#### References:

- (1) Absorption and Extraction by Sherwood.
- (2) Industrial Chemical Calculations—Hougen and Watson. Second Edition, page 430.
- (3) Article by Egloff and Nelson—Transactions of A. I. Ch. E., Vol. 32, 1936, page 221.
- (4) Industrial Chemical Calculations—Hougen and Watson. Second Edition, pages 338-339.

C. H. E. FORTIWON,

Development and Design Department

#### STUDENT CONTEST PROBLEM FOR 1942

#### FOREWORD

The sub-committee of the Student Chapters Committee handling the details of the 1942 Student Contest Problem consisted of the following: B. E. Thomas, Chairman, P. G. Marsh, E. T. Stehlby, A. J. Pastene, R. G. Hemminghaus, W. M. Cooper and J. W. McCrackin.

Council wishes here to record its appreciation of the work done by this committee and the efficient manner in which they carried on their arduous and time-consuming duties.

The first prize, the A. McLaren White Award of \$100.00 was presented to Mr. Charles C. Neas, University of Illinois, whose solution appears on the following pages. The other prize winners were as follows:

Gilbert R. Shockley, Missouri School of Mines, Second Prize-\$50.00

Karl H. Rothe, Cooper Union, Third Prize-\$25.00

Harold R. Frisbie, Oregon State College, Honorable Mention— \$10.00

Robert E. Deatz, Kansas State College, Honorable Mention— \$10.00

Herbert Kress, Pratt Institute, Honorable Mention-\$10.00

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## CONTEST PROBLEM

1942

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 16, 1942 (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, 1942. 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 counsellor, but a solution must be postmarked not later than midnight March 15, 1942, 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

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## A.I.CH.E. ANNUAL STUDENT COMPETITION

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

> Professor M. C. Molstad, Engineering Dept., University of Pennsylvania, Philadelphia, Pa.

ORGANIC CHEMICALS, INC.
PLANT AND EXECUTIVE OFFICES

St. Louis, Missouri

Office of the President

November 1, 1941

To: ARTHUR L. BROWN Production Manager

## SUBJECT: PROPOSED ANILINE PLANT

As you doubtless already know there is at present a serious world shortage of aniline oil, a product which is vitally needed in the defense effort both here in America and also in Britain. It is used chiefly in the manufacture of tetranitraniline, dinitromonoethyl aniline, dimethyl aniline for making explosive "tetryl," and in the preparation of a variety of pharmaceutical products. Inasmuch as our business is for the most part the synthesis of organic chemical compounds we have been asked to undertake a project to manufacture aniline oil from nitrobenzene on a scale of 5 tons per day, seven days per week. Since it is quite likely that our normal business may be seriously curtailed by the present priority situation on raw materials as well as on mechanical supplies and equipment and furthermore, since it is our desire to be of all possible assistance in the Defense Program, the company has already indicated its acceptance. Therefore, our technical staff must undertake the job at once.

The iron-muriatic acid reduction process is well known and amply described in the literature. However, since we have had no experience whatsoever with it, every detail of the proposed plant must be given careful thought as literature descriptions of manufacturing operations are often very misleading and incomplete.

I cannot emphasize too strongly the poisonous properties of both nitrobenzene and aniline in their effect on human beings. In addition to being very hazardous in vapor form they are both readily absorbed through the pores of the skin to create acute cyanosis in a short time. The plant must, therefore, be designed and laid out having this behavior constantly in mind. Complete cleanliness both of equipment and personnel will be imperative. Working conditions free of process vapors and accidental material spillage will be of paramount importance.

When drawing up the plans and specifying equipment careful consideration must be given to the fact that it will be a wartime plant of very uncertain subsequent utility or value.

All possible speed must be exercised in working up the project as urgent needs for the finished product make it impossible for us to do extensive development or pilot plant work. As you well know, work already piled up in the shops where chemical equipment is ordinarily fabricated may result in serious delay unless some processing units already on hand can be used to get construction started.

Our research chemist, Mr. Charles M. Davis, has already made a thorough search of the literature and has run a number of trial reductions and refining procedures in the laboratory in which he obtained a product of good quality with satisfactory yield. The data so obtained is contained in a report which he will submit directly to you. It is recognized that this is far from being as complete as might be desired but it nevertheless will have to serve the purpose.

I suggest you get in touch with our purchasing agent, Mr. Robert H. Jones, at once so that he can assemble cost, packing and quality data for you relative to the necessary raw materials, which information will be needed in planning the plant and making a tentative production cost estimate.

Shipment of the finished aniline will be made in 8000 gallon iron tank cars suitably equipped and in 55 gallon returnable iron drums in carloads of 36,000 lbs. net.

Since we have no available building in which to house the new plant that feature must be carefully studied bearing in mind that over a

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tical experience you have had adequate university training and we believe you have the requisite inherent ability which will be required. It is a remarkable opportunity for you to demonstrate that you can handle a really big job creditably.

The problem specifically is:

"To prepare the chemical engineering design of the proposed plant having in mind that you will be responsible for its operation after it has been built. The chemical engineering design includes: choice among process alternatives; listing of all process equipment including any instrumental control; preparation of a diagrammatic flow sheet in elevation (not required to be to scale); and brief description of any housing required. The equipment list must give a complete description of the features required to fulfill the process functions of the respective units. It must include all essential data from which the final capital cost estimate and construction and installation drawings can be made in our mechanical engineering department. It will not be necessary, however, to specify mechanical details not directly connected with the process functions of the respective units. Thus the flow sheet is the only drawing you must prepare. Calculations of pipe sizes for liquid transfers and supplies of utilities are not required but information necessary for design of piping layout must be included.

