

1955

PROBLEM

To the Contestant:

The Student Contest Problem for 1955 has been selected by the committee as a typical problem which faces a process engineer in industry. Frequently alternative processes must be evaluated before a significant number of experimental data have been obtained in order for the company to decide on expenditure for development work. In addition, many times decisions to proceed with a commercial plant are based on tentative evaluations of portions of the process which have an appreciable effect on the over-all economics.

In this particular problem a manufacturer of a high-priced protein product needs to know with reasonable accuracy the cost of recovering the solvent used in precipitating the product. Because of the large quantities of solvent relative to the amount of product, the cost of solvent recovery is an appreciable part of the total manufacturing cost. A new idea such as the one presented in this problem can often make the difference between a profitable venture and one which merely breaks even or perhaps is abandoned. When a project of this kind has been carried through the pilot plant stage, the management research committee must decide whether to carry out the necessary work to present the project to top management for appropriation of capital funds. In order to make this decision reasonably accurate, cost estimates must be available, and often, as in this case, the calculations can be based on available data and correlations from the literature, the necessity for costly experimental work at this stage of the project being thus eliminated.

An important part of an assignment of this kind is the presentation of material for use by management. The report must be clear and must summarize the results briefly to save management time, but must also present the detailed explanation of how the results were obtained so that suggested modifications may be readily evaluated by others.

The committee has tried to present the problem in a manner which might be used by a typical company in the chemical field through the use of interdepartmental memoranda. As in most technical activities in the chemical industry, no problem is entirely the work of one man, and this is demonstrated by the various sources of information which are used to provide the process engineer with the data necessary to do his job. Company policies vary widely, but the committee believes that the approach used is similar to that frequently encountered. The student will consider

himself to be C. E. Major, a process engineer in the Engineering Department of the Boontown Chemical Company. He reports to J. B. Smith, Chief Engineer and head of that department. The other members of the company staff will be identified in the various memoranda.

BOONTOWN CHEMICAL COMPANY
BOONTOWN, U. S. A.

INTERDEPARTMENTAL MEMORANDUM

DATE: April 15, 1955

SUBJECT: Solvent-recovery-
process Evaluation

TO: C. E. Major, Process Engineer

FROM: J. B. Smith, Chief Engineer

CC: 1) J. J. Baker, Vice President, Operations
2) S. M. Jones, Manager, Development
3) A. D. Norton, Manager, Estimating Section

A request has been received from Dr. S. M. Jones, Manager of Development, for an evaluation of a new solvent-recovery process which has recently been proposed in connection with the production of Protein X. Pilot plant studies of this product are nearing completion and the Research Committee is scheduled to meet on May 20 to review the data to decide whether the project is ready for an appropriation request for a commercial plant. The Development Department has made a brief review and believes that there is enough data available from their work and from the literature to make a sufficiently sound estimate of this new process so that the Research Committee may evaluate it without requiring experimental work. All the correspondence and data from the Development Department are attached.

You are requested to carry out the necessary process-design calculations to permit an economic comparison of the proposed recovery process with the simple distillation process which has been used in the pilot plant, and to submit a preliminary estimate of investment and operation costs for the more economical process. If you find it necessary to make assumptions, you should indicate in your report the nature of any experimental work which will be needed prior to commercial-scale plant design. This report must be in my hands on or before May 15, 1955. Since the report will be used directly by the Research Committee, it is important that it be carefully written in accord-

ance with the outline below, which is standard in this company for this type of presentation.

1. A brief letter of transmittal, addressed to Dr. Brown as Chairman of the Research Committee, identifying the report.
2. A one-page summary stating the general approach used, the results obtained, and the conclusions and recommendations.
3. An index of the balance of the report.
4. A brief introduction describing the problem and the approach which was used in the solution.
5. A presentation of the solution divided into logical sections, without detailed calculations but with procedures, results, flow sheets which present material and energy balances, and any tables or graphs which are needed to present the results.
6. An over-all discussion developing the conclusions and recommendations.
7. An appendix arranged by sections corresponding to those used in Part 5, which presents the detailed calculations plus tables and graphs used in the solution.

By copy of this memorandum, I am requesting that Mr. A. D. Norton, head of our Estimating Section, provide you with the necessary information for estimating the cost of the various types of equipment involved plus the factors we use for this type of estimate.

