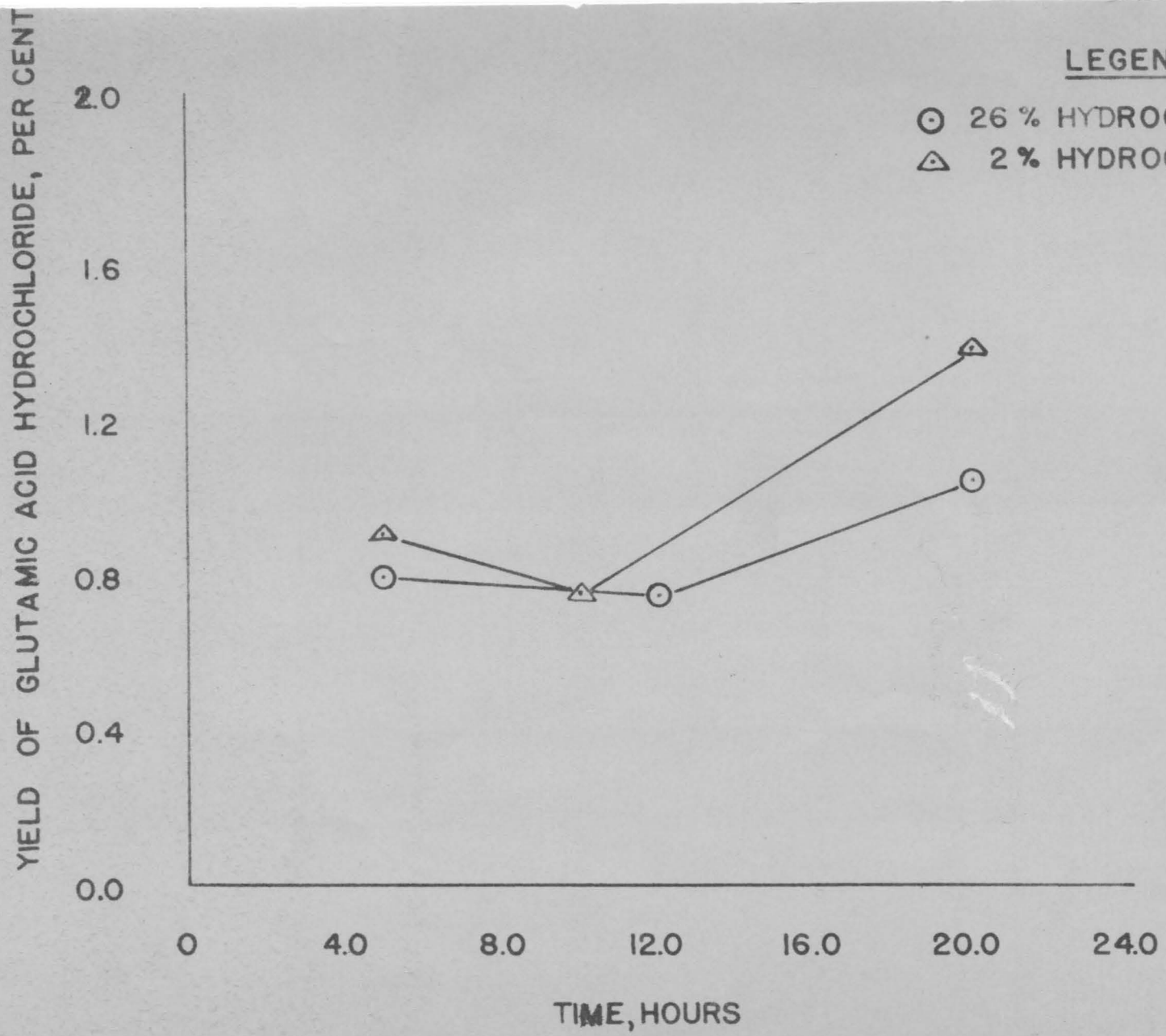


FIGURE 1. EFFECT OF TIME ON YIELD OF GLUTAMIC ACID HYDROCHLORIDE

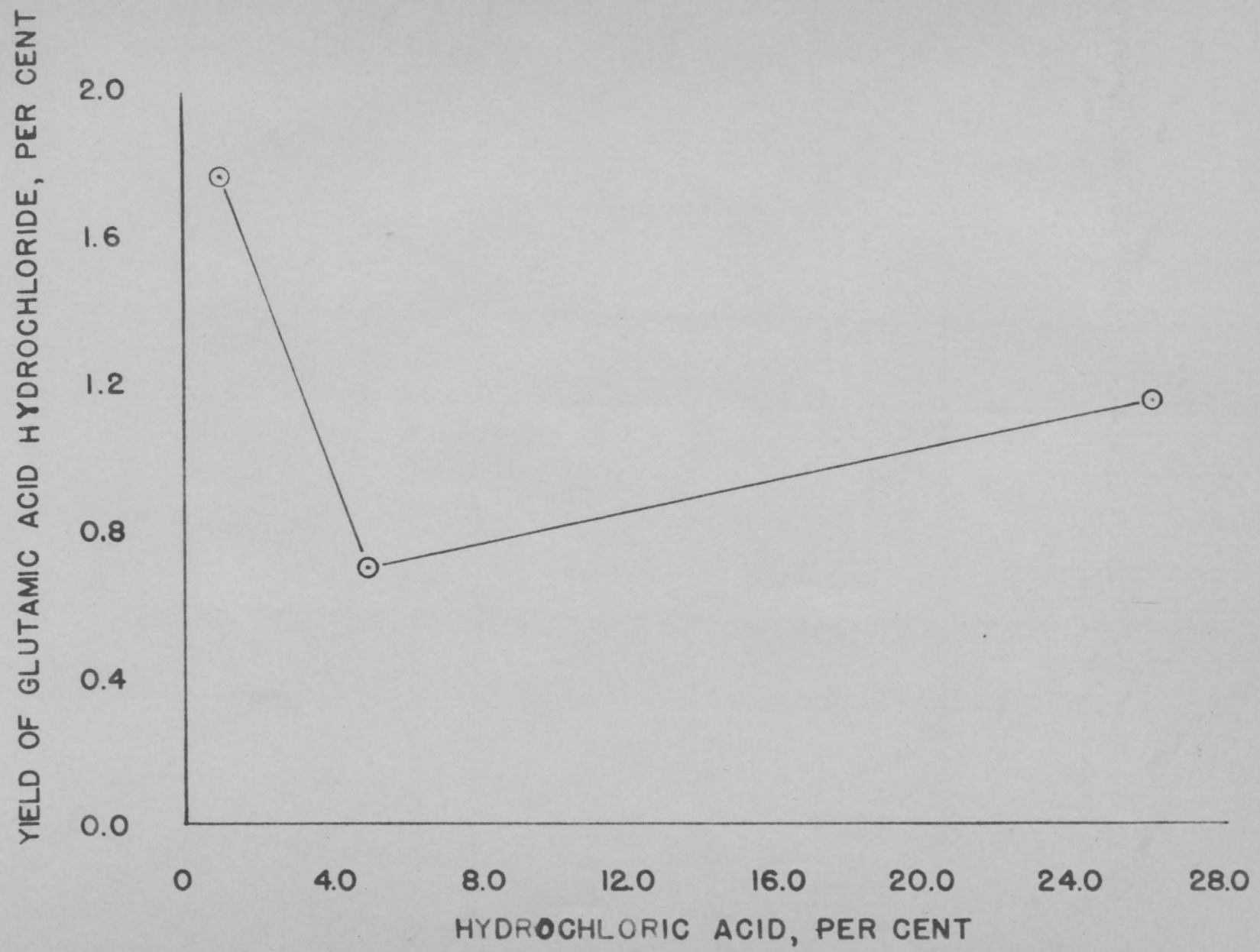


FIGURE 2. EFFECT OF ACID CONCENTRATION ON YIELD OF GLUTAMIC ACID HYDROCHLORIDE

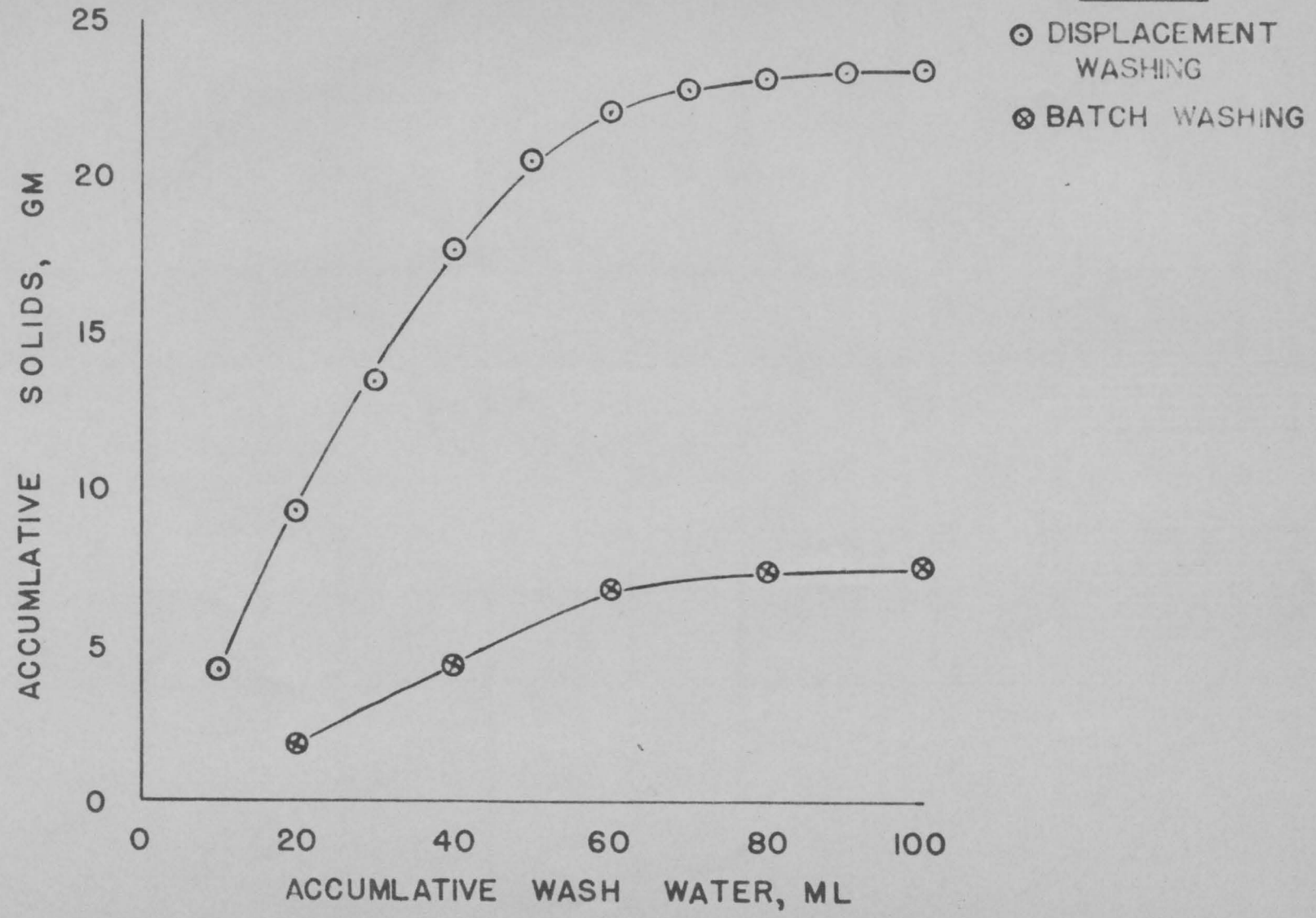


FIGURE 3. EFFECT OF WASH WATER ON REMOVING SOLIDS FROM HUMIN

Material Balance[47]

Basis: 100 lb of monosodium glutamate per 8-hr day.
All data are given in avoirdupois pounds.

