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Ferrelve Corporation
Technical Division

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Manual of Operations for the S-50 Plant (U)

June, 1945

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Reported by: Ralph T. Overman
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Arvin D. Quist 8/28/95
Technical Information Officer Date
Oak Ridge K-25 Site (B)



FOREWORD

It is recognized that the S-50 plant operations are far from standardized and therefore any manual will require continual revision to keep it up to date. Even during the period of writing this first edition, many changes occurred and were incorporated in the manual. Such revisions could delay indefinitely the issuance of the manual and it was felt that in its present form it could be useful in the training of new plant personnel. The undersigned was engaged in extensive editing of the manual when it became apparent that all the revisions he would like to make would delay unduly even the issuing of this first edition. It is therefore released at this time solely for use in connection with the training program.

Barnett F. Dodge
Technical Director



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MANUAL OF OPERATIONS

THE FERCLEVE CORPORATION

Section A. General Information

1. The Purpose of the Process

The specific purpose of the process employed by the Fercleve Corporation in the plant at Oak Ridge, Tennessee, is one of secret information and cannot be released here. It can be stated, however, that the plant operation involves the processing of a chemical substance. This substance is not identified in the plant or in the process but is referred to only as "material". Its code designation is C-616. The process in general involves the separation of the raw material into constituents with more desirable properties. It is not necessary to know the name of the material nor the exact nature of the process in order to carry out the operations so this information will not be given in this Manual.

The problem involved is to transport the material from the containers in which it is received by the plant into the equipment set-up in the plant for carrying out the operation, to remove the various products from the process equipment and to do other auxiliary operations.

2. The Plan of the Process Building

The S-50 Area consists of one principal building, called the process building with the following subsidiary buildings:

- (1) Pump House
- (2) Water Treatment Building
- (3) Power House
- (4) Laboratory No. 1
- (5) Laboratory No. 2
- (6) Fercleve Administration Office
- (7) Warehouse, Maintenance and Machine Shop
- (8) Time Office and Cafeteria
- (9) Change House

The process building contains 21 identical operating units called racks. Therefore, if one has an understanding of the function and operation of one of these racks with its auxiliary equipment, he will understand the whole operation of all of them. These 21 racks are arranged in groups of 7 which are designated by the term sections. Each of the sections is supplied with steam by a main steam line referred to as SF-1, SF-2, and SF-3. The sections are numbered according to this designation. Thus racks 1-7 are supplied by SF-3, racks 8-14 are supplied by SF-2 and racks 15-21 are supplied by SF-1. The sections are identical in operation but are merely arranged in this manner to simplify the distribution of steam to the racks. The floor plan of the process building is shown in Dwg. 9-1.

For each 2 racks there is a transfer room located on the first level above the floor along the west side of the building. Since there is an odd number of racks, rack 21 is served by one transfer room. This rack is considered to be an experimental rack; it and its transfer and control rooms are under the direction of the Technical Division.

For each pair of racks there is also one control room, making a total of 11 control rooms. These rooms are located next to the transfer rooms on the mezzanine level.

The process building consists then primarily of these racks, control and transfer rooms with the rest of the equipment being used to supply steam, water and air to the systems. These systems will be described in detail in other parts of this section.

3. Plan of Material System

Each of the racks in the process building consists of 102 columns arranged in rows of 51 columns each. Each of the columns is similar, in that it consists of concentric pipes through which is passed steam, material and water. The material is passed into the second of the three concentric pipes. Thus, the material has steam on one side of it in the center pipe and water on the outside in the outer pipe.

The material channels of the columns are then connected together with small diameter nickel tubing at the bottoms so that the columns are essentially a series of standpipes in a line. The material is passed from the scale tanks in the transfer room into the rack through nickel tubes called conduits encased by a steam jacket. The storage tanks are located on the second level in the transfer rooms. In the transfer room also are located the shipping tanks in which the material is brought into the plant and in which it is removed.

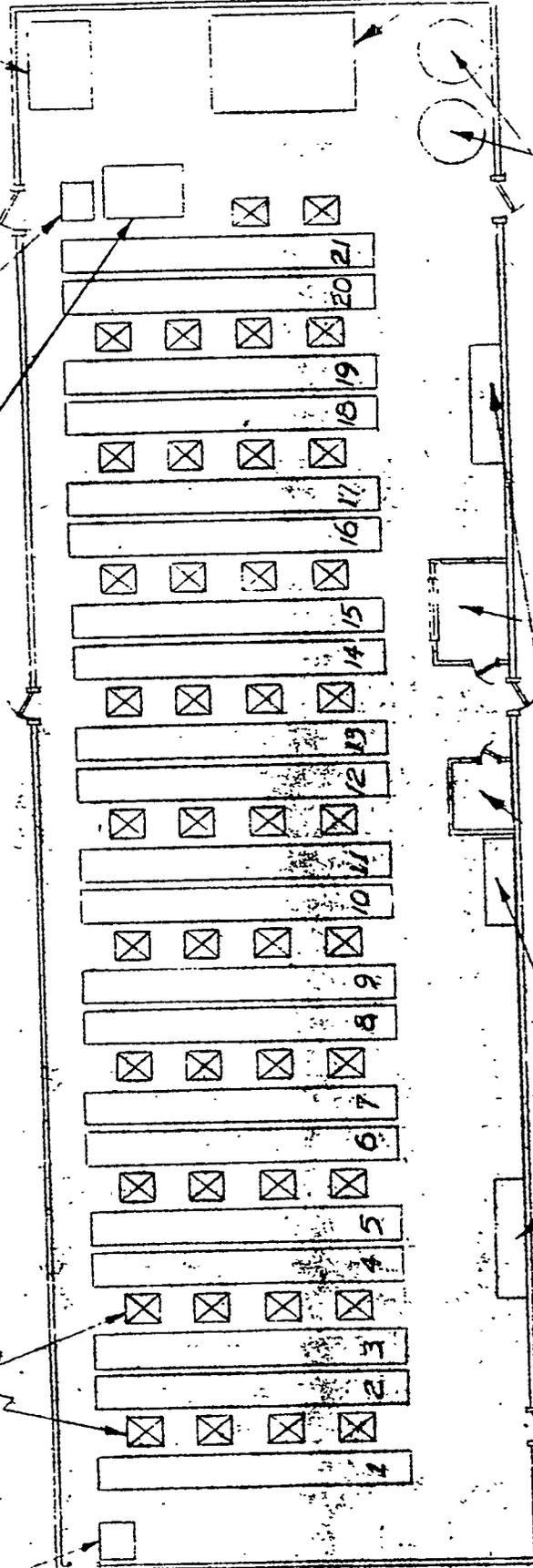
General Layout

Pacific Centrifugal Machine for Circulating Water

Blow-down Tanks 1000 lb Steam Cond.

Electrical

Elevator



Water Filters

Steam Pumps
&
Desuperheaters

Electrical Control Boards

Maintenance Office

Plant Operations Office

THE FERGLIVE CORP
 Technical Divn.
 FLOOR PLAN
 Process Building

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-3-

The Process. The operation of the plant is along a simple cycle of circulating the process material from a scale tank in the transfer unit through a column to a scale tank. It passes across the bottom of a column of material and back through another column into the scale tank. The material in the column exchanges with material in the column where the desired process takes place. At the top of the column the product is removed by one of several product withdrawal methods. After a certain period of time in which the material is circulated along the bottom of the column and material taken from the top of the column, the scale tank is emptied of depleted material while the rack is connected to a new scale tank so that the process in the column is continued.

Circulation. The actual process takes place in the column is not dependent on the circulation of the material through the bottom of the column. However, it is quite necessary for each particle to interchange with the material in the column. The material in the column is a nearly inert solid. Consequently, the only way material moves in the material treatment is the action of circulation. The material must be free to pass from the scale tank, through the column and back into the scale tank. This is the method for replenishing the supply of material in the column. The system is represented schematically in Fig. 3-2.

4. Properties of the Process Material

The chemical identity of the process material used in the 3-50 Area, the product produced, the quantities used and the nature of the process data are entirely secret and cannot be disclosed here. However, a knowledge of the properties of the fluid is essential for efficient plant operation; hence the following information is given to facilitate an understanding of the plant equipment and techniques.

Material. The primary material used in this process, as stated before, goes by code designation, 1-616. It is usually called process fluid or material. Throughout the plant and in this manual the term material always refers to this particular substance. The following statements give a few of the mechanical and physical properties of the substance:

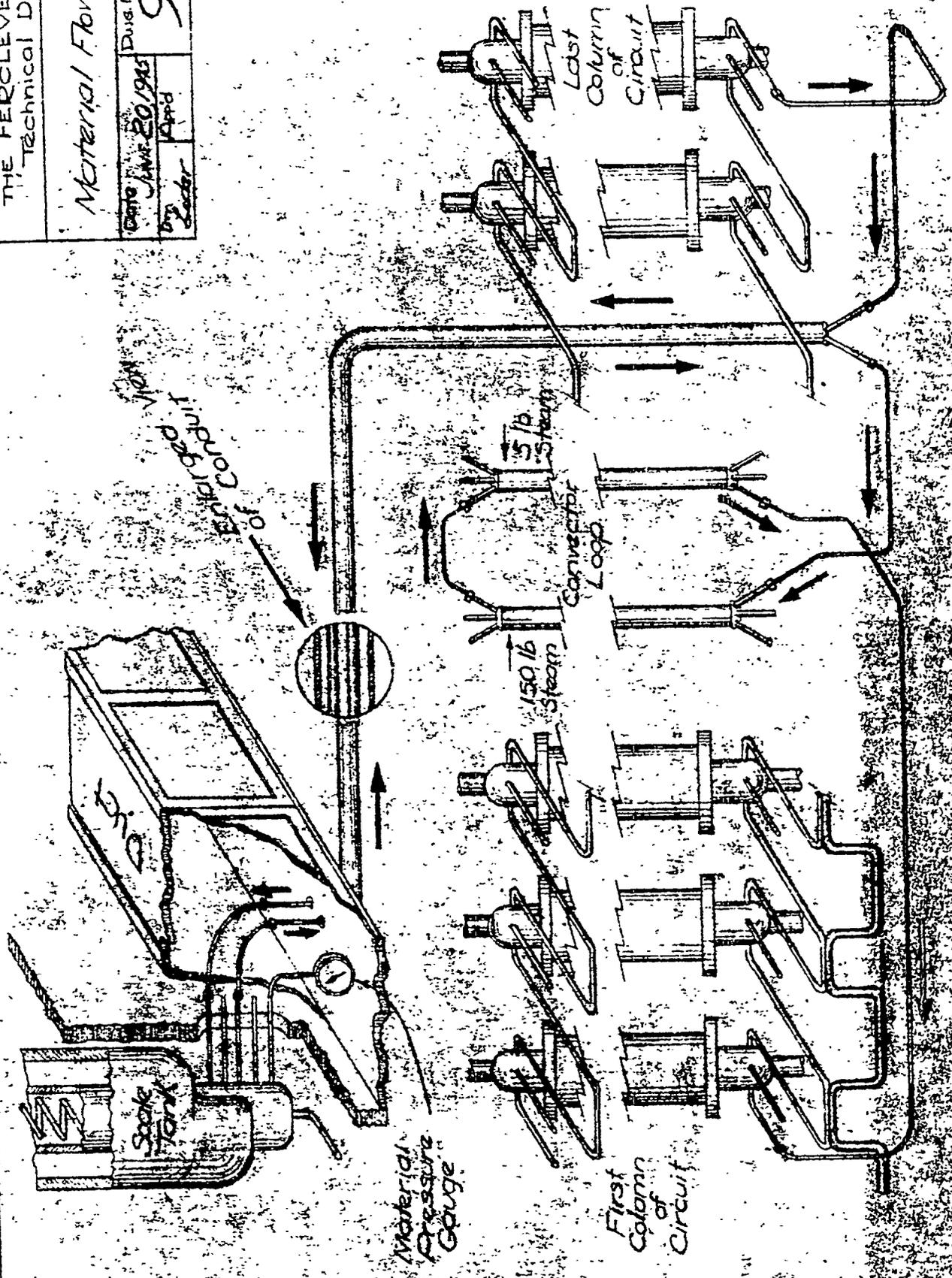
a. Physical State. The material is a white crystalline solid at room temperature. This property explains why all process lines must be heated in some way to keep the material in the liquid state. All ducts and conduits are steam heated.

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THE FERGEEVE CORP.
Technical Divn.

Material Flow System

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b. Melting Point. The material sublimes (changes directly from the solid to the gaseous state) at 57°C . at atmospheric pressure. Thus, if a tube is uncapped and the material exposed to the atmosphere, it will vaporize. If the tube is heated with the cap on so as to develop pressure, it will melt at 64°C . under a pressure of one and one-half atmospheres. Under the operating pressure used in the process, it melts at about 70°C . Therefore, all points in the process system must be maintained at temperatures higher than 70°C . so that plugs of solidified material will not be formed.

c. Critical Constants and State in the Process. The material has a critical temperature of about 232°C . and a critical pressure of approximately 700 p.s.i. The operating temperature of the process fluid in the columns lies between 270°C . on the hot side and 70°C . on the cold side; however, because of the high operating pressure, 1500 p.s.i., there is no separation of the process fluid into two states, the gaseous and liquid states. On the hot side the density of the material is more like that of a liquid than gas, yet its compressibility is much greater than that of a perfect gas. On the cold side, on the other hand, both the density and compressibility are characteristic of the liquid state. The process fluid, therefore, can be visualized on the cold side of the column as being a liquid which gradually and imperceptibly takes on some of the properties of gases as the hot wall is approached, but which never separates into two phases or states anywhere in the system as long as the high pressure of 1500 p.s.i. is maintained.