INTERDEPARTMENTAL MEMORANDUM

DATE: April 14, 1955

SUBJECT: Protein X — Solvent-Recovery Process Evaluation

TO: J. B. Smith, Chief Engineer

FROM: S. M. Jones, Manager of Development

CC: 1) Mr. E. S. Day
2) Dr. H. R. Brown, Vice President, Research and Development
Chairman, Research Committee

Our pilot plant operation for the production of Protein X by alcohol precipitation from aqueous solution is nearing completion, and the Research Committee has scheduled a meeting for May 20 to review the work to date with the idea of recommending commercialization. Mr. E. S. Day, who is in charge of our pilot plant, has just proposed a new extraction method for recovering the spent alcohol which he believes should be considered as an alternative to the distillation procedure we have been using in the pilot plant. There is nei-

ther time nor manpower available to study this new recovery method experimentally, but we believe that there are sufficient data in the literature combined with those which we already have, to permit a thorough evaluation and possibly commercial design without experimental work.

Therefore, we request that you assign to one of your process engineers the problem of comparing the two alternative recovery processes. Since time is relatively short, we shall not have time to incorporate your results in our reports. Therefore, the report from your department should be written for direct use by the Research Committee in making its study and should be addressed to Dr. Brown with copies for us.

Attached is a memorandum prepared for you by Dr. Day which describes the new and old processes and presents all the pertinent data which he has found in connection with his preliminary consideration. If in the course of the work additional information is needed, Mr. Day will be glad to assist in any way he can.

INTERDEPARTMENTAL MEMORANDUM

DATE: April 14, 1955

SUBJECT: Technical Data for Evaluation of Solvent-Recovery Process

TO: J. B. Smith, Chief Engineer (2)

FROM: E. S. Day, Development Department

CC: 1) Dr. S. M. Jones, Manager of Development

As requested by Dr. S. M. Jones, this memorandum presents the pertinent information available to us with regard to the recovery of alcohol in connection with the precipitation of Protein X. Wherever possible we have indicated references in this memorandum, but all items which we feel will have any bearing on the problem have been tabulated here.

PROCESS FOR PROTEIN X

The main process for the production of Protein X, as originally proposed by the Research Department and modified during the course of the pilot plant studies, starts with solubilizing a portion of vegetable protein from one of the products of our present operation. After chemical treatment and clarification the desired fraction is precipitated at room temperature by adding an 80% by weight solution of isopropyl alcohol to give a final concentration of 70% alcohol in the liquid phase. The results of our pilot plant work indicate that tertiary butyl alcohol will also effect a satisfactory precipitation if used in mole concentrations equivalent to the foregoing weight percentages of isopropyl alcohol. These concentration limits must be maintained in order to produce a firm, granular precipitate that can be filtered

readily. Because of the large excess of alcohol required for the precipitation, it is important that it be concentrated and recycled with a minimum of loss.

The treated solution fed to the precipitation process is 2% by weight of Protein X in water. A 96% yield is obtained in the precipitation and subsequent filtering and drying operations. The small amount of 80% alcohol used to wash the precipitate on the filter is added to the 70% alcohol stream from the precipitation and filtration operations and kept within the system. The drying is accomplished by using heated nitrogen cycled through a closed system. The alcohol vapors picked up by the nitrogen are removed by a condenser and returned to the main alcohol cycle. The quantities of alcohol involved in these operations are small and their effect on the main portion of the process can be neglected.

Based on the amount of raw material available from plant operations and the market study prepared by the Technical Economics Group, we are proposing that the commercial plant should be designed to have a capacity of 100 lb./hr. of Protein X. It is planned to operate the commercial plant on a 3 shift/day basis with 330 operating days/yr.

DISTILLATION PROCESS

Our pilot plant process has been based on the use of isopropyl alcohol, which is believed to be the more economical alcohol when recovery by distillation is used. A portion of the 70% alcohol from the precipitation step is concentrated to 86%, then blended with the remaining 70% alcohol to give the 80% solution required for precipitation. This procedure results in a lower steam cost for the distillation procedure than if all the 70% solution were concentrated to 80%. The bottoms concentration of the still was fixed at 0.05 mole % alcohol by our waste-disposal requirements and economic considerations.