M-1. Hydrolysis Tank.

Entering		Leaving	
From storage and scales		To pump, p-1	
Soybean oil meal	4,000	Unhydrolyzed meal	2,156
HCl	320	Hydrolyzed protein	2,102
Water	15,680	HCl(bound)	320
Total	20,000	Water	15,422
		Total	20,000

P-1. Pump.

Entering		Leaving	
From hydrolysis tank, M-1		To rotary filter, M-2	
Unhydrolyzed meal	2,156	Unhydrolyzed meal	2,156
Hydrolyzed protein	2,102	Hydrolyzed protein	2,102
HCl(bound)	320	HCl(bound)	320
Water	15,422	Water	15,422
Total	20,000	Total	20,000

M-2. Rotary Filter.

Entering		Leaving	
From pump, p-1		To pump, p-2	
Unhydrolyzed meal	2,156	Hydrolyzed protein	2,102
Hydrolyzed protein	2,102	HCl(bound)	320
HCl(bound)	320	Water	9,590
Water	15,422	Total	12,012
Total	20,000	To storage	
		Unhydrolyzed meal	2,156
		Water	5,832
		Total	7,988
		Total	20,000

P-2. Pump.

Entering		Leaving	
From rotary filter, M-2		To purifying tank, M-3	
Hydrolyzed protein	2,102	Hydrolyzed protein	2,102
HCl(bound)	320	HCl(bound)	320
Water	9,590	Water	9,590
Total	12,012	Total	12,012

M-3. Purifying Tank.

Entering		Leaving	
From pump, p-2		To pump, p-3	
Hydrolyzed protein	2,102	Hydrolyzed protein	2,102
HCl(bound)	320	HCl(bound)	320
Water	9,590	Water	9,590
Total	12,012	Bonechar	40
From storage and scales		Total	12,052
Bonechar	40		
Total	40		
Total	12,052		

P-3. Pump.

Entering		Leaving	
From purifying tank, M-3		To filter press, M-4	
Hydrolyzed protein	2,102	Hydrolyzed protein	2,102
HCl(bound)	320	HCl(bound)	320
Water	9,590	Water	9,590
Bonechar	40	Bonechar	40
Total	12,052	Total	12,052

M-4. Filter Press.

Entering		Leaving	
From pump, p-3		To pump, p-4	
Hydrolyzed protein	2,102	Hydrolyzed protein	2,102
HCl(bound)	320	HCl(bound)	320
Water	9,590	Water	9,590
Bonechar	40	Total	12,012
Total	12,052	To disposal	
		Bonechar	40
		Total	40
		Total	12,052

P-4. Pump.

Entering		Leaving	
From filter press, M-4		To evaporator, M-5	
Hydrolyzed protein	2,102	Hydrolyzed protein	2,102
HCl(bound)	320	HCl(bound)	320
Water	9,590	Water	9,590
Total	12,012	Total	12,012

M-5. Evaporator.

Entering		Leaving	
From pump, p-4		To condenser, M-6	
Hydrolyzed protein	2,102	Water	9,108
HCl(bound)	320	Total	9,108
Water	9,590	To pump, p-5	
Total	12,012	Hydrolyzed protein	3,537
From heat exchanger, M-10		HCl(bound)	595
Hydrolyzed protein	1,435	Water	1,738
HCl(bound)	275	Total	5,870
Water	1,256	Total	14,978
Total	2,966		
Total	14,978		

M-6. Condenser.

Entering		Leaving	
From evaporator, M-5		To drain	
Water	9,108	Water	9,108
Total	9,108	Total	9,108

P-5. Pump.

Entering		Leaving	
From evaporator, M-5		To cooling tank, M-7	
Hydrolyzed protein	3,357	Hydrolyzed protein	3,357
HCl(bound)	595	HCl(bound)	595
Water	1,738	Water	1,738
Total	5,870	Total	5,870

M-7. Cooling Tank.

Entering		Leaving	
From pump, p-5		To pump, p-6	
Hydrolyzed protein	3,357	Hydrolyzed protein	3,357
HCl(bound)	595	HCl(bound)	595
Water	1,738	Water	1,738
Total	5,870	Total	5,870

P-6. Pump.

Entering		Leaving	
From cooling tank, M-7		To centrifugal, M-8	
Hydrolyzed protein	3,357	Hydrolyzed protein	3,357
HCl(bound)	595	HCl(bound)	595
Water	1,738	Water	1,738
Total	5,870	Total	5,870

M-8. Centrifugal.

Entering		Leaving	
From pump, p-6		To mixing tank, M-11	
Hydrolyzed protein	3,357	Hydrolyzed protein	2,102
HCl(bound)	595	HCl(bound)	320
Water	1,738	Water	482
Total	5,870	Total	2,904
		To pump, p-7	
		Hydrolyzed protein	1,435
		HCl(bound)	275
		Water	1,256
		Total	2,966
		Total	5,870

P-7. Pump.

Entering		Leaving	
From centrifugal, M-8		To centrifugal storage tank, M-9	
Hydrolyzed protein	1,435	Hydrolyzed protein	1,435
HCl(bound)	275	HCl(bound)	275
Water	1,256	Water	1,256
Total	2,966	Total	2,966

M-9. Centrifugal Storage Tank.

Entering		Leaving	
From pump, p-7		To pump, p-8	
Hydrolyzed protein	1,435	Hydrolyzed protein	1,435
HCl(bound)	275	HCl(bound)	275
Water	1,256	Water	1,256
Total	2,966	Total	2,966

P-8. Pump.

Entering		Leaving	
From centrifugal storage tank, M-9		To heat exchanger, M-10	
Hydrolyzed protein	1,435	Hydrolyzed protein	1,435
HCl(bound)	275	HCl(bound)	275
Water	1,256	Water	1,256
Total	2,966	Total	2,966

M-10. Heat Exchanger.

Entering		Leaving	
From pump, p-8		To evaporator, M-5	
Hydrolyzed protein	1,435	Hydrolyzed protein	1,435
HCl(bound)	275	HCl(bound)	275
Water	1,256	Water	1,256
Total	2,966	Total	2,966

M-11. Mixing Tank.

Entering		Leaving	
From centrifugal, M-8		To pump, p-9	
Hydrolyzed protein	2,102	Glutamic acid	147
HCl(bound)	320	Other amino acid	1,955
Water	482	NaCl	78
Total	2,904	HCl(bound)	272
From storage and scales		Water	29,665
NaOH	52	Total	32,117
Water	121		
Total	173		
Process water	29,040		
Total	32,117		

P-9. Pump.

Entering		Leaving	
From mixing tank, M-11		To centrifugal, M-12	
Glutamic acid	147	Glutamic acid	147
Other amino acids	1,955	Other amino acids	1,955
NaCl	78	NaCl	78
HCl(bound)	272	HCl(bound)	272
Water	29,665	Water	29,665
Total	32,117	Total	32,117

M-12. Centrifugal.

Entering		Leaving	
From pump, p-9		To neutralization tank, M-13	
Glutamic acid	147	Glutamic acid	87
Other amino acids	1,955	Water	10
NaCl	78	Total	97
HCl(bound	272	To storage	
Water	29,665	Glutamic acid	60
Total	32,117	Other amino acids	1,955
Process water	19	NaCl	78
Total	32,136	HCl(bound)	272
		Water	29,674
		Total	32,136

M-13. Neutralization Tank.

Entering		Leaving	
From centrifugal, M-12		To pump, p-10	
Glutamic acid	87	Monosodium glutamate	100
Water	10	Water	1,000
Total	97	Total	1,100
From storage and scales			
NaOH	24		
Water	56		
Total	80		
Process water	923		
Total	1,100		

P-10. Pump.

Entering		Leaving	
From neutralization tank, M-12		To evaporator, M-14	
Monosodium glutamate	100	Monosodium glutamate	100
Water	1,000	Water	1,000
Total	1,100	Total	1,100

M-14. Evaporator.

Entering		Leaving	
From pump, p-10		To atmosphere	
Monosodium glutamate	100	Water	524
Water	1,000	Total	524
Total	1,100	To pump, p-11	
		Monosodium glutamate	100
		Water	476
		Total	576
		Total	1,100

P-11. Pump.

Entering		Leaving	
From evaporator, M-14		To cooling tank, M-15	
Monosodium glutamate	100	Monosodium glutamate	100
Water	476	Water	476
Total	576	Total	576

M-15. Cooling Tank.

Entering		Leaving	
From pump, p-11		To pump, p-12	
Monosodium glutamate	100	Monosodium glutamate	100
Water	476	Ethyl alcohol	530.3
Total	576	Water	503.5
From storage and scales		Total	1,133.8
Ethyl alcohol	5.3		
Water	0.5		
Total	5.8		
From pump, p-14			
Ethyl alcohol	525		
Water	27		
Total	552		
Total	1,133.8		

P-12. Pump.

Entering		Leaving	
From cooling tank, M-15		To centrifugal, M-16	
Monosodium glutamate	100	Monosodium glutamate	100
Ethyl alcohol	530.3	Ethyl alcohol	530.3
Water	503.5	Water	503.5
Total	1,133.8	Total	1,133.8

M-16. Centrifugal.

Entering		Leaving	
From pump, p-12		To pump, p-13	
Monosodium glutamate	100	Ethyl alcohol	525
Ethyl alcohol	530.3	Water	498.5
Water	503.5	Total	1,023.5
Total	1,133.8		
		To drier, M-18	
		Monosodium glutamate	100
		Ethyl alcohol	5.3
		Water	5
		Total	110.3
		Total	1,133.8

P-13. Pump.

Entering		Leaving	
From centrifugal, M-16		To fractionating column, M-17	
Ethyl alcohol	525	Ethyl alcohol	525
Water	498.5	Water	498.5
Total	1,023.5	Total	1,023.5

M-17. Fractionating Column.

Entering		Leaving	
From pump, p-13		To still condenser, M-19	
Ethyl alcohol	525	Ethyl alcohol	525
Water	498.5	Water	27
Total	1,023.5	Total	552
		To drain	
		Water	471.5
		Total	471.5
		Total	1,023.5

M-19. Still Condenser.

Entering		Leaving	
From fractionating column, M-17		To pump, p-14	
Ethyl alcohol	525	Ethyl alcohol	525
Water	27	Water	27
Total	552	Total	552

P-14. Pump.

Entering		Leaving	
From still condenser, M-19		To cooling tank, M-15	
Ethyl alcohol	525	Ethyl alcohol	525
Water	27	Water	27
Total	552	Total	552

M-18. Drier.

Entering		Leaving	
From centrifugal, M-16		To storage	
Monosodium glutamate	100	Monosodium glutamate	100
Ethyl alcohol	5.3	Total	100
Water	5	To drier condenser, M-20	
Total	110.3	Ethyl alcohol	5.3
		Water	5
		Total	110.3

M-20. Drier Condenser.

Entering		Leaving	
From drier, M-18		To drain	
Ethyl alcohol	5.3	Ethyl alcohol	5.3
Water	5	Water	5
Total	10.3	Total	10.3

Equipment Specifications[48]

S-1. Scale.

Number required: One

Type: Toledo portable scale

Capacity: 2000 lb

Platform Size: 30 in x 30 in

Available from: Toledo Scale Co., Toledo 1, Ohio[11].

S-2. Scale.

Number required: One

Type: Toledo bench dial scale, with tare and capacity beam

Capacity: 200 lb

Overall dimensions: Length 2 ft; width 1 ft 6 in; height 3 ft

Available from: Toledo Scale Co., Toledo 1, Ohio[11].

M-1. Hydrolysis Tank.