"In addition, using unit cost data on raw materials, labor, process steam, 'power-light-heat and water,' repairs, depreciation, fixed expenses, etc., to be furnished you separately, an estimate of manufacturing cost must be prepared. In order to determine the actual depreciation figure to be used in such a calculation a preliminary capital cost estimate must be made.

"Finally you will have to prepare a brief description of the proposed method of operation of the plant stating therein quantities involved in operational steps and the corresponding time cycles, means for control of operating conditions with emphasis on those especially devised, the number of workmen required and the supervision planned for them, and any other explanatory statements you may wish to include."

I will have our chief engineer, Mr. Thomas F. White, send directly to you a list of equipment we have on hand which might be utilized for this plant.

period of years we may sometimes have a few days of winter with atmospheric temperature below zero Fahrenheit, while we are very likely to have several days of summer when the atmospheric temperature is 100° Fahrenheit or more. That portion of our vacant property known as the Baker lot directly adjacent to building 17 has been designated for the plant location. Adequate supplies of steam, cooling water and electric current can be obtained merely by connection to the sources of supply leading to that building.

The characteristics of good commercial aniline oil as taken from advertisements of the leading manufacturers are:

# PRACTICALLY COLORLESS, HIGHLY REFRACTIVE, OILY LIQUID WITH A CHARACTERISTIC ODOR

| Molecular weight           | 93.12               |
|----------------------------|---------------------|
| Pounds per gallon at 25° C | 8.50                |
| Boiling point              | 184.2° C. at 760 mm |
| Freezing point             | −6.2° C.            |
| Flash point                | 70° C.              |

WILLIAM K. SMITH,

President.

#### ORGANIC CHEMICALS, INC.

St. Louis, Missouri

Production Department

To: Mr. T. Y. Rowe (Student Contestant's Nom de Plume) November 7, 1941

#### SUBJECT: PROPOSED ANILINE PLANT

We have been assigned the task of designing a plant as rapidly as possible to manufacture aniline from nitrobenzene at the rate of 5 tons per day seven days per week using the well known iron-muriatic acid reduction process. It is obvious that the pressure of regular daily duties will prohibit anyone on regularly assigned work from accomplishing this in the short time in which it must be completed. Therefore, it has been decided to turn the project over to you as a full time assignment. Though you have comparatively little prac-

A.I.CH.E. ANNUAL STUDENT COMPETITION 659

Miscellaneous data which will be required for your calculations have been tabulated by our development department and are also appended hereto.

A copy of a memorandum from our President, Mr. William K. Smith, giving a broad outline of the situation is enclosed for your information as well as a copy of a report by our Purchasing Agent, Mr. Robert H. Jones, on availability of the necessary raw materials together with essential data regarding specifications, packing, etc.

As a primary basis for your work I am enclosing a copy of a research report by Mr. Charles M. Davis covering such laboratory investigation and trial of the process as it has been possible to carry out. While all data actually needed for design of a large scale plant are embodied in this report care must be taken to accurately correlate the theoretical material with the practical application involved.

While making your plans and calculations, if you find it necessary to make any assumptions not covered by the data which are being furnished you, they should be listed and explained fully.

Please be sure that your calculations on the various phases of the problem are listed in your final report so that they may be checked. That report is to be submitted directly to me.

Certain characteristics of the two major chemical compounds which are to be handled in this proposed plant will have a direct bearing on the chemical engineering design and therefore deserve special attention before the work is started.

Neither nitrobenzene nor aniline is classed as a volatile solvent though both are flammable and may create explosive mixtures with air under certain unlikely conditions. Due regard should also be paid to the obvious hazard caused by evolution of hydrogen when making ferrous chloride from iron and muriatic acid.

From the standpoint of regular plant operation special attention must be paid to the fact that both nitrobenzene and aniline cause reactions on workmen which in acute form appear as cyanosis. No serious effects result if prompt treatment is given in the form of a cleansing bath, withdrawal from the area and possibly administration of a mild non-alcoholic stimulant. More advanced cases may result in collapse which may then prove fatal unless more vigorous measures are employed, specifically oxygen inhalation and possibly heart stimulants.

The effects of aniline are usually observed more rapidly after exposure than those of nitrobenzene. Cyanosis may be caused by

direct contact or by absorption through the pores of the skin from an atmosphere containing aniline vapor or by inhaling such an atmosphere. Hence, all accidental contact must be avoided both in vapor form or by skin contact with liquid. Obviously, the plant should be so designed that, when in operation, a visitor with an acute sense of smell will fail to detect either aniline or nitrobenzene.

> ARTHUR L. BROWN, Production Manager.