The process fluid in the scale tank is kept normally at temperatures above the critical temperature (the critical temperature is commonly defined as the temperature above which it is impossible to liquefy a gas regardless of the pressure); under this condition and the condition of high pressure it will expand rapidly when the pressure is suddenly released, and is to be regarded, therefore, as a gas under high pressure. When the temperature of the scale tank is reduced below the critical temperature, there will be no condensation of the material to the liquid state or separation of the material into a liquid and gaseous phase because of the high operating pressure. In other words, below the critical temperature no vapor of the liquid can exist because the high pressure would immediately compress it to the liquid state. The process fluid in the circulating system is always kept at pressures above the critical pressure, (the critical pressure can be thought of as the maximum vapor pressure of the liquid.)

d. Reaction with Water. The material reacts rapidly with water to produce a hard, yellow, non-volatile solid. For this reason it is extremely important to prevent water and air from coming into contact with process material. Fortunately, the hydrolysis product is very soluble in water which makes it easily removed from short link lines and small equipment.

Water must be kept out of the process system at all costs, as small amounts of water will react with the process fluid producing plugs or lumps of solid matter which quickly plug up the narrow diameter link lines and tits, thus completely stopping circulation. This is the reason why elaborate precautions are taken to dry completely all equipment before introducing material into it. The material will also react with the moisture in the air producing a characteristic dense, usually white fog and smoke. This smoke is usually evidence of a leak in the system as very small leaks produce great quantities of smoke. The smoke will be noticed occasionally when operators are taking samples from the columns. Since air contains moisture, solid material in cooled tits should be exposed to the air the minimum time necessary in changing link lines or taking samples.

e. Inflammability. The material is non-inflammable. It will impart a yellow color to a gas flame, however, this property is useful in testing for leaks. The yellow color results from the heating of solid smoke particles to incandescence.

f. Chemical Activity. In addition to its affinity for water, the process fluid reacts with organic compounds; hence oil and grease should not be allowed to come into contact with the fluid; otherwise the circulating system will plug as mentioned above in the case of water. All columns, gauges and link lines are cleaned with a grease solvent before use and vacuum pumps, which contain oil, are protected from material fumes by dry ice and charcoal traps.

g. Reaction with Metals. The material will react with most metals. Nickel is the most satisfactory from a corrosion standpoint and for this reason most of the equipment in the plant is made of pure nickel. Copper is also fairly good and is used. The high pressure material gauges used in the plant are made in part of beryllium-copper alloy. Steel should be avoided since its corrosion by the process fluid is relatively rapid.

h. Stability. The material is a very stable compound. It can be kept for long periods at high temperatures in contact with nickel without decomposition.

i. Density. The density of solid material is 4.68 g/cc. (292 lbs/cu ft.) at its melting point. The liquid at the melting point has a density of 3.7 g/cc. Thus there is a volume increase of 35% in going from the solid to the liquid state, a change in volume which is abnormally high. This fact must be remembered since it has an important bearing on many operations. Thus if one has a tube full of solid material and wishes to thaw the material with a burner, the tube would have to be uncapped on one end and heated very slowly at the uncapped end so that the material as it melts would have sufficient volume to expand into without creating excessive pressures. If it is being heated with steam, the heating must be begun by starting very slowly with warm water, making sure that the material is free to expand in both directions. Failure to observe these precautions has caused some serious accidents in the plant. There are other important

applications of this density property which are discussed in another section.

j. Vapor Pressure. The vapor pressure of the material at room temperature is about 100 mm. of mercury which explains the ability of the material to fume in air. It increases to 760 mm. at 54°C.

k. Safety. The material is poisonous, but does not have a high toxicity. Although the material itself or its vapors quickly produce skin burns, copious quantities of water promptly applied to exposed areas can make this hazard negligible. Material vapors or the smoke produced by reaction of the material in the moisture should not be inhaled; otherwise, lung irritation may be produced.

As the equipment has been designed and built to withstand the normal operating conditions of temperature and pressure, there is little hazard in working about the racks. In the transfer room the operators, however, have to work more carefully and take greater precautions because of the relatively large volume of highly compressed process gas in the scale tanks. Material hazards in transfer rooms arise only when the weight of material in each tank rises above 420 pounds.

l. Valves. It has been mentioned above that the material is very corrosive. This is one reason why metal valves are not used for the control of the material in the system. Development work on the valve, however, is being carried out. In the present system valves are formed by freezing the process fluid at the desired point, thus forming a plug of solid material. This freezing is accomplished by the use of dry ice at some points, and by cooling water flowing through pipes soldered to the nickel lines at others.

m. Pressure-volume-temperature relations. The material behaves quite like other gases in these relationships. The gas demonstrates essentially the same abnormalities existing in other heavy gases particularly near the critical point. Thus in this region there are large volume changes for very slight changes in pressure at constant temperature. Attached is a set of curves illustrating the pressure-temperature relations at several constant volumes. A modified form of this chart is in all of the transfer rooms for ready reference in working with the material. Fig. 9-3 shows the relationship between steam pressure on the scale tank and the material pressure developed in an average size scale tank of 77 liters volume.

The vapor pressure of the pure solid increases uniformly with temperature up to its melting point at 54°C. and the liquid exerts an increasing vapor pressure as the temperature increases. At 232°C in a closed system the pressure will be about 700 pounds per square inch. Above that temperature, the entire system becomes a single fluid state and a further rise in temperature merely increases the pressure of the system, roughly in conformity with Charles' law. If the volume of the original system is not sufficient to accommodate all of the fluid as it melts, the container may fill entirely with liquid due to the 35% expansion on melting. In this case, increase of temperature causes further liquid expansion and an increase of pressure far greater than that due only to the vapor pressure of the material.

Property of expansibility and rapid change of volume with pressure has a direct bearing on plant operations. The material is charged to a tank on a scale in weighed amounts and heated with steam of varying pressure up to 1000 p.s.i. If the container is filled with 420 pounds of material, the pressure will reach the neighborhood of 2500 pounds per square inch when the tank is heated with 1000 pounds pressure of steam. This corresponds to a temperature of steam of 545°F. If, however, additional material is put in the scale tank, the pressure can rise to much higher values when heated with 1000 pounds steam. Pressures greater than 2500 pounds per square inch are not considered in the range of safe operating pressures. The data for material pressure as a function of weights and temperature are given in the Section on the Transfer Room.

n. Properties of "Fresh Air". Another material used in the process building is identified by the name "fresh air" which has the code designation G-216. It is used in the conditioning process in which equipment is made ready for use but it is not actually used in the process.

Fresh air is supplied to the plant in large nickel cylinders at a pressure of about 40 pounds per square inch. It has a very distinctive and penetrating odor and can be detected in small amounts. The cylinders are painted grey with yellow bands. There are three cylinders storing the fresh air placed outside of the process building on the west side. The gas is piped in through copper tubing to the various transfer rooms from which it can be distributed further.

Fresh air is very active chemically, more so even than the process material. It will combine with practically all materials vigorously. Consequently, it is necessary to eliminate all leaks in the fresh air piping system. Leaks can be tested for by holding a burner through which illuminating gas is flowing but which is not ignited near the point in question. Even a small leak will usually cause the burner

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to ignite spontaneously. A small leak will also cause a lighted burner flame to appear bluish-violet in color. Also, since the gas is an active oxidizing agent, starch-iodide paper can be used as a test. Another test is performed by bringing an aqueous solution of ammonium hydroxide into the vicinity of the suspected leak. If fresh air is present, clouds of white smoke will develop.

Fresh air produces corrosive burns on the skin. The best remedy is to apply large quantities of water. It will also affect the respiratory system, but it gives definite warning of its presence by its odor. Because it reacts with water, oil or oxide surface films and because it forms an inert corrosion resisting film on the surface of nickel, it is used to pre-test or "condition" equipment before introduction of the process material.

5. The Plan of the Water System

The water system may be divided into three separate systems: (1) Circulating water for columns. (2) Freeze-off water for inter-column connectors and (3) Gland water for desuperheater pumps.

Circulating Water System. Circulating water is supplied to the jackets of all columns at a temperature of about 150°F. In removing heat from the columns, its temperature rises to about 160°F. A continuous supply of this water with accurate temperature control is absolutely essential to the operation of the plant.

Two Pacific centrifugal pumps, with a capacity of 10,000 g.p.m. each are provided for each rack. They are arranged in groups of four pumps, located between each pair of racks and arranged with manifold systems, so that the pumps can be interchanged between racks in case of breakdown. These pumps discharge into a common header which in turn delivers water through a rubber hose connector to the bottom of the water jacket on the column. The discharge water from the column jackets passes through rubber hose connectors to a discharge header and then returns to the suction side of the circulating pump. At the high point in the discharge line from each column a small vent line is connected which connects into a common header for the rack and then vents through the roof of the building. The pressure on the suction side of these pumps is normally about 25 p.s.i. and on the discharge side about 30 p.s.i.

Since the temperature in a closed circulating system would soon exceed the required upper limit of 160°F. it is necessary to add cold make-up water continuously. This make-up water is supplied by the pump house which contains five Pacific vertical pumps with a capacity of 15,000 g.p.m. each. The water supply for the pump house is in turn supplied by power house pumps which take raw water from the river and deliver to a sump at the pump house. This supply is so adjusted that excess water is continuously flowing over a weir on the outside of the pump house. Under these conditions, the sump contains about 29.6 feet of water, which should be sufficient for approximately 40 minutes operation, at full capacity in case of water failure. Constant watch must be kept over the water supply.

The discharge from the 15,000 g.p.m. vertical pumps goes into a header which passes underground to the process building and then branches off to the individual racks. The raw make-up water feeds through a control valve on the suction side of the circulating water system. This control valve is operated by a temperature recorder and controller just outside the transfer room. The controller is set to maintain a temperature in the suction side of the circulating water system of 150°F. If the temperature rises, the valve opens and admits more make-up water. If the temperature drops, the valve closes. Pressure of the make-up water is usually between 60-80 pounds. Since make-up water is added continuously, it is necessary to vent water from the system continuously. A line branches off the discharge header at the top of the racks on the east side of the building and conducts the water to the sewer.

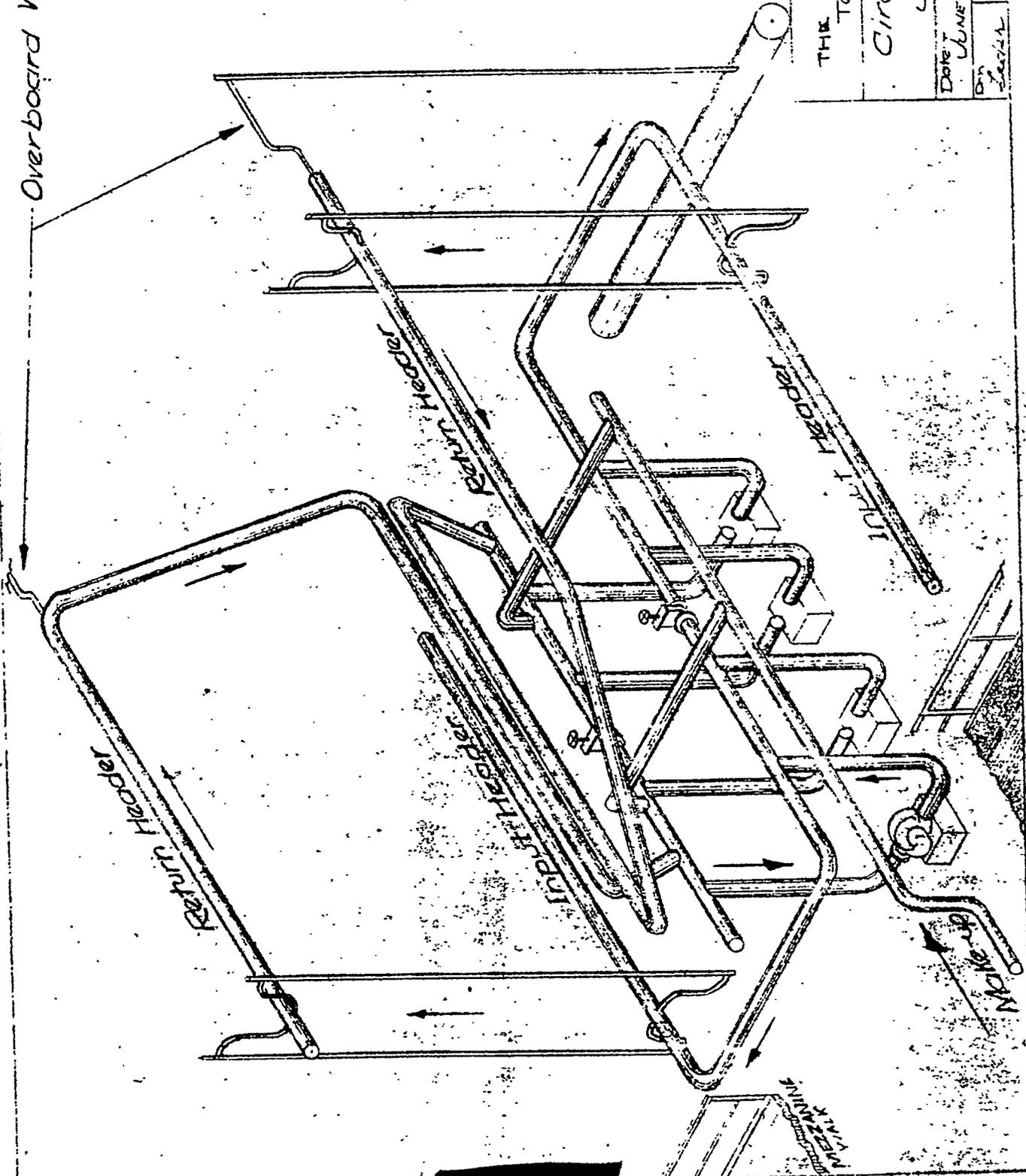
The hose connecting individual columns to the inlet and outlet headers have caused trouble in the past by slipping off the pipe during service. Since it is imperative to keep water in the circulating system at all costs, a special operating procedure is included in another section to cover this emergency. In general, it consists in adjusting the temperature controller so that the make-up water valve is wide open, thus supplying adequate water to the rack in spite of the leak at the hose connector. After this, the steam is shut off and after the rack has cooled down, the circulating pumps are shut off and the faulty hose replaced. If the rack contains material, the columns should be frozen off at the bottom of the rack before lowering the water temperature.