Number required: Three

Nominal capacity: 370 cu ft

Type: Cylindrical, removable cover plate, spherical bottom

Construction: Bronze

Overall dimensions: Diameter 5.5 ft; height 12.33 ft

Steam inlet: 4 in bronze tubing, 15 lb/sq in, gage

Condensate outlet: 4 in bronze tubing

Solution outlet: 4 in bronze pipe

Nature of contents: Soybean oil meal in 2 per cent hydrochloric acid solution at 248 °F

Heating coils: 88.5 ft of 4 in bronze tubing

Available from: The Pfaudler Co., Rochester 5, N. Y.[12].

M-2. Rotary Drum Filter.

Number required: Three

Filtering area: 500 sq ft

Type: FEinc pilot plant filter, compression dewatering, string discharge

Construction: Bronze

Drum diameter: 10.5 ft

Drum speed: 1 rpm

Cake characteristics: 1 in thick, 60 per cent moisture, unhydrolyzed meal at 150 °F

Filtrate characteristics: Hydrolyzed protein and water at 150 °F

Capacity: 2666 lb dry solids per day

Vacuum: 22 in of Hg

Filtrate receiver: Bronze

Overall dimensions: Length 21 ft 11 in; width 15 ft 2 in; height 13 ft 9 in

Available from: Filtration Engineers, Inc., Newark 4, N. J.[13].

M-3. Purifying Tank.

Number required: One

Nominal capacity: 1500 gal

Type: Cylindrical with hemispherical top and bottom

Material of construction: Bronze

Specifications: Actual capacity, 1630 gal; diameter, 78 in; cylinder height, 72 in; hemisphere height, 13 in; six 3 in pipe legs; 16 in manhole in top hemisphere; openings, 2 in top; 2 in bottom

Support: Six 3 in diameter pipe legs

Material handled: Hydrolyzed protein, bound HCl, bonechar, and water

Overall dimensions: Height, 103 in; diameter, 78 in

Condition of use: Temperature, 70 °F, pressure, atmospheric

Special feature: Blade type agitator, 150 rpm

Available from: Glascote Products, Inc., Cleveland, Ohio[14].

M-4. Filter Press.

Number required: One

Holding capacity: 0.125 cu ft

Type: Washing filter press

Size: 7 in with inside dimensions of frame 6 in x 6 in

Filtering area: 3 sq ft

Construction: Bronze

Nature of materials: Bonechar to be filtered from a solution composed of hydrolyzed proteins bound with HCl in a water solution

Available from: Independent Filter Press Co., Inc., Brooklyn 20, N. Y.(15).

M-5. Evaporator.

Number required: One

Type: Basket, vacuum evaporator

Nominal capacity: 1760 gal

Construction: Bronze

Overall dimensions: Diameter 6 ft; height 8 ft

Solution inlet: 2 in bronze pipe

Solution outlet: 2 in bronze pipe

Vapor outlet: 2 in steel pipe

Steam inlet: 1 in steel pipe

Condensate outlet: 1 in steel pipe

Nature of contents: Water solution of hydrolyzed protein with some bound HCl at 212 °F

Heating surface: 147 sq ft required, approximately 200 sq ft provided

Available from: The Colonial Iron Works Co., Cleveland 10, Ohio[16].

M-6. Condenser.

Number required: One

Type: Standard shell and tube condenser, single pass

Construction: Material - steel, 3/4 in od tubing; 16 in shell diameter

Overall dimensions: Length 11 ft 7 in; diameter 16 in

Cooling water inlet: 6 in steel pipe

Cooling water outlet: 6 in steel pipe

Vapor inlet: 8 in steel pipe

Condensate outlet: 3 in steel pipe

Cooling surface: One hundred and eighty 3/4 in od diameter steel tubes; area, 292 sq ft

Condensing material: Water, 212 °F

Available from: Doyle and Roth Manufacturing Co., Inc., Brooklyn 32, N. Y.[17].

M-7. Cooling Tank

Number required: One

Nominal capacity: 750 gal

Type: Cylindrical with a hemispherical top and bottom

Material of construction: Bronze

Specifications: Actual capacity, 780 gal; diameter, 60 in; cylinder height, 60 in; hemisphere height, 10 in; four 3 in pipe legs; 2 in opening in bottom

Support: Four 3 in diameter pipe legs

Material handled: Hydrolyzed protein with bound HCl and water

Cooling surface: Bronze jacket, 14.5 sq ft cooling surface; 70 ft of 2 in bronze tubing; 2 in cooling water inlet and outlet

Solution inlet: 2 in

Overall dimensions: Height, 72 in; diameter, 60 in

Special feature: Blade-type agitator, 50 rpm

Available from: Glascote Products, Inc., Cleveland, Ohio[14].

M-8. Centrifugal.

Number required: One

Type: Baker Perkins TypeHS-36 universal filtering centrifugal

Volume of drum: 4.15 cu ft

Basket diameter: 36 in

Nominal capacity: 4500 lb/hr

Construction: Bronze

Character of contents: Water solution of hydrolyzed protein

Solution inlet: 2 in

Available from: Baker Perkins Chemical Machinery Division, Saginaw, Michigan[18].

M-9. Centrifugal Storage Tank.

Number required: One

Nominal capacity: 300 gal

Type: Cylindrical with hemispherical top and bottom

Material of construction: Bronze

Specifications: Actual capacity, 318 gal; diameter, 42 in; cylinder height, 50 in; hemisphere height, 7 in; four 3 in pipe legs; 16 in manhole in top hemisphere; opening 2 in top; 1 in bottom

Support: Four 3 in diameter pipe legs

Material handled: Hydrolyzed protein, bound HCl, and water

Overall dimensions: Height, 69 in; diameter 42 in

Condition of use: Temperature, 70 °F; pressure, atmospheric

Special feature: Blade-type agitator; 150 rpm

Available from: Glascote Products, Inc., Cleveland, Ohio[14].

M-10. Heat Exchanger.

Number required: One

Type: Standard shell and tube heat exchanger, single pass

Construction: Material-bronze, 3/8 in od tubing; 4-1/2 in shell diameter

Overall dimensions: Length 4 ft; diameter 5-1/2 in

Cooling water inlet: 1-1/4 in bronze tube

Cooling water outlet: 1-1/4 in bronze tube

Vapor inlet: 2 in bronze tube

Condensate outlet: 1/2 in bronze tube

Cooling surface: Thirty-four 3/8 in od diameter bronze tubes; area, 5 sq ft

Materials handled: Water solution of hydrolyzed protein

Available from: The Patterson-Kelly Co., Inc., East Stroudsburg, Pa.[19].

M-11. Mixing Tank.

Number required: One

Nominal capacity: 4000 gal

Type: Cylindrical with open top and hemispherical bottom

Material of construction: Bronze

Specifications: Actual capacity, 4560 gal; diameter, 102 in; cylinder height, 110 in; eight 3 in. pipe legs; 1 in bottom opening

Support: Eight 3 in daimeter pipe legs

Material handled: Solution of water, amino acid salts, and sodium hydroxide

Overall dimensions: Height 144 in; diameter 102 in

Condition of use: Temperature, 70 °F; pressure, atmospheric

Special feature: Blade-type agitator, 150 rpm

Available from: Glascote Products, Inc., Cleveland, Ohio[14].

M-12 and M-16. Centrifugal.

Number required: Two

Type: Baker TypeHS450 universal filtering centrifugal

Volume of drum: 0.29 cu ft

Basket diameter: 12 in

Nominal capacity: 200 lb/hr

Construction: Bronze

Character of contents: Glutamic acid, other amino acids, sodium chloride, and water

Solution inlet: 2 in

Available from: Baker Perkins Chemical Machinery Division, Saginaw, Michigan[18].

M-13. Neutralization Tank.

Number required: One

Nominal capacity: 150 gal

Type: Cylindrical with open top and hemispherical bottom

Material of construction: Bronze

Specifications: Actual capacity, 169 gal; diameter, 36 in; cylinder height, 36 in; hemisphere height, 6 in; four 2 in pipe legs; 1 in opening in bottom

Support: Four 2 in diameter pipe legs

Material handled: Glutamic acid in a water solution treated with 30 per cent sodium hydroxide to obtain a solution of a pH of 7

Overall dimensions: Height, 54 in; diameter 36 in

Condition of use: Temperature, 70 °F; pressure, atmospheric

Special feature: Blade-type agitator, 150 rpm

Available from: Glascote Products, Inc., Cleveland, Ohio[14].

M-14. Pan Evaporator.

Number required: One

Type: Open jacketed, hemispherical, jacketed evaporating pan

Nominal capacity: 132 gal

Construction: Bronze

Overall dimensions: Diameter, 4 ft; height, 3 ft

Solution inlet: 1 in bronze pipe

Solution outlet: 1 in bronze pipe

Steam inlet: 1 in steel pipe, 15 lb/sq in, gage

Condensate outlet: 1 in steel pipe

Nature of contents: Water solution of monosodium glutamate

Heating surface: 7.3 sq ft required

Available from: The Colonial Iron Works Co., Cleveland 10, Ohio[16].

M-15. Cooling Tank.

Number required: One

Nominal capacity: 150 gal

Type: Cylindrical with an open top and hemispherical bottom

Material of construction: Bronze

Specifications: Actual capacity, 169 gal; diameter, 36 in; cylinder height, 36 in; hemisphere neight, 6 in; four 2 in pipe legs; 1 in opening in bottom

Support: Four 3 in diameter pipe legs

Material handled: Monosodium glutamate and ethyl alcohol in a water solution

Cooling surface: Bronze jacket, 8.4 sq ft cooling surface; 16 ft of 2 in bronze tubing; cooling water inlet and outlet, 2 in

Solution inlet: 1 in

Overall dimensions: Height, 54 in; diameter, 36 in

Special feature: Blade-type agitator, 150 rpm

Available from: Glascote Products, Inc., Cleveland, Ohio[14].

M-17. Fractionating Column.

Number required: One

Type: Bubble-cap, with stillpot, condenser, and reflux

Plates: Thirty-five 10 in diameter; height, 5 in; 1 in flange at top and bottom; downcomers - length, 5 in; diameter, 1 in; 1 bubble-cap per plate - height, 2 in; diameter, 2 in

Stillpot: Diameter, 2 ft; height, 2 ft; 23.5 ft of 2 in copper coils, heating area - 12.3 sq ft

Feed plate: Nineteen plates from top of column

Material of construction: Steel

Feed inlet: 1 in steel pipe

Bottoms outlet: 1 in steel pipe

Material distilled: Ethyl alcohol-water; maximum temperature, 210 °F

Steam inlet: 1 in steel pipe, 15 lb/sq in, gage

Condensate outlet: 1 in steel pipe

Vapor outlet: 1 in steel pipe

Reflux inlet: 1 in steel pipe

Overall dimensions: Height, 16 ft 7 in; diameter 1 ft

Available from: The Pfaudler Co., Rochester 4, N. Y.[12].

M-18. Drier.

Number required: One

Type: Vacuum shelf, with vacuum pump and surface condenser

Construction: Interior and shelves - bronze, heating shelves - 15 lb/sq in steam, exterior of steel

Supports: Concrete saddles

Characteristics of material: Monosodium glutamate; melting point, approximately 446 °F

Drying Agent: Ethyl alcohol; boiling point, 176 °F

Moisture content: 10 per cent

Quantity: 100 lb monosodium glutamate per 8 hr day

Vacuum: 23 in of Hg

Dimensions: Eight 40-in x 43-in shelves; 2-15/16 in between shelves; effective pan surface, 74 sq ft

Overall dimensions: Length, 5 ft 6 in; width, 5 ft; height, 5 ft

Condenser: Shell and tube surface - shell diameter, 5 in; surface area, 10 sq ft

Available from: J. P. Devine Mfg. Co., Inc., Pittsburgh 1, Pa.[20].

