A Continuous Process and Cost Estimate for the Production of Isopropenyl Stearate

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ABSTRACT

A continuous process for the commercial production of isopropenyl stearate (IPS) from triple pressed stearic acid and a stabilized form of propyne has been developed. Cost estimates, including capital costs, operating costs, and profitability, for commercial scale plant production which show the process to be economically feasible are presented. This potentially profitable process offers the advantages of reliable raw material sources, minimal external thermal requirements, and usable process waste streams. For a plant producing 5 million pounds of IPS per year, the selling price range is 80 to 107 cents/lb IPS, corresponding to a raw material cost range of 27 to 54 cents/lb of IPS. For a 20 million pound per year plant, the selling price range is 58 to 85 cents/lb IPS. The selling prices include a 20% annual return on fixed capital investment. Fixed capital requirement ranges from 2.7 to 10.9 million dollars (3rd quarter, 1975) for plants ranging in size from 5 to 50 million pounds of IPS per year, respectively.

INTRODUCTION

Isopropenyl stearate (IPS) is a versatile acylating reagent (1). It has been demonstrated to react readily with OH or NH compounds (2,3) under mild reaction conditions with only acetone as by-product.

Rothman and Serota (4) disclosed a method of producing IPS by the direct addition of propyne to stearic acid (HST), with zinc stearate ($ZnST_2$) as catalyst. Subsequently, an effort was begun to develop a commercially feasible process for the production of IPS. This effort was intended to develop equipment, an integrated process line, product specifications, data for scale-up, a process flow sheet, and data required to calculate a cost estimate on the process and a selling price for the product.

A previous paper described the research, development, and scale-up of a continuous pilot plant process (5). This process was based upon the addition reaction of equation E-1.

$$\underset{\text{HST}}{R-\overset{O}{\overset{\|}{C}}-OH} + \underset{\text{propyne}}{HC\equiv C-CH_3} \xrightarrow{ZN^{++}} \underset{\text{IPS}}{R-\overset{O}{\overset{\|}{C}}-O-\overset{CH_2}{\overset{\|}{C}}-CH_3} \qquad \text{E-1}$$

Triple pressed stearic acid was used as the source of stearic acid. However, for safety and economic reasons, this process used a stabilized welding gas, MAPP ®, for the source of propyne. The propyne concentration of MAPP is 30-35%. Yields of 92% IPS, based upon HST, were obtained when the reactors were operated at 550 psi and 415 F with 10 min residence time and 52% catalyst by weight.

This paper presents a continuous commercial process and its cost estimate.

RESULTS

Continuous Process

The continuous process is illustrated in Figure 1, a schematic flow sheet based upon the pilot plant process reported by Craig et al. (5). The process includes feed, reaction, quench, product recovery, solvent recovery, and catalyst recovery and recycle.

HST is melted in jacketed, agitated kettles, No. 1, (Fig. 1) and pumped to heat exchanger, No. 3. MAPP liquid is heated to 400 F in heat exchanger, No. 6, and then mixed with recycle catalyst in a mixing chamber, No. 25. The mixed catalyst and MAPP is heated to 400 F before being mixed with HST at 400 F.

The feed streams are pumped at a rate to produce a reaction mixture consisting of 52% $ZnSt_2$ of total weight of fatty material and a molar ratio of propyne to HST of 0.9. The dwell time of the reaction mixture is 8.5 min in the jacketed, continuous stirred tank reactor (CSTR), No. 10. The reactor is maintained at 415 ± 5 F and 550 psig. Since a CSTR will not produce equilibrium conversion, a plug flow finishing reactor, No. 11, is included to complete the reaction. The volume and flow characteristics of these two reactors should be such as to give approximately the same residence time distribution found in the continuous pilot plant process or greater plug flow residence time (2.9 min plug flow). The residence time in the plug flow finishing reactor must be a minimum of 36 sec. Any decrease in residence time in the finishing reactor will reduce the yield of IPS (5).