If the supply of make-up water fails, the circulating water will heat up to the boiling point in about two or three minutes and will turn completely into steam if service is not resumed. In the columns there are several silver-soldered joints in the copper tube. These joints are susceptible to failure due to annealing of the work hardened joint when heated by the 1000 pound steam which flows through the inner nickel pipe and when subjected to process pressure of 1500 p.s.i. Also the copper tube itself will anneal and being under stress due to differential expansion of the two metals, may fail.

A schematic diagram of the circulating water system is shown in Dwg. 9-4

Freeze-off Water System. Water for the circulating system is river water treated with Calgon. River water itself is not satisfactory for use as freeze-off water since it contains sediment which would plug up the small valves and small diameter copper pipes soldered to the inter-column connectors. Consequently, a separate water system is used. Water is pumped from a well to the water treatment plant where it is given a coagulation and settling treatment. This well water is considered to be better than the raw river water for this purpose. The water is then pumped to a supply header at the top of the process building which provides a constant head for the freeze-off water lines. The header is closed so that a pressure at the bottom of the rack is developed of about 80 pounds per square inch by means of an air pump. An auxiliary supply controlled by a float valve is available so that if the well water supply fails, the head can be maintained by the automatic introduction of city water into the system. It is imperative that freeze-off water be available at all times.

Overboard Water



THE PERCULVE, Inc.
Technical Div.

Circulating Water
System

Date	JUNE 20, 1945	Dwg No	9-4
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The supply of water to the freeze-off header is adjusted so that there is a constant overflow. Branches from this header lead to the individual racks, where they again divide into a supply for the top and bottom of the rack. Three of the four water headers can be controlled by a solenoid valve, operated by a timer or toggle switches located in the control room. The solenoid control is provided in connection with the automatic removal of product. The water lines may be operated by manually controlled valves on the rack. The water, after passing through the copper freeze-off lines goes to the sewer.

Gland Water for Desuperheater Pumps. Gland water is required in connection with the operation of the desuperheater pumps which are described under the section on the steam system. It was originally intended to take water from the water treatment plant and pass it through Permutit filtration tanks before supplying to the small gland pumps which would in turn pump water to the glands of the desuperheater pumps. The Permutit tanks are the tanks located in the southwest corner of the process building. The system now in use, however, takes condensate from the low pressure steam and uses it as the gland water in the large pumps. This water then flows to a tank directly above the desuperheating pumps where it flashes into steam, which is used as the low pressure heating steam in the process building.

6. Plan of the Steam System

Steam is supplied to the process building from the power house through three 10" lines. Each line serves 7 racks and these 7 racks form a section. The steam comes from the power house at about 1325 pounds per square inch and 935°F. It is the duty of the steam and power department to control the desuperheating of this steam to our process requirement which is 545°F. The pressure must also be reduced to 1000 p.s.i. as this is approximately the saturation pressure of water at 545°F.

Desuperheating. Desuperheating is accomplished by the injection of water condensate from the low p.s.i. steam used in the columns into the steam at four different nozzles. One nozzle is located just outside the process building, one just inside the building in the south end and one along the west wall near Rack 21. These three nozzles are controlled by one controller on the steam control board and constitute the initial desuperheating system stage. The fourth nozzle, which is located just beyond the steam control board, constitutes the final desuperheater.

Water for the desuperheating is injected into the steam from three desuperheater pumps located at the south end of the building. These pumps supply the condensate for injection at a pressure in the neighborhood of 1500 pounds per square inch.

[REDACTED]

[REDACTED]

- 11 -

Process Steam Supply. Steam at 1500 pounds pressure is taken from the header and led to the individual coils through 3" lines controlled by gate valves. Each valve is equipped with a "ball" type by-pass to permit gradual warming up of the coils. The 3" lines then pass to a steam header and is admitted to individual columns through expansion loops and flap valves. After passing through the column and condensing, it discharges at the bottom through another flap valve and expansion loop into a condensate header.

It is necessary, of course, to control the liquid level of the condensate at the bottom of the column. If condensate is allowed to build up in the column, heat transfer immediately drops and the natural pressure falls permitting a flow of more material into the column. Furthermore, the column performance will deteriorate. Finally, if steam is allowed to blow through a column, the liquid film of condensate is removed in addition which increases the heat transfer considerably. This also causes the natural pressure of the column to increase, sending a surge of fluid out of the column and into the waste tank. Therefore, to control the condensate level each column is equipped with a float control, a liquid level gauge glass and a valve which is actuated by the float. When the level rises above a predetermined value, the valve opens and allows condensate to flow to the header.

During the warm-up period of the rack, the pressure of the condensate is low up to the condensate pressure in the header so condensate must be expanded to the low pressure condensate line. This produces flashing and consequently loss of condensate. This warm-up period should not be extended any longer than necessary. When the pressure reaches approximately 500 pounds, the condensate is returned to the high pressure condensate header.

Importance of Condensate Level Control. As mentioned above, the operation of the condensate removal system at the bottom of the rack may have important effects on operation. Suppose, for example, that the control valve on the condensate line did not open, so that the columns filled with condensate. The stoppage of the flow of steam coupled with the lower heat content of the condensate (lower by the amount of the heat of condensation) would allow the circulating water to cool down the process material in the column space to the extent that it would contract and allow more material to flow into the column. Thus although the normal amount of material per column is about 4.6 pounds, it is possible to get 5.5 to 6 pounds per column under these conditions. Now, suppose that the valve opened automatically or that the condition was observed by an operator who opened the valve manually. This sudden removal of condensate would allow steam to blow through the column again, which would cause a rapid increase in the heat transfer and a rapid rise in material temperature and pressure. The rise in the pressure is due to the fact that the ability of the process fluid to expand with rising temperature is limited by the small size of the conduit lines leading back to the transfer rack. Dangerously high pressures have been developed, pressures which have caused columns to burst.

[REDACTED]

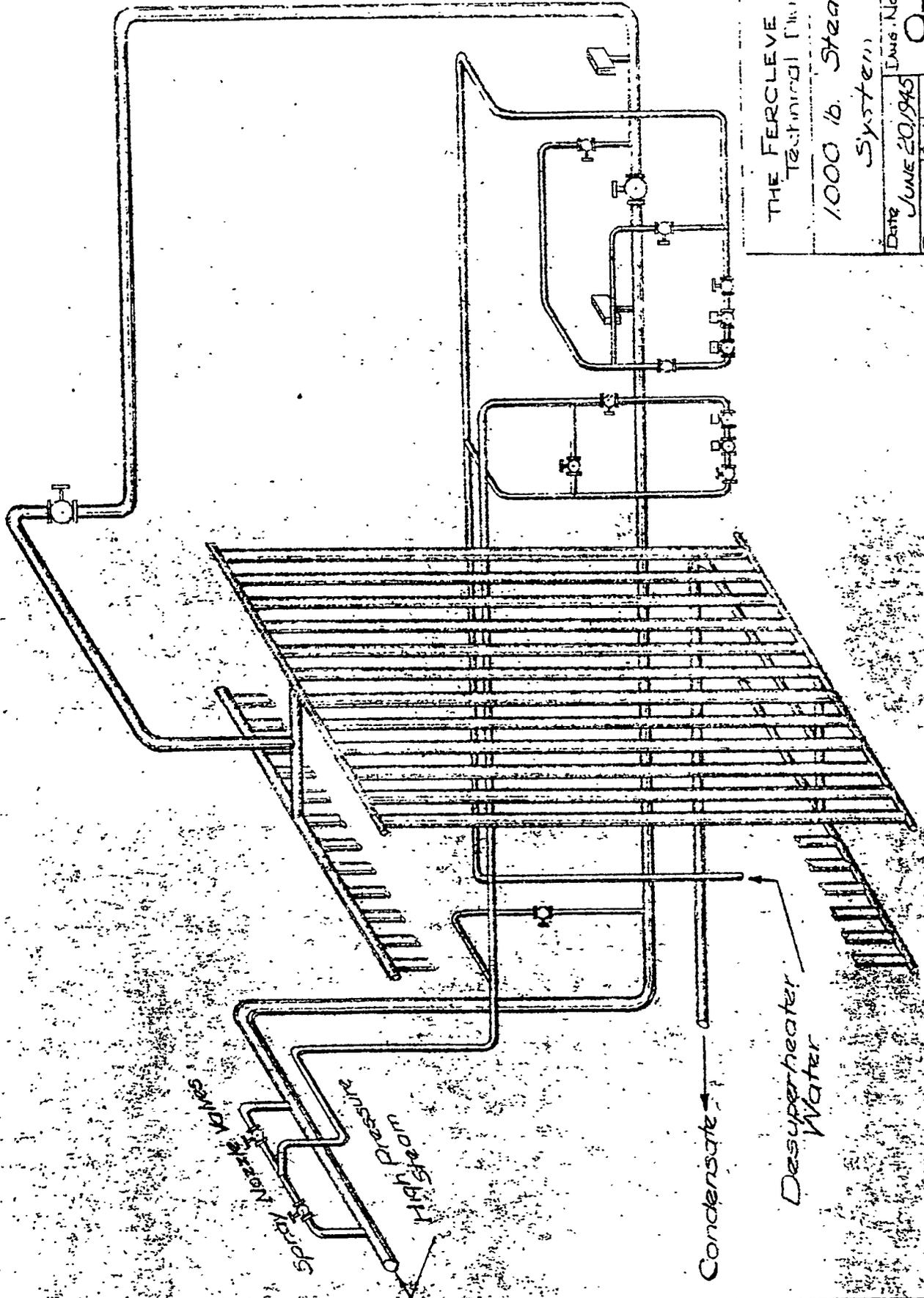
The transfer room operator should be able to detect quickly abnormal flows of material from the scale tank to the rack by means of his scale readings. He can confirm the condition of excessive condensate in the columns by consulting the circulating water temperature recorder located outside the transfer room. Normally, the temperature differential on this recorder is about 10°F. If the columns are filled with condensate, heat transfer drops off and the temperature differential decreases. If this is the case, the operator should notify his rack foreman who will arrange to drain the condensate from the columns very slowly, permitting material to flow back slowly into the scale tanks. During this period the operator should make sure that the columns are not isolated from the transfer room by means of freeze-off connections. If the temperature differential has not decreased and if the scale readings on the scale tank continue to drop off rapidly, an internal leak in the system has probably occurred. In this case the Transfer Room should be isolated from the rack by freezing off.

Steam up to 1000 p.s.i. pressure is also used, for heating purposes in the coils of the scale tank in the transfer room. Since it is necessary to have an accurate control of this steam pressure, each tank is equipped with an air-loaded reducing valve. To increase the steam pressure up to a predetermined value and to maintain it automatically at this pressure, it is merely necessary to set the air pressure in the valve at the desired value. Air at 1000 pounds pressure is available for this purpose. It is supplied from a small compressor located in control No. 11.

Diagrams of the high pressure steam systems and condensate are given in Dwg. 3-5 and Dwg. 9-6.

Low Pressure Steam Systems. Three Pacific desuperheater pumps are provided for injection of water into the superheated steam, one for each S.F. line. These pumps are designed to take the high pressure condensate from the racks and use a portion of it as injection water, returning the surplus to the power house. Instead of packing the glands on these pumps, the leakage through the gland is sealed by pumping cold water into it.

