

**1961
Problem
The Design of a Hydrogen Recovery Plant**

FOREWORD

This year the solutions were devised and judged by a committee from E. I. duPont de Nemours and Company, Incorporated, Wilmington, Delaware: C. H. Collier, chairman; E. L. Mongan; and E. A. Ransom. The judges wrote:

The Committee of Judges found their work rewarding as well as entertaining. The following comments are offered in the hope that the many contestants who could not receive direct recognition will find them instructive and possibly even of some use in their future chemical engineering assignments.

We found uniform evidence of much hard work having been expended by the contestants. It was also gratifying to observe that the entries gave strong evidence that the contestants competently read and understood necessary background articles (e.g., the Colburn-Hougen procedures for sizing partial condensers) and successfully applied the technical material to the problem at hand. Considering the advanced nature of some of this material for engineers at the senior college level, we were agreeably surprised at the success most contestants experienced in this area.

The judges were sharply disappointed, on the other hand, at the lack of imagination displayed by the entries as a whole. The problem was deliberately selected (with relatively minor changes from a real process) because it offered opportunity to display ingenuity as well as a test of the ability of the contestant to carry out a successful process design at the evaluation level of precision. For example:

1. *Sizing Partial Condenser* - Surprisingly, only about a third of the entries offered a treatment which took cognizance of the central fact that the most economical choice of this condenser requires that the cooled gas exit at a relatively low temperature (ca. 21° to 22°C.). Most papers chose 30°C. as the exit temperature, though loss of carbon tetrachloride becomes uneconomical at this temperature. Further, investment savings on the condenser from use of this exit temperature are more than offset by additional investment required for other equipment, principally the compressor.
2. *Caustic Scrubber* - Although the problem statement stipulated 15% caustic solution for this scrubber, the contestants were invited to offer alternative proposals for later consideration by their employer. A further clue was provided in that they were asked to comment on the feasibility of using 50% caustic. Although most contestants correctly rejected the latter alternative, only a handful observed that even 15% is an uneconomically high concentration, and no one suggested the use of sodium carbonate or scrubbing with water, followed by neutralization with calcium hydroxide (or sale of the hydrochloric acid solution!) as further economies.
3. *Purge Stream* - Even if the partial condenser is operated at 21°C., there remains a substantial loss of carbon tetrachloride to the purge. Again, only a handful of entries suggested that further study be made of means of reducing this loss. All of these proposed use of an adsorbent such as activated carbon for the purpose. None of the papers suggested that the use of a refrigerated purge condenser might be practicable.

A few other errors were observed which, while found in only a minority of the entries, were encountered often enough to warrant comment. Many contestants assumed that the compressor would operate at 100% efficiency, and a few even assumed a motor efficiency of 100%. Some contestants recommended use of cooling coils within the ceramic scrubbing tower but failed to suggest an economical material of construction for the coils capable of coping with this environment or to note the difficulty with which they could be inserted. Some papers assumed operation for 24 hours a day, 365 days a year. There was a general tendency to assume unreasonably high labor requirements, although the judges recognize that many students have little or no background for proper choice in this area.

Finally, most papers were too long to be submitted to an industrial process development executive and many did not contain a suitably concise and clear summary. While excessive length can perhaps be temporized with, the judges consider that a clear, factual summary is mandatory. Failure to meet this requirement was weighed heavily in selection of the winning papers.

The first prize of \$200, the A. McLaren White Award, went to Robert L. Rettig, Oregon State University; the second prize of \$100, the A. E. Marshall Award, to Allen Clamen, McGill University; the third prize of \$50 to Steven F. DiZio, Oregon State University; and honorable mention to Michael G. Cape, McGill University, and Richard P. Fogaroli, Villanova University. The first-prize-winning solution follows on page 76.

TO THE CONTESTANT

The chemical engineer is frequently asked to evaluate alternative process concepts based upon laboratory or pilot plant data. The objective usually is to assess the practicability of the concepts under study in order to select the most promising for further experimental study. A secondary objective often is to provide guidance for the experimental work.