The reaction mixture is flashed in flash tank, No. 14, to about 25 psig. Flashing separates the unreacted MAPP gas and acetone from the IPS product and catalyst. The liquid is cooled and solidified on the rolls of the flaker, No. 16, at about 60 F and is transferred via a conveyor to a counter-

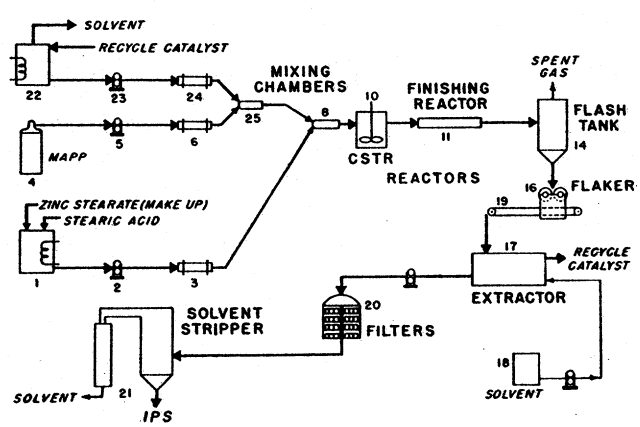

FIG. 1. Process flow sheet.

TABLE I

Fixed Capital Cost – Isopropenyl Stearate

	5×10^6 lb IPS per year	20×10^6 lb IPS per year	50×10^6 lb IPS per year
1. Land, site preparation, buildings, and structures	$ 149,200	$ 307,900	$ 532,400
2. Utilities	99,400	224,800	386,200
3. Purchased equipment and installation	701,900	2,012,300	3,487,600
4. Direct materials and labor	742,700	1,467,900	2,541,400
5. Engineering, construction, and fee	824,600	1,785,700	3,091,600
6. Contingency	220,000	497,200	860,800
7. Total fixed capital	$2,737,800	$6,295,800	$10,900,000

TABLE II

Fixed Capital Cost of Plant Sections

	5×10^6 lb/yr		20×10^6 lb/yr	
	$	%	$	%
1. Raw material	658,900[a]	24.07	2,033,100	32.29
2. Reaction	363,200	13.27	490,200	7.79
3. Solvent recovery	456,400	16.67	917,800	14.58
4. Solids separation	326,600	11.93	888,400	14.11
5. Product separation	110,700	4.04	238,900	3.79
6. Product storage	335,900	12,27	608,200	9.66
7. Refrigeration unit	124,300	4.54	278,000	4.42
8. Heating unit	110,500	4.04	165,000	2.62
9. Utilities and facilities	99,400	3.63	224,800	3.57
10. Land	7,000	0.26	15,400	0.24
11. Catalyst	4,000	0.15	16,600	0.26
12. Interest	141,100	5.15	420,000	6.67
13. Total	2,738,000	100.0	6,296,400	100.0

[a]More than two significant figures are used for purposes of checking calculations and internal consistency.

current extractor, No. 17.

A single-pass extractor was used in the pilot plant process; but it was demonstrated that multi-pass extraction is better. The undissolved solids (recycle catalyst) are fed to jacketed tanks, No. 22, for recovery of residual solvent and recycling of the $ZnSt_2$ catalyst. The catalyst containing 20% IPS is melted and pumped through a heat exchanger, No. 24, to a mixing chamber, No. 25, where it is mixed with MAPP gas feed. The liquid IPS solution is clarified in pressure filters, No. 20, for removal of small amounts of solids which are also sent to jacketed tanks, No. 22. The solvent is removed from the IPS stream in solvent stripper, No. 21, and pumped to the solvent recycle tank, No. 18. The product is a technical grade IPS with a composition of 90-95% IPS with the difference composed of HST and stearic anhydride (St_2O).