M-19. Still Condenser.

Number required: One

Type: Standard shell and tube condenser, single pass

Construction: Material - steel, 5/8 in od tubing; 8-5/8 in shell diameter

Overall dimensions: Length, 7 ft 3 in; diameter, 8-5/8 in

Cooling water inlet: 3 in steel pipe

Cooling water outlet: 3 in steel pipe

Vapor inlet: 4 in steel pipe

Condensate outlet: 2 in steel pipe

Cooling surface: Forty 5/8-in od diameter steel tubes; area 29 sq ft

Condensing material: 95 per cent ethyl alcohol, 178 °F

Available from: Doyle and Roth Manufacturing Co., Inc., Brooklyn 32, N. Y.[17].

P-1, P-2, P-3, P-4, and P-9. Process Pumps.

Number required: Five

Type: Self priming, centrifugal, single stage

Capacity rating: 200 gal per min

Materials handled: Hydrolyzed protein, unhydrolyzed meal, glutamic acid, other amino acids, and water

Total head: 270 ft max

Material of construction: Bronze

Size inlet: 4 in

Size outlet: 3 in

Motor: Squirrel-cage induction, 3-hp, 3-phase, 60-cycle, 220 to 440-volt, ac, V-belt drive

Overall dimensions: Length, 5 ft; width, 4 ft; height, 2 ft

Available from: Worthington Corp., Harrison, N. J.[21].

P-5, P-6, P-7, P-8, P-10, P-11, P-12, P-13, and P-14. Process Pumps.

Number required: Nine

Type: Self priming, centrifugal, single stage

Capacity rating: 30 gal per min

Materials handled: Water, hydrolyzed protein, monosodium glutamate, and ethyl alcohol

Total head: 50 ft

Material of construction: Bronze

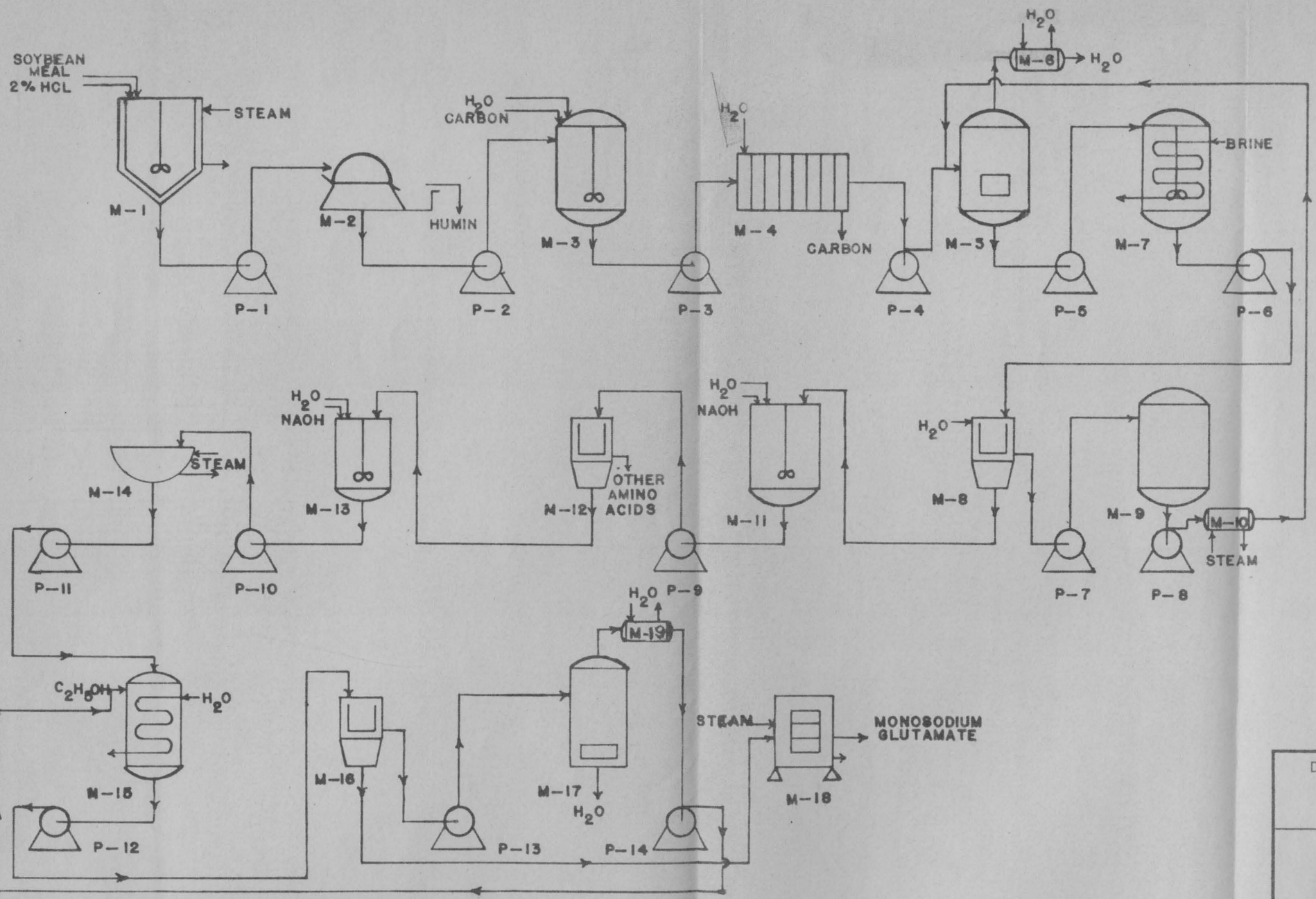
Size inlet: 1 in

Size outlet: 1 in

Motor: Squirrel-cage induction, 1/4-hp, 3-phase, 60-cycle, 220 to 440-volt, ac, 1750 rpm, V-belt drive

Overall dimensions: Length, 2 ft; width 1 ft 6 in; height, 1 ft 6 in

Available from: Worthington Corp., Harrison, N. J.[21].



LEGEND

M-1 HYDROLYSIS TANK
M-2 ROTARY FILTER
M-3 PURIFYING TANK
M-4 FILTER PRESS
M-5 EVAPORATOR
M-6 CONDENSER
M-7 COOLING TANK
M-8 CENTRIFUGAL
M-9 STORAGE TANK
M-10 HEAT EXCHANGER
M-11 MIXING TANK
M-12 CENTRIFUGAL
M-13 NEUTRALIZATION TANK
M-14 PAN EVAPORATOR
M-15 COOLING TANK
M-16 CENTRIFUGAL
M-17 STILL
M-18 DRIER
M-19 STILL CONDENSER
P-1 – P-14 PROCESS PUMPS

DEPARTMENT OF CHEMICAL ENGINEERING
VIRGINIA POLYTECHNIC INSTITUTE
BLACKSBURG, VIRGINIA

FLOWSHEET OF MONOSODIUM GLUTAMATE PILOT PLANT

SCALE:	NONE	DATE		CASE NO:	
DRAWN BY:	RSL	4/5/56		FILE NO:	
CHECKED BY:	RSL	4/5/56		FIGURE NO:	4
APPROVED BY:	FCV			SHEET NO:	

Equipment Costs[22,37]

Item No	No Required	Description	Installed Cost
S-1	1	weighing scale	$ 1,000
S-2	1	weighing scale	100
M-1	3	hydrolysis tanks	58,500
M-2	3	rotary drum filter	62,600
M-3	1	purifying tank	5,850
M-4	1	filter press	390
M-5	1	evaporator	5,850
M-6	1	condenser	4,600
M-7	1	cooling tank	4,500
M-8	1	centrifugal	24,100
M-9	1	storage tank	2,930
M-10	1	heat exchanger	975
M-11	1	mixing tank	8,800
M-12 & M-16	2	centrifugals	21,400
M-13	1	neutralization tank	1,950
M-14	1	pan evaporator	1,750
M-15	1	cooling tank	2,930
M-17	1	still	7,250
M-18	1	drier	5,060
Subtotal I			$ 220,535

Item No	No Required	Description	Installed Cost
M-19	1	still condenser	$ 400
P-1,2,3,4,&9	5	process pumps	8,800
P-5,6,7,8,10, 11,12,13,& 14	9	process pumps	8,800
Subtotal II			18,000
Subtotal I			220,535
Total			$ 238,535

Total Manufacturing Machinery and Equipment Costs[23]

Total numbered equipment (I)	$ 238,535
Piping: at 64 % of (I)	152,900
Wiring: at 20 % of (I)	47,800
Instruments: at 20 % of (I)	47,800
Insulation: at 4 % of (I)	9,550
Foundations, supports, and platforms: at 16 % of (I)	38,200
Site preparation, painting, and cleanup: at 6.3 % of (I)	15,050
Miscellaneous: at 3.8 % of (I)	9,075
Subtotal	558,910
Project overhead and contingencies: at 40 % of Subtotal	223,000
Total Manufacturing M & E	$ 781,910

Raw Materials Costs[23]

Material	Unit	Quantity/yr	Unit Cost	Cost/yr
Soybean meal	lb	967,000	$ 0.025	$ 24,200
Hydrochloric acid	lb	233,000	0.034	7,940
Bonechar	lb	9,670	0.0325	315
Sodium hydroxide	lb	36,800	0.0275	1,010
Ethyl alcohol	lb	1;805	0.455	820
Total				$ 34,285

Labor Costs[a]

	No Required	Hr Rate	Salary	Total
Supervision	1	--	$ 7,000	$ 7,000
Foreman	1	$ 2.58	5,000	5,000
Operator, skilled	2	1.90	3,952	7,904
Operator, unskilled	6	1.70	3,536	7,072
Watchman	1	1.05	2,184	2,184
Subtotal				29,160
Payroll charges: at 20 % of Subtotal				5,830
Total				$ 34,990

[a] Annual salary based on 242 operating days per year at 8 hours per day.

Manufacturing Cost Estimate[23]

Product: Monosodium glutamate

Production rate: 24,200 lb/yr

Total M & E: $ 781,910

Building cost (at 18 % of equipment and piping): $ 70,500

	Unit	Quantity/yr	Unit Cost	Cost/yr
Direct Conversion Expense				
Labor				$ 29,160
Payroll charges				5,830
Steam	M lb	15.3	$ 0.90	14
Electricity	kwh	24,700.0	0.01	247
Cooling water	1000 gal	8,260.0	0.01	83
Process water	1000 gal	1,700.0	0.03	51
Plant supplies	1 % (M&E)	--	--	7,819
Repairs	10 % (M&E)	--	--	78,191
Laundry	man/wk	630.0	1.00	630
Royalty	lb	24,200.0	0.01	242
Containers	25 lb cartons	970.0	0.10	97
Total				$ 122,264

Indirect Conversion Expense

Depreciation, equipment: at 10 % of M & E	$ 78,191
Depreciation, building: at 3 % of building cost	2,120
Taxes: at 5 % of M & E	39,100
Insurance: at 0.5 % of M & E	3,910
Controllable indirect conversion expense: at 50 % of labor costs	17,500
Total	$ 140,821
Bulk conversion cost: direct plus indirect costs	$ 263,085
Total manufacturing cost: raw material plus bulk conversion costs	$ 297,370

Estimated Capital Requirements[23]

Product: Monosodium glutamate

Production rate: 24,200 lb/yr

Fixed Capital	
Land: at $5,000/acre	$ 5,000
Building	70,500
Manufacturing M & E	781,910
Nonmanufacturing M & E: at 40 % of M & E	313,000
General service facilities:	
Steam: at $5/lb used in one hour operation	39,500
Power: at $100/kwh used in one hour of operation	1,280
Others: at $30 for each $100 of manufacturing M & E	234,573
Total fixed capital	1,545,763
Working Capital	
Raw material inventory: 30 day inventory	4,250
In-process and finished goods inventory: In-process at one week's raw material cost; finished goods at one week's production at manufacturing cost	1,485
Others (credit extended and operating expenses): at one month's production at selling cost	4,650
Total working capital	10,385
Total fixed plus working capital	$ 1,556,148

Selling Price and Return on Capital[23]

Product: Monosodium glutamate		
By-product: Hydrolyzed protein		
Annual sales: Monosodium glutamate	24,200 lb ($1.55/lb)	
Hydrolyzed protein	1,095,000 lb ($0.05/lb)	
Received from annual sales, net		$ 92,300
Research subsidy		500,000
Total		592,300
Deduct - annual production cost		297,370
Gross profit		294,930
Deduct - other expenses, selling, administration: at 10 % of production cost		29,737
Net profit - before taxes		265,193
Deduct - income and excess profit taxes: at 30 % of sales		35,000
New earnings		230,193
Total fixed capital		$ 1,545,763
Total fixed plus working capital		$ 1,556,148
Annual return on capital		
On total fixed capital		
Net profit, before taxes		17.3 %
New earnings		15.0 %
On total fixed plus working capital		
Net profit, before taxes		17.0 %
New earnings		14.8 %

Sample Calculations[49]

The sample calculations presented in the following paragraphs were used in the calculation of the results of this investigation. The pilot plant was designed on the basis of a 7 per cent of the theoretical yield. The conditions calculated for the pilot plant are not static and may be varied to fit the pilot plant experimental conditions. These conditions were chosen for the design, as they were considered to be the maximum that would be required for pilot plant experimentation.