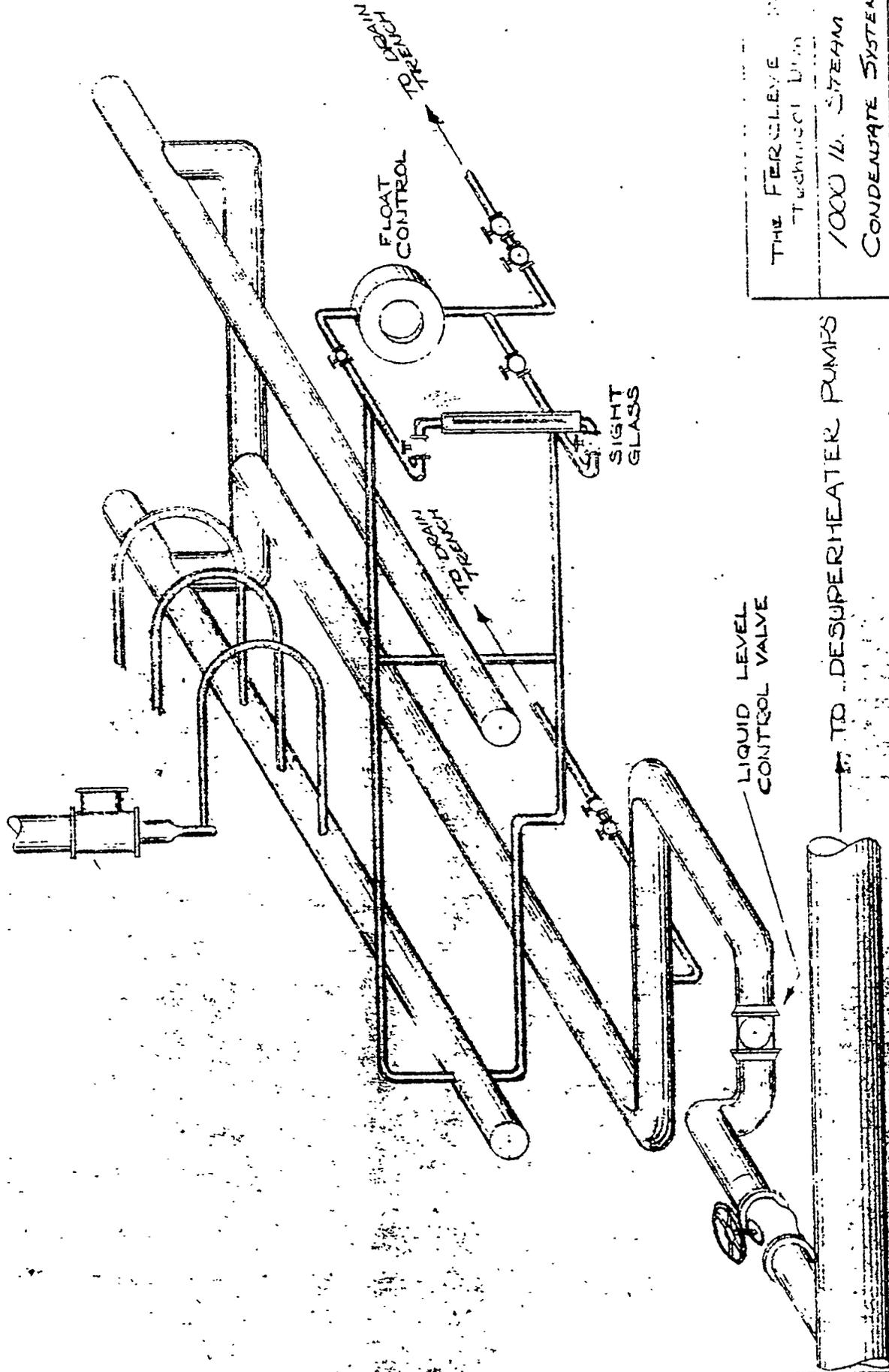
The flashing high pressure condensate as it leaks past the gland into a low pressure area, plus the cold gland water, produces 125 pound steam. Since this steam contains droplets of water, a separator has been installed over the desuperheater pumps to remove this moisture. The 125 pound steam is then used to heat ducts in the transfer room and at the top and bottom of each rack. After being discharged by a trap, the condensate from the 125 pound system is collected in the condensate sump tank in the south end of the building and pumped back to the power house.



THE FERGLEVE CORP.
 Technical Division

1000 lb. Steam
 System

Date	JUNE 20, 1945	Draw. No.	9-5
By	APRIL		



THE FERGUSON
 Technical Dept

1000 LB. STEAM

CONDENSATE SYSTEM

DATE JUN 20 1946

Dr. Back

Dwg No

9-6

TO DESUPERHEATER PUMPS

Condensate Traps. At present many of the traps on the 125 pound system as well as on the high pressure system are not operating correctly so it is necessary occasionally and briefly to by-pass the traps. These traps should be by-passed for the minimum time required to have them inspected and repaired by the Maintenance Department, since frequently condensate is lost when traps are by-passed. The importance of preserving all condensate applies equally to all other operations, as the plant was designed to return essentially all of the condensate to the power house. Efficient operation of the power house is dependent upon this fact.

5# Steam. 125 pound steam is also used to heat the high temperature side of the convector loops. Part of the 125 pound steam is reduced to 5 pounds per square inch pressure and used to heat the low pressure convector pipes and the conduits leading from the transfer room to the racks. Condensate from the 5 pound system is collected with the 125 pound steam condensate.

7. Plan of the Air System.

Air is supplied to the plant at two different pressures, namely (1) Low pressure air supplied at a pressure of about 100 pounds per square inch, and (2) high pressure air at a pressure of 1000 pounds.

Low Pressure Air. The low pressure air is supplied by a large Gardner-Denver compressor located in the southeast corner of the main building. There is also an emergency Ingersoll-Rand compressor located in control room #11 along with the high pressure compressors. The low pressure compressors are both connected to the low pressure header which runs along the top of the east side of the main building. The air which is first filtered at the top of Rack 21 passes by a mercoid switch which is connected to the alarm drops in the control rooms. If the pressure drops below 70 pounds per square inch, the signal is given in the control room.

The main header runs the length of the building on the east side of the plant with sectionalizing valves located between racks No.7 and No.8 and between racks No.14 and No.15. There are outlet valves for each rack running to the four air pipes on each rack. In some of these racks the air has been supplanted by steam but the valves are turned off manually at the header on these racks. At the north end of the building the line runs across to the west side of the building to the transfer room header.

At the end of the building where the numbering of racks begins is located the transfer room header valve. The line then returns along the mezzanine level with four lines with valves leading to each pair of racks. Another sectioning valve is located between transfer rooms No.7 and No.8. Air is supplied to the laboratories from a line located between transfer rooms No.3 and No.4. Also located near Rack No.1 is an air line to a low pressure steam reducer which furnishes low pressure steam to the plant.

Of the two lines per rack in the transfer room header, one line supplies air to the temperature controller, located on the mezzanine level, which controls the make-up water valve and the other line passes to the Swartwout Float Control valve which regulates the condensate level.

One other use of the low pressure air is to reduce the 125 pound steam to a variable steam pressure in the transfer room for heating the transfer pots in transferring material from the shipping container to the seal tank.

Effects of an Air Failure. The air is quite important in the continuity of production in the plant. The automatic air-operated valves are all spring opening, air closing type valves. In this connection, it is interesting to note that air failure would affect the plant in the following ways:

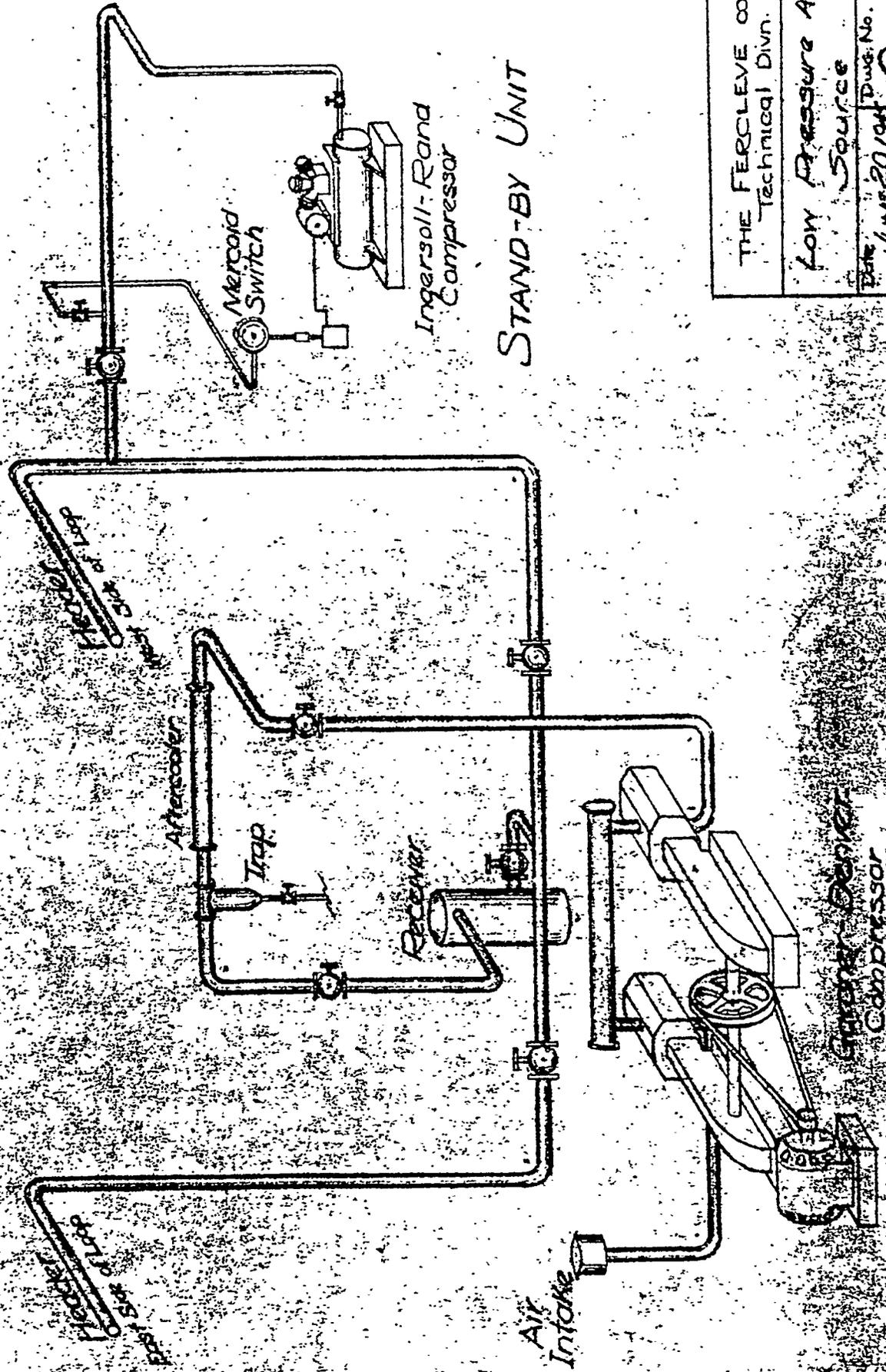
- (1) The desuperheating water regulators would open and flood the steam lines which would result in a steam hammer.
- (2) The temperature controller on the circulating water inlet would open and cool down any racks operating.
- (3) The valve reducing the steam pressure to 125 p.s.i. would open, allowing steam at 1000 pounds pressure to flow into the low pressure steam system,
- (4) The condensate return valves would open and blow steam back to the power house.

The layout of the low pressure air system is given in Dwg. 9-7 and 9-8.

High Pressure Air System. The high pressure air is supplied by one of two Worthington compressors located in control room No.11. The air is passed through a water cooled unit into a reservoir and from thence it passes into the 1000 pound header to the transfer rooms. There are two sectionalizing valves located on the header, one located in control room #8 and the other located in control room #3. There is also a valve on an open line at the end of the header at the north end of the building. The air is used to operate the Foster valves which give variable steam pressures up to 1000 pounds per square inch on the storage tanks.

Each transfer room has a shut-off valve located in it to shut the air off from any given transfer room. They are located in the transfer room except in the following cases:

- (1) The valve for transfer room No.7 is in control room No.7.
- (2) " " " " " " No.6 " " " " No.6.
- (3) " " " " " " No.5 " " " " No.5.
- (4) " " " " " " No.4 " " " " No.4.
- (5) " " " " " " No.3 " " " " No.3.
- (6) " " " " " " No.2 " " " " No.1.



THE FERGLEVE COEF
Technical Divn.

Low Pressure Air
Source

Date: June 20, 1944
Dwg. No. 9-7
D.D. 999d
B. 2666

The high pressure air is introduced from the transfer room supply line into the bowl of the Foster valves where it then regulates the amount of steam permitted to pass into the storage tank coil by means of a balancing diaphragm.

The high pressure air system is shown schematically in Dwg. 9-9 and 9-10.

8. Plan of the Electrical System.

High Voltage System. Electrical current is supplied to the 8-30 area from the switch house of the power plant. It is supplied at a voltage of 2300 volts to the switch house on the area located at the southwest corner of the building near the pump house. Current is used at this voltage to operate the four circulating water pumps in the pump house and is also fed through underground conduits into the process building. It passes to four feeder boxes located along the west side of the building. From one feeder box located farthest south, lines go to the low pressure Gardner-Denver air compressor, and to the three Silver Queen desuperheating pumps. From the other feeder boxes lines go to the groups of four circulating water pumps located between each pair of racks. This makes a total of 50 pumps which operate on 2300 volts in the main process building.