#### ORGANIC CHEMICALS, INC.

St. Louis, Missouri

Purchasing Department

November 5, 1941

Mr. ARTHUR L. BROWN. Production Manager

SUBJECT: RAW MATERIALS FOR PROPOSED ANILINE PLANT

In reply to your telephone request I submit the following purchase specifications on commercial grade nitrobenzene, muriatic acid, iron borings and hydrated lime which are now available in tonnage quantities. I wish to point out in this connection that, with conditions as they are everywhere in industry at the present time, deliveries of these materials on a firm schedule may not be wholly dependable. While it is fairly certain that the quantities required will be available, nevertheless, sufficient storage should be provided so that latitude may be allowed on incoming shipments. Some of the materials at times may have to be shipped long distances thus introducing a further factor of uncertainty. Also, due to an anticipated shortage of railroad rolling stock, it will not be permissible to hold any tank cars or box cars more than two or three days. Therefore, plans must be made to unload incoming shipments and to load outgoing shipments very promptly.

Nitrobenzene.-Light yellow liquid, crystallizing point 5.7° C., boiling point 210.9° C. at 760 mm., flash point 77° C., assay essentially 100%.

Cost = 7¢ per lb. delivered in properly equipped 8000 gallon iron tank cars on siding adjacent to manufacturing site.

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steam distillation the condensate from which separates into two phases -an aqueous phase, called "aniline water" and an oil phase, called

Aniline remaining in the excess water left over from all process operations may be removed by distillation or by extraction with nitrobenzene.

The crude aniline is refined by distillation.

## Apparatus and Raw Material Used

The laboratory reductions were carried out in a small insulated iron reducer of 1500 cc, capacity equipped with stirrer and jacket for heating. The top was closed with the exception of necessary outlets for a vented reflux condenser, iron addition, nitrobenzene addition, thermometer well, decantation, etc. Nitrobenzene was added from a glass charging funnel. The crude aniline layer was decanted by suction.

Steam distillation of the residue was carried out in the reducer and distillation of the aniline water from the resulting distillate was carried out in 1000 cc. round bottom glass flasks equipped with suitable glass condensers and glass Erlenmeyer flask receivers. Alternative handling of such aniline water by extraction with nitrobenzene was carried out in 1000 cc. separatory funnels:

Distillation of the crude aniline was carried out in a 1000 cc. glass distillation flask equipped with suitable condenser and receiver.

Tests made on copper, monel metal and cast iron indicate that they are all sufficiently resistant to the reduction so that their life would be determined more by mechanical than by chemical action. Besides the metals mentioned, steel was found to be suitable for all subsequent operations. Cuprous alloys cause discoloration of aniline.

Nitrobenzene, iron borings and lime of the same quality as proposed for large scale operations were employed.

Reagent grade concentrated HCl was used as no commercial grade material was immediately available. There is no reason to anticipate that variation in behavior would be caused by use of commercial quality acid.

#### Experimental Reductions

From the experimental charges run in the laboratory the following procedure is recommended for large scale application.

Muriatic Acid 18° Bé.-Light yellow liquid, assay 27.92% HCl, specific gravity at 15° C. = 1.1417.

Cost = \$20.00 per ton delivered in 24 ton rubber lined tank cars on siding adjacent to manufacturing site.

Iron Borings.-Appearance-clean cast iron borings free from large lumps and also free from oil and grease. Packed in 100 lb. cloth bags.

Fineness:

Retained on

0% maximum

Assay from 88% to 92%.

Thirty 100-lb. bags occupy space 2'6" x 4'0" x 2'6".

Cost = \$35.00 per ton delivered in 40 ton box cars on siding adjacent to manufacturing site.

Hydrated Lime.-Good commercial grade free from sand and other gross contaminant. Packed in 50 lb. paper bags.

Assay-90% minimum.

Cost = \$12.00 per ton delivered in 40 ton box cars on siding adiacent to manufacturing site.

Sixty 50 lb. bags occupy space 4'2" x 3'9" x 5'0".

ROBERT H. JONES. Purchasing Agent.

#### ORGANIC CHEMICALS, INC.

St. Louis, Missouri

Research Department

October 30, 1941

REPORT ON INVESTIGATION OF THE PROCESS FOR MAKING ANILINE FROM NITROBENZENE BY THE IRON MURIATIC ACID

#### REDUCTION PROCESS

Outline of Process

Nitrobenzene is reduced to aniline in the liquid phase by the action of iron and ferrous chloride in aqueous solution.

Most of the aniline is then separated from the iron sludge by decantation and the aniline remaining in the sludge is removed by

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To the 1500 cc. iron reducer 390 gms. of aniline water recovered from previous reduction charge are introduced. The ferrous chloride catalyst is then made up by adding with stirring 90 gms. fresh iron borings together with 60 gms. of iron recovered from a previous reduction and treating with 32.0 cc. of HCl having a specific gravity of 1.1885 at 15.6° C. The mixture is then heated with continued stirring until evolution of hydrogen ceases and reflux starts. Steam is then turned off the reducer jacket and continuous nitrobenzene feed is started at such a rate that 500 gms, are added over a period of four hours. 450 gms. more iron are added in 12 gm. portions every five minutes starting simultaneously with the addition of nitrobenzene. At the end of the nitrobenzene addition the reaction is 90% complete.

#### Finishing Reductions

After the addition of nitrobenzene has been completed a reflux of at least 88 cc. per hour at a vapor temperature between 95° C. and 100° C. must be maintained continuously in order to finish the reduction in three hours. Completeness of reduction may be judged by testing purity of the aniline in a sample withdrawn from the reducer using color and solubility in dilute HCl as criteria.