The vapor-liquid equilibrium data for the systems isopropanol - water and tertiary butanol - water are given in Table 1. The isopropanol data are those of Schumacher and Hunt (1) and the tertiary butanol data were calculated by use of the van Laar equation and the azeotropic composition as reported by Young and Fortey (2).

EXTRACTION PROCESS

The proposed extraction process would treat the dilute alcohol from the filtration of Protein X by countercurrent contact at room temperature with a strong salt solution in order to concentrate the alcohol to a strength at least as great as that needed for the precipitation. A series of articles by Ginnings et al. (3, 4, 5) presents equilibrium data for the system salt-alcohol-water for both isopropanol and tertiary butanol with a number of salts. Table 2 contains the data for salts that can be considered for use in this process. We are quite sure that the amounts of any of the salts listed in Table 2 which might return to the precipitation step with the recovered alcohol will not affect product quality; therefore, the choice

of both salt and alcohol is entirely dependent upon the economics of the situation. Our Purchasing Department has provided the data on reagent cost presented in Table 3.

We have briefly checked two of the possible systems and find that our experiments agree with the equilibrium data. We have also determined experimentally that the reconcentrated alcohol can be used directly for the precipitation of the product. We have not, however, given any consideration to the problem of handling the diluted salt solution from the extraction step.

From our experience with other solvent-extraction processes, we believe that the data on flooding presented in Figure 1 by the Colburn

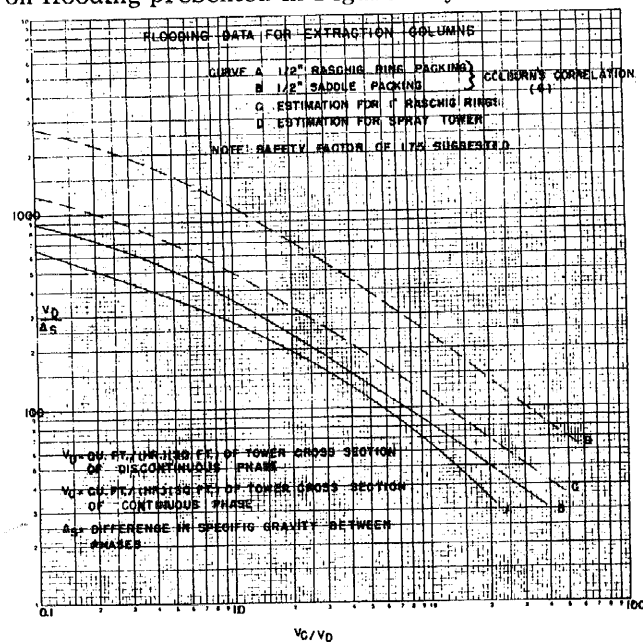


Fig. 1

correlation (6) are applicable. It is also believed that the height of a theoretical stage at maximum allowable fluid velocities will be about 5 ft. for a tower packed with 1-in. Raschig rings and 7 ft. for a spray tower. We have not found any data on the density of mixtures of water and the alcohols but feel that for this situation a straight-line interpolation will be sufficiently accurate.

LITERATURE CITED

- Schumacher and Hunt, *Ind. Eng. Chem.*, 34, 701 (1942).
- Young and Fortey, *J. Chem. Soc. Trans.*, 81, 717 (1902).
- Ginnings et al., *J. Am. Chem. Soc.*, 52, 2282, (1930).
- Ibid.*, 53, 3765 (1931).
- Ibid.*, 55, 875 (1933).
- Perry, J. H., "Chemical Engineers' Handbook," 3 ed., Fig. 52, p. 753, McGraw-Hill Book Company, Inc., New York (1950).

TABLE 1
VAPOR-LIQUID EQUILIBRIA AT 1 ATM.

Isopropanol - water (1)
Azeotrope composition -
87.4 wt. % alcohol (t = 80.3°C.)
A = Wt. % alcohol in liquid
B = Wt. % alcohol in vapor

Tertiary butanol - water
(Data calculated by use of the Van Laar equation
with constants evaluated at the azeotropic com-
position)

Azeotropic composition -
11.76 wt. % water (79.9°C.)
x = mole fraction alcohol in liquid
y = mole fraction alcohol in vapor.