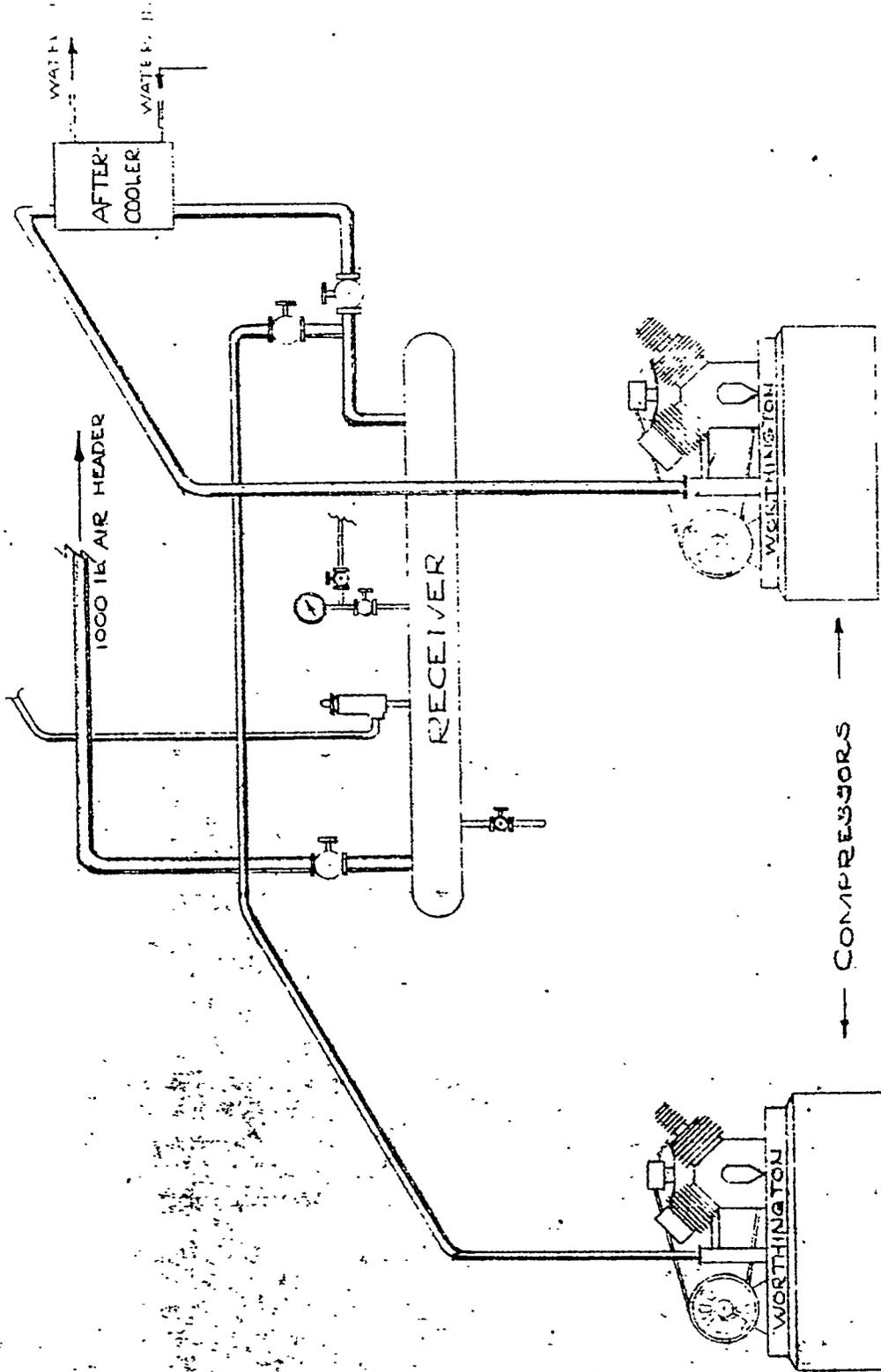
Low Voltage System. Most of the electrical current in the plant is used at a voltage of 440 volts with, of course, the lighting system using only 110 volts. The main equipment operating on this 440 volt line are duct fans, the ventilation fans in the transfer room, the gland seal water pumps and condensate return pumps together with various sump pumps and miscellaneous pumps located in the plant. The main switch for the building is located near the southwest corner of the main building.

Since the 440 volt system is a three phase system, the lighting system which is 110 volt single-phase system the current is supplied from the same source. The primary function of the low voltage supply is for lighting and for the operation of the various solenoid valves in the plant. There is also a 24 volt battery system for emergency operation of the solenoid system.

9. Technique of Operation.

A number of operational techniques are employed in the plant which are extremely important, in that they must be known and performed properly. These techniques are basic throughout the plant and are among the first things that an operator must be familiar with. Simple enough in themselves, they contribute much to the safety and production of the plant. The following paragraphs give a short discussion of several of these techniques which outline some general principles to be followed. The only adequate way for them to be learned, however, is actually to perform them under the tutelage of a competent instructor.

[REDACTED]



THE FERGUSON CORP
 TECHNICAL DIV.

High Pressure Air

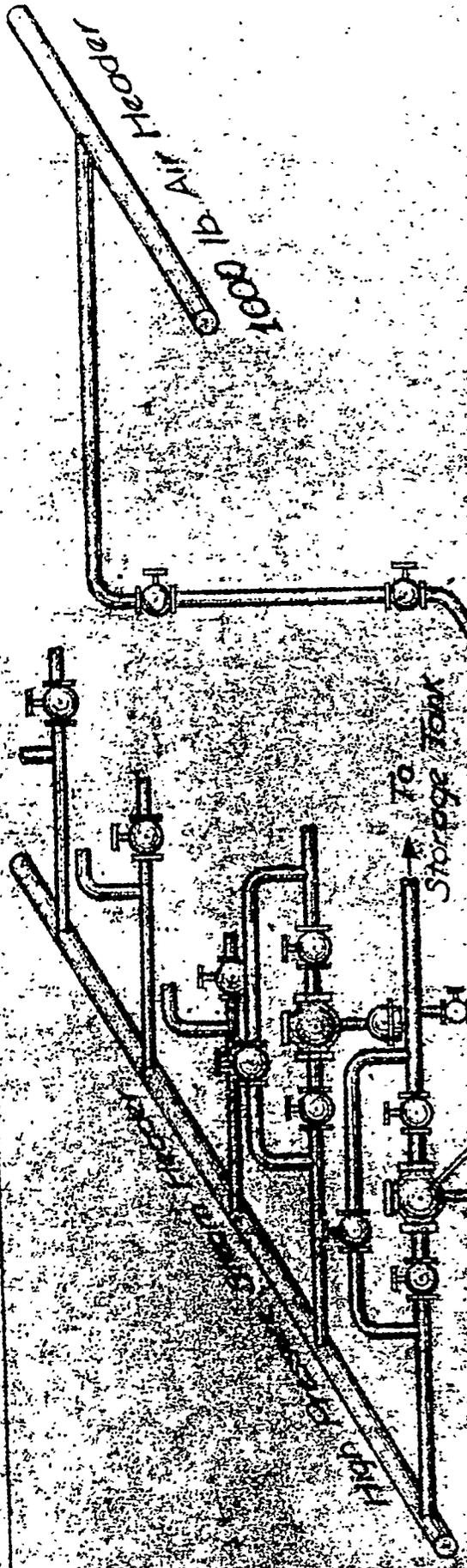
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9-9



THE FERGUSON CORP.
 Technical Divn.

High Pressure Air
 Distribution

DATE: JUNE 20, 1948
 Dwg. No. 9-10

Freezing off Connections etc. One of the most common operations is the simple operation of freezing off a line or a tit in order to change connections. The internal diameter of most of the tubing and openings in the material system is in the range of $1/16"$ to $3/32"$. This small diameter opening permits the formation of a plug of frozen material to form which is of sufficient length to hold the operating pressure of 1500 pounds. The cross sectional area of the plug is such that the total force operating to blow the plug out of an open tit is only about three or four pounds. This means that there is adequate protection in plug formation in the normal operation.

It is only if the plug is not properly formed or if the plug for some reason is porous and not well packed, that the plug blows out with resultant loss of material into the atmosphere.

The freezing off is done with dry ice and can be done simply by starting with the dry ice on the end of the tit away from the column, leaving about one inch of uncovered metal between the dry ice and the threads of the tit. This exposed inch of stem is felt from time to time with the bare fingers until it is cooled to room temperature. Then, the block of dry ice is gradually worked backward toward the column until it has been pushed back as far as it can go and will remain in good contact with the tit. There is a tendency in the plant merely to place a piece of ice on the tit and leave it there. This practice is not considered satisfactory, since the gas film around the tit may retard the freezing off operation; furthermore, the dry ice has been known to fall off. It is also important to note that no frost should collect on the tip of the tit. This will avoid the possibility of plugging the end of the tit in the hydrolyzed material while making the change.

At this point the whole tit will be filled with a solid plug of material of sufficient length to prevent it from blowing out. This should be a perfectly adequate method of preventing material breaks at the point, unless, as in some cases, there is "fresh air" in the tit which will not freeze with the consequent lack of formation of a good plug.

Disconnecting the Fitting. In order to make sure that the plug is formed another point in which great care is necessary should be noted carefully. In opening up a tube which has been frozen off, particularly one at the top of the column which has more probability of containing fresh air, one must be extremely careful to note any evidence that the tit might not be frozen off completely. To do this, the Lunkenheimer nut must first be "cracked" or loosened from its seat while still holding the dry ice on the tit. The right angle cap or the link line must then be bent back and forth slightly to break the seat between the ferrule and the tit. If there is any fresh air in the tit it will leak out around the nut. It will be noticeable by its odor.

[REDACTED]

If it is found to be present, one must let it bleed out around the nut until the fresh air or material fumes stop coming out. Then this indicates that the tit is full of solidified material, the operator can continue to open the connection slowly by alternately loosening the nut and moving the ferrule connection to make sure that any uncondensed gases will leak out before opening the tit to the atmosphere.

Breaking of a Link Line when Changing Connections. It can be pointed out that the nickel tubing forming the link lines is of such strength that it cannot be bent many times through a wide angle without breaking. NO LINK LINE MAY EVER BE TWISTED TO remove it from a tit. The torque produced by the twisting will break a link line off in the tit in nearly all cases. Should a link line or cap break off in the tit, it can frequently be removed by using a pair of electrician's pliers. Again, do not twist the tubing. By grasping the broken end of the tube which is stuck into the tit firmly and pulling directly outward, it is usually possible to remove it relatively easily. It may be necessary to use a vertical or horizontal vibration movement of the pliers to facilitate the removal.

It is important that a steady pull with a very slight up-and-down motion is much more effective than pulling with a wide-angle bending of the link line section. If this technique is not effective, the column must be drained from another tit and the tit with the broken line in it must be drilled out. Care must be taken not to ruin the threads of the tit. If any contact has been made with the threads so that a fitting will not screw on easily, the Maintenance Department must be called on to rethread the tit.

Changing the Connection. Another extremely important technique is the simple process of placing another connection of some sort on the open end of any line or tit from which a connection has been removed. It has been estimated that the time during which a tit is open to the atmosphere should be no longer than 5 seconds. In other words, the connection or line which is to be attached must be ready to be placed over the opening immediately. This is necessary to cut down the possibility of having a plug blow out and also to prevent water from hydrolyzing the material in the end of the tit forming a permanent plug. It is also good practice not to let the end of the tit get frosted while one is working on the tit, since moisture condenses on the end of a frosted tit. The cooling of the tit should be continued with dry ice on the back of the tit at all times, but the front end should be cooled only to a lukewarm temperature.

Before placing another connection on any line or tit, the fitting should be examined to make sure that the nut is not stripped and that the ferrule is on properly and in the right place.

[REDACTED]

Warming up a Line. The remaining step of making a change in a connection is the process of warming up the fit after the connection is changed. Usually it is considered better to allow a connection to thaw out from the heat of the duct or surrounding atmosphere in order to avoid any sudden expansion. However, sometimes it is necessary to speed up the process which can be accomplished by the judicious use of a gas burner. The flaming process again is a step that must be carried out very carefully, since the properties of the material are such that a tremendous expansion takes place in the melting of the solid.

The important factor, then, is to have sufficient volume for the liquid material to expand into as it changes its state. In other words, ALWAYS FLAME ANY LINE FROM A DIRECTION WHERE THE MATERIAL IS LIQUID OR FROM AN OPEN END. The operator must be very careful to note that at no time should the material be heated above its melting point without some space to expand into since it has been estimated that, if the center of a closed tube full of solid material were heated to 100°C. without this expansion region, pressures would be developed in excess of 10,000 pounds. This is in excess of the safety factor in the plant.

Moisture Tests. Another of the common procedures in the plant is the process of determining whether or not a system or part of a system is dry. One of the older methods was the drying of a system by pumping it down to a high vacuum while heating the system as hot as possible. After drying as completely as possible, a glass trap was inserted in the line and the trap immersed in liquid nitrogen. This was done usually by hooking rubber connections to the trap and allowing the vacuum to be pulled through the trap for several minutes. After equilibrium in the tubing with respect to the water content was reached, liquid nitrogen in a Dewar flash was used to freeze out on the walls of the trap the water emerging from the system. The trap which had previously been tared could then be reweighed and the moisture determined. It was usually expressed as grams of water collected per hour under a vacuum of 29".

It was found that the evacuation of the system was not adequate in drying out the metal properly and that the moisture test was often spoiled by humid air leaking into the evacuated system, so the plan was then adopted of blowing dry nitrogen gas through the system to sweep out the moisture. This moisture was then collected in the same manner as given above by freezing the moisture out in a glass trap by liquid nitrogen.

Qualitative Tests. The method used at present for quick qualitative tests on individual columns is a modification of the flowing nitrogen test. In this test the nitrogen is passed through the bottom of an individual column or pair of columns and a one minute test is run with the liquid nitrogen and trap. A visual determination of the water collected serves to eliminate any obviously leaking columns. This is not satisfactory for small leaks of moisture into a system whose existence can only be definitely proved by a careful quantitative test lasting a half to one hour.

Quantitative Tests. These tests are usually made on a circuit or part of a circuit after preliminary tests have shown large leaks to be absent. The test conducted in the same manner as the others except that the nitrogen is introduced at a point in the circuit where it has to flow through the whole circuit or part of the circuit before entering the testing trap. If the nitrogen is introduced, for example, at the bottom of column 1, it is taken out and the moisture collected at the top of column 25. Usually the circuit is blown out for some time before running the test, then the trap assembly is inserted in the line at a convenient point and after passing the nitrogen through the trap at room temperature for 15 minutes, the trap is immersed in the liquid nitrogen. This test is usually carried out for 30 minutes. If the water collected is less than 0.04 g. per circuit per 30 minutes, the system is considered dry enough to introduce the fresh air and material for the process.


