THE CONSTRUCTION OF A FORTY-TRAY BUBBLE CAP DISTILLATION COLUMN

By

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THE CONSTRUCTION OF A FORTY-TRAY

BUBBLE CAP DISTILLATION COLUMN

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Further indebtedness is acknowledged to the Administration of the Oklahoma State University and the Dow Chemical Company for the fellowship grant which enabled the author to carry out this project.

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CHAPTER I

INTRODUCTION

The need for a pilot plant size distillation unit has long been felt in the School of Chemical Engineering at Oklahoma State University.

The unit would need to be versatile enough for research on a wide range of problems of multi-component systems.

Some of the requirements for such a unit would be:

1. To allow the study of both high and low boiling feedstocks.
2. To be large enough to allow operation under actual operating conditions.
3. To allow maximum interchange of feed plates.
4. To allow for interchanging and cleaning of individual plates.
5. To allow maximum variations of feed rates, feed temperatures, reflux rates, reflux temperatures, and bottom boil-up rates.

In September, 1956, Cities Service Oil Company shut down and prepared to dismantle a small chemical plant at Tallant, Oklahoma. The Company offered to donate to the School any equipment from the plant that would be useful. It was learned that a twelve-inch bubble cap column was available from the plant and was thought that with suitable modifications, the column could be made to serve the purposes of the School of Chemical Engineering.

Several trips were made to Tallant by members of the faculty of the School of Chemical Engineering to inspect the column. Although most of the piping and accompanying equipment to the column were found to be badly corroded and unserviceable, it was felt that enough equipment could be salvaged from the rest of the plant to replace the unserviceable items.

In late September of 1956, the dismantling of the column and the preparation for moving the column to Stillwater, Oklahoma was begun.

CHAPTER II

STATEMENT OF THE PROBLEM

The primary object of this project was to provide the School of Chemical Engineering with a pilot plant size distillation unit. The problem resolved to the following:

1. Dismantling of the column and integral equipment at Tallant, Oklahoma.

2. Gathering of substitute equipment for that found to be unserviceable.

3. Moving of all equipment and supporting structures to Stillwater, Oklahoma.

4. Design and construction of suitable foundations for the column, supporting structures, pumps, and tanks.

5. Reassembly and repair of equipment at Stillwater, Oklahoma.

6. Design and construction of suitable instrumentation and piping.

CHAPTER III

PROCEDURE

Dismantling of Column and Supporting Structure

The column as constructed was supported in a two-inch, welded-pipe structure. As a substitute for the existing, welded, pipe frame, a bolted, angle-iron structure, supporting several adjacent columns, was torn down and used. The integral piping and control instruments were removed along with several exchangers and rotameters from other columns in the plant. A 1700-gallon tank with internal steam coils was dismantled along with two other smaller tanks for use as storage and receiving tanks for the reconstructed unit.

In order to remove the column from the supporting structure, it was necessary to break it into seven parts. Each part, thus formed, consisted of three or four flanged sections which, complete with insulation, weighed approximately three hundred pounds. Each part, along with an overhead condenser, was lowered to the back of a truck by the use of a block and tackle; which was rigged to the overhead-crossbar of the supporting structure.

The total weight of the equipment brought to Stillwater, Oklahoma was approximately seven tons. Of this equipment, approximately four tons were later found unserviceable and disposed of as scrap.

Design of Foundation

The site selected for the construction of the Unit is located directly behind and adjacent to the Quonset Building which houses the School of Chemical Engineering at Oklahoma State University.

The foundation was required to support the weight of the twelve-inch column, approximately 2000 pounds, and a supporting structure which weighs approximately 800 pounds.

The soil on which the unit was to be constructed complicated the design of the foundation. Because it is a soft, clay type soil, it is subject to an expansion of one-eighth volume when it is wet. The frost line in the Stillwater area varies from three to five feet below the surface of the ground during seasonal changes of the year. For this reason, any foundation built on this surface would experience a rise and fall of from one to three inches with each change of season, making it very unstable.

Professor Jan J. Tuma, of the School of Civil Engineering at Oklahoma State University, recommended that footings of six feet depth be placed under any foundation built on the surface. At this depth, the soil is permanently wet and the footings would be supporting the surface foundation on stable soil. The designed foundation has five supporting legs which reach to a depth of six feet.

The complete foundation was designed to withstand 100 mile-per-hour winds and for a soil with a load-bearing strength of 5000 pounds per square foot at a six-foot depth.

A set of complete foundation calculations is shown in Appendix B.

A concrete slab, four inches thick, eighteen feet long, and nine feet wide was poured as foundation for the feed, overhead product, and bottom product tanks and pumps. This slab was poured on a two-inch layer of loose sand and gravel. One-half inch reinforcing rods and one-half inch scrap pipe were used as reinforcing in the concrete slab.

A separate foundation was poured for the salt-bath heater because the reboiler weighed approximately 5000 pounds. For this unit, two pillar type foundations were poured. Each was nine inches tall and covered a ground surface of nine square feet.

Construction of Supporting Structure

Since the column to be constructed would, with skirt and overhead condenser, be over twenty-five feet high, some type of supporting structure was required.