Cost Estimate

Fixed capital: Plant costs: The fixed capital costs for three plant sizes, 5, 20, and 50 million lb per year of IPS, are given in Table I. The fixed capital costs were calculated by use of a commercially available computer program for the 5 and 20 million lb plant sizes. The program contains a data bank of equipment and installation costs to compute complete process plant costs. The fixed capital costs are based on the third quarter of 1975. The 50 million lb plant cost was scaled from the costs of the 5 and 20 million lb plants by the relationship:

$$\text{Cost}_2 = \text{Cost}_1\,(\text{Capacity}_2/\text{Capacity}_1)^{0.60}$$

The 0.60 exponent was calculated from the costs obtained for the 5 and 20 million lb plants. Fixed capital cost may also be estimated by the following equation: $\text{Cost}_1\,\$ = 262\,(W_1\ \text{lb/yr})^{0.60}$ where W is lb/yr plant capacity.

Fixed capital cost is broken down by plant sections in Table II. Product recovery, comprising sections 3, 4, 5, and 7, accounts for about 37% of the fixed capital cost. The raw material and product storage sections account for a total of about 36-42% of the cost, depending on plant size. The reaction sections 2 and 8 account for about 17 and 10% of the cost, the lower figure being for the 20 million lb plant. Equipment costs for the 5 and 20 million lb per year plants are available.

Table III lists the equipment by process section. A list of individual equipment specifications and the material balance for the 5 million lb per year plant are available. The plant equipment is sized for operation 24 hr/day, 350 days/yr. The makeup rate for solvent is 10.8 lb/hr. Solvent loss is assumed equal to the sum of 1% of the weight flow plus 3% of the equipment volume per hr leakage (vapor phase). No unusable waste streams are produced by the process. The gas stream, consisting of propane, propylene, butane, propadiene, acetone, and propyne, is burned to supply heat required for the process. The heat of combustion from the gas stream is ca. 85×10^6 btu/day. The heat required for process heating is ca. 54×10^6 btu/day. Assuming an efficiency of about 0.67, 57×10^6 btu/day is available for the process. Therefore, little or no heat is available for the nonprocess heating, and no credit was taken for it.

Selling price: The selling price range versus raw material cost and versus HST cost for profitable plants producing 5 to 50 million lb per year of isopropenyl stearate is shown in Figure 2. The selling prices assume a 20% net annual return on fixed capital investment. The actual product is nearly 92% by weight isopropenyl stearate. Therefore, on a product basis the selling price shown on Figure 2 should be multiplied by 0.92.

DISCUSSION

The flow sheet for a commercial process is a scaled-up version of the pilot plant process except for recovery and recyle. Although single pass leaching was used in the pilot plant scale process, obviously it would not be used in a commercial process since it is far too inefficient. Craig et al.