1. General Description of the Transfer Room

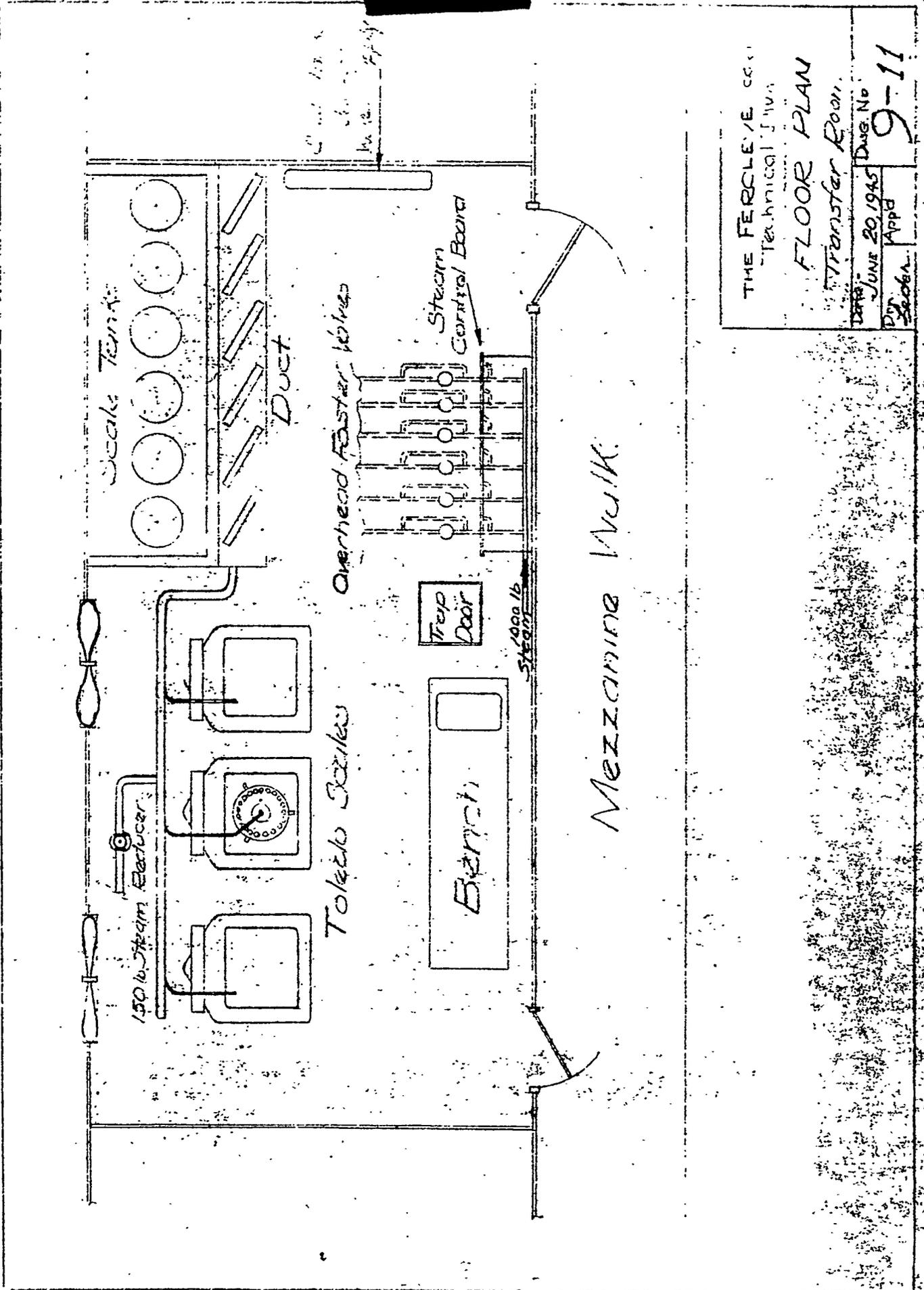
The transfer room is located on the mezzanine level which is along the west side of the plant. The rooms are eleven in number, which means that each room serves two racks, except transfer room eleven which is only used for Rack 21. All of the double rooms are similar in construction and layout, whereas transfer room eleven has the same equipment and design but only half as much as the other rooms. The double rooms differ in that some of the rooms are reversed in their design from the others. In other words, in some of the rooms the valve system for the transfer tanks is on the south side of the room and in others it is on the north side. The relative layout of the rooms is the same except for this reversal. See Dwg. No. 9-11.

However, before describing the room, the scale tank itself will be described in order to clarify the function of the various parts of equipment in the room.

The Scale Tank. The scale tank is essentially a tank in which material can be stored and from which it can be placed in the columns of the rack, and from which the rack can replenish its supply as the product is taken off. The tank itself consists of three parts: The nickel pressure vessel containing material, a steel jacket surrounding it and a high pressure steam coil in the center of it. The purpose of this arrangement is to allow the material to be heated with low pressure steam in order to melt it, to be heated to high temperature by 1000 pound steam thus developing the operating pressures, and also to allow the tank to be cooled off by introduction of cold water into the outer jacket in order to solidify the material. The other equipment in the transfer room is largely auxiliary to the scale tank

(Description of scale and mounting, also description of barricade will follow in revised copy)





THE FERGLER CO.
 Technical Division
FLOOR PLAN
 Transfer Room

DATE	JUNE 20 1945	DWG. No.	9-11
DR.	SCOTT	APP'D.	



125 Pound Steam. Probably the most extensive system in the room is the 125 pound steam system since it is used to facilitate the transfer of material and also to heat up a number of lines. There are three steam lines entering the room. One of these is located behind the main door of the room and enters near the ceiling. This line enters and splits into two sections. One of the lines passes through a reducing valve which reduces it to a 5 pound pressure, fitted with a by-pass and gauge on the downstream side of the valve, and from there passes through one of the six valves into the jacket of the scale tank. It is used for heating the material up slowly. The other branch passes through a reducing valve and by-pass assembly into the line which is used for heating the duct. The reducing valve is not adjusted to reduce the pressure since it is necessary to obtain as high a pressure of steam as possible to heat the duct properly. The steam from this line, after passing through the duct, goes to a condensate line which contains another trap-by-pass assembly and empties into the condensate return line.

Another line enters from the end of the transfer room farther from the main door and goes to a pressure reducing valve located near the west wall of the room close to the scale tank vault. This goes through the reducing valve and gauge line to the transfer pot where it is used to heat up the shipping tanks when transferring material into the scale tank.

The condensate line from the transfer pot enters a return header and passes through a trap-bypass assembly into the low pressure condensate return line.

5 Pound Steam. Another 125 pound steam line comes down from the top of the plant and passes through a pressure reducing valve located just above the roof of the transfer room where it is reduced to 5 pounds. It enters the transfer room at a pressure of 5 pounds at the end farther from the main door. It separates into two branches with one branch going to the transfer conduit connecting the transfer pots and the terminal box in the duct. It is used for heating this line and goes through the line into the usual trap-bypass unit and thence to the low pressure condensate return line. The other branch passes along close to the ceiling of the room to the corner of the room by the scale tank vault where it goes into the conduit and is used for heating the conduit. The condensate return for this branch is on the rack at the other end of the conduit.

1000 Pound Steam. This steam line enters from the 1000 pound header outside the transfer room and passes to the Foster pressure reducing valves. There is a 1000 pound steam gauge on the control board. The Foster valve has an inlet for steam on one side of a diaphragm and an inlet for air on the other side.



[REDACTED]

This steam pressure is regulated by controlling the air supply by a valve on the board. This valve regulates the air supply which in turn regulates the amount of steam which is permitted to pass through the valve. All of the rooms have the Foster valves equipped with upstream and downstream out-off valves and by-passes and most of them now have lines leading to gauges on the board which are located in such a position as to show the pressure which is in the line even when the valve is on the by-pass. The steam pressure is registered on the left of the two gauges attached to each Foster valve line. See Dwg. 9-10.

The controlled steam pressure passes from the reducing valve into the top of the scale tanks where it passes through the coils running through the center of the material chamber. It leaves at the bottom through the trap-bypass assembly and into the 1000 pound condensate return.

Water System. The water line enters the room in the corner behind the main door. It then passes through a solenoid assembly which is connected to the general freeze-off system on the racks. These solenoid valves are bypassed and are usually operated with the bypass open without relying on the solenoid system. This allows more flexibility of operation when working with different tanks. One branch of the water line goes to the header along the top of the ducts which furnishes water to freeze off the tits on the scale tanks. There is a valve on each tank which permits the water to flow through the copper coil which is in contact with each tit on the tank. They are all frozen off together at the present time but some experimental work is being done to develop a method of freezing them separately, particularly since it is desirable to know the pressure of the tank when the rest of the tits are frozen off.

The other branch of the water line goes to the valves on the side of the room near the scale tanks through which the water goes to the outer jacket of the scale tanks. The assembly is arranged so that water and steam pass into a common pipe so that either water or steam may be passed into any of the jackets to either thaw or freeze that particular tank. The water and steam pass into the condensate return line through a rubber connection at the bottom of the tank. Another water line goes to the sink in the cabinet in the room. It drains to the sewer.

Air System. The 1000 pound air line enters near the 1000 pound steam line and goes to the header furnishing air to the Foster valves. There is also a gauge on this line located on the control board. The air goes to one side of the diaphragm which controls the steam pressure. This pressure is held until the air vent valve is opened on the control board which releases the air pressure on the valve and correspondingly lowers the steam pressure on the scale tank.

[REDACTED]

[REDACTED]

The right gauge of the pair for each storage tank is the air pressure that is on the diaphragm. The gauges showing steam pressure and air pressure should be very close together in readings or the stress on the diaphragm may be great enough to break the diaphragm. See Dwg. 9-10.

The low pressure air line runs along the west wall of the room and has an outlet in the neighborhood of the transfer pot pressure reducing valve. This is used for blowing out equipment and for cleaning operations involving the forcing of liquids through equipment under pressure.

2. Methods of Conditioning Equipment.

a. Conditioning Scale Tanks

The fundamental objective of scale tank conditioning is to obtain a clean vessel free from dirt, nickel oxide and material decomposition products. Unless these foreign materials are completely removed, the storage chamber will act as a source of plugging substances that will interfere with proper operation of the rack.

- (1) Storage chambers should first be completely dried. Rough drying can be accomplished by opening all tits and introducing through the top tit a stream of air while maintaining 5 pound steam on the outer jacket and 150 pound steam on the inner coil. The tits should also be checked to make sure that all of them will pass air through them and are not welded shut. Removal of the bulk of the moisture can be checked by holding a cold mirror next to one of the tits. All tits should then be capped except one serving as a lead to the vacuum pump.
- (2) The chamber should be evacuated through a rubber hose attached to the tit until the gauge has shown a vacuum of 29" for at least one hour. All tits should be flamed from time to time to guarantee that water has not accumulated in them. It is essential that moisture be thoroughly removed because later in the procedure when the material is introduced obnoxious plugs will form in the tits if the drying technique has not been sufficiently rigorous.

- [REDACTED]
- (3) Connections to the chamber should be changed to permit the introduction of fresh air. A line should be run between the fresh air valve to the chamber and connections to the vacuum pump altered so that when fresh air is introduced the gas can only come in contact with an all-metal system. The chamber should be then re-evacuated to 29", the valve to the pump closed, and fresh air introduced into the chamber to a pressure of 10 p.s.i. The chamber should remain filled with the gas for an hour at which time the pressure can be relieved to atmospheric conditions. The connections to the fresh air valve and to the vacuum system should be removed and the tits employed for this purpose should be capped.
- (4) The next step is a step that has been followed throughout the conditioning of nearly all of the scale tanks. In conditioning the last several tanks that were conditioned the operators did, however, eliminate this step with no apparently bad results. This item is to introduce into the tank about 8 pounds of material. The material itself is one of the best conditioning substances there is so the principle was to let the material condition the metal in this step. However, there was some thought, that the fresh air would be satisfactory so it is possible that this step can be eliminated in the future.

During the introduction of this material, the central steam coil need not be heated and the temperature of the outer jacket can be maintained at a satisfactory value by heating with 5 pound steam. The tit through which material has been introduced should be frozen off and capped and the central heating coil activated with 1000 pound steam for at least 6 hours.

- (5) The material should then be removed. It is advisable to use some sort of a trap for catching the larger part of the material but, while several traps have been developed, none of them is particularly satisfactory for saving an appreciably large quantity of the material. It has been necessary to blow the material out into the duct and lose the major part of it. This is not economically or healthfully advisable but if a link line is run some distance up into the duct and the material let out through that line, it is possible to remove most of the material without a great quantity coming out into the room.
- [REDACTED]

[REDACTED]

When the pressure in the tank has dropped to atmospheric pressure, the remainder of the vapor should be thoroughly blown out by introducing dry nitrogen through one tit while a second is uncapped. The nitrogen should be blown through the chamber until the outgoing gas no longer shows any signs of material. All tits should then be opened and flamed in order to get a free passage of nitrogen through them. The operator must remember all seven of the tits, including the one in the top and also the one in the bottom, both behind the armor plate in the vault. If these tits are not freed of material considerable difficulty will be experienced later when water is introduced, because the water will form plugs with the material.