When finished the mass is neutralized with 5 grams of hydrated lime. It is then allowed to settle for one hour at a temperature not less than 75° C. A supernatant crude aniline layer containing 80% of the aniline produced separates practically free of sludge. This aniline layer is decanted and set aside for refining.

Reductions were made with as little as 390 grams and as much as 600 grams of aniline water in all cases obtaining a decantable layer.

The yield in the reduction stage is 99% of theory.

It was particularly noticeable that whenever the reduction reaction was sluggish in the early stages due to low temperature, poor agitation or deficiency of catalyst there was a tendency for unreduced nitrobenzene to accumulate in the reducer. Later in the cycle of these sluggish charges a rapid acceleration of reaction took place in some cases creating enough pressure in the reducer to blow out one of the stoppers and forcibly eject much of the contents.

## Recovery of Residual Aniline from the Reducer Sludge

Aniline remaining in the sludge may be recovered in several ways all of which are described in the literature. The recommended method is simple steam distillation. While laboratory measurements of the steam distillate volumes were made from several typical experiments it would be impracticable to base the economics of plant design upon this data. It should rather be derived from theoretical considerations.

Up to the solubility limit of aniline in water the partial pressure of water follows Raoult's law and that of aniline, Henry's law. Up to the solubility limit of water in aniline the partial pressure of aniline follows Raoult's law and that of water, Henry's law.

In this connection the settling rate of aniline from mixtures obtained by such steam distillation was noted. An aniline and water mixture containing a large excess of aniline was steam distilled and samples of distillate were caught in a cylindrical vessel to a depth of 16 inches. By varying the temperature of the distillate the following data were obtained:

| Distillate<br>Temperature | Time Required to Settle into Distinct Layers |
|---------------------------|--|
| 55° C.                    | 139 seconds                                  |
| 50° C.                    | 150 seconds                                  |
| 48° C.                    | 160 seconds                                  |
| 40° C.                    | 400 seconds                                  |
| 35° C.                    | 900 seconds                                  |
| 30° C.                    | 1500 seconds                                 |
| 25° C.                    | 2800 seconds                                 |

After the steam distillation is complete the spent sludge is poured into a 100 cc. beaker from which it overflows to the drain. The unreacted iron settles to the bottom of the beaker and is washed by swirling a water hose down in the beaker close to the surface of the settled iron until salts, iron oxide, etc., are washed away. The iron recovered is used in a subsequent reducer charge.

## Recovery of Aniline from the Aniline Water

Two alternative methods for recovering aniline from the aniline water were investigated, namely, distillation and extraction with nitrobenzene. As both methods were employed quite successfully in the laboratory, choice must be made between them for plant application.

The apparent distribution coefficient for aniline in the system nitrobenzene-water between 25° C. and 55° C. was found to be 18 assuming no dissociation of aniline in either layer.

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#### ORGANIC CHEMICALS, INC.

St. Louis, Missouri

Development Department

November 6, 1941

To: Mr. Arthur L. Brown, Production Manager

Subject: Design Data for Proposed Aniline Plant

Following our conversation of a few days ago I have assembled the miscellaneous information you requested for use in designing a plant to manufacture aniline. Included are data for chemical engineering calculations as well as the generally accepted factors needed for preparation of a preliminary capital cost estimate and manufacturing cost estimate.

TABLE I.—MUTUAL SOLUBILITY OF ANILINE AND WATER \*

|                  | Weight Per Cent Aniline         |                                 |  |  |  |  |  |  |  |  |  |
|------------------|---------------------------------|---------------------------------|--|--|--|--|--|--|--|--|--|
| Temperature ° C. | Water Saturated<br>with Aniline | Aniline Saturated<br>with Water |  |  |  |  |  |  |  |  |  |
| +10              | 3.38                            | 95.18                           |  |  |  |  |  |  |  |  |  |
| 15               | 3.43                            | 95.06                           |  |  |  |  |  |  |  |  |  |
| 20               | 3.49                            | 94.93                           |  |  |  |  |  |  |  |  |  |
| 25               | 3.55                            | 94.77                           |  |  |  |  |  |  |  |  |  |
| 30               | 3.63                            | 94.62                           |  |  |  |  |  |  |  |  |  |
| 40               | 3.83                            | 94.29                           |  |  |  |  |  |  |  |  |  |
| 50               | 4.05                            | 93.86                           |  |  |  |  |  |  |  |  |  |
| 60               | 4.40                            | 93.40                           |  |  |  |  |  |  |  |  |  |
| žŏ               | 4.91                            | 92.75                           |  |  |  |  |  |  |  |  |  |
| 80               | 5.59                            | 91.95                           |  |  |  |  |  |  |  |  |  |
| 90               | 6.36                            | 91.00                           |  |  |  |  |  |  |  |  |  |
| 100              | 7.18                            | 89.70                           |  |  |  |  |  |  |  |  |  |
| 110              | 7.95                            | 88.05                           |  |  |  |  |  |  |  |  |  |
| 120              | 9.15                            | 86.0                            |  |  |  |  |  |  |  |  |  |
| 130              | 11.05                           | 83.6                            |  |  |  |  |  |  |  |  |  |
| 140              | 13.60                           | 80.6                            |  |  |  |  |  |  |  |  |  |
| 150              | 17.00                           | 76.4                            |  |  |  |  |  |  |  |  |  |
| 160              | 23.90                           | 69.1                            |  |  |  |  |  |  |  |  |  |
| 165              | 30.60                           | 61.8                            |  |  |  |  |  |  |  |  |  |
| 167.5            | 46.16                           | 46.16                           |  |  |  |  |  |  |  |  |  |
|                  |                                 |                                 |  |  |  |  |  |  |  |  |  |

<sup>\*</sup> International Critical Tables, vol. 3, p. 389, McGraw-Hill.

