Capacity of the still pot:

Weight of aniline to be charged in each of two runs per day	5,380 lbs.
Volume occupied (5,380/62.4 × 1.02)	86 cu.ft.
Doubling to allow for the heating coils and for the separation of entrainment	172 cu.ft.

Diameter—6′; Height—7′

Thickness of stillpot:

The stillpot must withstand an external pressure of approximately one atmosphere.

$$t = \sqrt{\frac{P \times d \times L}{89{,}600}}$$

P = External pressure (lb./sq.in.) = 14.69
d = Diameter of tank (inches) = 72″
L = Length of tank (feet) = 7′

Solving, $t = 0.288$ or, ⅜″ steel.

Diameter of column:

This column must have three theoretical plates as estimated by Mr. Davis, and verified in the Material Balance. The maximum vapor velocity in the column occurs during the final distillation under reduced pressure.

Volume of vapor (doubling to allow for reflux)

during the 16 hour distillation

$$2 \times 140 \times 359 \times \frac{760}{50} \times \frac{323}{273}\ 1{,}812{,}000 \text{ cu.ft.}$$

or ....113,500 cu.ft./hr.

Limiting the vapor velocity to five feet per second,

Cross sectional area (113,500/3,600 × 5) ....6.26 sq.ft.

$$\text{Diameter} = \sqrt{\frac{6.26 \times 4}{\pi}} = \ldots\ldots\ldots\ldots 2.83 \text{ ft.}$$

Hence, a distilling column three feet in diameter must be used.

Pump to remove uncondensable gases:

The leakage is estimated to be 1% of the total volume distilled.

Capacity (0.01 × 113,500) ....1,135 cu.ft./hr.

Converting to one atmosphere,

Capacity (1,135 × 50/760) = 74.5 cu.ft./hr. or 1.24 cu.ft/min.

A size five two stage non-condensing pump must be used.

Pump to remove the distillate.

Approximately 86.0 cu.ft. are to be removed in 16 hours or,

Capacity (86.0 × 7.48/16) .... 40.3 gal./hr.

Hence, the smallest pump available may be used.

Head (760 × 13.6/10 × 2.54 × 12) .... 32.2 ft. of water

The condenser size has been calculated under the classification of heat exchange equipment.

CALCULATION OF EQUIPMENT COSTS

The tank costs were calculated on a basis of the total weight of steel with factors supplied by Mr. Curtis.

$$\text{Tank Cost} = f\,\frac{T}{12}\,62.4 \quad 7.2\left(\frac{D^2}{2} + DH\right)\pi \text{ Dollars}$$

T = Tank thickness in inches
D = Tank diameter in feet
H = Tank height in feet
f = Cost of steel per pound

The values substituted in this equation and the values obtained are listed below,

Nitrobenzene storage tank: f, $0.14; D, 15′; H, 15.5′; T, ¼″. Cost, $1,300.

Aniline storage tank: f, $0.14; D, 15′; H, 15′; T, ¼″. Cost, $1,110.

Hydrochloric acid storage (rubber lined tank): f, $0.34; D, 8.5′; H, 8.5′; T, ¼″. Cost, $1,220.

Settling tank: f, $0.20; D, 5.25′; H, 10′; T, ¼″. Cost, $604.

Aniline water storage: Item 21 in list of second hand equipment supplied by Mr. White. f, $0.12; D, 6.0′; H, 4.0′; T, ¼″. Cost, $149.

Item 19: f, $0.12; D, 5′; H, 6′; T, ¼″. Cost, $180.

Aniline oil storage: f, $0.20; D, 6′; H, 7′; T, ¼″. Cost, $354.

Stillpot, Item 20: f, $0.12; D, 6′; H, 7′; t, ⅜″. Cost, $440.

The calculation of the remaining equipment costs involve no special equations, and will not be given since the cost is obvious from the cost per unit as supplied by Mr. Curtis and from the number of units as listed.

LITERATURE CITED

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Mr. Charles M. Davis, Report dated 10/30/41.
Mr. Richard P. Curtis, Report dated 11/6/41.
Mr. Thomas F. White, Memorandum dated 11/10/41.

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General criticism is as follows:

1. This solution of the problem requires the use of the finished goods distillation still for distillation of both finished goods and water layer. The idea, in itself, is sound and no fault was found with its use. However, if all errors in calculation are corrected it is easily seen that time cycles do not allow this equipment to perform both functions. A separate still would be necessary for one use, or else the size of the one still would have to be increased.

2. The use of a copper steam coil in the reducer would have cut down water layer to be reworked by a considerable quantity.

