American Institute of Chemical Engineers

STUDENT CONTEST PROBLEM

1966

345 East 47 Street • New York, New York 10017

CONTEST PROBLEM

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AMERICAN INSTITUTE OF CHEMICAL ENGINEERS STUDENT CHAPTERS

Open Only to Undergraduates or Those
Without a Degree in Chemical Engineering

DEADLINE FOR MAILING

Solution must be postmarked not later than midnight, June 15, 1966

RULES OF THE CONTEST

Solutions will be graded on (a) substantial correctness of results and soundness of conclusions, (b) ingenuity and logic employed, (c) accuracy of computations, and (d) form of presentation. Accuracy of computations is intended to mean primarily freedom from mistakes; extreme precision is not necessary.

It is to be assumed that the statement of the problem contains all the pertinent data except for those readily available in handbooks and similar reference works. The use of textbooks, handbooks, journal articles, and lecture notes is permitted. In cases where there is disagreement in the data reported in the literature, the values given in the statement of the problem have been chosen as being most nearly applicable.

The problem is not to be discussed with any person whatever until June 15, 1966. This is particularly important in cases where neighboring institutions may not begin the problem until after its completion by another chapter. Submission of a solution for the competition implies adherence to the foregoing condition.

A period of not more than 30 consecutive days is allowed for completion of the solution. This period may be selected at the discretion of the individual counselor, but in order to be eligible for an award a solution must be postmarked not later than midnight, June 15, 1966.

The finished report should be submitted to the chapter counselor within the thirty-day period. There should not be any variation in form or content between the solution submitted to the chapter counselor and that sent to the A.I.Ch.E. office. The report should be neat and legible, but no part need be typewritten.

The solution should be accompanied by a letter of transmittal giving only the contestant's name, school address, home address, and student chapter, lightly attached to the report. This letter will be retained for identification by the Secretary of the Institute. The solution itself must bear no reference to the student's name or institution by which it might be identified. In this connection, graph paper bearing the name of the institution should be avoided.

Each counselor should select the best solution or solutions, not to exceed two in number, from his chapter and send these by registered mail to

Mr. F. J. Van Antwerpen, Secretary
American Institute of Chemical Engineers
345 East 47 Street
New York, New York 10017

TO THE CONTESTANT

In the development of the 1966 Student Contest Problem an attempt has been made to emphasize the importance of time. The contestant, like his industrial counterpart, has a limited time to spend on a study of process alternatives. Process engineers must decide early where the decisive issues lie and strive toward reaching the correct decisions on the issues. Time represents money to the chemical company; the quicker the feasibility and economics of a process can be determined, the more profit a company can make.

STATEMENT OF THE PROBLEM

The subject of the 1966 problem is the preliminary economic process design of a plant to manufacture a monomeric chemical. The manufacturing company does not know very much about the process and needs a preliminary evaluation. The Sales Department approached the Research Laboratories about developing a process to produce the monomer for captive use. Continually increasing use of the material in a specialty coating is expected. The Research Department has attempted several exploratory experiments with encouraging results. These results form the basis of the preliminary process design and economic evaluation. The following memoranda represent the total correspondence concerning the monomer. The student is to assume the role of Mr. Walker in solving the problem, which is the preparation of a preliminary process-design and manufacturing cost estimate for t-butyl methacrylate.

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cc: V. R. Field
P. Rinceton
I. Tower

April 1, 1966

MEMORANDUM

To: V. P. Walker, Process Engineer
From: J. H. Sign, Chief Process Engineer
Subject: t-Butyl Methacrylate: Manufacturing Cost Estimate

The Coatings Sales Department has requested the preparation of a manufacturing cost estimate for production of t-butyl methacrylate. Current usage of this raw material in Coating 262-X is 200,000 lb./mo. with requirements expected to increase to 1,000,000 lb./mo. by the second quarter of 1969. Because of this predicted rapid growth in the use of the chemical, consideration is to be given to captive manufacture. Please prepare preliminary process designs and comparative manufacturing cost estimates for production of t-butyl methacrylate by both batch and continuous processes.