Note: Assume that the salting out effect of inorganic salts in the reducer solution will reduce aniline solubility in the water layers in above table by 20%; also that data on aniline rich layer is unaffected.

## Refining of Crude Aniline

Crude aniline obtained from the three steps in the process just described was combined and distilled in glass. Pure aniline was collected after first removing water and wet aniline. An end fraction of impure aniline was finally separated.

Distillations were carried out at atmospheric pressure and at reduced pressure. Boiling points of aniline at the various pressures were in substantial agreement with the data in the literature covering aniline and water.

The first experiments were carried out using direct distillation with no fractionation. Under these conditions it was found that the distilled aniline was appreciably colored and its crystallizing point was slightly low unless unduly large foreruns and end fractions were separated. A small fractionating column packed with glass helices (calculated to be equivalent to three theoretical plates) was then used and a larger proportion of aniline of satisfactory quality was obtained. A reflux of half the forward flow was maintained while the pure aniline was being distilled. Fractions were separated as nearly as possible by boiling point and color of distillate. It is suggested that on the plant scale these fractions can be more accurately controlled and separated by crystallizing point while giving proper consideration to color.

It was found possible to carry the pure aniline fraction to a point where only 5% of the charge was left in the still. At this point distillation was stopped and a fresh charge of crude was added without removing the residue. After some five successive charges had been run the residue was stripped, by-passing the fractionating column. A quantity of colored impure aniline was thus obtained amounting to 1% of the total crude charged. This material should be added to a reducer charge because of unreduced material present.

The non-volatile residue was dry powdery material consisting largely of iron oxide and amounting to 0.25% of the crude aniline charged. The net distillation loss was found to be 2% of the aniline charged. Laboratory data on the size of fractions taken off for elimination of water is not considered reliable for use in plant design which should preferably be based on calculated values.

CHARLES M. DAVIS,

Research Chemist.

A.I.CH.E. ANNUAL STUDENT COMPETITION

TARLE II -VAPOR PRESSURE OF ANTIME \*

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| INDEE II. VAIOR I RES | SORE OF TINILINE |
|-----------------------|------------------|
| Temperature ° C.      | Mm. Hg.          |
| 50                    | 2.4              |
| 60                    | 5.7              |
| <i>7</i> 0            | 10.6             |
| 80                    | 18.0             |
| 90                    | 29.2             |
| 100                   | 45.7             |
| 110                   | 69.2             |
| 120                   | 06.6             |

\* International Critical Tables, vol. 3, p. 221. McGraw-Hill.

For values from 145° to 185° calculate from formula—  $\log_{10}p_{mm}=\frac{-0.05223}{T}A+B$  where A=45951, B=8.1278, and T=° C.

TABLE III.—MUTUAL SOLUBILITY NITROBENZENE AND WATER \*

|                  | Weight Per Cent Nitrobenzene         |                                      |  |  |  |  |  |  |  |  |  |
|------------------|--------------------------------------|--------------------------------------|--|--|--|--|--|--|--|--|--|
| Temperature ° C. | Water Saturated<br>with Nitrobenzene | Nitrobenzene<br>Saturated with Water |  |  |  |  |  |  |  |  |  |
| 10               | _                                    | 99.82                                |  |  |  |  |  |  |  |  |  |
| 20               | .19                                  | 99.78                                |  |  |  |  |  |  |  |  |  |
| 30<br>55         | .22                                  | 99.73                                |  |  |  |  |  |  |  |  |  |
| 55               | .27                                  | 99.47                                |  |  |  |  |  |  |  |  |  |
| 100              | .80                                  | 98.60                                |  |  |  |  |  |  |  |  |  |
| 120              | 1.6                                  |                                      |  |  |  |  |  |  |  |  |  |
| 130              | 2.1                                  |                                      |  |  |  |  |  |  |  |  |  |
| 140              | 2.7                                  | -                                    |  |  |  |  |  |  |  |  |  |
| 160              | 4.0<br>5.2                           | 93.0                                 |  |  |  |  |  |  |  |  |  |
| 180              | 5.2                                  | 92.0                                 |  |  |  |  |  |  |  |  |  |
| 200              | 8.2                                  | 88.0                                 |  |  |  |  |  |  |  |  |  |
| 220              | 15.8                                 | 82.0                                 |  |  |  |  |  |  |  |  |  |
| 230              | 24.0                                 | 74.0                                 |  |  |  |  |  |  |  |  |  |
| 235              | 49.0                                 | 49.0                                 |  |  |  |  |  |  |  |  |  |

<sup>\*</sup> International Critical Tables, vol. 3, p. 389, McGraw-Hill.