Calculation of Theoretical Yield. The calculation and equation utilized in calculating the theoretical yield are presented below. The calculation is based on 60 grams of soybean meal and 240 grams of 2 per cent hydrochloric acid. The protein content of the soybean meal used was 46.1 per cent.

$$\left[NH - CH(CH_2)_2 - \overset{O}{\overset{\|}{C}} \right]_n + nHCl = nH_2O =$$

$$n[HCl][NH_2 - CH(COOH)(CH_2)_2 - COOH]$$

mol weight	mol weight	mol weight	=	mol weight
129	36.5	18		183

Mols of protein in soybean meal

$$= \frac{\text{grams of protein}}{\text{mol weight of protein}}$$

$$= \frac{60 \times 0.461}{129}$$

$$= 0.214$$

Mols of hydrochloric acid

$$= \frac{\text{grams of hydrochloric acid}}{\text{mol weight of hydrochloric acid}}$$

$$= \frac{240 \times 0.02}{36.5}$$

$$= 0.132$$

Mols of water

$$= \frac{240 \times 0.98}{18}$$

$$= 13.1$$

Theoretical yield

= mols of protein x mol weight glutamic acid hydrochloride

= 0.214 mols x 183 gm/mol

= 39.2 grams of glutamic acid hydrochloride

Calculation of Per Cent Yield. The following sample calculation was used to determine the per cent of the theoretical yield, obtained in the study of synthesis variables.

$$\text{Per cent yield} = \frac{\text{grams of product} \times 100}{\text{grams, theoretical yield}}$$

$$= 3.43 \times 100/39.2$$

$$= 8.75\ \%$$

The data for this computation were from test 17, Table III, page 36.

Material Balance Calculations. The following calculations were employed to determine the quantity of soybean meal, 2 per cent hydrochloric acid, bonechar, sodium hydroxide, and ethyl alcohol necessary for the production of 100 pounds of monosodium glutamate per day.

Mols of protein in soybean meal

$$= \frac{\text{weight of protein}}{\text{mol weight, protein}}$$

$$= \frac{4000 \text{ lb} \times 0.461}{129 \text{ lb/lb-mol}}$$

$$= 14.3 \text{ lb-mol}$$

2 per cent hydrochloric acid

= 4 times weight of soybean meal

= 4 x 4000 lb

= 16,000 lb

Bonechar

= 1 per cent of weight of soybean meal

= 0.01 x 4000 lb

= 40 lb

Sodium hydroxide to adjust pH to 3.2-3.5

= 0.162 per cent of total weight in mixing tank

= 0.00162 x 32,065 lb

= 52 lb

Sodium hydroxide to adjust pH to 3.2-3.5

= 2.18 per cent of total weight in neutralization tank

= 0.0218 x 1100 lb

= 24 lb

Ethyl alcohol

= for every pound of product add 0.92 pound of ethyl alcohol

= 0.92 x 576 lb

= 530.3 lb

Heat Capacity of Reaction Mixture. The heat capacity of the reaction mixture was determined by the use of Knopp's rule[33]. Experimental data showed that the temperature of the reaction mass increased from 70 to 248 °F without cooling.

Heat capacity of soybean meal

= 1.8 x No carbon atoms +
2.3 x No hydrogen atoms +
6.2 x No nitrogen atoms +
4.0 x No oxygen atoms

= 1.8(5) + 2.3(7) + 6.2(1) + 4(3)

= 43.3 cal/gm-mol °F

Heat capacity of reaction mixture

= C_p hydrochloric acid +

= C_p soybean meal

= 0.185 Btu/lb °F + $\frac{43.3 \text{ Btu/lb-mol °F}}{129 \text{ lb/lb-mol}}$

= 0.521 Btu/lb °F

Design of Hydrolysis Tank. The design of the hydrolysis tank involved the determination of the capacity, heat requirement, and heating area.

Capacity of Hydrolysis Tank. The following calculation was used to determine the capacity of the tank.

Volume

= volume of soybean meal + volume of hydrochloric acid

$$= \frac{4{,}000 \text{ lb of soybean meal}}{35 \text{ lb/cu ft}} + \frac{16{,}000 \text{ lb of 2 \% hydrochloric acid}}{62.4 \text{ lb/cu ft}}$$

= 370 cu ft

Heat Requirement. The quantity of 15 pounds per square inch, gage, steam that was required to elevate 6,675 pounds of soybean meal and 2 per cent hydrochloric acid from 70 to 248 °F was determined by the following calculation. The heat capacity of the mixture was assumed to be equal to the sum of the heat capacities of soybean meal and hydrochloric acid according to Knopp's rule of mixtures[33].

$$q = wcdt \qquad (1)$$

where:

q = quantity of heat to be added, Btu

w = weight of reaction mass, lb

c = heat capacity of reaction mass, Btu/lb-°F

dt = temperature rise, °F

q = 5,340 lb x 1 Btu/lb-°F x (248 - 70) °F +
1,335 lb x 0.58 Btu/lb-°F (248 - 70) °F

q = 1,088,000 Btu

therefore:

Pounds of steam

= heat required/latent of 15 lb/sq in., gage steam

= 1,088,000 Btu/945 Btu/lb

= 1,150 lb

Heating Area. The heating area required to elevate 6,675 pounds of soybean meal and 2 per cent hydrochloric acid from 70 to 248 °F was determined by the following calculation. The overall coefficient was assumed to be 60 Btu/hr-sq ft-°F[50].

$$Q = U A dt_m \quad (5) \qquad (2)$$

where:

Q = quantity of heat transferred, Btu

U = overall heat transfer coefficient, $\frac{\text{Btu}}{\text{hr-sq ft-°F}}$

A = area of heat flow, sq ft

dt_m = log mean temperature difference, °F

$$A = \frac{Q}{U\, dt_m}$$

$$A = \frac{1{,}088{,}000 \text{ Btu/hr}}{60 \text{ Btu/hr-sq ft-°F} \times \frac{(300-70)-(300-248)}{\ln 130/52}}$$

A = 92.5 sq ft or 88.5 ft of 4 inch bronze tubing

Capacity of Rotary Filter. The assumption in this calculation was that a drum filter with 500 square feet of filtering area was available. The calculation was to determine the number of revolutions required for filtering the hydrolyzed mass from the hydrolysis tank assuming the drum speed was one revolution per minute[6]. Solvent content was 67 per cent water.

Bulk Density of Wet Cake. The bulk density of the dry cake was 15.2 pounds per cubic foot. The following calculation was used to approximate the bulk density of the wet cake.

Density

= density of wet cake

= 0.33(15.2) lb/cu ft + 0.67(62.4) lb/cu ft

= 46.8 lb/cu ft

Revolutions of Filter. Assuming a one-inch cake on the filter, the following calculation was utilized to determine the revolutions of the filter.

Quantity filtered per revolution

= area of filter x cake thickness x cake density

= 500 sq ft x 1/12 ft x 46.8 lb/cu ft

= 1,950 lb

Revolutions

$$= \frac{\text{quantity of wet cake}}{\text{quantity filtered per revolution}}$$

= 6,675 lb/1,950 lb/rev

= 3.4 revolutions

Fractionating Column Design. The following calculations were used to design the fractionating column. The calculations involved the determination of the terminal stream conditions, calculation of q-line, calculation of minimum reflux, determination of slope of operating line, and determination of the actual number of plates[2,10].

Terminal Stream Conditions. The feed stream and the tops and bottoms compositions were determined by the following calculations. Ethyl alcohol here denotes 100 per cent.

Mol fraction ethyl alcohol in feed stream

$$= \frac{\frac{\text{lb ethyl alcohol}}{\text{mol wt ethyl alcohol}}}{\frac{\text{lb ethyl alcohol}}{\text{mol wt ethyl alcohol}} + \frac{\text{lb water}}{\text{mol wt water}}}$$

$$= \frac{525 \text{ lb}/46 \text{ lb}}{525 \text{ lb}/46 \text{ lb} + 498.5 \text{ lb}/18 \text{ lb}}$$

$$= 0.515$$

Mol fraction ethyl alcohol in tops

$$= \frac{\frac{\text{lb ethyl alcohol}}{\text{mol wt ethyl alcohol}}}{\frac{\text{lb water}}{\text{mol wt water}} + \frac{\text{lb ethyl alcohol}}{\text{mol wt ethyl alcohol}}}$$

$$= \frac{525 \text{ lb}/46 \text{ lb}}{525 \text{ lb}/46 \text{ lb} + 27 \text{ lb}/18 \text{ lb}}$$

$$= 0.88$$

Mol fraction ethyl alcohol in bottoms

$$= \frac{\frac{\text{lb ethyl alcohol}}{\text{mol wt ethyl alcohol}}}{\frac{\text{lb water}}{\text{mol wt water}} + \frac{\text{lb ethyl alcohol}}{\text{mol wt ethyl alcohol}}}$$

$$= \frac{1 \text{ lb}/46 \text{ lb}}{1 \text{ lb}/46 \text{ lb} + 471.5 \text{ lb}/18 \text{ lb}}$$

$$= 0.8 \times 10^{-3}$$

Calculation of q-Line. The following calculation was used to determine the slope of the q-line.

$$= \frac{\text{heat to make lb-mol of feed, vapor}}{\text{molal latent heat of feed}}$$

$$= \frac{46 \text{ lb} \times 0.473 \text{ Btu/lb-°F} \times (172 - 70) \text{ °F}}{46 \text{ lb} \times 371 \text{ Btu/lb}} +$$

$$46 \text{ lb} \times 371 \text{ Btu/lb}$$

$$= 1.13$$

Slope of q-line

$$= q/(q-1)$$

$$= 1.13/(1.13 - 1)$$

$$= 8.7$$

Calculation of Minimum Reflux. The minimum reflux was calculated by the following method. The actual reflux was 1.5 times the minimum reflux.