A	B	A	B	A	B	x	y	x	y
91.3	89.7	72.7	81.8	33.0	77.7				
90.0	89.0	68.0	80.9	24.0	77.0				
88.4	87.9	65.0	80.5	17.3	75.2				
86.8	87.0	62.8	80.1	17.5	74.8	0.020	0.201	0.350	0.561
84.7	85.9	61.6	79.9	11.6	71.8	0.050	0.442	0.400	0.565
82.5	84.9	59.3	80.0	10.7	70.8	0.100	0.528	0.450	0.573
80.0	83.9	58.1	79.7	7.20	63.3	0.150	0.548	0.500	0.586
78.0	83.3	52.0	79.2	5.00	52.6	0.200	0.555	0.550	0.601
75.8	82.5	44.0	78.8			0.250	0.557	0.600	0.621
73.5	82.2	41.8	78.3			0.300	0.559	0.644	0.644

TABLE 2
EQUILIBRIUM DATA FOR SALT-WATER-ALCOHOL SYSTEMS

I. Tertiary butanol - salt-water at 30°C. (3)
Points on binodal curves* - composition in weight per cent

Alcohol	Na ₂ CO ₃	Alcohol	NaCl	Alcohol	K ₂ CO ₃	Alcohol	KCl
52.3	0.7	83.8	0.3	57.8	0.7	67.3	1.4
50.0	1.0	57.3	1.5	39.3	1.8	58.4	2.0
41.3	1.3	44.4	2.4	26.9	3.1	50.9	2.6
35.4	1.8	36.4	3.2	18.9	4.8	43.8	3.3
30.8	2.1	27.7	4.3	17.7	5.1	36.6	4.5
26.2	2.8	26.0	4.5	16.1	5.8	33.3	4.8
22.4	3.3	19.4	5.1	12.8	7.2	28.3	5.6
19.7	4.0	14.5	6.7	9.1	9.8	23.9	6.2
18.1	4.6	11.5	9.0	7.5	11.4	20.5	7.4
16.6	5.0	10.0	10.0	5.9	13.3	18.2	8.1
15.3	5.4	8.5	11.2	4.5	15.4	16.5	9.0
11.9	6.0	7.5	12.5	3.5	17.0	12.5	11.4
9.9	7.2	6.7	14.1	0.9	26.7	11.5	12.2
8.6	8.0	5.8	15.6	0.4	34.2	10.5	13.3
6.8	9.7	5.6	15.7			9.4	14.6
6.1	10.4	4.2	18.4			7.9	15.9
5.2	11.2	3.6	19.8			7.0	17.6
4.0	12.9	2.7	22.4			6.4	18.6
3.3	14.1					5.9	20.0
3.1	15.3					4.8	22.6
2.5	16.0					4.5	23.3
1.5	18.9						
0.6	25.2						

Conjugation data

A = Wt. % tertiary butanol in alcohol-rich layer

B = Wt. % salt in salt-rich layer

PP = Plait point

A	B	A	B	A	B	A	B
	Na ₂ CO ₃		NaCl		K ₂ CO ₃		KCl
97	25.2	84	22.4	69	34.2	90	23.3
70	7.7	60	13.9	62	24.4	57	7.1
55	4.5	48	10.7	53	14.8	53	6.7
35	2.0 PP	23	4.6 PP	27	3.2 PP	39	4.2 PP

* The binodal curve, or saturation isotherm, is the boundary between the one-phase region and the two-liquid-phase region of the phase diagram.

TABLE 2 (continued)

II. Isopropanol - salt-water at 25°C. (4)

Points on binodal curve - composition in wt. %

Alcohol	Na ₂ CO ₃	Alcohol	NaCl	Alcohol	K ₂ CO ₃	Alcohol	KCl
63.50	0.10	75.20	1.70	69.60	0.10	53.95	5.12
47.60	0.91	64.35	2.95	42.20	2.80	42.56	7.65
35.80	2.34	49.50	5.20	28.40	6.04	37.50	8.93
19.34	6.54	25.45	10.17	17.25	9.65	36.14	9.17
12.80	9.35	23.90	10.62	10.10	13.86	29.34	11.04
8.96	11.70	15.70	13.70	4.42	19.83	22.87	12.95
5.61	14.50	5.90	21.10	1.93	25.50	17.64	15.17
3.24	17.33			0.65	33.20	13.72	17.48
2.15	19.18			0.23	52.67		
1.60	19.60						

Conjugation data

A = Wt. % alcohol in the alcohol-rich layer

B = Wt. % salt in the salt-rich layer

PP = Plait point

A	B	A	B	A	B	A	B
	Na ₂ CO ₃		NaCl		K ₂ CO ₃		KCl
63.5	19.6	75.2	21.1	69.6	52.7	53.9	17.5
19.9	6.3 PP	49.5	5.2 PP	27.8	6.2 PP	37.5	8.9 PP
1.6	0.10	5.9	1.7	0.23	0.10	13.8	5.1

Note: The first pair of conjugated compositions represent the termination of the two-liquid-phase regions.