TABLE III

Equipment Cost – 5 x 10^6 lb/yr Plant

Item	Item no.	Cost ($)	Quantity	Total cost ($)
A. Raw material storage				
1. Solvent storage pump	CP-1	800	1	800
2. Zinc stearate storage tank	VT-3	7,200	1	7,200
3. Stearic acid storage tank	VT-2	12,700	3	38,100
4. MAPP storage tank	HT-2	66,400	2	132,800
5. Solvent storage tank	HT-1	4,300	1	4,300
B. Reaction				
6. MAPP liquid pump	P-4	2,000	1	2,000
7. Stearic acid feed pump	P-2A	2,000	1	2,000
8. Reactor	APT-10	22,600	1	22,600
9. Feed tank	APT-1	35,450	2	70,900
10. Recycle catalyst heater	HE-8	1,000	1	1,000
11. Plug flow reactor	HE-11	1,100	1	1,100
12. MAPP recycle heater	HE-9	1,200	1	1,200
13. Stearic acid feed heater	HE-3	700	1	700
14. MAPP heater	HE-5	1,100	1	1,100
C. Solvent recovery				
15. Catalyst solvent conveyor	CO-19	4,500	1	4,500
16. Solvent condensate pump	CP-7	640	2	1,300
17. Solvent condensate pump	CP-22	800	1	800
18. Solvent pump	CP-17	800	1	800
19. Recycle pump	P-2B	1,990	1	2,000
20. Recycle tank	APT-7	20,540	2	41,100
21. Solvent storage and recycle tank	VT-17	6,350	1	6,400
22. Solvent recovery condenser	HE-7	6,170	2	12,300
23. Solvent condenser	HE-22	35,400	1	35,400
D. Solids separation				
24. IPS - catalyst conveyor	CO-14	4,500	1	4,500
25. Flaker feed pump	P-12	2,000	1	2,000
26. Extractor	CR-16	11,600	1	11,600
27. Flaker	FL-13	22,300	1	22,300
28. Dry feeder	FE-15	2,900	1	2,900
29. IPS solution pump	CP-20	800	1	800
30. Surge tank	VT-21	9,600	1	9,600
31. Flash tank	VT-12	6,700	1	6,700
32. Solvent cooler	HE-18	1,200	1	1,200
33. IPS solution filter	F-21	12,000	2	24,000
E. Product separation				
34. Evaporator feed pump	CP-21	800	1	800
35. Solvent evaporator	E-22	25,300	1	25,300
F. Product storage				
36. IPS storage pump	CP-3A	900	1	900
37. IPS storage tank	HT-3	16,500	2	33,000
38. IPS heater	HE-31	1,570	2	3,100
G. Refrigeration unit	RU-1	31,300	1	31,300
H. Heating unit	HU-1	33,800	1	33,800

(5) showed that the extraction could be made twice as efficient by using two passes to cut the solvent requirements in half. Although other recovery methods are technically feasible, multipass countercurrent leaching, No. 17, (Fig. 1) is included in the recommended process flow sheet.

The solid flakes from the flaker resemble moist soap flakes. They are free flowing in the dry feeder or conveyor but clump readily if compressed. It was found that continuous operation is crucial to successful leaching. If the flakes are not processed continuously, they change both physically and chemically. The chemical change is associated predominantly with the very slow conversion of by-product stearic anhydride to stearic acid. The structure of the solid changes adversely, affecting the leaching. The solids clump, and the filtrate, after clarification, is still cloudy.

For an economical process the catalyst must be recycled. The recycle catalyst contains about 20% IPS. The recycle cannot be returned to the fresh HST feed tanks, No. 1, because the HST would react with the IPS in the recycle, as shown in equation E-2, producing stearic anhydride.

$$\underset{\text{HST}}{R-\overset{O}{\overset{\|}{C}}-OH} + \underset{\text{IPS}}{H_3C-\overset{CH_2}{\overset{\|}{C}}-O-\overset{O}{\overset{\|}{C}}-R} \xrightarrow{Zn^{++}} \underset{\text{stearic anhydride}}{R-\overset{O}{\overset{\|}{C}}-O-\overset{O}{\overset{\|}{C}}-R} + \underset{\text{acetone}}{H_3C-\overset{CH_3}{\overset{|}{C}}=O} \quad \text{E-2}$$

Catalyst was not recycled in the pilot plant process. One potential method to minimize or eliminate reaction E-2 is to minimize contact or reaction time between HST and IPS. In the flow sheet of Figure 1, a third feed line for recycle catalyst is included. The catalyst is recylced to separate recycle catalyst feed kettles, No. 22. The recycle catalyst is melted and mixed with MAPP gas feed in mixing chamber, No. 25. Propyne and $ZnSt_2$ are present in quantities needed to form IPS, but the HST necessary for stearic anhydride formation is present only in trace amounts. This stream is then mixed with HST feed in the mixing chamber, No. 8. At no time are HST and IPS in contact unless MAPP gas and catalyst are also present under reaction conditions which should drive the reaction to IPS instead of stearic anhydride, since the reaction to form stearic anhydride is much slower than the reaction to form IPS.