A punched, angle-iron type of structure was used. The legs were of 2" x 2" x 3/16" angles, with 1 1/4" x 1 1/4" x 1/8" bracing. The main cross-braces were also of 2" x 2" x 3/16" angles. The four legs were bolted to the concrete foundation that was poured to support the column.

The structure was four feet, six inches from center of leg to center of leg and had an over-all height of twenty-nine feet. The column was centered in the structure by a series of cross-braces.

The overhead condenser was bolted to the top of one of the legs with the vapor inlet four feet above the top of the column.

The main cross-braces at the top were reinforced by bolting a second angle-iron so as to form a channel-iron of the two-angle-irons. A piece of two-inch I-beam, six feet long, was placed across the top of the structure so as to extend one and one-half feet over the side. An eight-inch diameter ring was placed over the I-beam to provide a lifting hook for a block and tackle to be used for assembly and disassembly of the one-foot column sections.

The structure was strong enough to permit hooking a chain hoist in the top ring and using the hoist to lift the entire column and skirt off its foundation.

Reassembly and Repair of Equipment

Before reassembly of the column, the insulation was stripped off and each section was taken apart, cleaned, and inspected. Three of the sections and six plates were found to be badly corroded and were discarded. The overhead condenser was found to be unserviceable and was also discarded as junk.

A skirt for the column was made from a section of twelve-inch, Schedule 40 pipe, three feet long. The skirt was welded to a steel base plate that was thirty inches in diameter and three-fourths of an inch thick. The product "take-off" pipe, from the bottom of the column to product storage, came vertically out of the bottom of the column and out through the side of the skirt. The base plate was bolted to the concrete foundation by six three-quarter inch bolts which were set in the foundation as it was being poured.

The bottom section of the column was revised by the addition of a chimney tray. The chimney tray was installed to trap the liquid that flows down through the column. The liquid is drawn from the chimney tray by a pump and passes through the reboiler and back into the column directly below the chimney tray. The reboiler temperature is adjusted until the desired amount of liquid passes, unvaporized, through the reboiler and can be drawn from the bottom of the column as product.

The column was reassembled, one section at a time, by use of a block and tackle that was hooked to the supporting structure.

The overhead condenser was bolted to a leg of the supporting structure in a vertical position with the vapor inlet about four feet above the top of the column.

The top section of the column was eighteen inches long in order to provide for complete vapor-liquid disengagement. A ROSS, all copper and brass, shell and tube-exchanger, with twenty-five square feet of surface, was used as the overhead condenser in place of the original one which had been scrapped.

The overhead vapors are passed through the shell of the exchanger. The liquid from the condenser is then split, part being returned to the column, and the remainder going to a pump on the ground which pumps to the two receiver tanks as overhead product. Each of the two streams is metered to provide accurate measurement of the reflux to the column and the L/D ratio.

Design of Instrumentation

Since the primary use of the column would be for training purposes in the fundamentals of distillation, the column was not designed for automatic operation. Also, to provide the desired amount of flexibility in the column, instrumentation for automatic control would be very difficult and very expensive. For these reasons, the control and instrumentation of the unit was made as simple as possible. Most of the controls are manually operated.

The flow of the feed to the column is measured by an orifice in the feed line at such a place so as to be easily replaced to change the range of the orifice. The flow of the reflux, bottom take-off, overhead product and bottom circulation rate are also measured by similar orifice plates. The pressure-drop across each orifice is measured by 24-inch mercury manometer. Manual, globe-type control valves were placed in all the lines to control the rates as determined by the orifice meters. Cooling water rates to the overhead condenser and bottom product cooler are controlled by manual globe valves to maintain outlet temperatures as desired. Thermometer wells were placed in the outlet nozzle from the overhead condenser and the bottom product cooler. A liquid level is maintained in the bottom of the column by a control valve in the bottom take-off line. This control valve is actuated by a Foxboro "Differential Pressure Cell". The Foxboro D/P cell measures the liquid head in the bottom of the column and operates the valve to maintain a constant liquid head.

The arrangement of the bottom section of the column with the installed chimney tray can be seen in Figure 1.

The burner on the salt bath heater is controlled by a temperature-indicating controller with the temperature-sensing element extending through the side of the heater into the molten salt. The burner was provided with a second safety controller which shuts off the gas flow if the maximum temperature of the heater is exceeded and the temperature-indicating controller should fail. As an added safety feature, the burner was equipped with a "thermal latch" similar to that found on all home hot-water heaters that automatically shuts off the gas flow if the pilot light was not burning. In case of a fire or spill of the distillate, the gas line to the heater was provided with block valves at the control panel and also inside the Chemical Engineering building. In an emergency the gas-flow to the burner is safely turned off.

Design of Piping and Layout of Equipment

Most of the column accessories, i.e. pumps, preheater, product receivers and feed storage tanks, were placed on a concrete pad about 15 feet from the base of the column. All the orifice meter runs except the reflux flow meter were placed at ground level between the concrete pad and the column structure. The reflux flow meter run was in the vertical section of pipe running from the overhead condenser through the O. H. product withdrawal pump to the product storage tanks on the concrete pad. The reflux ratio is determined by the amount of product withdrawn by the product pump in ratio to the amount of condensate returned to the column.