Minimum reflux

$$= \frac{x_D - y'}{y' - x'}$$

where:

x_D = mol fraction ethyl alcohol in product

y' = mol fraction ethyl alcohol in vapor at point of tangency of the operating line with the equilibrium curve

x' = mol fraction ethyl alcohol in liquid at point of tangency of the operating line with the equilibrium curve

Minimum reflux

$$= \frac{0.88 - 0.821}{0.821 - 0.798}$$

$$= 2.56$$

Actual reflux

$$= 2.56 \times 1.5$$

$$= 3.84$$

Calculation of Slope of Operating Line. The slope of the operating line was determined by use of the following equation.

$$\text{Slope} = \frac{R}{R + 1}$$

where:

R = actual reflux

$$\text{Slope} = \frac{3.84}{3.84 + 1}$$

$$= 0.795$$

Determination of Actual Number of Plates.

The theoretical number of plates was determined by the McCabe-Thiele method, as shown in Figure 5, page 104. The average plate efficiency was assumed to be 60 per cent.

Actual number of plates

$$= \frac{\text{theoretical plates}}{\text{plate efficiency}}$$

$$= \frac{21.0 - 1}{0.60}$$

$$= 34$$

The total number of plates is 35, including the stillpot, which is considered a perfect plate.

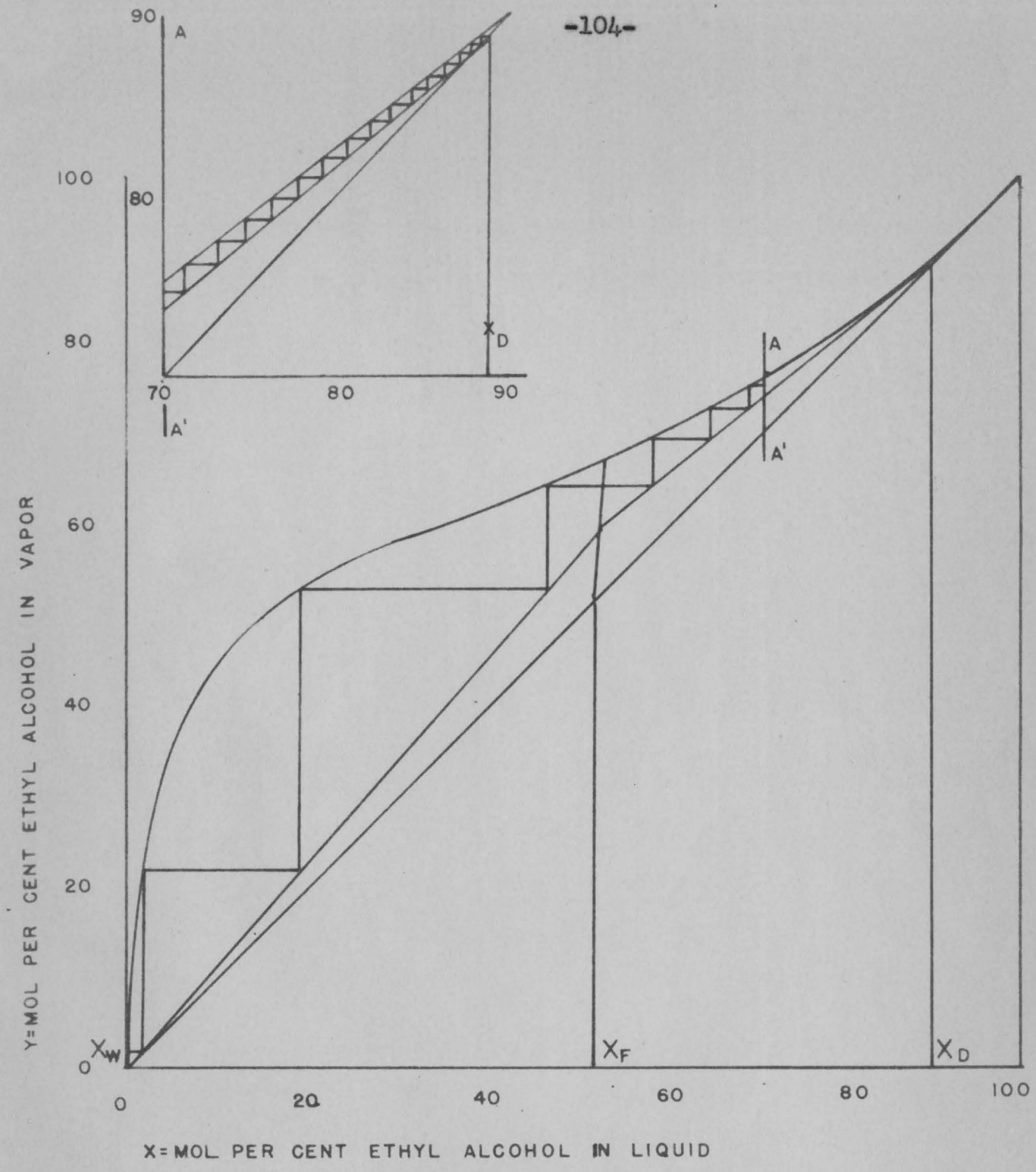


FIGURE 5. McCABE-THIELE DETERMINATION OF THEORETICAL PLATES IN FRACTIONATING COLUMN

Determination of Column Diameter. The column diameter was determined by using a maximum vapor load in the column of 552 pounds per hour and assuming a C of 400[55].

$$G = C[p_v(p_l - p_v)]^{0.5}$$

where:

G = the mass velocity of the vapor of the column cross-section, lb/hr-sq ft

C = a parameter depending upon the plate spacing and the surface tension of the liquid on the plate, ft/hr

p_l and p_v = the densities of the liquid and vapor, respectively, at the column conditions, lb/cu ft

$$G = 400 \text{ ft/hr}[0.103 \text{ lb/cu ft}(46 \text{ lb/cu ft} - 0.103 \text{ lb/cu ft})]^{0.5}$$

$$= 870 \text{ lb/hr-sq ft}$$

Cross-section area of column

$$= \frac{870 \text{ lb/hr-sq ft}}{552 \text{ lb/hr}}$$

$$= 0.636 \text{ sq ft}$$

Diameter of column

$$= \sqrt{\frac{4 \times \text{area}}{\pi}}$$

$$= \sqrt{\frac{4 \times 0.635}{\pi}}$$

$$= 0.9 \text{ ft}$$

Calculation of Capacity of Drier. The following calculations were employed to determine the capacity of the drier. A drier with six 40-inch by 43-inch trays and an effective heating area of 74 square feet was selected to perform the operation. The drier operates at 23 inches of mercury, vacuum[7]. The width of the filter drum is 1.06 feet and the circumference of the 10 square foot filter is 9.45 feet.

Area required

= circumference of drum x revolutions x width of cake

= 9.45 ft/rev x 6.6 rev x 1.06 ft

= 66 sq ft

The drier of 74 square feet area will fulfill the requirements. The tray of 40 inches in width will allow three cakes to be placed side by side on the tray.

Design Assumptions. The purpose of the design of this pilot plant was to design equipment, capable of handling materials at variable conditions, so that their effect upon the production of the material may be determined. The capacity of all of the equipment was designed so as to make the entire day's production in one step. Also, the water rates were calculated for each piece of equipment, on an assumed operation time. The water rates were also determined on the assumption that the entire day's production would be produced in one batch operation. The heating and cooling areas were determined on the assumption that the heats required or liberated for a day's production would have to be added or removed in one hour. This calculation was necessary, in order that the heat transfer surface would fit the pilot plant size equipment.

IV. DISCUSSION

This section of the thesis is a discussion of the results, recommendations, and limitations involved in the investigation of the design of a pilot plant for the production of monosodium glutamate.

Discussion of Results

The discussion of results obtained in the investigation of the design of a pilot plant for the production of monosodium glutamate is presented under the headings of synthesis method, synthesis conditions, pilot plant design, preliminary cost analysis, and proposed pilot plant studies.

Synthesis Method. Due to inadequate presentation of the reaction procedure in the literature, it was necessary to investigate the synthesis method. The synthesis method was investigated from the standpoint of variables of concentration, temperature, time, and purification. Monosodium glutamate was not obtained in the study of the variables listed but glutamic acid hydrochloride was obtained to give a basis of per cent yield. The discussion of the synthesis method is presented with reference to Table III, page 36.

Concentration Effect. Test 1 was performed in the manner interpreted from the literature[30]. In this case soybean meal was hydrolyzed with 26 per cent hydrochloric acid. This synthesis resulted in the formation of a product called glutamic acid hydrochloride. The yield obtained was 1.43 per cent glutamic acid hydrochloride based on the 60 grams of original soybean meal. The reaction was exothermic in the beginning of hydrolysis but heat had to be applied to maintain the 120 °C temperature required for the 20-hour hydrolysis period.

Temperature Effect. It was then thought that the 120 °C temperature was not necessary to obtain the glutamic acid hydrochloride. In test 13 the hydrolysis was carried out using 26 per cent hydrochloric acid for 20 hours hydrolysis at ambient temperature. The yield obtained was 0.48 per cent glutamic acid hydrochloride based on the 60 grams of original soybean meal as against 1.43 per cent yield using 120 °C temperature during hydrolysis.

Time Effect. It was thought that a shorter time of hydrolysis at the reflux temperature of

120 °C might produce a greater yield of glutamic acid hydrochloride because it was thought that the 20 hours hydrolysis was too long a period or that hydrolysis was complete before 20 hours. But, by decreasing the time the yield of glutamic acid hydrochloride decreased from 1.43 per cent to 0.94 per cent for 20 and 2 hours, respectively.

Purification Effect. It was stated in the literature[30] that carbon purification of the product from the hydrolysis of soybean meal with hydrochloric acid would give greater yields. The reason for the greater yields was stated to be that the carbon removed some impurities but no specific impurity was indicated. As a comparison of carbon purification and no carbon purification, tests 1 and 17 gave yields of 1.43 and 5.72 per cent, respectively. Test 1 being no carbon purification and test 17 using carbon purification.

Synthesis Conditions. After the optimum synthesis method had been determined, the next step in the investigation was the determination of the effect of time, the effect of acid concentration, effect of charcoal purification, and the effect of washing the humin.

Time of Hydrolysis. Figure 1, page 38, is a graphical presentation of the effect of time on the yield of glutamic acid hydrochloride. The graph has both 26 and 2 per cent hydrochloric acid represented. At 5 hours hydrolysis time the yields of glutamic acid hydrochloride for 26 and 2 per cent hydrochloric acid were 0.81 and 0.91 per cent, respectively. The yields are based on 60 grams of original soybean meal charged. Increasing the time of hydrolysis to 10 hours decreased the yield to 0.76 per cent using 2 per cent hydrochloric acid. Increasing the time of hydrolysis to 12 hours decreased the yield to 0.77 per cent using 26 per cent hydrochloric acid. Increasing the time of hydrolysis to 20 hours increased the yields of glutamic acid hydrochloride using 26 and 2 per cent hydrochloric acid to 0.92 and 1.49 per cent, respectively. The optimum hydrolysis time for both 26 and 2 per cent hydrochloric acid was 20 hours.

Hydrochloric Acid Concentration. Figure 2, page 39, is a graphical presentation of the effect of acid concentration on the yield of glutamic

acid hydrochloride. Using 26 per cent hydrochloric acid during the hydrolysis reaction of the soybean meal, gave a yield of 1.16 per cent glutamic acid hydrochloride. The amount of hydrochloric acid required by stoichiometric calculation was approximately 5 per cent hydrochloric acid using the same liquid to solids ratio of 4 to 1. Therefore, the 5 per cent hydrochloric acid gave a yield of 0.70 per cent glutamic acid hydrochloride. Then, trying a smaller amount of acid, 2 per cent hydrochloric acid. The yield obtained using the 2 per cent was 1.77 per cent glutamic acid hydrochloride. Accordingly, the optimum quantity or concentration of hydrochloric acid to be used during hydrolysis would be 2 per cent hydrochloric acid.