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## TABLE IV.—TEMPERATURE—SPECIFIC GRAVITY CURVES: ANILINE IN WATER

#### Specific Gravity

| Temperature ° F. | Aniline in Water | Water in Aniline |
|------------------|------------------|------------------|
| 32               | 1,003            | 1.035            |
| 40               | 1.002            | 1.034            |
| 50               | 1.001            | 1.031            |
| 60               | 1.000            | 1.026            |
| 70               | .999             | 1.022            |
| 80               | .998             | 1.017            |
| 90               | .997             | 1.012            |
| 100              | .996             | 1.008            |
| 110              | .994             | 1.003            |
| 120              | .992             | .999             |
| 130              | .989             | .994             |
| 140              | .987             | .989             |
| 150              | .984             | .985             |
| 160              | .982             | .982             |
|                  |                  |                  |

#### Density-Temperature Relation

 $d_t = d_s + 10^{-8}a(t - t_s)$ 

where:

 $d_t = \text{density of material at temperature t}^{\circ} C$ .

 $d_s$  = density of material at temperature  $t_s$  and

| Material     | $d_s$   | а       | t <sub>a</sub> | Range           |
|--------------|---------|---------|----------------|-----------------|
| Nitrobenzene | 1.22300 | 0.98721 | 0° C.          | 0-boiling point |
| Aniline      | 1.03893 | 0.86534 | 0° C.          | 0-boiling point |

Note:  $d_a$  for nitrobenzene is an extrapolated value.

#### Miscellaneous Physical Data

Assume specific heat of aniline constant for all temperatures involved at  $0.52~\mathrm{gram}$  calories per gram.

Latent heat of vaporization of aniline is 103.68 gram calories per gram at its boiling point at 760 mm.

Assume specific heat of nitrobenzene constant for all temperatures at 0.34 gram calories per gram.

Latent heat of vaporization of nitrobenzene is 79.08 gram calories per gram at its boiling point at 760 mm.

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For external pressure, dished heads (convex side) maximum allowable working pressure is 60% of that for heads of the same dimensions with the pressure on the concave side.

Note: Consider internal vacuum as equivalent to external pressure.

## Unit Costs

The figures for basic units given here are the relative installed costs including foundation, immediate supporting steel, etc.

## 1. Tank Units

a. Steel tanks without agitators, per pound of metal in shell, heads and jacket.

| 500  | gal. |   |   |   |   |   |   |   |   |   |   |   |   |   |   |   |   |   |  |  |   |   |   |   |   |   |   |   |   |   | \$0.20 |
|------|------|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|--|--|---|---|---|---|---|---|---|---|---|---|--------|
| 5000 | gal. | • | • | • | • | • | • | • | • | • | • | ٠ | • | • | • | • | ٠ | • |  |  | • | • | ٠ | • | ٠ | ٠ | • | ٠ | • | ٠ | 0.17   |
|      |      |   |   |   |   |   |   |   |   |   |   |   |   |   |   |   |   |   |  |  |   |   |   |   |   |   |   |   |   |   |        |

b. For additional features multiply the above base figure by the following factors and add to the base figure.

| Rubber lined steel 0.8 Copper or bronze 1.5 Haveg 2.0 Glass enameled, stainless steel (18-8), nickel or monel metal 2.5 | ţ | In place of steel |
|---|---|-------------------|
| For agitator and motor drive  |   |                   |

#### 2. Cast Iron Flat Bottom Reducers Including Agitator and Motor Drive

For 10 lbs. per sq. inch internal operating pressure, bottom and lower portion of side wall jacketed for 50 lbs. per sq. in. operating pressure. All reducers are equipped with a six inch discharge outlet extending through the jacket at the bottom of the side wall and they may be equipped with any specified number of cover openings consistent with the size of the cover.

| Nominal Size       | 1000 Gal.    | 1500 Gal.   | 2000 Gal.   | 2500 Gal.   |
|--------------------|--------------|-------------|-------------|-------------|
| Tank diam. x depth | .5'3" x 6'6" | 5'9" x 7'6" | 6'6" x 8'0" | 7'0" x 8'6" |
| Jacketed height    | 2'10"        | 3'3"        | 3'6"        | 3'9"        |
| Installed value    | . \$4500     | \$6000      | \$7000      | \$7800      |

Take heat of reaction liberated in the reduction of nitrobenzene to aniline at 1900 B.t.u. per pound of nitrobenzene charged.

The critical temperature of aniline is 426° C. and the critical pressure is 52.4 atmospheres.

# Maximum Allowable Working Pressure (For tank calculations, etc.)

d = inside diameter in inches

L = straight side length of shell between supports in feet P = maximum allowable working pressure in lbs./sq.in.

gauge  $r_d$  = radius of dish in inches (= diameter of shell for standard dish)

S = maximum allowable working stress in lbs./sq.in. (take as 20% of TS)

t = minimum metal thickness in inches

TS = minimum tensile strength of material in lbs./sq.in.

Values of TS are: steel or enameled steel ... 55,000 copper or bronze ..... 30,000 stainless steel .... 70,000

For internal pressure, cylindrical tanks, shell:

$$P = \frac{2 \times S \times t \times E}{d}$$

where E, the efficiency of longitudinal welded joint,

| for | new tanks is         | 0.80 |
|-----|----------------------|------|
| for | second hand tanks is | 0.70 |

For external pressure, cylindrical tanks, shell:

$$t = k \sqrt{\frac{P \times d \times L}{89600}}$$

where k for steel, enameled steel, stainless steel and monel metal is 1.0 and for copper or bronze is 1.7.

Note: Consider internal vacuum as equivalent to external pressure. For internal pressure, dished heads (concave side):

$$t = \frac{8.33 \times P \times r_d}{2 \times TS \times E}$$

where E for seamless bumped heads = 1.00.