One of the problems facing all engineers engaged in process evaluations is allocating study time. The contestant, like his industrial counterpart, has limited time to spend on a study of process alternatives. It is essential that he decide early where the important issues lie and work toward reaching the correct decisions in these areas. Less important areas should be given less complete treatment. Evidence of good judgment in allocation of effort will be considered when the solution is judged. Each contestant should constantly bear in mind that this problem is aimed at selecting designs and estimating costs for the given process. The results will be used in choosing the most profitable course of action from among several other competing alternatives not specified herein. The calculations should, therefore, be made only with sufficient rigor to assure adequate resolving power in choosing from among the alternatives.

Clear and adequate presentation of conclusions, together with supporting material, is essential. While a formal report is not desirable, the contestant is expected to state his conclusions and recommendations clearly for quick understanding by research and development management. Supporting material should include a discussion of how these conclusions were reached and an appendix conveying calculations, figures, tables, and appropriate references.

The problem consists of evaluating a process for recovering hydrogen from a waste stream and disposing of the unwanted impurities. The purified hydrogen, with suitable make-up, is to be returned at 600 lb./sq. in. gauge to a reaction area for reuse. Impurities to be removed are nitrogen, hydrogen chloride, and carbon tetrachloride. Several process conditions, such as temperatures, flows, and pressures, are left open. The contestant is asked to specify his choice of these alternatives, keeping in mind the desirability of achieving a minimum-cost process in the limited study time available. Should he detect other promising process changes, he is expected only to bring these to the attention of

his supervision with minimum supporting calculations, since they, together with suggestions from other sources, will constitute the next stage of process evaluation work.

The problem is assigned to you, a chemical engineer, by your immediate supervisor, Mr. Boyd, as follows:

STATEMENT OF THE PROBLEM

Mr. Boyd: Al, as you know from your last assignment, one of the principal economic factors in our proposed process for Q is the necessity for recovery and reuse of the hydrogen in the off-gas from the Q reactor. While it is essential that we recover the hydrogen in this stream, all our laboratory work has been conducted on a "once-through" basis for hydrogen. We therefore plan to demonstrate the feasibility of recycling the purified hydrogen in the pilot plant to be built this fall.

Several means of purifying and recycling the Q reactor waste gas have been proposed. One of these is shown on this rough flow sheet (Figure 1). I'd like you to evaluate this proposal at fullscale plant conditions so that your results will be comparable with several other ideas we are now studying for the ultimate commercial plant. Your evaluation should include selection and sizing of the major equipment: the partial condenser, the scrubber, the compressor, and the dryer. We shall also need your conclusions concerning a number of alternative operating conditions. Finally, you should compute the cost of including this recovery area in the plant. Since results on other flow sheets will be compared with yours, wrap up the economics as the cost of providing hydrogen to the Q reaction by this process in cents per thousand standard cubic feet.

Mr. Engineer: Mr. Boyd, the partial condenser appears to constitute a difficult problem. What method shall I use to calculate the heat transfer coefficient?

Mr. Boyd: I suggest you use the method of Colburn and Hougen (1) for calculating the area. The inlet gas is saturated with carbon tetrachloride. You can use the Nusselt relation (2) for the condensate film coefficient. Neglect the correction for vapor velocity in this preliminary work. Use a vertical, tubular exchanger with 3/4-in., 18-BWG tubes; process stream down through the tubes. Our plant standards require tube lengths to be a multiple of 4 ft., with 24 ft. the maximum length. We expect to have 16°C.

cooling water available at the site. Since the water becomes excessively corrosive at elevated temperatures, do not design for water-side temperatures above about 50°C. Use these values (Table 1) for physical properties of this system. It will be satisfactory to use carbon steel for this service, since the inlet gas is expected to be dry.

Mr. Engineer: Removal of the condensed carbon tetrachloride will depend on, among other things, the inlet velocity to the cyclone. What removal efficiency is required?

Mr. Boyd: We customarily use velocities between 50 and 100 ft./sec. I suggest that you use a value of about 70 ft./sec., as this will give negligible liquid carry-over (3).