The bottoms product pump takes suction from the bottom of the column, and pumps through a bottoms cooler and control valve to a set of two receiver tanks, also in parallel. The skirt on the bottom of the tower gives three feet, six inches of head to the bottom pump. The control valve in the bottoms take-off line is actuated by a Foxboro D/P Cell, which controls the liquid level in the bottom section of the column.

The four receiver tanks are piped so that the overhead and bottoms product pumps may be used to return the products from the tanks to the feed tank when the run is completed.

Four receiver tanks were provided to give adequate capacity for holding the charge in the feed tank.

The feed pump takes suction from the feed tank and discharges through a feed pre-heater and orifice to one of three feed plates. The feed may enter the column on the second plate from the bottom, the second plate from the top, or in the center of the column. The feed tank is a copper-walled vessel with a volume of 230 cubic feet. The overhead product tanks have a combined volume of 157 cubic feet and the bottoms product tanks have a combined volume of 183 cubic feet.

CHAPTER IV

DISCUSSION

The use of a direct-fired heater using internal coils immersed in a heat-transfer salt for fractionator reboiler applications is relatively new. The most prominent advantage of this technique being that no high pressure equipment is needed. Temperature up to 800 degrees Fahrenheit may be maintained with only atmospheric pressure on the salt. Other advantages are the simplicity and convenience of this means of high temperature heating. The molten salt bath mixture is a safe, non-volatile, non-corrosive, eutectic mixture of salt compounds. The salt melts at approximately 228 degrees Fahrenheit and is fluid above that temperature. These properties result in excellent heat transfer.

The main disadvantage may lie in the danger of having an open-fired heater only a few feet away from a column that contains flammable organic liquids. This would limit its use in large petroleum refineries and chemical plants where the fire danger is acute. However, this will pose no problem for small isolated operations that involve only a single fractionation operation.

Another disadvantage would be the unfavorable economics of the use of a separate salt bath heater for multiple column fractionations as contrasted to the use of a centrally located boiler with a condensible vapor piped to exchangers on each column. The direct-fired heaters also require a ready supply of natural gas for the burner where a steam boiler, for example, may use practically any available flammable substance.

The use of a chimney tray in the bottom of a column is not new. It is common to find this type of reboiler arrangement in use with thermosyphon reboilers. This type of arrangement has a distinct advantage over other types when the feed to the fractionator is of constant composition and temperature. Once the column operation has steadied out after start-up, the heat input to the column is easily balanced with the temperature in the reboiler. However, once the material has passed through the reboiler, that part which does not vaporize is lost to the system. For a feed that is variable or subject to frequent upsets, this would mean the level of heat input to the column must be varied to maintain the desired fractionation.

Liquid sample ports were provided on every other tray with vapor sampling ports on at least every fourth tray up the column. The reason for ample sampling of the component distribution in the column was to provide information for checking against the IBM 650 Computer. Plans have been made to run a series of known components through the column and to check the product distribution found in the column against that calculated with the IBM 650 Computer. In this way, it is expected that important data on increasing the accuracy of the calculations used in standard IBM 650 Distillation Programs and on new methods of computation will be obtained. Also, it is expected that the sampling ports will be a means for obtaining data which tray efficiencies can be obtained if it is desired to evaluate various type of plates. The column could easily be adapted for quick plate changes from one type to another.

It was planned that the primary use of the column would be for training students in the fundamentals of distillation. Therefore, an attempt was made to design the column for maximum flexibility of operation with automatic controls only where necessary for proper operation of the column.

The feed tank is large enough to provide feed to the column for approximately twelve hours of operation at low L/D ratios. Provisions were made for recycling the overhead and bottoms product to the feed tank if desired, so continuous operation could be maintained. However, it was expected that for a class in unit operations, a run of six to twelve hours is as long as would be practical. A typical class run might be as follows:

1. The desired feed mixture would be mixed in the feed tank.
2. The salt bath heater would be fired up and set approximately at the desired temperature to give the required bottoms product.

3. Cooling water would be started to the overhead condenser and bottoms cooler at this point. Steam would be started to the feed preheater.

4. Since the pumps are all of self-priming type, they could be started and the manual control valves closed.

5. The valve line-up should now be checked to be sure that the column is on total reflux and that the feed will enter on the desired tray.

6. A small feed stream is now allowed to flow to the column through the feed pump.

7. As soon as the column begins to load-up, the reflux ratio should be slowly decreased so as to pull off a small amount of overhead product. Also, the bottoms temperature in the salt bath heater should be adjusted so as to give a small amount of bottoms product. The feed should be slowly increased as the overhead and bottoms take-offs are increased. If it is desired to operate at a fixed reflux ratio, it should now be set and the column allowed to come to steady operating conditions.

8. Since there are no automatic controls on the column, it will be necessary to continuously adjust the feed rate, feed temperature and reboiler temperature until the column lines out. As soon as the column is stabilized, the desired samples can be taken and the column shut down.

This outlines a typical run to determine overhead and bottoms product compositions at a set reflux ratio for a certain feed mixture. However, since the column was designed to operate under a variety of conditions it would be possible to run under an almost infinite number of problem conditions.