## A.I.CH.E. ANNUAL STUDENT COMPETITION

3. Pump Units Made of Cast Iron with Motor, Installed

| Capacity | 50  | gal. | per | min. | <br>\$600.00 | each |
|----------|-----|------|-----|------|--------------|------|
| Capacity | 200 | gal. | per | min. | <br>700.00   | each |
| Capacity | 500 | gai. | per | min. | <br>800.00   | each |

For alloy construction such as bronze, monel metal, nickel, etc., multiply by 2.0.

## 4. Instruments

For temperature, pressure or depth:

| Po   | pint of Use       |
|--|-------------------|
| Glass factory thermometers and indicating pressure   | ***               |
| Distant indication of temperature, depth or pressure | \$20.00<br>100.00 |
| Recording and control instruments                    | 150.00            |

## 5. Ventilating Equipment with Motor, Installed

Installed cost per cu. ft. per min. of air moved:

| 1000  |     |     |  |  |  |  |  |  |  |  |      | , |  |  | , |  |  | \$0.2 | :5 |
|-------|-----|-----|--|--|--|--|--|--|--|--|------|---|--|--|---|--|--|-------|----|
| 5000  | cu. | ft. |  |  |  |  |  |  |  |  | <br> |   |  |  |   |  |  | 0.1   |    |
| 20000 | cu. | ft. |  |  |  |  |  |  |  |  | <br> |   |  |  |   |  |  | 0.0   | 6  |

#### 6. Vacuum Pumps

The following standard steam jet exhausters are available having capacities as indicated in cu. ft. free air (atmospheric pressure, 60° F.) per minute, exhausted from a system at indicated absolute pressure operating with 75 lb. steam pressure.

a. Single Stage in 3 Sizes. Price, \$250.00 each for any size.

|      |                   |            | Capacity at |         |
|------|-------------------|------------|-------------|---------|
| Size | Steam Consumption | 80 mm.     | 100 mm.     | 200 mm. |
| 1    | 275 lbs. per hr   | 1.5 cu.ft. | 7.5         | 20.0    |
| 2    | 190 lbs. per hr   | 0.5        | 3.0         | 13.0    |
| 3    | 05 the ner hr     | 0.4        | 1 5         | 60      |

b. Two Stage Non-condensing. Price, \$500.00 each for any size.

|             |                   | C      | apacity at        |                   |
|-------------|-------------------|--------|-------------------|-------------------|
| Size        | Steam Consumption | 15 mm. | 25 mm.            | 50 mm.            |
| 4<br>5<br>6 | 305 lbs. per hr   | 0.5    | 2.5<br>1.5<br>0.6 | 4.5<br>3.2<br>1.3 |

c. Three Stage Non-condensing. Price, \$750.00 each for either size.

|        |                   | C                   | apacity at  |             |
|--------|-------------------|---------------------|-------------|-------------|
| Size   | Steam Consumption | 4 mm.               | 6 mm.       | 22 mm.      |
| 7<br>8 | 325 lbs. per hr   | 0.15 cu.ft.<br>0.15 | 0.5<br>0.37 | 1.2<br>0.95 |
| J      |                   |                     |             |             |

#### 7. Heat Exchangers, Steel Shell and Tube Type

On the basis of tubular heat exchange surface:

| 100 | sq. | ft | \$5.00 | per | sq. | ft. |
|-----|-----|----|--------|-----|-----|-----|
| 250 | sq. | ft | 3.00   | per | sq. | It. |
| 500 | sa. | ft | 2.50   | per | sq. | Įτ, |

(For copper or copper alloy multiply the above by 2; for stainless steel, monel metal or nickel by 3.5.)

# 8. For Steel Pipe Coil Heating Surface in Tanks, etc. \$4.00 per sq. ft.

Factors for other metals as under heat exchangers.

#### 9. Fractionating Columns, Steel

|   | 2' diam. | 3' diam. | 4' diam. | 5' diam. |
|---|----------|----------|----------|----------|
| Bubble cap units, per plate                     | \$150.00 | \$240.00 | \$350.00 | \$450.00 |
| Packed column units, per foot of packing height | 80.00    | 150.00   | 220.00   | 300.00   |

#### 10. Filter Presses, Cast Iron

| 100 sq. ft | \$10.00 per sq. ft. filtering area |
|------------|------------------------------------|
| 300 sq. ft | 7.00 per sq. ft. filtering area    |
| 500 sq. ft |                                    |

11. Manufacturing Building Cost-\$0.20 per cu. ft. total volume.

#### 12. Miscellaneous Data

Figure second hand equipment from surplus equipment list at 60% of new installed value unless otherwise stated.

Add 100% to the total of installed costs of basic equipment (not including building) for piping, structural steel platforms, floors and sewers, equipment for handling solid materials and storage of them, electric wiring, lights, etc.

## 674 AMERICAN INSTITUTE OF CHEMICAL ENGINEERS

## ORGANIC CHEMICALS, INC.

St. Louis, Missouri

Engineering Department

November 10, 1941

To: Mr. T. Y. Rowe

## SUBJECT: IDLE EQUIPMENT IN MACHINERY STORES

The following items of used equipment in machinery stores are not on reserve for specified uses and are consequently available for the proposed aniline project. All equipment listed is in sound condition.