3. Continuous separation of aniline-water distillates in small overflow type separators would have materially reduced the aniline water and crude aniline storage space necessary.

4. A combination of coil in the reducer and continuous return of aniline water from the distillate to the reducer during the reducer steaming can be calculated which eliminates the necessity for reworking any aniline water.

CONTEST PROBLEM

1943

STUDENT CHAPTERS—AMERICAN INSTITUTE OF CHEMICAL ENGINEERS

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

DEADLINE FOR MAILING—

Must be Postmarked Not Later than Midnight, March 15, 1943
(See Rules)

RULES OF THE CONTEST

Solutions will be graded on (a) ingenuity and logic employed, (b) conclusions reached, (c) accuracy of computations, and (d) form of presentation.

It is to be assumed that the statement of the problem contains all the pertinent data except for those readily available in steam tables and similar reference works. Your instructor is not to be consulted in regard to doubtful points. The problem is not to be discussed with any person whatever until after March 15, 1943. This is particularly important in cases where neighboring institutions may not begin the problem until after its completion by another chapter. 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. Submittal of a solution for the competition implies adherence to the above conditions.

A period of not more than 24 consecutive days is allowed for completion of the solution. This period may be selected at the discretion of the individual counselor, but a solution must be postmarked not later than midnight March 15, 1943, in order to be eligible. Each 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 Chairman of the Committee on Student

Chapters. 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, from his chapter, not to exceed two in number and send these by registered mail to

Dr. J. Henry Rushton
Department of Chemical Engineering
University of Virginia
Charlottesville, Va.

A.I.CH.E. STUDENT CONTEST PROBLEM

To the Contestant:

The committee has selected a problem which calls for the application of engineering judgment and perspective rather than routine calculation. At first study, the contestant may fear the problem to be too vague for proper solution. It is, however, a very real problem taken from a recent actual case. It was presented in essentially this form by a chemical manufacturer to one of his young chemical engineers. This engineer presented a report which was a start in guiding the company executives to a decision.

In brief, the problem calls for an analysis of four proposed processes for the production of magnesium. You will assume that you are working for a company which hopes to go into the production of magnesium and is faced with the problem of choosing a process. You will be furnished with an outline of each of four processes along with a short bibliography of the subject. While other processes exist, the committee has restricted the number to four so as not to unduly complicate the problem.

Your first reaction may be that insufficient data have been furnished to permit a thorough analysis of the subject. While this may be true, you will find that such difficulties are often faced in industry. Because of the scarcity of information, your company will have other engineers, executives and consultants using their contacts and experience. You should, however, be able to prepare a report which will aid the company executives in arriving at their decision.

Because of the nature of this problem, your papers will be judged on the basis of the completeness of your consideration of the subject matter, the logic behind your conclusions, and the manner in which your report is presented.

IMPORTANT NOTICE

The manufacturers who have been consulted during the course of the preparation of this problem have requested that the contestants refrain from writing to them for information regarding the manufacture of magnesium. You are urged to confine yourself to the literature available in your libraries and the bibliography given at the end of the problem.

For the purpose of the contest, you will assume that you are Mr. R. U. Wright, a process engineer in the Research and Development Department of the Progress Chemical Corporation. You will receive the rest of your instructions in a memo from your Department Director.

Mr. R. U. Wright
Process Engineer, R & D Dept.

Dear Mr. Wright:

I have received word from our main office that we might be interested in adding magnesium to the already large list of products manufactured by our company. An additional production of twenty million pounds of magnesium per year is needed to meet an expansion in the war program. The production must be available at the earliest possible date, and since many other parts of the war program will depend upon having this new material, it is important that promised deliveries be met.

Realizing the importance of the problem and the difficulties involved in undertaking such a program, the company has employed a well-known firm of consulting engineers to help us decide upon a process, and help us later with all detailed problems of design. However, in order to aid our executives in reaching a necessarily hurried decision involving one of four commercially feasible processes, the Research and Development Department has been asked to contribute its analysis of the problem.

I am asking you to undertake this analysis, leaving all details of design and plant layout up to our Engineering Department and our consultants. However, I am requesting of you an analysis which is backed by sound engineering judgment and preliminary calculations which will aid us in finally deciding upon one process.

Four commercially feasible processes have been proposed for our consideration. I am enclosing a brief description of each, namely,

the electrolytic, carbon reduction, calcium carbide reduction and ferrosilicon reduction processes. I would like you to evaluate these processes on the basis of the information available to you. We are particularly interested at this point in costs, engineering problems, and availability of raw materials, power and construction materials. Where you find that lack of data makes it difficult to draw definite conclusions, you may discuss alternatives and point out what information would be needed to enable you to arrive at a conclusion.