The results of the initial laboratory experiments by Dr. Tower's group are attached. Although Dr. Tower's experiments have found 60% conversion to be optimum for the batch reaction, this will not necessarily be the case in a continuous process.

By copy of this memorandum, Mr. Rinceton is requested to forward to you a summary of pertinent design data and cost-estimation factors. He should also include the specifications for the available 6,000-gal. batch reactor and associated distillation column which would be used for batch production. All the equipment for a continuous process and auxiliary equipment for a batch process must be purchased.

Will you compare the costs of the two processes as a function of the product output per month. In particular, what is the maximum capacity of a batch process employing the available equipment and at what level of output does it become economically advantageous for the company to change processes? Any recommendations you may have for reducing costs will be welcome. Because of the urgency of this cost estimate and the minimal data available from the laboratory, all necessary design information may not be included. Therefore, state clearly in your final report all important design assumptions employed in your calculations.

Since it will require some time to put a process on stream, your final report is needed as soon as possible. Therefore you may not be able to optimize both designs; however, we hope that you can outline in sufficient detail how such optimizations (particularly of the continuous process) would be carried out for our final design.

For your further information, background memoranda are attached.

cc: J. H. Sign,
Chief Process Engineer

March 1, 1966

MEMORANDUM

To: Dr. V. R. Field, Director of Research
From: R. Vail, Sales Manager
Subject: t-Butyl Methacrylate, Monomer for Specialty Coating

Following is a summary of our discussions of t-butyl methacrylate held in my office on February 28.

The sales volume for Coating 262-X increased dramatically last year to an equivalent t-butyl methacrylate usage of 200,000 lb./mo. Market analysis leads us to believe monomer requirements will increase to more than 350,000 lb./mo. by the end of this year and to 1,000,000 lb./mo. by 1969.

You speculated that the monomer could be manufactured for captive use at a cost significantly below the 50 cents/lb. we are now paying and that a process could be developed which would not infringe on existing patents.

I encourage you to initiate process studies aimed at captive manufacture of the monomer and to report the results to Mr. Sign for an early evaluation by his group. I believe it important to determine manufacturing feasibility and to assess the economics of your processing schemes at the earliest moment.

cc: R. Vail

March 15, 1966

MEMORANDUM

To: J. H. Sign, Chief Process Engineer
From: V. R. Field, Director of Research
Subject: t-Butyl Methacrylate: Laboratory Process

At the request of Mr. Vail, several laboratory experiments were made to synthesize t-butyl methacrylate. In the attached memorandum Dr. I. Tower summarizes the experimental results, which we find encouraging. The laboratory process described therein is a batch process. A corresponding continuous process has not been explored, but we think that such a mode of operation is feasible.

In the final design and cost report that your department will issue, we would seek your assistance in selecting those areas in each process where significant cost reduction might be achieved. In particular, we want to know at what level of production it would be economically advantageous for the company to change from one process to the other. In light of the growing demand for Coating 262-X, the results of your comparison will be important in determining the research path we should pursue.

March 11, 1966

MEMORANDUM

To: Dr. V. R. Field, Director of Research
From: I. Tower, Senior Chemist
Subject: t-Butyl Methacrylate: Preliminary Laboratory Scouting Experiments

SUMMARY

Several exploratory experiments have been carried out to synthesize t-butyl methacrylate. The results of these preliminary experiments have been very encouraging. The chemistry of the reaction, the laboratory procedure, and pertinent results are outlined below.

CHEMISTRY

t-Butyl methacrylate is the reaction product of methacrylic acid and isobutylene:

$CH_2=CCH_3COOH$ +

methacrylic acid

$$(CH_3)_2CCH_2 \xrightleftharpoons[25^\circ C.]{H_2SO_4} CH_2CCH_3COOC(CH_3)_3$$

isobutylene t-butyl methacrylate

The reaction is acid-catalyzed and is reversible. The heat of reaction (ΔH) is -15,000 cal./g.-mole. An unavoidable side reaction produces the undesirable compound diisobutylene:

$$2(CH_3)_2CCH_2 \longrightarrow (CH_3)_2C=CHC(CH_3)_3$$

isobutylene diisobutylene

Heat of reaction (ΔH) is -10,000 cal./g.-mole

LABORATORY PROCEDURE

The methacrylic acid and 100% sulfuric acid catalyst are charged to a stirred, jacketed 1-liter autoclave (pressure rated at 100 lb./sq. in. gauge). The temperature of the material fed to the autoclave is 25°C.; the isobutylene (IB) is fed to the unit at a constant rate for 20 min., the batch temperature being maintained at 25°C., with the maximum pressure of 35 lb./sq. in. gauge observed at the end of the IB addition. After the reactants have been held at 25°C. for 2 to 7 hr., 50% (weight) caustic soda is added to the reaction mass in a quantity to neutralize the sulfuric acid catalyst. The methacrylic acid is essentially unaffected by the caustic, but neutralization is required to prevent reversal of the reaction when the isobutylene is recovered.

When the autoclave agitator is stopped, the contents of the autoclave separate into two phases: an organic phase containing t-butyl methacrylate (t-BMA), isobutylene (IB), methacrylic acid (MAA), and diisobutylene (DIB), and an aqueous phase containing only dissolved salts of neutralization. After separation of the aqueous phase, pure isobutylene is recovered by venting the reactor to atmospheric pressure and then heating to 50°C. The remainder of the IB, DIB, and t-BMA are recovered by distillation.

The residue in the autoclave is primarily methacrylic acid. Actual experimental results and comments are contained in the next section. Percentage of conversion per time for the reaction is tabulated under Comments. As noted in the Comments, 60% conversion has been found to be the best batch operating point.

EXPERIMENTAL RESULTS

A typical laboratory material balance for 60% conversion:

In	MAA	344.4 g.
	IB	448.8 g.
	H_2SO_4	11.3 g.
	50% caustic solution	18.5 g.
		823.0 g.
Out	Aqueous phase	29.9 g.
	IB	287.0 g.
	DIB	27.0 g.
	t-BMA	341.2 g.
	MAA	137.8 g.
		822.9

Reaction conditions:

Reaction temperature	25°C.
Reaction pressure (maximum)	35 lb./sq. in. gauge
Time to feed IB	20 min.
Conversion	See Comments for conversion table

COMMENTS ON LABORATORY EXPERIMENTS

1. Operation of reaction to 60% conversion is suggested for batch process.
2. DIB formed during reaction appears to be related to product formed. Use ratio of 0.1 mole of DIB per mole of t-BMA formed.
3. Both neutralization of catalyst and phase separation of organic-aqueous layers are extremely fast.
4. Negligible quantity of organic is lost to water phase. Nil water is carried out with organic phase. The salts of neutralization remain dissolved in the aqueous phase.
5. The batch distillation of DIB from t-BMA and MAA requires reflux ratio of at least 30 to 1.
6. Temperature of materials at any time must

not exceed 80°C. to avoid polymerization of MAA and t-BMA.

7. Weight percentage composition of distillates and residues at various stages of separation is as follows:

	Distillate (average)	Bottoms
At end of IB strip	99.8 (IB)	0.003 (IB)
At end of DIB strip	0.5 (t-BMA)	0.001 (DIB)
At end of t-BMA strip	99.9 (t-BMA)	0.1 (t-BMA)

8. Corrosion data obtained during reaction and distillation experiments indicate stainless steel (SS304) to be a suitable material of construction.

9. Losses of t-BMA during neutralization, decantation, and purification should be less than 1%.

10. Conversion of MAA vs. time:

Time (hr.)*	% MAA converted
1	34
2	47
3	55
4	59
5	61
6	63
7	64

*Time measured from start of IB feed.

April 4, 1966

MEMORANDUM

To: V. P. Walker, Process Engineer
From: P. Rinceton, Process Cost Analyst
Subject: Design Factors and Cost Information for t-BMA Cost Estimate

At Mr. Sign's request of April 1, 1966, the following information is forwarded to you for use in the preparation of the manufacturing cost for t-BMA.