- (6) The storage chambers should be washed with water to remove decomposition products. The volume of the vessels should be determined by weighing them full of water. The water should be run out of all the tits until it is colorless and free of all decomposition products. The chamber should then be drained through the bottom tit recording the weight of water removed. The weight of the water removed should be the same as the weight full. If these figures do not check satisfactorily the tank should be filled again and the volume and weight determined.
 - (7) The tank should now be thoroughly dried. First, rough dry the tank by blowing dry air through it while the chamber is being heated as was done in the first step of the conditioning. Rough drying should be followed by a careful pumping procedure in which the chamber is evacuated to a vacuum of at least 29". Air is introduced into the chamber periodically to aid in flushing out any residual water vapor. At the end of 6 hours dry nitrogen should be admitted into the tank. After several minutes of flowing nitrogen through the tank by passing the nitrogen in through the top tit and out the bottom, attach a glass trap to the bottom tit and immerse the trap in liquid nitrogen in order to test for the presence of moisture. Pass the nitrogen through for 5 minutes and weigh to the nearest centigram. If the moisture test shows the presence of less than 0.04 grams of water, the tank may be considered dry enough to fill. Also make certain that all the tits are dry.
- [REDACTED]

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- (8) The chamber should be re-evacuated and fresh air immediately introduced to a positive pressure of 10 pounds per square inch. The 5 pound steam should be turned off the outer jacket and 1000 pound steam run through the central heating coil. Fresh air should remain in the chamber under positive pressure until operators are prepared to introduce process fluid.
 - (9) Fresh air connections should be removed and a suitable jumper to permit the introduction of material should be connected.
 - (10) With 1000 pound steam still on the inner coil and no flow of fluid, steam on the outer jacket, the reading of the scale of the storage chamber should be tared to zero.
 - (11) The high pressure steam should be cut off and the chamber held at a temperature of 90°C. by introducing a mixture of 5 pound steam and cold water into the outer jacket. When the outer jacket has been filled with this hot water the weight on the scales should be noted. The reading will approximate 169 pounds.

At this point the tank is ready for the introduction of material.

3. Transferring Material

a. Filling Scale Tanks

The first step is to make sure that the Scale Tank is empty, the tits flamed out and free and that the scale is properly tared. Also the operator must make sure that the full transfer pot is ready on the platform scale in the jacket.

The steps to be followed are outlined as follows:

- (1) Connect inlet and outlet steam lines to the transfer pot jacket.
 - (2) Remove valve cap from the transfer pot and put on the adapter. Make sure that the lead gasket in the valve cap does not stick to valve plugging the line. The adapter and gasket should be well flamed before putting on the transfer pot valve.
 - (3) Connect transfer jumper to transfer pot valve and transfer conduit tit.
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- (4) Connect link line in duct to scale tank tit and to transfer conduit tit. Be sure that the freeze off water is running on the scale tank tits.
 - (5) Turn steam on the transfer pot through the low pressure P.R.V. Start with 5 pound steam to warm the pot and to check the traps. Increase slowly a half hour later to about 30 pounds pressure.
 - (6) Put low pressure steam on the scale tank jacket to bring the temperature to 100° C. Check the traps to see if they are operating properly.
 - (7) Open valve on pot and flame the whole line after unfreezing the scale tank tits.
 - (8) Keep accurate records of the process while transferring the material. In no case should more than 420 pounds be allowed in the scale tank.
 - (9) When transferring is finished out the transfer pot valve off, disconnect the link line from the scale tank, cap the scale tank tit off and plug the link line end.
 - (10) Admit water into the scale tank jacket after turning the steam off and the tank is ready for operation.

b. Emptying Scale Tanks into Depleted Tanks (Acetylene Tanks)

- (1) Connect a new link line to the scale tank tit and to the empty depletion tank.
 - (2) Put high pressure steam on the scale tank. Warm the tank up slowly at first then increase pressure gradually. Unfreeze the scale tank tits.
 - (3) Start the transfer by heating the line with a burner. Open the cylinder valve and continue heating until the material moves freely into the cylinder. Increase the steam pressure to 1000 pounds near the end of the transfer.
 - (4) When empty blow out remainder of material into ducts. This should not be more than a pound or two. Make certain the fan is on.
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- (5) Have scales tared and the tank is ready for re-filling.

4. General Instructions for Routine Operation

a. Starting up Operations

With all the storage tanks full of material (420) pounds and circuits of the racks filled with material, the transfer room is now ready to operate. After determining the configuration of the circuits, the rack foreman will notify the transfer room man which conduits are to be used with each circuit. The conduits will be designated by number. The conduits from the rack emerge in the duct in the transfer room and are numbered from left to right.

This holds true when there are 36 conduits per rack. In some transfer rooms where temporary or replacement conduits have been put in, the conduits are stamped or tagged with their respective numbers. The transfer room man will then complete the circuits by connecting the conduits to a storage tank by means of link lines.

b. Arrangement of Circuits

Two circuits are to be connected to one storage tank--this means that the four circuits of the rack will be served by two storage tanks and the third tank will be held in reserve. It is preferable that circuit No. 1 and No. 2 be on one tank and circuits No. 3 and No. 4 be on the other unless otherwise ordered by the rack foreman. A material pressure gauge must then be attached to the fifth tit of the storage tank. It is a good policy that the conduits be attached so that the lowest number conduit be connected to the top tit of the storage chamber and the other three in the like manner, thus leaving the bottom tit for the pressure gauge.

c. Establishing Communication with Rack

The circuits having been completed, the transfer room is ready to communicate with the rack. There are two methods which may be used in establishing communication:

(1) When Circuits are Frozen off on Each and at Bottom of Rack

The freeze-off water should be turned off of the tank tits and low pressure steam turned on at the mixing valves to the jacket of the storage tank for warming up and melting the material.

[REDACTED]

No high pressure steam should be used until the material has been completely melted. This can be checked by means of a thermometer inserted in the well of the storage tank. It should read 80° C. before adding high pressure steam. If there is no hurry, it is advisable to use low pressure steam only until a material temperature of 100° C. is reached.

When the material is completely melted, about 100 pound high pressure steam may be turned on the coil of the tank by proper manipulation of the valves controlling the Foster Regulating valve for this particular tank. The jacket steam should now be turned off and only high pressure steam used. Gradually build up the steam pressure until the desired material pressure (1500 pounds/in² unless otherwise ordered) is reached as indicated by the material pressure gauge attached to the storage tank. The transfer room is now ready for the rack to be unfrozen.

(2) When Circuits are not Frozen on Bottom of Rack and Tits of Scale Tank Remain Frozen

Proceed as in (1) for warming up and turning on high pressure steam. At this time, since the gauge is also frozen off, the necessary steam pressure needed to obtain the required material pressure must be determined by use of the material pressure-steam, pressure-material weight chart shown in Dwg. 9-3

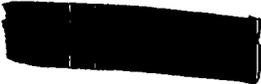
Adjust the steam pressure accordingly giving adequate margin for safety. Of course, if the pressure is too low, when the rack is unfrozen the material will flow back into the scale tank. This can possibly make the rack pitch. Endeavor to have the pressure as close as possible to the pressure in the rack to avoid a surging of material.

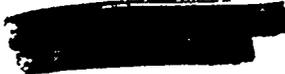
When the material pressure is judged to be satisfactory, the freeze-off water may be turned off the tank tits.

d. General Notes and Precautions

- (1) There shall always be two operators in each transfer room.
- [REDACTED]

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- (2) In dealing with long link lines in the duct, remember that they may cool off when the duct doors are opened, thus causing the material to solidify. Such link lines containing solidified material constitute a considerable hazard, as they might be accidentally heated in the middle of the length of solid material by nearby steam pipes or by a Bunsen burner flame used in the duct to heat other link lines. This intermediate heating can easily cause the link line to burst. A material break in the transfer room is most serious.
- (3) Always use heavy walled tubing for link lines. Be careful that they are not heated above 1000° C. When the lines contain material under high pressure, or heated at all, except carefully at the open ends when containing solid material.
- (4) Never put more than 5 pound steam or water pressure on the storage tank jacket as the rubber hose connection on the drain may break loose.
- (5) When changing a link line or material gauge connection on a storage tank:
- (a) Freeze off tits by means of the water freeze off system.
 - (b) Feel of tits with the finger to make sure that they are cold (always check a link line this way when you think that it is frozen off.)
 - (c) Bring the high pressure steam on the storage tank to zero pressure.
 - (d) FLOOD THE SCALE TANK JACKET WITH COLD WATER. (Most of the bad breaks in the transfer room have been caused by attempting to save time by eliminating this precaution. It is a false economy.)
 - (e) When the temperature of the storage tank has fallen below 80° C. it is permissible to disconnect Lunkenheimer fittings from storage tank tits.
 - (f) As a final security measure, before starting up have another operator check connections before renewing operation.
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- (6) In the rare case that the conduit lines leading to the rack have been frozen off by flooding the conduit jacket with water, the following procedure should be used in thawing out the conduit lines:
- (a) Make sure that the storage tank or tanks to which the conduits are connected are heated; that the storage tank tits are thawed out, that duct link lines are hot. In other words, there should be free movement of process fluid from the conduit lines to the storage tanks. Also, make sure that the rack ducts are hot and that circulation along link and conveyor lines can take place.
 - (b) Turn down the cold water valve on the conduit freeze-off system, slowly open the steam valve and mix the two so that the temperature of the water flowing through the conduit pipe rises gradually to 70-75° C.
 - (c) Take half an hour to bring the temperature up to 75° C.
 - (d) Check the temperature by measuring it where the water flows out of the end of the drain pipe below the rack duct.
 - (e) As the thawing of the conduits is an extremely delicate undertaking it should always be done under the direct supervision of the Technical Superintendent.
- (7) Material should be added to storage tanks up to 420 pounds. Make certain that at no time does the weight in the tanks exceed 440 pounds. Operate a rack only from tanks containing a maximum of 420 pounds.
- (8) Do not operate more than 51 columns on one storage tank at any one time.
- (9) Maintain the operating pressure of the material in the rack at 1500 pounds. When milking raise the pressure to 1700 pounds but since the circuit drops to about 1500 pounds while being milked, be certain to drop the tank pressure to 1500 before unfreezing again. This is to make sure that no surge takes place from the tank into the rack.
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- (10) It is strictly forbidden for any person to crawl into the storage tank vault, unless all the storage tanks are cold and all the low and high pressure steam is turned off.
 - (11) A large differential of steam and air pressures should never be allowed across the pressure reducing valves. When operating on the by-pass of the steam reducing valve, close the air valve leading to the Foster valves so the pressure reads on the steam gauge without going to the Foster valve. Special valves have been installed in some of the Transfer rooms to allow this to be done without affecting the Foster valve.
 - (12) Never operate for any length of time using the condensate trap by-pass. This is dangerous in that the high pressure steam may get through to the condensate line and also the pressure of the steam pushes the water into the condensate return tank and out over the roof.
 - (13) All transfer room operators should know at all times which circuits on which racks are being operated from a particular storage tank.
 - (14) Any unexpected change in storage tank weight and material, steam or air pressures should be noted as quickly as possible by the operators and reported to the Rack Foreman, or if he cannot be found, to the Assistant Technical Superintendent, or the Technical Superintendent.
 - (15) It is absolutely necessary that each transfer room operator should know the location of all valves and controls well enough so that each one can be found by the operator quickly without delay, in the darkness in case of an electric light failure, or in a dense smoke in case of a material break.
 - (16) Whenever steam is turned on, the steam trap valves should be checked as to whether they are open or closed.
 - (17) No change is to be made in the transfer room operating procedures and no valves are to be opened or closed on order of any people outside the transfer room crew, except the Rack Foreman, Chief Rack Foreman or the Technical Superintendents.
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- (18) In spare time, work on cleaning up the transfer room and transferring material. It is a good idea to label all spare equipment, link lines, hoses, etc. and hang on the walls.

5. General Emergency Instructions

In general, as in the case of all of these operating instructions, emergencies arise when the operator is careless or attempting to be too hasty. The best emergency procedure is to follow the methods given here with care and the emergencies will not arise. In case of an accident in the transfer room, the following steps should be taken, preferably in the order in which they are listed:

- a. Put on face mask, holding breath until adjusted.
 - b. Close duct doors.
 - c. Turn on duct fan and ventilator fan in room.
 - d. Turn on all water freeze-off line. (the water valves should be kept normally in position so that opening the one main valve will start all the freeze-off water flowing.)
 - e. Turn off high pressure steam on all storage tanks.
 - f. Flood all storage tanks with water.
 - g. Send a message to the rack to freeze off top and bottom freeze off coils.
 - h. The details of any accident, material break or leak as well as all other operations should be fully described on the rack chart and in the transfer room log book.
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Section C. The Columns and the Rack