In attempting to determine costs, keep in mind factors such as raw materials, power, labor, auxiliary operations, amortization, maintenance, etc. While your limited experience will handicap you on the subject of costs, you should be able to make comparative estimates of items such as labor, power, maintenance, materials, handling, etc., on the basis of the nature of the process, size of units involved, material preparation required and the degree of complexity of the operations. Maintenance and replacement of units may become a considerable cost item and should, therefore, be given careful consideration. Consider the possibility of recovering by-products and residues and the value of such residues. Construction material requirements should be studied with an eye towards costs and availability of critical materials during the present war period. Remember, however, that we would like this plant to be capable of competing with others under peace conditions after the war.

Power will be an important item and this factor should be studied with a view towards cost and availability. Plant locations as they will be affected by the availability of power, raw materials, etc., should be discussed.

You will find most of your material prices in current periodicals. If you wish, you may derive your own prices, but the method of doing so and all sources should be shown.

You may come as close to recommending any particular process as you feel the available information will permit.

Sincerely yours,
M. G. Hope,
Director of Research and Development.

Electrolytic Process

The electrolytic process for the production of magnesium involves primarily the electrolysis of dehydrated magnesium chloride. The electrolytic decomposition is carried out in cells in which the steel pot serves as the cathode while the anode consists of graphite bars. The

electrolytic process is a continuous operation, the metal being removed daily. The cell bath is maintained at approximately a constant level, by either a continuous or intermittent feed of dehydrated $MgCl_2$. NaCl is added from time to time to reduce the melting point and increase the conductivity of the bath. Exterior heat helps to maintain the proper cell temperature. The Mg metal formed is lighter than the cell bath, and, therefore, rises and floats on the surface of the same, but does not burn because of the protecting action of a thin film of molten salt bath. The sludge which is formed, due largely to the small percentage of MgO present in the cell feed, sinks to the bottom. Many factors combine to yield a metal of such purity that subsequent refining is unnecessary. The average analysis of this Mg direct from the cells reveals a purity of 99.9 per cent.

The preparation of the anhydrous $MgCl_2$ for use in the cell is dependent upon the raw material most easily available. Many processes present themselves starting with either magnesite or a natural brine. A typical process involves the use of a brine containing approximately 14 per cent NaCl, 9 per cent $CaCl_2$, 3 per cent $MgCl_2$ and 0.5 per cent bromine. For the purpose of simplifying the problem, we will ask you to restrict yourself to this method of obtaining the raw material for the electrolytic process. After the bromine is removed, the brine is treated with magnesium hydrate slurry to precipitate the iron and other impurities which are separated in continuous thickeners and sedimentation tanks. The decanted liquor is then evaporated until the NaCl has crystallized. The salt is removed. The $MgCl_2$ and $CaCl_2$ are then separated from each other by fractional crystallization. After a series of crystallizations, approximately one-half of the $MgCl_2$ originally present in the brine has been removed, the other half being returned to the process for reworking. The final steps in preparing feed for the electrolytic cell consist in a series of dehydration operations to remove the 6 molecules of water of crystallization which correspond to 53 per cent of the weight of the hydrated flaked chloride. This work has shown the existence of a complete series of hydrated salts containing respectively 6, 4, 2, and 1 molecules of H_2O. Air drying on the counter-current principle is practical to a composition corresponding approximately to $MgCl_2 \cdot 2H_2O$, provided the temperature is carefully controlled to prevent incipient fusion. The last two molecules of water are removed by heating to still higher temperature in an HCl atmosphere which is necessary to prevent hydrolysis and the formation of MgO.

Summarized data on operation details are as follows:

Raw material	anhydrous $MgCl_2$
Melting Pt. material ° C.	$MgCl_2$ 708
Melting Pt. metal ° C.	Mg 651
Bath material	$MgCl_2$ + NaCl
Voltage across cell	6–9
Current density, amp/in²	16–35
Operating Temp. ° C.	670–730
Raw material consumption	4–5 lb. $MgCl_2$ per lb. Mg
Current Eff.	70–80%
Energy Eff.	20–30%
Energy consumption	8–13 KWH per lb. Mg.

CARBON REDUCTION PROCESS

This process depends basically on the reduction of magnesium oxides to magnesium using finely divided carbon as the reducing agent. The reduction is effected in an electric furnace at a temperature of approximately 2000-2100° C. Under these conditions the reaction proceeds according to the equation ($MgO + C = Mg + CO$); but unless the products of the reaction are suddenly cooled the equation quickly reverses itself and little or no magnesium is produced.