1. Batch reactor is normally operated at 85% of full volume, continuous reactor at 100% of full volume.

2. All components are assumed to obey Raoult's and Dalton's laws.

3. All distillations may be taken as binary separations.

4. Pressures through any one distillation column may be considered uniform.

5. Columns normally purchased by the company have a 12-in. tray spacing with 1-in. liquid seals.

6. Overall plate efficiency of columns is 50%.

7. Overall heat transfer coefficients (U) normally employed for designs:

	B.t.u./hr. (sq. ft./°F.)
Condensers	U = 75
Jacket reactors	U = 40
Steam reboilers	U = 100

8. All distillation columns will be operated at their maximum vapor rate (3). All batch distillations will be run with a constant reflux ratio.

9. 160-lb. saturated steam is used in the reboilers.

10. For simplicity, I suggest that you evaluate the continuous process at an operating level of 1,000,000 lb./mo. and employ the "six-tenths factor" to determine the equipment and plant investment at other levels of capacity; that is,

$$I_2 = I_1 \left(\frac{C_2}{C_1}\right)^{0.6}$$

where I_2 = fixed investment in plant and equipment at capacity 2
I_1 = fixed investment in plant and equipment at capacity 1
C_1 = capacity 1
C_2 = capacity 2

11. Manpower requirements (each process to be run 7 days/wk. around the clock):

Continuous process	2 operators/shift
Batch process	4 operators/shift

12. The following equipment is available for the batch process:

Reactor—6,000-gal. SS reactor topped with a 36-in.-diameter, 22-plate bubble-cap column (12-in. tray spacings). Equipment rated for 100 lb./sq. in. gauge, 5 yr. old with original installed cost of $500,000 including allocated building costs. Building is serviced with steam, water, and brine.

13. Storage tank sizing:
Final product: 1 wk. storage
Raw materials: 1 wk. storage
Intermediates and recycles: 1 day

14. Disposal of waste at no cost.

15. Quality control and supplies for similar materials usually run about $0.50/100 lb. product.

16. Raw material overhead is 1% of raw material cost.

17. Equipment costs can be found in references 1 and 2, all values being adjusted to an ENR = 1,000.

18. Plant cost equals 3.5 times the installed equipment costs.

LITERATURE CITED

1. Chilton, C. H., ed., "Cost Engineering in the Process Industries," McGraw-Hill, New York (1960).
2. Vilbrandt, F. C., and C. E. Dryden, "Chemical Engineering Plant Design," 4 ed., McGraw-Hill, New York, (1959).
3. Perry, J. H., ed., "Chemical Engineers' Handbook," 4 ed., Sect. 18, P. 6, McGraw-Hill, New York (1963).

TABLE 1. Cost Factors

Labor	$3.00/manhour.
Labor overhead	35% of labor
Supervision	25% of labor
Supervision overhead	35% of supervision
Depreciation (straight-line method)	10% of investment/yr.
Repairs and maintenance	5% of investment/yr.
Property overhead	2% of investment/yr.

TABLE 2. Utilities

		Cost
Steam	160 lb./sq. in. abs. sat'd.	$1.00/1,000 lb.
Cooling water*	68°F.	$1.00/10,000 gal.
Chilled water*	40°F.	$2.00/10,000 gal.
Brine*	-50°F.	$15.00/10,000 gal.

*Maximum allowable temperature rise of 20°F.

TABLE 3. Raw Materials and Products

	MAA	IB	DIB	t-BMA	H_2SO_4(100%)	50% NaOH
Molecular weight	86.1	56.1	112	142	98.1	
Density, g./ml.	1.015	0.59	0.723	0.80	1.83	1.525
Heat of vaporization, cal./g.-mole	7,200	5,275	9,500	16,000		
T_B,°C. (1 atm.)	163	-6.9	102	134		
T_B,°C. (300 mm. Hg)	134	-29.0	73	105		
C_P, B.t.u./(lb.) (°F.)	0.45	0.50	0.50	0.45	0.35	0.77
Cost, $/100 lb.	40.00	7.70	....	....	1.20	1.50

TABLE 4. Heat Transfer Area of Jacket Agitated Reaction Vessels

Reactor volume, gal.	Heat transfer area, sq. ft.
100	25
1,000	140
10,000	840