Charcoal Purification. From Table III, page 36, the use of carbon purification increased the yield of glutamic acid hydrochloride. As shown in tests 17, 18, and 19 using carbon decolorizing treatment, no carbon, and activated carbon, respectively, the yields were 5.72, 2.64, and 4.95 per cent glutamic acid hydrochloride.

As was determined the yields were approximately doubled when using some type of carbon purification. The amount of carbon used was 0.1 per cent by weight of the charged soybean meal. Exactly what impurities were involved in the carbon purification was not determined; only the effect on the yield of glutamic acid hydrochloride by carbon purification was studied. Of the types of carbon studied, the carbon decolorizing gave the greatest yield.

Washing the Humin. Figure 3, page 40, is a graphical presentation of the effect of washing the humin by both batch washing and displacement washing. The amount of solids washed out during displacement washing using 100 milliliters of wash water was 24 grams of soluble solids. The amount of solids washed out during batch washing using 100 milliliters of wash water was 7.5 grams of soluble solids. It was thought that batch washing would be the best type of washing but as the results indicated, displacement washing was the best. To try and explain this difference each method of washing was examined thoroughly. It

was concluded that the humin absorbed the soluble solids; because the other effects such as losing material during washing and operational differences could be duplicated to check the effects. Therefore, the results showed that displacement washing of the humin would give the best washing operation.

Pilot Plant Design. The pilot plant was designed to produce 100 pounds of purified monosodium glutamate per day. The pilot plant design is discussed under the headings of material balance, capacity of equipment, equipment heat transfer equipment, and pilot plant operation.

Material Balance. The material balance was calculated on the assumption that the minimum yield would be 7 per cent of the theoretical. The solids to liquid ratio (soybean meal to 2 per cent hydrochloric acid) was 4 to 1 by weight; the soybean meal contained 46.1 per cent protein. The amount of bonechar or decolorizing carbon was 0.1 per cent of the solids charged or the soybean meal. The only impurities that have any effect are sodium chloride, any excess sodium hydroxide,

and ethyl alcohol. As for the sale of the final product, monosodium glutamate, these so-called impurities have no great effect except that the product cannot be sold as 99.9+ per cent. The quantity of ethyl alcohol in the reaction mass was 50 per cent, which was considered the average quantity of ethyl alcohol to be used in the pilot plant studies. The use of these quantities in the design of the pilot plant was to design equipment which would be capable of handling the maximum quantities of materials required in the pilot plant investigations. These quantities are not representative of the amount of materials which the pilot plant actually will process. These amounts will be determined by the variables studied in the pilot plant.

Capacity of Equipment. In pilot plant investigations it is assumed that varying syntheses and operating conditions will result. In designing the capacity of the equipment, it was assumed that at least one test per day would produce product that was of the required specifications. This necessitated equipment capacities which would

be capable of handling the materials in one batch that were required for the production of 100 pounds of purified monosodium glutamate. In all cases the equipment was designed to hold the required amount for one batch operations, and in many cases the equipment was capable of holding up to one and one-half the required amount. All of the equipment that would be in contact with corrosive materials was made of bronze and the rest of the equipment was made of steel. All supports were constructed of steel and the piping was of bronze for corrosive materials and steel for noncorrosive materials. The equipment can be obtained from standard, chemical process equipment manufacturers, as presented in the equipment specifications.

Equipment Heat Transfer Requirements. The assumption of overall coefficients of heat transfer in the low range and overdesign of the heating areas will allow the equipment to perform the operation in one batch. The cooling medium rates were determined on an assumed operating time for each piece of equipment, for a single batch production

of the product. This calculation revealed the maximum cooling medium rates that would be necessary. The steam requirements were calculated on the basis of the total amount of heat necessary for the production of 100 pounds of product. The steam employed in pilot plant operation was 15 pounds per square inch, gage, process steam.

Pilot Plant Operation. Figure 4, page 78, is the flow sheet of the monosodium glutamate pilot plant. The soybean meal and 2 per cent hydrochloric acid are hydrolyzed in the hydrolysis tank, M-1. The hydrolyzed material is then pumped to the rotary filter, M-2. After the unhydrolyzed meal (humin) is filtered out, the filtrate is then pumped to the carbon purification tank, M-3. The carbon is then removed in the filter press, M-4, with the filtrate being pumped to the evaporator, M-5. The concentrated filtrate is then cooled in the cooling tank, M-7. During the cooling of the filtrate, the glutamic acid hydrochloride is formed and filtered out in a centrifugal, M-8. To the solids filtered out,

water and sodium hydroxide are added in the mixing tank, M-11, to obtain a pH of 3.2-3.5, thus allowing the glutamic acid to form and be filtered out in the centrifugal, M-12. Again, water and sodium hydroxide are added forming monosodium glutamate with this solution being concentrated in the pan evaporator, M-14. The solution is cooled down in the cooling tank, M-15, and then the monosodium glutamate is filtered out in the centrifugal, M-16. The monosodium glutamate filter cake is then dried in the drier, M-18. The hydrolysis operation is carried out by the skilled operator and two unskilled operators. One of the unskilled operators above can carry out the filtering operation and carbon purification. The evaporation and cooling is carried out by one unskilled operator. The centrifugals are handled by one skilled operator and one unskilled operator can handle the drying for purification of the product. The skilled operator during hydrolysis can handle the fractionation of the ethyl alcohol. During the pilot plant operations, data can be recorded by the foreman and the

skilled operators. The studies and setup of the operation of the equipment for the particular pilot plant study can be determined by the engineers involved in the pilot plant investigation process. The pilot plant would operate eight hours a day for 241 days a year. The 241 days a year allow for Saturdays and Sundays off and also the holidays which are usually observed, nationally.

Preliminary Cost Estimation. The preliminary cost estimation of the pilot plant was determined in order to ascertain the economic feasibility of a research project of this type. The pilot plant should be built before completion of the commercial plant to determine the type of process to use in manufacturing the product. Pilot plant studies should be carried on during the operation of the commercial plant to determine the adjustments that should be made in the existing process, which would reduce the cost of manufacturing the product. The preliminary cost estimation is discussed under the headings of pilot plant cost estimation, economic feasibility of construction, and economic feasibility of producing only hydrolyzed meal.

Pilot Plant Cost Estimation. Pilot plant cost estimation is similar to commercial plant cost estimation, but different assumptions are required in the pilot plant cost estimation[37]. In building pilot plants it is usually assumed that the commercial plant will furnish a building for housing the pilot plant. In the cost estimation this eliminates the estimation of building costs, taxes on the building, and depreciation on the building. This also eliminates the estimation of real estate costs and taxes. There are several different ways of estimating the cost of pilot plant equipment[37]. The cost of pilot plant equipment can be estimated from the standard curves for commercial plants, but the small size places the particular piece of equipment at the lower end of the curve and many times must be approximated by extrapolation. This method is usable, but not reliable. Also, the cost of pilot plant equipment is increased by the necessity of building the equipment of corrosion resistant materials, such as bronze. One method of estimating the cost of pilot plant equipment

is by the weight and the type of equipment. This method is also usable, but not reliable due to the approximation of the weight of the piece of equipment. The cost estimation of this pilot plant was accomplished by using a combination of the cost graphs for commercial plants and the estimation by weight. The choice of this method was determined to be the best. The method of approximating the capital requirements for the plant was a standard method of approximating capital requirements for commercial plants[23]. Some of the approximations used in the calculation were adjusted so that they would satisfy the requirements of a pilot plant construction. It was also assumed that the packaging of the product would be handled by the commercial plant and that the only storage of the product would be in 25-pound drums. In determining the return on the capital investment, it was assumed that the pilot plant would have to share in the administration costs and that the profits obtained from sale of the pilot plant product would be taxable, the same as in the commercial plant.

Economic Feasibility of Construction. The pilot plant was designed to operate 241 days a year. During this time it was assumed that approximately 10 per cent of the yearly production, or 27 days' production, would be unsalable due to varying pilot plant conditions and obtaining a product, which did not fit the required product specifications. From the sale of 24,200 pounds of monosodium glutamate and 1,095,000 pounds of hydrolyzed protein produced per year the pilot plant could realize a net income of $92,300 per year. This net income would require a research subsidy of $500,000 a year to yield a 14.8 per cent return on the "investment." The capital investment required for the pilot plant was $1,556,148. This capital investment was the total fixed capital plus the working capital. This means that the company manufacturing monosodium glutamate must invest $1,556,148 in the construction and operation of the pilot plant. The company must also invest $500,000 a year in order to correlate the 14.8 per cent return on the investment. Within 10 years the income from

the pilot plant and the $500,000 a year subsidy would have paid off the original investment. After the first 10 years the company would only have to subsidize the pilot plant approximately $375,000 per year, if no net profits are to be realized from the pilot plant after the first 10 years. From this cost analysis it is evident that the process data obtained from the pilot plant would justify the investment required for this pilot plant. Further calculations were made to determine what the return on investment would be if the pilot plant was operating 24 hours a day and not as above, which was 8 hours a day. The net income was $276,900 and the research subsidy was $500,000 a year yet the return on investment was determined only to be 6.5 per cent. Therefore, the previous calculation gave the greatest return on investment which was 14.8 per cent.

Economic Feasibility of Producing Only Hydrolyzed Meal. The pilot plant was designed to produce only hydrolyzed protein meal. From the sale of 1,095,000 pounds of hydrolyzed protein

produced per year the pilot plant could realize a net income of $54,750 per year, as shown in Table V, page 125. This net income, coupled with a research subsidy of $250,000 a year, would yield a 11.6 per cent return on the investment. The capital investment required for the pilot plant was $762,700. This capital investment was the total fixed capital plus the working capital. This means that the company manufacturing only the hydrolyzed protein meal must invest $762,700 in the construction and operation of the pilot plant. The company must also invest $250,000 a year in order to correlate the 11.6 per cent return on the investment. Within 10 years the income from the pilot plant and the $250,000 a year subsidy would have paid off the original investment. After the first 10 years the company would only have to subsidize the pilot plant approximately $188,000 per year, if no net profits are to be realized from the pilot plant after the first 10 years. From this cost analysis it is evident that the process data obtained from the pilot plant would justify the

TABLE V

Selling Price and Return on Capital for Pilot Plant Producing Only Hydrolyzed Protein

Product: Hydrolyzed protein	
Annual sales: Hydrolyzed protein 1,095,000 lb ($0.05/lb)	
Received from annual sales, net	$ 54,750
Research subsidy	250,000
Total	304,750
Deduct - annual production cost	177,332
Gross profit	127,418
Deduct - other expenses, selling, administration: at 10 % of production cost	17,733
Net income - before taxes	109,685
Deduct - income and excess profit taxes: at 38 % of sales	20,800
New earnings	88,885
Total fixed capital	752,700
Total fixed plus working capital	762,700
Annual return on capital	
On total fixed capital	
Net profit, before taxes	14.5 %
New earnings	11.8 %
On total fixed plus working capital	
Net profit, before taxes	14.3 %
New earnings	11.6 %

investment required for this pilot plant of producing only hydrolyzed protein meal.

Proposed Pilot Plant Studies. The purpose of this pilot plant is to investigate the effects of the various process variables on the semi-commercial plant scale. The pilot plant studies are divided into investigations into the reaction conditions, operation of purification equipment, and operation of recovery equipment.

Reaction Conditions. The reaction conditions to be studied are the same as in the development laboratory in this investigation. These variables consist of the determination of optimum time for hydrolysis, per cent of hydrochloric acid, quantity of carbon for carbon purification, quantity of water in redissolving, quantity of sodium hydroxide for complete conversion to monosodium glutamate, and temperature of reaction. The study of the quantity of hydrochloric acid to soybean meal will be instrumental in increasing the yield because the correct rate of molecular contact will be obtained. Also, the effect of using sulfuric acid or phosphoric acid in hydrolysis

should be studied on the pilot plant scale. Another important variable is the determination of the effect of various amounts of soybean meal, hydrochloric acid, carbon, and sodium hydroxide on the yield and the properties of the product. Another variable to study is the determination of the effect of cooling the solution to 0 °C after evaporation. To study the effect of the other amino acids on the product is another important variable. Another important variable is the determination of what impurities are removed in carbon purification.