The spent MAPP gas was not analyzed but should contain very little propyne. The nominal composition of the spent gas is assumed to be ½ allene, ½ stabilizing gases, and a trace of propyne and acetone. It is used as a fuel gas in the process, but there is an alternative. The equilibrium conversion to IPS increases with the propyne content of the MAPP gas. The allene in MAPP gas could be isomerized to propyne before being fed to the process. This would utilize twice as much MAPP as reactant instead of fuel gas and may give higher conversions. The safety of such a gas mixture has not been checked under reaction conditions (415 F and 550 psig).

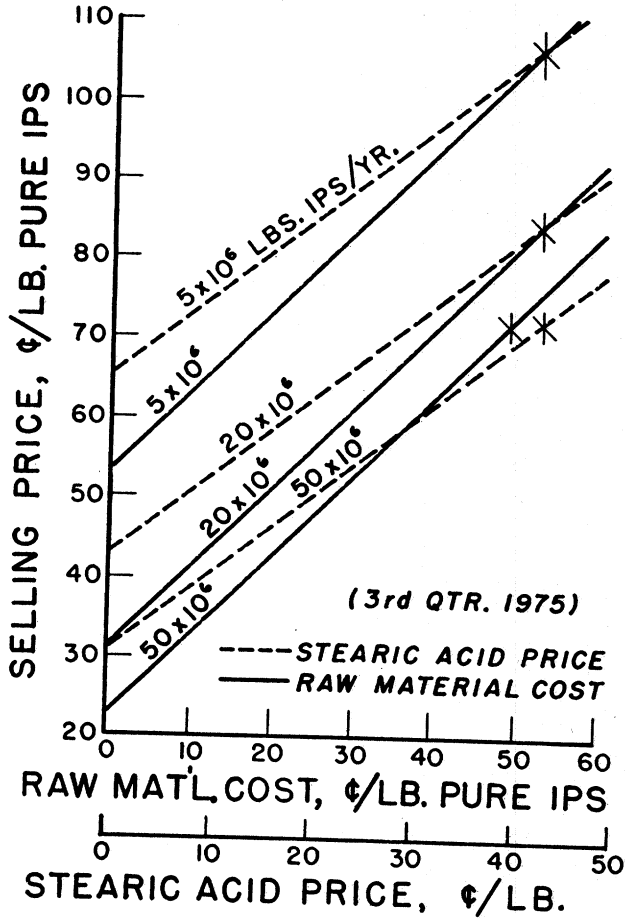

FIG. 2. Selling price versus raw material cost and stearic acid price.