TABLE 3

COST OF CHEMICALS DELIVERED
TO BOONTOWN AREA

INTERDEPARTMENTAL MEMORANDUM

DATE: April 17, 1955

SUBJECT: Equipment cost data

Chemical	Cost	Freight \$/ton
Tertiary butanol		
Tanks*, \$/lb.	0.12	12
L.c.l.†, \$/lb.	0.145	15
Isopropanol		
99% Tanks, \$/gal.	0.39	Costs
L.c.l., \$/gal.	0.58	are on
95% Tanks, \$/gal.	0.37	delivered
L.c.l., \$/gal.	0.52	basis
91% Tanks, \$/gal.	0.34	
L.c.l., \$/gal.	0.50	
Sodium carbonate		
58% Na ₂ O		
Bulk, c.l.‡, \$/100 lb.	1.35	14
Bags, c.l., \$/100 lb.	1.65	17
Sodium chloride		
Rock, bags		
C.l. and l.c.l., \$/100 lb.	1.10	6
Potassium carbonate		
Calcined, 99% bags		
C.l. and l.c.l., \$/100 lb.	9.00	8
Liquid, 47% drums		
C.l. and l.c.l., \$/100 lb.	4.60	8
Liquid, 47% tanks, \$/100 lb.	3.50	6
Potassium chloride		
Granular drums, \$/lb.	0.18	8

* Tanks = tankcar lots

† L.c.l. = less than carloads

‡ C.l. = car lots or carloads

TO: C. E. Major, Process Engineer

FROM: A. D. Norton, Estimating Section

CC: 1) J. B. Smith, Chief Engineer

As requested in Mr. Smith's memorandum of April 15, we have prepared some equipment-cost information that may be needed for evaluating the solvent-recovery systems for the Protein X process. It is believed that sufficient data are given in Table 4 to estimate the cost of all the major items of equipment that may be needed.

Utilities and Fixed Charges

Utility and labor costs at the rates now in effect at our Boontown plant, where it is proposed to locate the Protein X operation, are listed in Table 5.

Current company practice is to use straight-line amortization of equipment at the rate of 10% of the installed cost per year. Local taxes and insurance can be estimated at 2% of the installed cost per year. Overhead charges are currently 100% of the direct operating labor cost. It is believed that the total maintenance charges on the type of equipment involved in this process will be 4% of the installed equipment cost per year.

Heat Transfer Factors

In an estimation of the cost of heat transfer surface, the following over-all coefficients can be used.

	U, (sq. ft. / °F.) B. t. u. / (hr.)
Liquid to liquid	200
Liquid to boiling liquid	75
Liquid to condensing vapor	500
Condensing vapor to boiling liquid	600
Minimum terminal temperature difference	15°F.

The pressure loss through heat exchanges can be neglected.

TABLE 1

EQUIPMENT COSTS

The costs given are for the equipment installed in place and include delivery and erection costs. Piping and insulation costs are estimated to be 50% of the total installed equipment cost. Electrical wiring and instrumentation can each be taken at 10% of total installed equipment. Building space and auxiliary facilities are available so that no factor need be allowed for these items.

Distillation towers - bubble cap

Diameter, in.	\$/plate
8	120
10	140
25	300
50	630
100	1,570
350	15,700

Extraction columns (excluding packing)

	\$/ft. height
6	65
10	90
25	240
50	380
100	820
200	2,360

Cost of 1-in. Raschig rings

Porcelain	- \$6.95/cu.ft.
Stoneware	- \$5.65/cu.ft.
Carbon	- \$10.10/cu.ft.