The rapid chilling may be obtained by the use of large quantities of hydrogen, or natural gas, if the latter is available. The magnesia obtained either from calcining magnesite or brucite is mixed with petroleum coke and formed into briquets using pitch as the binder. The briquets are then fed continuously into the reduction furnace. The latter is a totally enclosed carbon-brick lined 3-phase electric arc furnace. An atmosphere of hydrogen is maintained in the furnace to prevent the entrance of magnesium powder to the isolated electrode glands.

The reaction products from the furnace are drawn off on one side through a stainless steel nozzle and condensed immediately by the blast of natural gas injected through the condenser at the rate of approximately 25 volumes of natural gas to each volume of furnace gas. After this initial quenching, the magnesium along with certain impurities is carried as fine dust into the cooling chamber—a large drum equipped with revolving scrapers to prevent caking on the side walls and with a reaming mechanism to prevent plugging the furnace nozzle. Some of the magnesium powder drops to the bottom of the condenser and is caught by a screen conveyor that takes it to a dust storage bin. In the meantime, the temperature of the gas has been lowered to about 150–200° C. and most of the dust is carried by the cooling gases to an electrostatic precipitator where the particle size of the magnesium dust is increased. The balance is removed in bag filters.

The dust from this primary reduction consists of 60–65 per cent metallic magnesium contaminated with some magnesium oxide and carbon. The dust is compressed without a binder into tablets. These tablets are then charged into totally enclosed electrically heated retorts that operate at temperatures of about 750° C. and under extremely high vacuum. The metal rises to the upper part of the retort and is deposited there on the water- and oil-cooled steel walls of a removable shell. After the distillation is completed, the top of the retort furnace is removed and the upper shell on which the metallic magnesium has condensed into a crystalline ring is lifted out. It is then dumped on the floor of a cooling room and transported from there to conventional furnaces for remelting and casting.

The estimated power consumption for this process per pound of magnesium is approximately as follows:

Reduction furnace	6.6 KWH
Distillation	1.1 "
Re-melting	0.2 "
Auxiliaries	0.2 "
Hydrogen supply	1.4 "
	9.5 KWH

FERRO-SILICON PROCESS

This process comprises the reduction of dolomite with ferrosilicon at high temperature and vacuum to produce magnesium. The basic equation may be written as follows:

$$2MgO \cdot CaO + Si \rightarrow 2Mg + 2CaO \cdot SiO_2$$

The dolomite used must be of the crystalline and not of the amorphous variety and it should contain equi-molecular proportions of MgO and CaO. There should not be more than 2 per cent silica present in the dolomite. Ferrosilicon of about the 75 per cent grade has been used as the reducing agent.

The dolomite is quarried and then calcined in a kiln so that the residual CO_2 is less than 2 per cent. The calcined dolomite is then ground to 90 per cent through 200 mesh. The ground, calcined dolomite is intimately mixed with the ferrosilicon which has been crushed to pass through a 65 mesh screen with a minimum of fines. The mixed powder is briquetted at several tons per square inch.

The dolomite-ferrosilicon briquets are charged into retorts of suitable size and construction to permit heating to 1150° C. for 6 hours at an absolute pressure of 0.3 min. or less.

The magnesium is vaporized during the reaction and collects at one end of the retort which is water cooled so as to condense the magnesium. After being collected this magnesium is re-melted and cast into ingots, etc. The magnesium produced is pure enough to require no redistillation.

Yields of 75 to 80 per cent based upon silicon may be expected from a ferrosilicon of 75 per cent grade from 6 hour runs at 1150° C. and 0.3 min. absolute pressure.

Energy requirements for reduction are 6 KWH per pound of magnesium. This does not include energy for auxiliary operations.

CALCIUM CARBIDE PROCESS

This process comprises the reduction of magnesium oxide by calcium carbide to yield magnesium. The fundamental reaction is:

$$MgO + CaC_2 \rightarrow Mg + CaO + C.$$

Raw materials used are magnesium oxide from magnesite and commercial calcium carbide. The calcium carbide is crushed and ground to 95 per cent through 200 mesh.

Pressed briquets of magnesium oxide and calcium carbide are reacted in retorts under the same conditions as those used in the ferro-silicon reduction process.

Magnesium oxide conversion efficiencies of 75 to 80 per cent may be obtained from 6 hour runs at 1150° C. at 0.3 min. absolute pressure.

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This contest problem for 1943 was prepared by Lincoln T. Work chairman, F. H. Hirschland and R. Ricklin. They awarded first prize to S. Y. Wong.