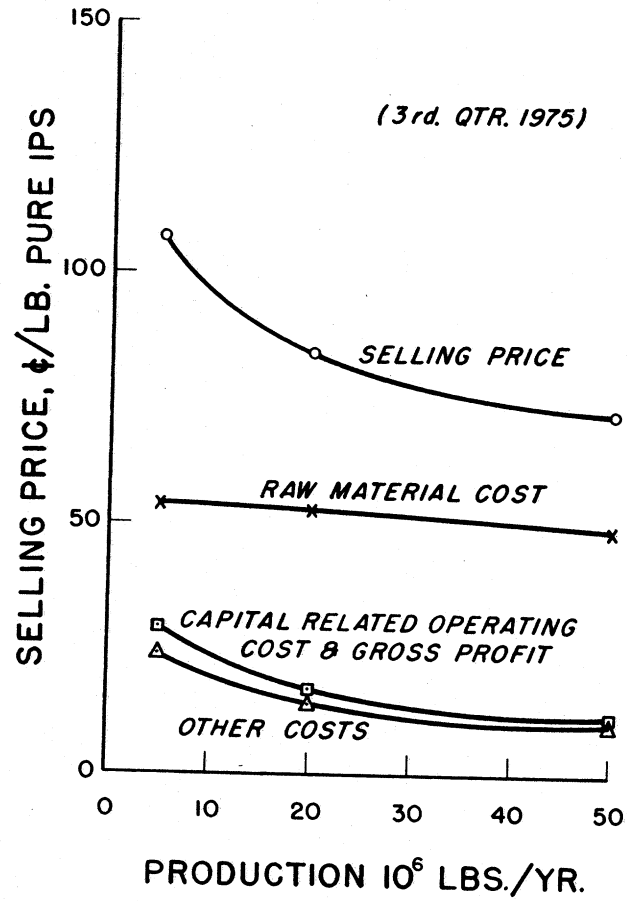

FIG. 3. Selling price and major selling price components versus production.

TABLE IV

Effect of Cost Components on Selling Price

Component	Variation in component (%)	Variation in selling price (%)	
		5×10^6 lb/yr	50×10^6 lb/yr
Fixed capital	±15	±4	±2.5
Raw material	+9, -32	+5, -16	+6, -24
Other costs	±10	±2	±2
Total (selling price)	—	+11, -22	+10.5, -28.5

TABLE V

Financial and Economic Summary

IPS production, lb/yr	5×10^6	20×10^6	50×10^6
1. Fixed costs, $/year	985,700	2,510,400	4,749,500
2. Variable costs, $/yr	3,286,600	11,820,400	27,004,500
3. Total costs (1 + 2), $/yr	4,272,300	14,330,800	31,754,000
4. Federal income tax, $/yr	505,000	1,162,000	2,012,000
5. Net profit, $/yr	548,000	1,259,000	2,180,000
6. Total sales (3 + 4 +5), $/yr	5,325,000	16,752,000	36,000,000
7. Fixed capital, $	2,738,000	6,296,000	10,900,000
8. Working capital, $	620,000	2,230,000	5,000,000
9. Total capital (7 + 8), $	3,358,000	8,526,000	15,900,000
10. Depreciation, $/yr	235,000	544,000	942,000
11. Cash flow (5 + 10), $/yr	782,000	1,803,000	3,122,000
12. Capital recovery rate (11÷7) (per year)	0.2856	0.2864	0.2864
13. Return on investment (disctd. cash flow uniform earnings), %	26.4	26.5	26.5
14. Project life, years	11	11	11
15. Annual earnings on fixed capital, %	20	20	20
16. Annual earnings on total capital, %	16.30	14.76	13.71
17. Turnover ratio (6÷7)	1.94	2.66	3.30
18. Net profit on sales (5÷6), %	10.3	7.51	6.06
19. Selling price, cent/lb IPS	107	84	72
20. Break-even point, pounds IPS	2.39×10^6	10.08×10^6	26.4×10^6