## Tubular Heat Exchangers

without baffles.

Steel shell, tubes, and tube sheets; cast iron water ends; without baffles.

| Item | 1   |   | One of 65 sq. ft. tube surface.  |
|------|-----|---|----------------------------------|
| Item | 2 & | 3 | Two of 165 sq. ft. tube surface. |
|      |     |   |                                  |

Item 4 One of 260 sq. ft. tube surface.

Copper shell and tubes, bronze tube sheet; cast iron water ends;

Item 5 One of 300 sq. ft. tube surface.

Motor driven cast iron pumps; 40 ft. head.

Item 6 One of 50 g.p.m. capacity.
Item 7 One of 150 g.p.m. capacity.

Bubble-cap fractionating column, steel, suitable for atmospheric or reduced pressure work at 60% estimated plate efficiency:

Item 8 One 3' diameter, 8 plates on 15" centers.

Chemical stoneware jars, with three 2'' cover openings and one 1'' side opening at bottom.

Item 9 One 100 gal. capacity, reinstalled value \$25 Item 10 One 300 gal. capacity, reinstalled value \$75 For manufacturng cost calculation assume the following:

Depreciation at 20% of capital value per year.

Repairs and maintenance at 5% of capital value per year.

'Power-light-heat and water' exclusive of process steam, at 5% of capital value per year.

Fixed expenses including factory overhead, taxes, insurance, etc., 10% of capital value per year.

Cost of saturated steam at 75 lbs. pressure—\$0.35 per 1000 lbs.

Labor, including labor overhead—\$1.25 per hour

Supervision and analytical control, including overhead—\$3.00 per hour

Maximum summer temperature of cooling water is 85° F. It should be discharged from heat exchangers at a maximum of 140° F.

#### OVERALL HEAT TRANSFER COEFFICIENTS

B.t.u./hr./sq.ft./° F. for Metal Thicknesses in Common Use for Such Equipment as Is Involved in This Problem

#### In steel

|  | Aqueous | Organic        |
|--|---------|----------------|
| Condensing steam to boiling liquid Water to condensing vapor Water to liquid | . 100   | 80<br>75<br>50 |

In copper, 110% of above; nickel, 125%; monel metal, 75%; stainless steel, 60%.

In cast iron reducer

|                            | Steam | w ater |
|----------------------------|-------|--------|
| To agitated reducer charge | 20    | 10     |

#### Radiation from metal

|                                | Uninsulated | Insulated  |
|--------------------------------|-------------|------------|
| Quiet air, as within enclosure | 2           | 1/4<br>1/2 |

RICHARD P. CURTIS,

Development Director

## ALCHE, ANNUAL STUDENT COMPETITION

|  |                                   | A.I.CH.E. ANN  | NUAL STUDI   | ENT COMPETITION   | 675   |
|--|-----------------------------------|--|--|---|---|
| lanks (welded construction and heads internally concave unless otherwise noted). | Comments                          | Cone bottom, flat top. Cone bottom, flat top. Loubber linke steel; with agi- tator, manhole, and three 2 <sup>n</sup> flanged nozzlas in one head; Ly, center flanged nozzla in other head; drive and motor. | Flat bolted cover. Flat bolted cover with inserted agitator, manhole, and three 2" nozzles; drive and motor. | Tank dimension. Jacket dimension. Jacket dimension. Vertical tank; bottom head jacketed; jacket extends 3' up side wall 4" fanged note to populing into tank at center of jacketed head; three fanged note and the fanged note is a fanged not be and the fanged not side the fanged not interpret of jacketed head; three head not please in other head. | THOMAS F. WHITE,  |
| inless other   | Heads<br>Thick-<br>ness<br>Inches | %/\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\   | 5/16   | *****   | 5/16"<br>5/16"<br>14"<br>TH                             |
| concave u  | Dish                              | Sed. ***   | Std.<br>Std.<br>Std.<br>Std.   | 4 5 5 5<br>5 5 5 5<br>5 5 5 5 5<br>5 5 5 5 5 5 5  | SS SS G   |
| internally   | Shell<br>Thick-<br>ness<br>Inches | 5/16"<br>5/16"   | %%%%   | ~~~~~   | 5/16.<br>7/2  |
| and heads  | Straight<br>Side<br>Length        | 5'6"<br>5'0"<br>4'0"   | 9.5.8<br>0.0<br>0.0<br>0.0   | 30,0°,0°,0°,0°,0°,0°,0°,0°,0°,0°,0°,0°,0°   | 5'0"<br>12'0"<br>18'0"                                  |
| construction   | Internal<br>Diamete:              | 20,0<br>20,0<br>20,0<br>20,0   | 044470<br>1044400  | 200 0.00<br>0.00<br>0.00<br>0.00<br>0.00<br>0.00<br>0.00<br>0   | teel 6'6"<br>teel 7'6"<br>teel 9'0"<br>† One head only. |
| ( welded   | Material<br>of Con-<br>struction  | copper<br>steel<br>steel<br>steel  | copper<br>steel<br>steel   | steel<br>steel<br>steel<br>(jacketed)   | N N N   |
| Ianks  | No. of<br>Units                   |  | 2-   |   | 1<br>1<br>1<br>* Depth of cone.                         |
|  | Item<br>No.                       | 12221  | 15<br>17,18<br>19,18   | 82 2  | 22 22 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2                   |