It is important, Al, that the HCl content of the gas entering the compressor be held to not over 0.001 mole %. As you can see from the flow sheet, this is to be accomplished with a caustic scrubber to remove the acid. The tower should be a packed tower fed by 15% caustic. Because of corrosion, assume that we shall require ceramic towers with ceramic Raschig ring packing. As such tower sections are limited to a maximum diameter of 5 ft., multiple towers may be required. All necessary design information is available in Perry (3). I'd also like you to comment on the feasibility of using 50% caustic, since we shall receive our caustic as a 50% solution. As you know, caustic dilution facilities for R area can readily supply the 15% caustic needs for the H₂ recovery area. The solution will be available at 30°.

Mr. Engineer: Mr. Boyd, I note that an unspecified stream is extracted from the flow sheet and flared. Is the quantity to be removed at this point unknown?

Mr. Boyd: The purpose of this purge is to maintain nitrogen at a level below 10 mole %. The only source of nitrogen is the make-up hydrogen, which contains 1/2 mole % N₂. The Q reactor consumes 1,000 lb./hr. of hydrogen. Use the plant flare stack which has adequate capacity to handle this purge stream.

Remember that moist carbon tetrachloride is quite corrosive to cast iron and steel. For this reason we plan to dry the scrubbed gas to a -30°F dew point before compression. Use silica gel dryers for this operation. I believe that Perry will provide all the needed information for sizing these dryers and calculating utility requirements. Steam is available at 150 lb./sq. in. gauge.

Mr. Engineer: The flow sheet does not indicate how many stages of compression are required. Will this be determined by the permissible compression ratio or by the corrosiveness of HCl at the exhaust temperatures?

Mr. Boyd: If you hold the HCl concentration below 0.001 mole % leaving the scrubber, corrosion should not be a problem. The economical limit on interstage temperatures. In any case, remember that mechanical considerations and thermal decomposition of CCl₄ will limit interstage and discharge temperatures to 400°F. No after cooler will be required.

TABLE 1. GAS COOLER—PHYSICAL PROPERTIES

Viscosity	0.03 centipoise @ 100°C.
Off-Gas:	0.04 centipoise @ 150°C.
	0.012 centipoise @ 40°C.
	0.015 centipoise @ 100°C.

Solvent-free gas:	
Prandtl numbers	Hydrogen 0.68
	Nitrogen 0.70
	CCl ₄ 0.88
	HCl 0.90

Assume that the Prandtl number is independent of the temperature.

For mixtures, assume that the Prandtl numbers and viscosities are additive on a mole fraction basis.

The Schmidt number of carbon tetrachloride in gas mixture = 2.8

See reference 3 for additional data.

Mr. Engineer: Do we know the acceptable amount of CCl_4 in the recycle stream?

Mr. Boyd: The Research Section has told us that 5 mole % will be acceptable. I suggest you use this figure as a maximum concentration.

That about wraps it up, Al. Talk to Mr. Cone of our Estimating Section about estimating the capital costs of the recovery process and to Mr. Downey of the Accounting Section to obtain costs for utilities and for guidance in converting investments and operating costs to the final criterion, which is cost per thousand standard cubic feet for the recovered hydrogen. Have you any questions?

Mr. Engineer: When will you require my report, Mr. Boyd?

Mr. Boyd: In order to use your results in conjunction with the other studies now in progress, I must have your finished report by May 15.

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You next discuss sources of investment costs with Mr. Cone of the Estimating Section.

Mr. Engineer: Mr. Cone, I have been asked to evaluate costs for the process shown on this flow sheet. As you can see, I shall require estimates for heat exchange equipment, cyclone separators, a packed scrubber, silica gel dryers, and a compressor. Can you suggest appropriate sources?

Mr. Cone: Since you are working at evaluation-level precision, I suggest that you use correlations generally available in the literature, with appropriate adjustments to fit our local conditions. It should not be necessary to obtain manufacturers' quotations for this type of study.

For heat exchangers, I suggest that you use Sotnick's correlation (7). Installed costs, that is, cost in place with supports, insulation, piping, etc., will run about 3.0 times the costs quoted in this article.

For the cyclone separators, useful cost information appears on page 2-64 of "Chemical Business Handbook" (4). Multiply by about 3.5 to cover overhead, piping, etc.

The scrubber may be estimated from data found in Perry (3), page 709. I suggest that you scale these 1946 costs up by about 70% to represent today's costs. Use a factor of 4 to obtain installed cost from these estimates to cover shipping, erection, piping, and other costs associated with putting the tower in operating condition.