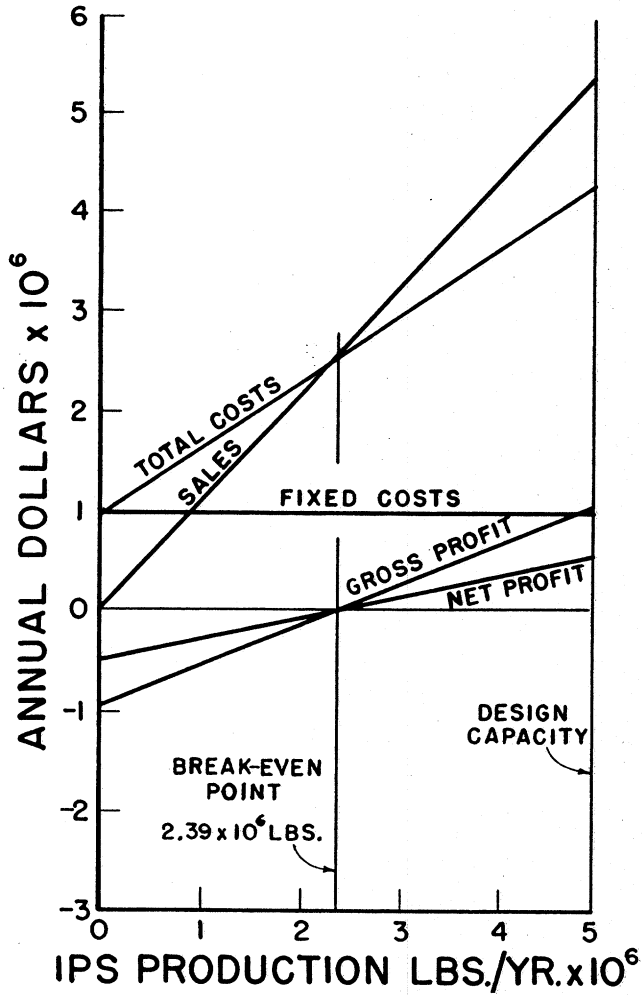

FIG. 4. Economic production chart.

Details of the calculation of selling price are available. The basic quantities required for the estimation of selling price are as follows:

a. Raw materials -		lb/lb IPS	
MAPP		0.4231	
Stearic acid		0.9698	
Zinc stearate		0.0130	
Skellysolve-B		0.0179	
b. Production rate, x 10^6lb/yr	5	20	50
Men per shift -	4	5	8
Utilities -			
Electricity			
kw-hr/day	4,390	16,985	41,100
cents/kw-hr	4.0	4.0	2.0

c. Maintenance - 4% of fixed capital per year

The IPS selling price and costs versus plant size are given in Figure 3. Raw material is the major cost item, as shown in Figure 3, and is also subject to the greatest cost variation for the following reasons. MAPP gas is purchased at quantity discounts. The stearic acid price is probably very slightly dependent on quantity. However, it is subject to significant market price variations on the order of ±25%. This cost estimate was made near a peak price for stearic acid (3rd quarter 1975). Figure 2 shows the effect of stearic acid price (in cents/lb of stearic acid) on the selling price of IPS. The abscissa corresponding to the "X" on each line is the raw material cost used in calculating selling price for the 3rd quarter of 1975.

The overall maximum range in the selling price when all components are at their extreme values is roughly +10% to -25% of the base price. The effect on the selling price of variation in the major cost components is shown in Table IV for production rates of 5 and 50 million lb per year of IPS.

The selling price range indicates that isopropenyl stearate is in the low to medium volume chemical class. The largest volume uses are probably in the plasticizer and rubber processing chemicals classes. A process plant to make IPS will probably sell to more than one customer, and the IPS will probably be used in more than one market area (type of application).

Financial and Economic Summary

The cost and economic factors for the 5, 20, and 50 million lb per year plants are summarized in Table V. The economic factors indicate that the process is economically feasible. The economic production chart for the 5 million lb per year plant is shown in Figure 4. This chart may be used to see the effect of a change in costs or sales on the break-even point and profit. For instance, total yearly sales are shown as \$5,350,000 (\$1.07 x 5 x 10^6 lb). Total costs at this production are \$4,280,000. Profit is \$5,350,000 - \$4,280,000 = \$1,070,000 before federal income tax. The break-even point, at the intersection of the total costs and sales lines, is 2.39 x 10^6 lb of IPS.

Economic production charts for the 20 and 50 million lb per year plants are available. These charts show break-even points for these plants occurring at ca. 50% of design capacity. The low break-even points for these plants allow the plants less risk on market entry than plants with higher break-even points because of (a) increased allowable deviations for equipment failure and other emergencies, and (b) lower profitable selling prices, making it possible to meet competition.

A process development package and a cost estimate package are available from the authors. The cost estimate package includes equipment cost for the 20 x 10^6 lb per year plant, material balance, equipment specifications, equipment flow diagram, selling price calculation, break-even charts for 20 and 50 x 10^6 lb per year plants, and unit cost chart. The process development package includes a pilot plant process flow sheet with a detailed equipment list and operating procedure.

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