CHAPTER V

SUMMARY

This project was concerned with the construction of a forty-tray, twelve-inch diameter, distillation column.

The column and accessories were moved from the Cities Service Oil Company chemical plant at Tallant, Oklahoma upon its shut-down in September, 1956. Upon dismantling, most of the component exchangers and piping were found to be completely unserviceable and had to be replaced by equipment salvaged elsewhere in the Tallant plant.

The entire unit was moved to the School of Chemical Engineering of Oklahoma State University at Stillwater, Oklahoma. There it was repaired, cleaned and reassembled on prepared foundations. The piping for utilities was run and a "salt-bath" heater, for use as a reboiler, was moved in.

The column, as constructed, contained thirty-four bubble-cap trays on a six-inch tray spacing. Each plate contained three standard three and one-half inch bubble-caps.

A supporting structure was provided to support the column and overhead-condenser and to allow for easy dismantling of the column.

The unit was complete with a 1700-gallon feed-storage tank, and all necessary exchangers, pumps and tanks for continuous operation. Since it is expected that the primary use of the column will be for instruction in a Unit Operations class, an attempt was made to design and layout the equipment for easy access for inspection and data collecting.

As the column is easily adapted for making quick plate changes, it is expected that the sampling ports will be used to give data for calculating and comparing plate efficiencies for various types of plates.

It has also been planned to use data from the column, when operating on a known system, to compare with the data for improving existing programs set up for the computer and the testing of new programs.

Future Work

The following expansion and modification of the project carried out thus far may be suggested:

1. Operation of the column using a known system of components as feed to study the operating characteristics of the unit.

2. Increasing of tray spacing to twelve inches by the removal of every other plate.

3. Substitution of various types of trays to obtain a comparison of tray characteristics under similar operating conditions. Once the riveted plates are removed, it will be relatively easy to change from one type of plate to another.

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APPENDIX A

Auxiliary Equipment Specifications

1. Reboiler:
   Black, Sivalls and Bryson model 70S2-SBH Salt Bath Heater, rated at one-million BTU hr, direct gas fired and containing 2800 pounds of Du Pont HY-TEC heat-transfer salt.

2. Overhead Condenser:
   Ross, BCF 603 all copper and brass exchanger with inside packed floating head, two passes on the tube side, and one pass on the shell side. The exchanger contains 116 tubes, 5/8 inch in diameter, and 31.5 inches long.

3. Bottom Product Cooler:
   Ross, BCF 300-8 all copper and brass exchanger single pass on the tube side and single pass on the shell side. The exchanger contains 72 tubes 5/8 inch in diameter and 12 3/4 inches long.

4. Feed Preheater:
   The feed preheater consisted of a cylindrical shell, 14 inches in diameter and 3 1/2 feet long, containing 190 feet of 3/4 inch copper tubing, bent in a square winding, and having an area of 37.4 square feet. An inlet was provided for introducing steam to the shell with a steam trap outlet on the bottom of the shell.

5. Feed Pump:
   A.C. Pump, Type SS, with 1 H.P. motor, rated capacity of 30 G.P.M. at 35 feet of head.

6. Bottoms Pump:
   Magna Flux Corp., Model 25, with 1/4 H.P. motor, rated capacity of 48 G.P.M. at 4.6 feet head, and 40 G.P.M. at 9.2 feet head.

APPENDIX A - continued

7. Overhead Product Pump:
   Reulands Electric Corp., with 1/4 H.P. motor; rated capacity of 30 G.P.M. at 10 feet of head.

8. Feed Storage Tank:
   A horizontal, cylindrical tank, six feet in diameter and eight feet long, was used as a feed tank. The tank had a volume of 1730 gallons.

9. Overhead Product Tank:
   Two vertical, cylindrical tanks were used to receive the overhead product. The combined volume of the two tanks was 1180 gallons.

10. Bottoms Product Tank:
    Two vertical, cylindrical tanks were used to receive the bottoms product. The tanks had a combined volume of 1370 gallons.

11. Liquid Level Controller:
    A Foxboro D/P Cell was used in conjunction with a flow controller to maintain a constant level in the bottom of the column. This D/P Cell was a Foxboro, Type 20, with a range of 20 inches of water.

APPENDIX B

Calculation of Column Foundation

Method of design from Petroleum Refining, April, 1943.

Maximum wind: 100 m.p.h.

Maximum soil bearing pressure: 5000 p.s.f.

Tower diameter: 16 inches (allowing for a two-inch coating of insulation).

Tower weight: 2100 pounds (with insulation).

Soil Contact Surface of foundation: 4.22 square feet.