We have found that the costs of silica gel dryers are correlated by the relationship

$$C = 4,000 (W)^{0.6}$$

where C represents cost of the delivered dryer in dollars and W is the weight of the contained silica gel in thousands of pounds. Multiply the resulting costs by about 150% to give installed costs, including piping, foundations, electrical supply, and other related items.

Data for estimating the compressor will be found in Chemical Engineering for June, 1949. Note that these prices include interstage coolers. Scale up these figures by 50% to cover advances in costs. Use a value of \$12.50/hp. in estimating the cost of an electric motor drive plus gear reducer for the power range 2,000 to 6,000 hp. Installed cost may be taken as 2.0 times the basic equipment (compressor, intercoolers, motor, and speed reducer). This will provide for piping, interstage separators, instrumentation, electrical switchgear, wiring, building, and foundations.

Mr. Engineer: As I understand it, use of the foregoing factors will give me estimates for each piece of major equipment, which will sum to the cost of the entire process. Is that correct?

Mr. Cone: Not quite. You must also add appropriate allowances for the investment tied up in providing utilities. I suggest that you assume that an allowance of \$12.00 be made to cover investment to provide each pound per hour of steam. An allowance of \$40.00/kw. would also be appropriate. Use about \$125/(gal.) (min.) for cooling-water supply. Since only a part of a plant is being considered, you may neglect other usual capital requirements, such as cafeterias, roadways, laboratories, and so on. The cost factors I have given you should be ample to include buildings, foundations, controls, process, and service piping. Add about 15% to cover unrecognized contingencies.

Mr. Engineer: Thank you, Mr. Cone. If I run into trouble, I hope that I may get in touch with you again.

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Finally, you call on Mr. Downey of the Accounting Section to get information needed to compute costs in cents per thousand standard cubic feet of hydrogen.

Mr. Engineer: This process is being considered for recovery of hydrogen from the proposed

Q area, Mr. Downey. Mr. Boyd suggested that I see you to obtain appropriate cost information needed to evaluate the proposal.

Mr. Downey: You will, of course, require unit costs for utilities. Our average values are set forth in this table (Table 2).

TABLE 2

Electricity	\$0.009/kw.-hr.
Steam - 150 lb./sq. in.	0.90/1,000 lb.
Water	0.10/1,000 gal.

Carbon tetrachloride is one of the basic ingredients required by the Q reactor area. I suggest that you charge your recovery process with the losses of CCl_4 in the purge stream. Use a price of \$0.11/lb. for this commodity. No credit should be taken for the recovered CCl_4 in assessing the economic attractiveness of this particular recovery scheme, since the Q reactor waste gas delivers this material to your process at no charge. A proper comparison of each of the competitive recovery processes will result if each is charged with the losses it incurs. Caustic will cost about \$2.90/cwt. of contained NaOH.

Are any other raw materials required?

Mr. Engineer: How should I handle the make-up hydrogen?

Mr. Downey: Hydrogen is available at 50 lb./sq. in. abs. from the H area at a total cost of \$1.05/1,000 std. cu. ft.

I suggest you use the following factors in computing the final cost per pound of the recycle gas:

1. Operating labor @ \$2.85/hr.
2. Supervision—10% of operating labor per year
3. Maintenance—8% of plant investment
4. Overhead, insurance, property taxes—6% of investment per year
5. Depreciation—10% of investment per year

You can ignore sales expense in this evaluation. It will also be acceptable to ignore admin-

istrative expense, research costs, and working capital allowances under the special conditions of this study.

When we compare the alternatives, we prefer to use the cost-plus-return basis. This means that to the mill cost we add an allowance which represents the amount of money we should have to earn to make it worthwhile to make the investment required. Since all our earnings are subject to the 52% corporate tax, the gross earnings (in this case a cost to be added to your project) must be great enough to provide a 15% return on your investment after the taxes have been deducted.

An excellent guide for these earnings calculations is available to you in Perry's "Chemical Business Handbook" (4), pages 2-55 through 2-75. This, together with the numbers I have suggested, should give you all the information you will require.

Mr. Engineer: Thank you, Mr. Downey.

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