Heat exchangers

Shell-and-tube heat exchangers, heat transfer surface

Sq. ft.	\$
25	860
50	1,330
100	2,040
500	5,000
1,000	7,500
5,000	20,400
10,000	28,300

TABLE 1 (Continued)

Shell-and-tube calandrias or reboilers, heat transfer surface

Sq. ft.	\$
25	1,330
50	2,040
100	2,980
500	7,850
1,000	12,300

Evaporator

Basket type, total heating surface

Sq. ft.	\$
100	3,630
250	14,100
500	20,400
1,000	29,800
5,000	70,700

Long tube vertical, total heating surface

Sq. ft.	\$
100	2,980
250	5,500
500	9,440
1,000	15,700
5,000	50,300

Pumps

Gal./min.	Head, ft.	\$
25	50	300
25	100	795
50	50	450
50	100	795
100	50	490
100	100	975
200	50	540
200	100	975
500	50	1,160
500	100	1,160

Motors

Hp.	\$
1/2	70
3/4	95
1	110
2	140
5	220
10	350

Storage tanks

Volume, gal.	\$
100	380
200	570
500	910
1,000	1,310
2,000	2,040
5,000	3,150
10,000	4,400

TABLE 1 (Continued)

Agitated tanks	
Volume, gal.	\$
100	1,410
200	2,040
500	2,830
1,000	4,100
2,000	5,500
5,000	8,200
10,000	11,000
Thickeners - single compartment	
Diameter, ft.	\$
15	9,100
20	13,000
25	14,000
30	18,000
Rotary driers - peripheral area	
Sq. ft.	\$
100	4,700
200	8,300
500	20,400
1,000	34,600

TABLE 2

UTILITY AND LABOR COSTS

Electrical energy	\$0.012/kw.-hr.
Steam, 300 lb./sq. in. sat.	\$0.75/1,000 lb.
Cooling water*	\$0.03/1,000 gal.
Process water	\$0.10/1,000 gal.
Labor	
Operators	\$2.00/man-hr.
Helpers	\$1.75/man-hr.

* Cooling water from well available at 70°F. and must be sent to existing cooling tower at a temperature no higher than 115°F. for cooling and subsequent use in other portions of the process.

SOLUTION

Arthur L. Baron, Cooper Union

SUMMARY

A report has been prepared on an investigation of an economic comparison of two methods which have been proposed for recovering the alcohol used in the production of Protein X. Based on the observation that steam costs would probably be large relative to equipment costs, it was tentatively concluded that extraction would be the more economical process, since it does not depend primarily upon heat for separation, as does distilla-

tion. A complete process design was made for extraction, and the final cost estimates were \$56,960 for installation and \$441.55 daily operating costs. Brief consideration of the distillation method indicated a daily operating cost of about \$575. It was therefore recommended that extraction be considered for the commercial plant.

Suggestions for corrections and refinements which can be applied to the process design contained in this report for the purpose of a commercial design were made with reference to the dimensions of the distillation tower, the solvent composition, the storage tank requirements, and the variation of the height of a theoretical stage in a packed tower with relative tower dimensions.

INTRODUCTION

In order to solve this problem, three basic questions had to be answered:

1. Which process, extraction or distillation, is the more economical?
2. What are the optimum operating conditions for the more economical process?
3. How much will it cost to install and operate the recommended process?

The approach used in the solution of the problem was based primarily on two observations which were made concerning the stoichiometry and equilibria involved. First of all, owing to the small concentration of protein in solution, it was obvious that a large amount of material had to be processed. Second, the liquid-vapor equilibrium data for distillation and the ternary equilibrium data for extraction indicated that in terms of degree of separation the job was not a difficult one. From these observations it was tentatively concluded that the daily operating costs, consisting largely of steam costs, would be large in comparison with the fixed charges on the equipment. This conclusion favored the possibility of extraction being the better process since it does not depend primarily upon heat for the separation. Therefore, the extraction process was considered first.

Out of the eight possible ternary systems to be used, a selection was made on the basis of certain features of the relative shapes of the equilibrium diagrams. Similarly, the fixing of operating conditions for the extraction column was based indirectly on cost considerations and mainly on practicalities.

There were two problems associated with the salt solution from the extraction. The solution had to be brought back to the proper concentration, and it was necessary to reclaim as much as possible of the alcohol in the extract. Two steps were used. Evaporation was used to re-concentrate the solvent, and the vapors from this operation were sent to a distillation column where most of the alcohol was salvaged. Since the feed to the distillation step was already in