A. Wind pressure on column surface is computed from wind velocity.

$$\text{Wind Pressure} = p = 0.0025\ V^2$$

where V = wind velocity

$$p = 0.0025\ (100)^2 = 25 \text{ p.s.f.}$$

B. Wind load

$$\text{Wind Load} = P = pDH$$

where D = effective diameter of column

where H = height of column exposed to wind

$$P = (25)\ (16/12)\ (25) = 835 \text{ lbs}$$

C. Overturning Moment

$$\text{Overturning Moment} = Mo = Ph$$

where h = lever arm from midpoint of column to base

$$Mo = (835)\ (12.5) = 10{,}430 \text{ lb-feet.}$$

APPENDIX B - continued

D. Estimate weight of column and foundation.

Column =	2100 pounds
Supporting Structure =	800
Foundation =	4410
	7310 pounds

E. Calculate Safety Factor

$$S.f. = \frac{Ms}{Mo}$$

where Ms = moment of stability and is equal to RW

R = shortest radius of foundation

W = weight of column and foundation

Ms = (2.5) (7310) = 18,300

$$S.f. = \frac{18,300}{10,430} = 1.75$$

F. Check to see maximum soil bearing pressure is not exceeded.

$$\frac{Ms}{A} = \text{force on ground}$$

$$\frac{18,300}{4.22}$$

$$= 4,330 \text{ lbs/ft}^2$$

APPENDIX C

PHOTOGRAPHS AND DRAWINGS

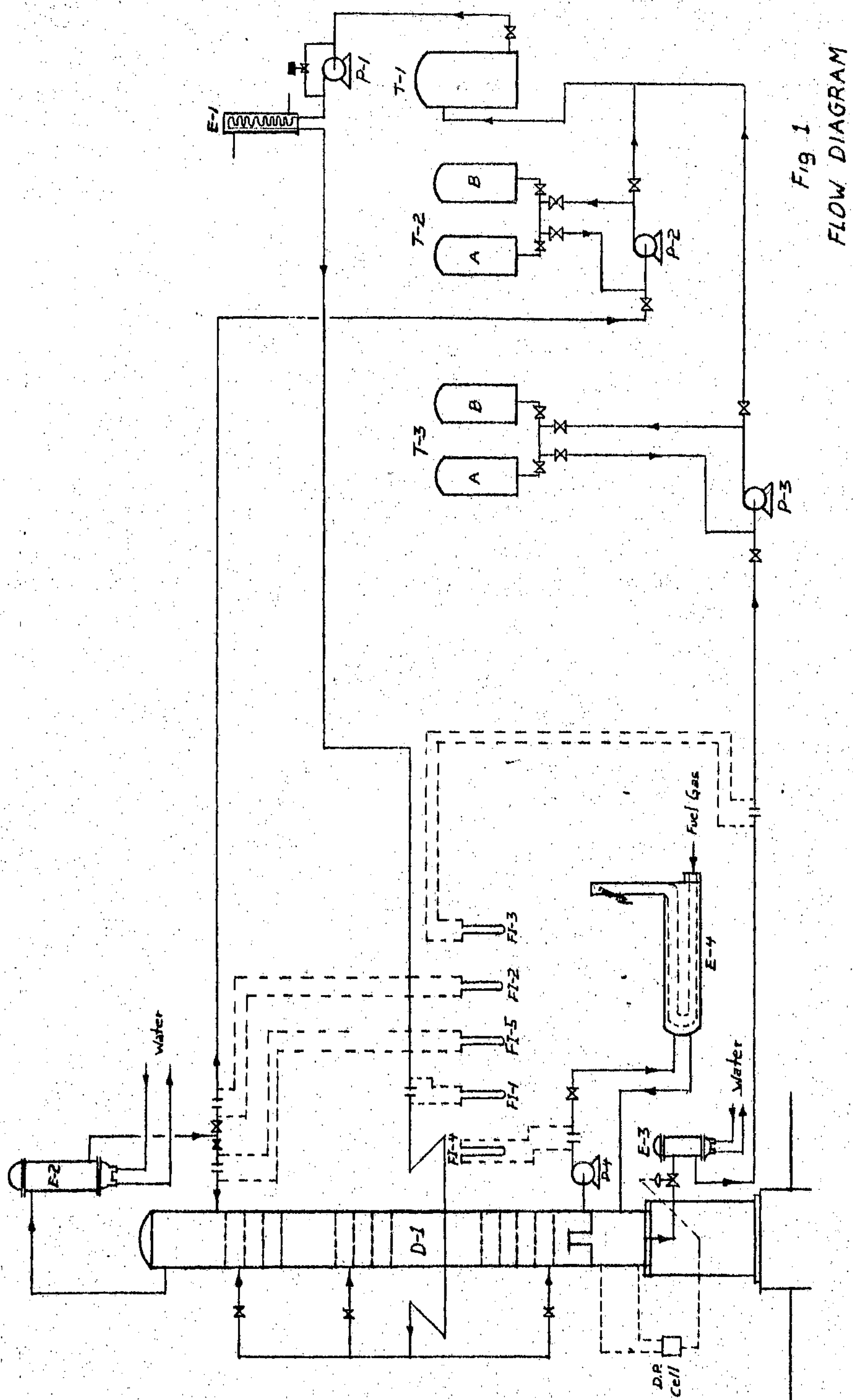


Figure 1. Flow Diagram

Figure 2. Typical Bubble Cap Plate

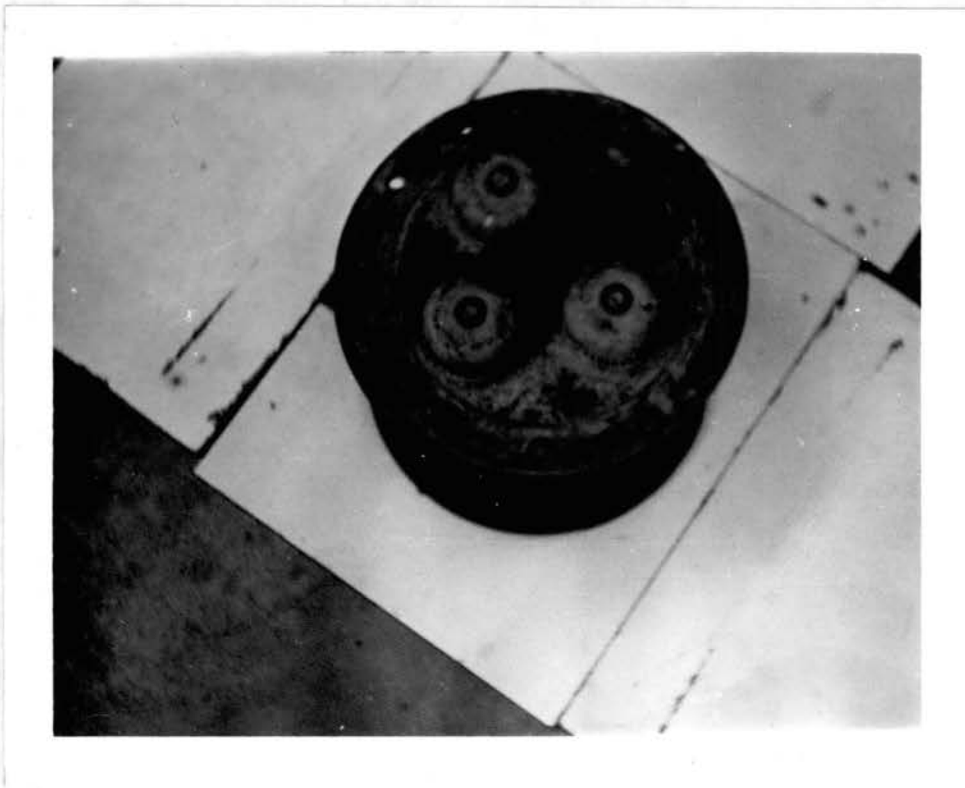
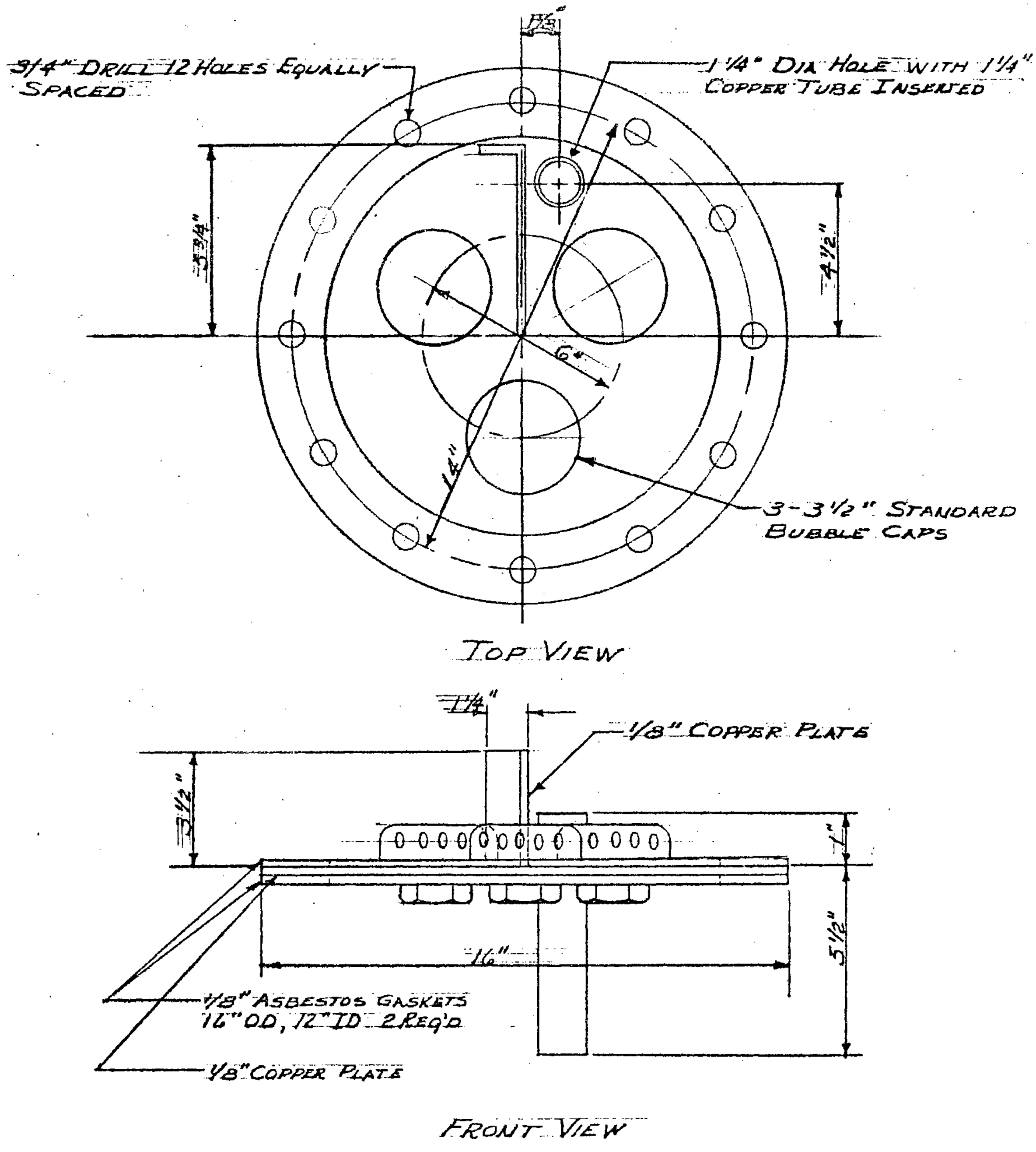
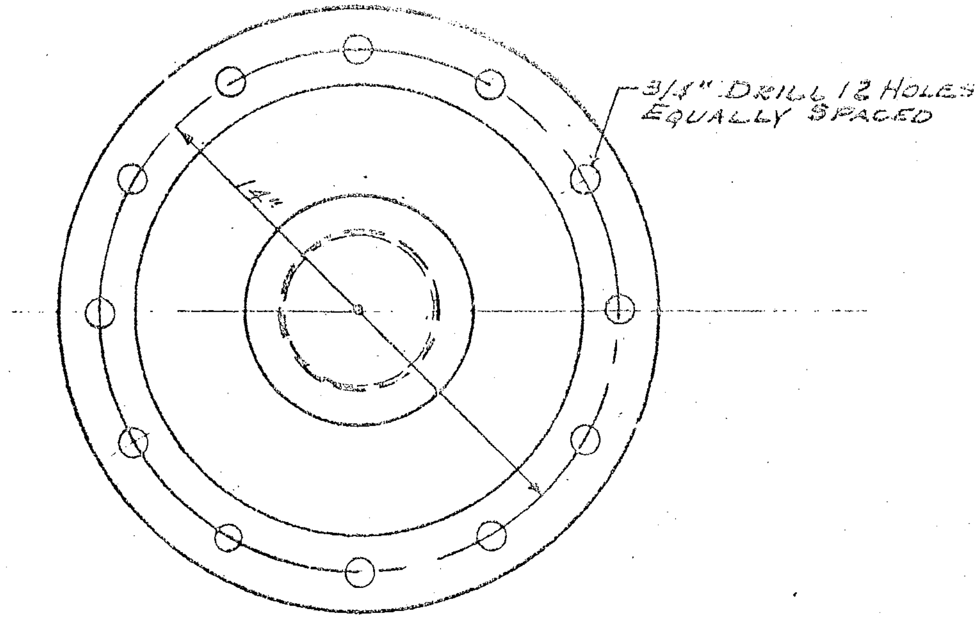