Operation of Purification Equipment. The purification of the crude monosodium glutamate is accomplished by filtering, drying, dissolving in water, concentration, crystallization, filtering, and drying. The operation of the filters is very important. More data concerning the characteristics of the slurries to the filters, the filtering characteristics of the slurries, the properties of the cakes, properties of the filtrates and filter operational conditions should be determined. These data should be obtained

for filtering the hydrolyzed protein slurry, carbon purification slurry, glutamic acid hydrochloride slurry, separating out other amino acids slurry, and the purified monosodium glutamate slurry. Also, the drying characteristics of the crude and purified monosodium glutamate slurry and humin should be determined. This will reduce the amount of water to be evaporated in the concentration process. The exact amount of water to be evaporated in the concentration processes should also be determined. The time for recrystallization of the glutamic acid hydrochloride and the monosodium glutamate should be studied. The rate of crystallization, conditions for crystallization, the type of crystals, and the type of crystal growth of glutamic acid hydrochloride and monosodium glutamate should be considered in this study. The time requirements for operation of each piece of equipment in the process should also be determined.

Operation of Recovery Equipment. The most important recovery in the pilot plant is the filtrate from the glutamic acid hydrochloride

filtration step. The filtrate is recycled to the concentration step where it is combined with new mother liquor to be concentrated. Once the pilot plant is put into operation this will be a continuous recycle operation. The filtrate should be analyzed to determine the amount of glutamic acid hydrochloride being recycled. Because it will become an economic factor when the larger commercial plant is built whether or not the concentration of the filtrate is rich enough in glutamic acid hydrochloride to warrant a recycle system. Another important recovery in the pilot plant is the fractionation of the filtrate from the monosodium glutamate filtration step. The composition of the filtrate to the fractionator was 51.5 mol per cent ethyl alcohol. The material to be distilled was composed of the filtrate and the water mixture from the centrifugal. The necessity of obtaining 95 weight per cent as the tops from the fractionator required a large number of plates, namely, 35 plates. The conditions under which the fractionator is

to be operated should be determined. The quantity of heat to be removed in the still condenser for optimum still steam requirements is another pilot plant study. Also, the bottoms from the fractionator should be carefully analyzed to determine if any monosodium glutamate is present.

In the design of the pilot plant, it was assumed that due to the long hours of hydrolysis (20 hours), all of the protein in the soybean meal was used up in the reaction. The possibility of the presence of unreacted protein in the filtrate from the hydrolysis reaction should be carefully studied. The filtering and drying characteristics of the monosodium glutamate and humin is another point that should be studied on the pilot plant scale. For all of the equipment, corrosion studies should be made to determine the materials of construction for the commercial plant.

Recommendations

The design of a pilot plant for the production of monosodium glutamate necessitates recommendations for pilot plant studies. In the analysis of the problem, it has been revealed that further development laboratory data are required. This development laboratory data should be obtained for the effect of using phosphoric acid or sulfuric acid in the hydrolysis reaction, the effect of recycling filtrate from glutamic acid hydrochloride filtration step, and the effect of recycling filtrate from glutamic acid filtration step.

Phosphoric Acid or Sulfuric Acid in Hydrolysis Reaction. Only hydrochloric acid was used in obtaining the development laboratory data for the production of monosodium glutamate. The possibility of having no impurities in the final product by using phosphoric acid should be investigated on the development laboratory scale. The use of sulfuric acid should also be studied and an economic study made to determine which acid would be the best to use in the hydrolysis reaction. The sulfuric acid will contaminate the humin to be sold for hydrolyzed protein but a method

of treating the humin may be determined if further studies were made.

Glutamic Acid Hydrochloride Filtrate Recycle. The presence of glutamic acid hydrochloride in the filtrate was not determined but it was the opinion of the author that a large amount of the glutamic acid hydrochloride passed through the first time filtered. If studies were made to determine the increase in yield of glutamic acid hydrochloride, the final yield of monosodium glutamate would be increased. This filtration step was where the change in the bulk quantity of mother liquor occurred. Thus, a loss of product at this point meant a decrease throughout the rest of the process in obtaining monosodium glutamate.

Glutamic Acid Filtrate Recycle. The glutamic acid is removed from the other amino acids at a pH of 3.2 to 3.5. The filtrate from this filtering process contains other amino acids. Studies should be made to determine how much glutamic acid passed through with the other amino acids. It may be economical to recycle this stream to increase the yield of glutamic acid. Thus, obtaining a greater yield of monosodium glutamate from the original soybean meal.

Pilot Plant Studies. In the event of the construction of this pilot plant, the pilot plant studies presented on pages 126 through 130 should be performed. Particular attention should be paid to the investigation of (1) reaction conditions, (2) filtration characteristics, (3) drying characteristics, (4) crystallization conditions, and (5) fractionation of the ethyl alcohol. Other problems which present themselves in the operation of the actual pilot plant, should be analyzed and studied.

Limitations

The limitations of the design of a pilot plant for the production of monosodium glutamate are presented under the headings of reactants, synthesis conditions, and design of the pilot plant.

Reactants. Soybean meal was employed in the laboratory investigation of the synthesis of monosodium glutamate. The soybean meal was 44 per cent protein solvent-extracted, obtained from A. E. Staley Manufacturing Company. The hydrochloric acid used in the reaction was CP grade, obtained from Allied Chemical and Dye Corporation. The sodium hydroxide used in the reaction was 100 per cent chemically pure grade, obtained from the Allied Chemical and Dye Corporation. The carbon used in purification was decolorizing code 1551, obtained from Allied Chemical and Dye Corporation. The 95 per cent ethyl alcohol used in the purification of the monosodium glutamate was scientific grade material, obtained from the U. S. Industrial Alcohol Company.

Synthesis Conditions. The optimum synthesis method was the hydrolysis of soybean meal with hydrochloric acid. The quantities used in hydrolysis were

60 grams of soybean meal reacted with 240 grams of 2 per cent hydrochloric acid. The hydrolysis times of 2, 5, 10, 12, 20, and 30 hours were studied. The concentrations of the hydrochloric acid studied were 2, 5, and 26 per cent. The amounts of wash water studied for washing the humin were 50, 100, 200, and 240 milliliters. The amount of carbon used in the purification step was 0.6 gram. The temperature of hydrolysis was studied for 20 and 120 °C.

Design of Pilot Plant. The pilot plant was designed to produce 100 pounds of monosodium glutamate per day. The pilot plant was to operate 241 days a year and eight hours a day. It was assumed that 27 days' production was unfit for sale. It was also assumed that the company, building the pilot plant, would furnish a pilot plant building for housing the pilot plant. The equipment was designed to hold the materials for one batch per day operation. The cooling water rates were calculated on the basis of an assumed operation time for each piece of equipment would occur in one hour's time for the entire day's production. The cost estimation was determined by a combination of

cost estimation for commercial plants, cost estimation for pilot plants, and the author's analysis of the cost estimation process.

V. CONCLUSIONS

The design of a pilot plant for the production of monosodium glutamate was approached from the standpoint of the determination of the optimum synthesis method and conditions, pilot plant design, and preconstruction cost estimation. The materials employed in this investigation were soybean meal 44 per cent protein solvent-extracted, chemically pure hydrochloric acid, 100 per cent chemically pure sodium hydroxide, carbon decolorizing code 1551, and scientific grade 95 per cent ethyl alcohol. The time of reaction was studied for 2, 5, 10, 12, 20, and 30 hours. The concentrations of hydrochloric acid studied were 2, 5, and 26 per cent. The amounts of wash water studied for washing the humin were 50, 100, 200, and 240 milliliters. The amount of carbon used in the purification step was 0.6 gram. The temperature of hydrolysis was studied for 20 and 120 °C. The pilot plant was designed to produce 100 pounds of monosodium glutamate per day. The following conclusions were drawn from this investigation.

1. The optimum synthesis method is the reaction of soybean meal with hydrochloric acid in water. For maximum yields of monosodium glutamate, there should be agitation.

2. The optimum hydrolysis time for the synthesis of monosodium glutamate is 20 hours.

3. The optimum concentration of hydrochloric acid to use in the synthesis of monosodium glutamate is 2 per cent.

4. The optimum type of wash for washing the humin is displacement washing.

5. The optimum temperature of the synthesis of monosodium glutamate is 120 °C.

6. Carbon purification will give a greater yield of monosodium glutamate.

7. A total fixed plus working capital of $1,556,148 is required for the construction of a minimal pilot plant for the production of monosodium glutamate. An additional research subsidy of $500,000 per year, coupled with the income from the sale of the product, will result in a 14.8 per cent return on the investment.

8. A total fixed plus working capital of $762,700 is required for the construction of a minimal pilot plant for the production of hydrolyzed protein. An additional research subsidy of $250,000 per year, coupled with the income from the sale of the product, will result in a 11.6 per cent return on the investment.

VI. SUMMARY

The purpose of this investigation was to design a pilot plant for the production of monosodium glutamate. The investigation involved the determination of optimum synthesis method and conditions, design of the pilot plant, and preconstruction cost estimation of the pilot plant.

The first attempts at the synthesis of monosodium glutamate resulted in a very poor yield of glutamic acid hydrochloride, the first product formed in the synthesis of monosodium glutamate. It was determined that the acid concentration was too great and that during the hydrolysis the excess hydrochloric acid hindered the hydrolysis reaction rather than assisted the hydrolysis reaction. It was determined that the optimum synthesis method that favored the formation of the glutamic acid hydrochloride was the reaction of soybean meal with a dilute solution of hydrochloric acid.

The next step in the investigation was the determination of the optimum synthesis conditions. It was determined that the optimum hydrolysis time was 20

hours. The optimum concentration of hydrochloric acid to be used in the hydrolysis reaction was 2 per cent. The humin should be washed by the displacement washing method. The carbon purification step gave a greater yield of glutamic acid hydrochloride. The basic reaction temperature was 120 °C. The basic reaction that was employed to determine the effect of synthesis variables was the hydrolysis of the soybean meal in a dilute solution of hydrochloric acid. The basic quantities of materials employed in the reaction to determine the effect of synthesis variables were 60 grams of soybean meal and 240 grams of hydrochloric acid. The amount of carbon used in carbon purification was 0.6 gram, and the amount of wash water used in washing the humin was determined to be 100 milliliters. After the determination of each optimum condition, the condition was added to the basic reaction as a synthesis condition.

The pilot plant was designed to produce 100 pounds of purified monosodium glutamate per day. The pilot plant was to operate 241 days a year and eight hours a day. The total output of the pilot plant was 24,200 pounds of monosodium glutamate and 1,095,000

pounds of humin per year. The capacity of the equipment was designed so that the production for one day could be produced in a single batch. The cooling water rates were determined for an average operation time for each piece of equipment. The cooling water rates and steam requirements were based on a single batch operation. The heat transfer surfaces were determined on the assumption of one hour heat transfer time for each piece of equipment. The assumption of low overall heat transfer coefficients and overdesign probably will allow the heat transfer surfaces to handle a one batch a day operation.

The cost estimation of the pilot plant revealed that $1,545,763 was required as fixed capital for construction of the pilot plant. The total fixed plus working capital, required to construct and operate the pilot plant, was $1,556,148. A research subsidy required to operate the pilot plant each year was calculated to be $500,000. The research subsidy of $500,000 and the income of $92,300, derived from the sale of the product and by-product, yielded a 14.8 per cent return on the total fixed plus working capital investment.

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