Figure 3. Internal Riveted Plate (midway in section)

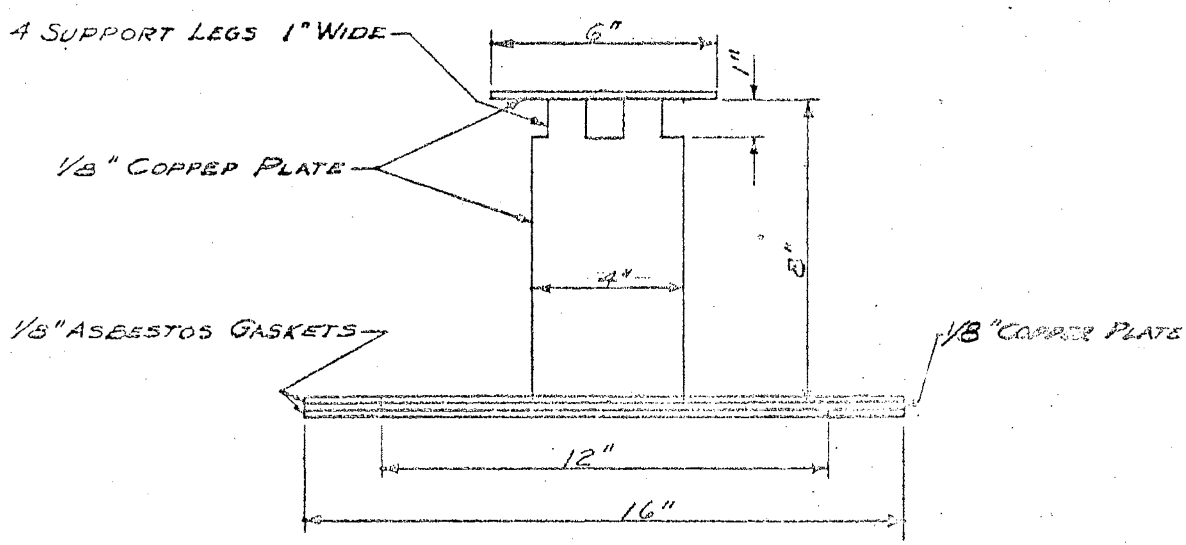


Bubble Cap Tray Details

Figure 4



TOP VIEW



FRONT VIEW

CHIMNEY TRAY DETAILS

FIGURE 5

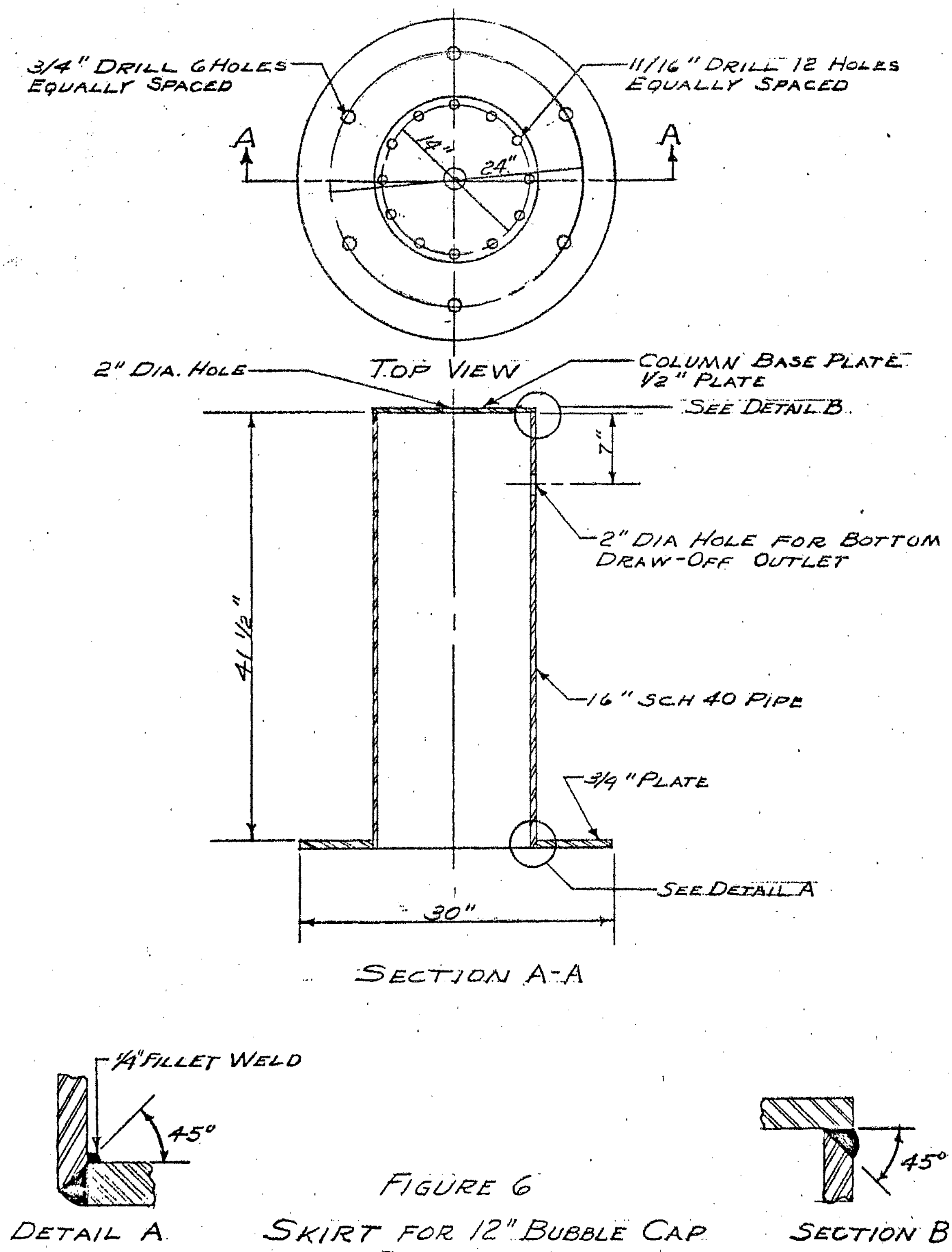


FIGURE 6

SKIRT FOR 12" BUBBLE CAP COLUMN

MAT'L: CARBON STEEL

VITA

Tommy Edwin Graham

Candidate for the Degree of

Master of Science

Thesis: THE CONSTRUCTION OF A 40-TRAY BUBBLE-CAP DISTILLATION COLUMN

Major Field: Chemical Engineering

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Education: Attended Central State Demonstration school in Edmond, Oklahoma; graduated from the Britton High School; received his Bachelor of Science Degree from the Oklahoma State University, with a major in Chemical Engineering, in January, 1956; completed the requirements for the Master of Science Degree in 1960.

Experience: While attending Oklahoma State University, worked Summers with The Texas Company, Robberson Steel Company and Dow Chemical Company. He served six months active duty in the Army and, upon discharge in August, 1957, went to work for Monsanto Chemical Company, Texas City, Texas. He is now serving as a Research Engineer in the Pilot Plant at Texas City.