1. Description of the Columns and the Racks

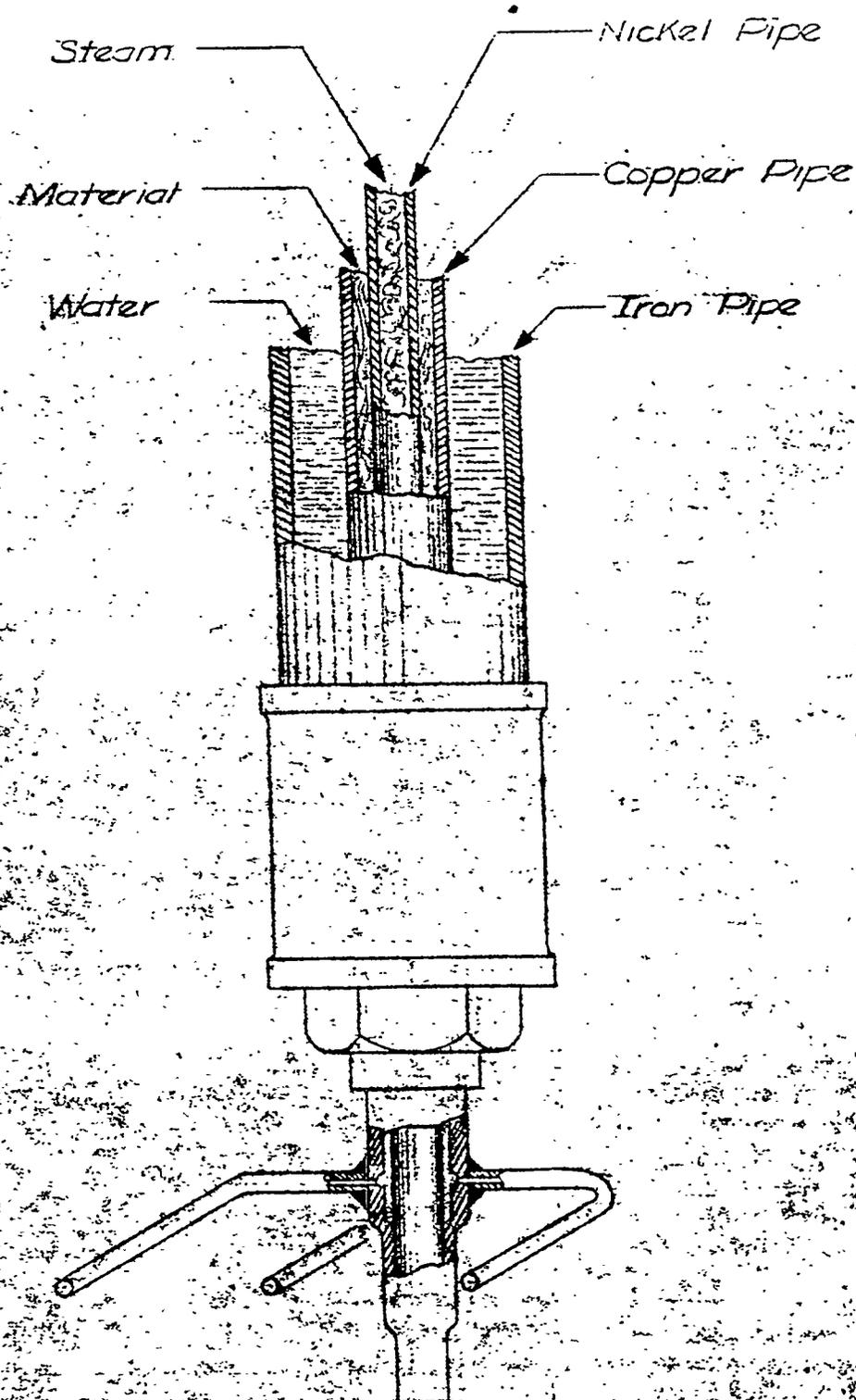
The vital process in the plant is brought about in the column. The operating procedure, then, is to introduce the material into the columns where the desired change occurs. The remainder of the plant functions to bring about the conditions which allow the columns to function in the most efficient manner. A rack consists of 102 columns, but since the columns are identical in construction a column will be described first.

The Columns

The column consists of a nickel pipe about 48' long surrounded by copper tubing. This tubing is then inserted into an iron jacket making three concentric pipes. The internal diameter of the copper and the external diameter of the nickel are selected to produce a small annular space between them and it is in this annular space that the material is introduced. Around the copper tubing is the galvanized steel jacket. Steam at about 1000 pounds per square inch pressure and 545° F. passes through the central nickel pipe and provides the necessary heat for the process. Water passes through the outer iron jacket and provides the necessary cooling. See DWG. 9-12. This water enters the jacket at the bottom at 150° F. and leaves the jacket at the top at a temperature around 160° F. having been heated up by the heat from the steam passing through the fluid in the annular space. Therefore, we have a temperature differential of about 545-165 = 390° F. across the column over its entire length. This temperature differential is essential to the process and cannot be allowed to vary. The equipment for controlling both steam and water is discussed in another Section.

Construction of the Column

The 48' column is not made up of single, continuous lengths of nickel and copper. The nickel inner tube is made by welding 24' 12' and 6' pieces together. The copper tube is made by silver soldering 24' lengths together. The silver solder joint is made by placing a copper sleeve over the lengths to be joined together and inserting a ring of silver solder in the recess in the sleeve. Heat is applied, the silver solder melts and is drawn by capillary action into the joint, thus sealing it. At each end of the column a short nickel sleeve is similarly silver-soldered to the copper so that the outer tube projecting from the water jacket is nickel, rather than copper. This arrangement permits the use of copper in a large part of the column and is desirable since copper is more economical than nickel and can be fabricated to close tolerance with ease, but it still allows the nickel tits to be welded to nickel. This is important since the tolerance in the annular space is probably the most critical in the entire system. The annular space is maintained by small nickel buttons which are spot welded to the inner nickel pipe and act as spacers.



THE FERGLIVE CORP
 Technical Divn
 COLUMN
 Sectional View
 Date: June 20, 1943
 Dwg. No. 9-12
 Dwg. by: Leder Appd

Copper is used on the cold side and nickel on the hot, because the high inside temperature would cause a slow annealing of the copper with a consequent reduction in mechanical strength.

Connections Between Columns

At the top and bottom of each column there are three small nickel tubes which project from column. These communicate with the annular space in the column and are used to introduce the material into the column and to remove it from the column. One of these tubes or tits as they are called is used to remove samples from the system for analysis and is capped off when the column is in operation. The two are used to connect columns to one another. Columns are connected by intercolumn connectors (frequently referred to as ICC's) These are small nickel lines ($1/8" \times 3/32"$) equipped with Lunkenheimer fittings so that they can be attached to the nickel tits. To the nickel link lines are soldered copper tubes through which water can be passed. This is in effect, then, a valve between individual columns since by passing water through the copper tube the process fluid is cooled until it solidifies and a plug of solid material formed, thus stopping the flow of fluid. On turning off the water the heat of the duct causes the plug to melt, thus allowing material to flow. These intercolumn connectors are used to connect adjacent columns on both the top and bottom of the rack.

The Conduit System

Also located at various regions of the rack are other nickel tits which are the ends of the conduit lines through which the material flows from the transfer room to the rack. Some of the racks have 36 conduit lines arranged in groups of six with 18 on each side of the rack. In the groups of six the conduits on the ends of each group are of a larger diameter than the other four in the group. These larger lines have an internal diameter of $3/32"$ while the other four in the group have an internal diameter of $1/16"$. Where possible it is advisable to use the larger tubing. Several other racks have only 18 conduits arranged in groups of three with nine on each side of the rack. The change was made to the smaller number in order to have only three lines in a single steam jacket. This was necessitated by the discovery that if any one of the conduit lines broke inside the conduit the material and its hydrolysis products corroded the conduit lines and plugged up the steam jacket. All of the lines in these racks are large bore with an I. D. of $3/32"$.

The Material Circuit

The material itself flows to the rack through one of the conduits. From there it passes through a nickel line or "jumper" to one end of the rack. At this point, the nickel line is introduced into the center of a coil of $3/8"$ copper tubing through which water can flow.

[REDACTED]

This arrangement is used in order to be able to freeze the material between the conduit and transfer rooms and the rack so that in the case of an emergency both will not be emptied. From the freeze-off coil the material is introduced into the side tit of the end column of the rack. The material then passes from the first column in the circuit out through the nickel line in the intercolumn connector and into the side tit of the next column and so on. The material thus follows a circuit through the bottoms of all of the columns, filling the column which stands in the circuit like a standpipe. Placed in series with this circuit somewhere in the loop is a convector system through which the material passes and from there it goes back into the columns. After passing through the bottoms of 25 or 51 columns it passes through another freeze-off coil into another conduit through which the material returns to the scale tank in the transfer room. Such a system of conduits, convectors, columns, freeze off coils and jumpers through which the material can circulate is called a circuit. Actual details on the building of a circuit and rules to be followed are given in another part of this Section.

The Convector Loop

It was stated in the section on properties of the material that one of the important properties from the standpoint of the plant is the tremendous change in density with temperature. This is important both in respect to the fact that flaming of lines must be done very carefully but also in the effect which is utilized in the circuit on the rack.

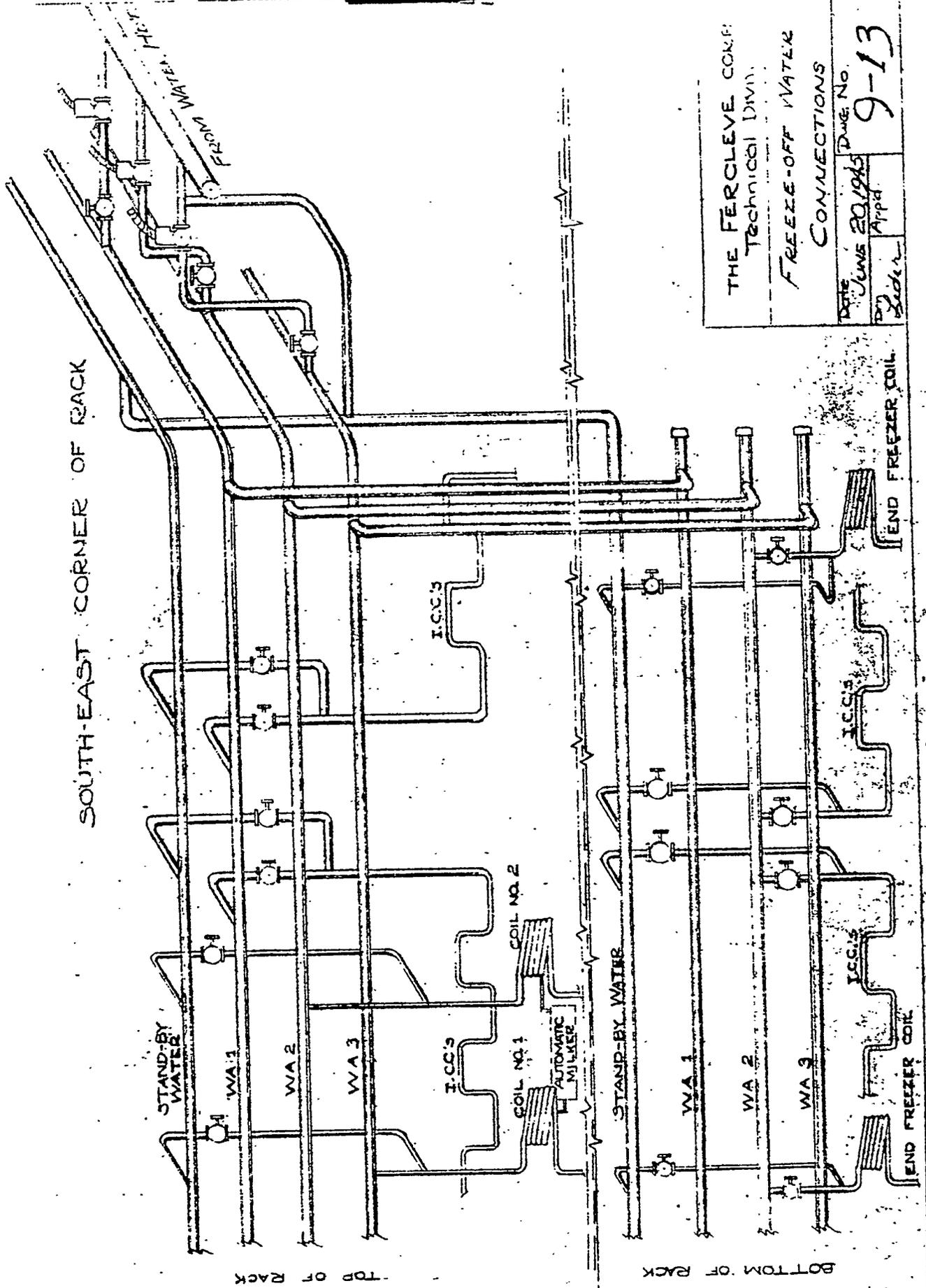
The temperature of steam at 100 pounds pressure is approximately 175° C. and the temperature of steam at 5 pounds pressure is approximately 110° C. The density of the material at 175° C. is about 3.0 g/cc. The density at 110° C. is about 3.6 g/cc. This difference is so great that use is made of it in the system. The material is passed through one tube which is heated by 100 lb. steam and then through another which is heated by 5 lb. steam. The tubes are located in twelve pairs of pipes with six pairs on each side of the rack. Since the density is less in the 100 lb. pipe than in the 5 lb. side, the material rises in the 100 lb. line and falls in the 5 lb. line giving the pumping action necessary to have the circulation desired. While the whole system is at a pressure of 1500 pounds or higher there is no differential in pressure except that given by this convection current occurring in the convectors. The total difference in pressure is of the order of only three or four pounds per square inch but is sufficient in the system to keep the material flowing around the circuit. However, it is not sufficient to push any material through the system if there is any type of material plug or constriction in the circuit. For this reason, "plug-hunting" is one of the most important parts in the operation of the plant. A separate section is devoted to looking for trouble of this type.

Valves

The essential parts of the rack itself of interest to the operator are the columns and their connections, the convector systems and the freeze-off system. See Dwg. 9-13

[REDACTED]

SOUTH-EAST CORNER OF RACK



TOP OF RACK

BOTTOM OF RACK

THE FERCLEVE COMP.
 Technical Divn.
 FREEZE-OFF WATER
 CONNECTIONS

DATE	JUNE 20 1945	DWG. NO.	9-13
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In addition to the parts of interest in the material system, the columns have five valves on each one. At the bottom of the rack is a four inch water valve on each column. These valves are located behind the columns inside the rack. Another set of valves are the condensate outlet valves which are located under the ducts in the front of the row of columns. This is a smaller valve on the 2" steam line. On top of the column is the steam inlet valve located in back of the column and inside the rack and also a four inch water outlet valve leading to the upper water header.

At the top of the water outlet elbow is another line running up to the air vent header. This is a one-inch line which permits the air released from solution in the circulating water by the increase in temperature as it rises to be vented to the roof. This line would also take care of an excess of water in case the make-up water valve should open and the system be unable to care for the increase in water. See Draw. C-14. On most of the racks this header is tiped so that 8 pound steam could be introduced into the four-inch water jacket but since it was found that the high temperature produced was detrimental to the rubber hose in the water system, this has been discarded.

Other aspects of the rack construction such as the condensate return system, the trap systems for the convectors and conduits and the condensate float control are included in the description of the Steam and Power Department.

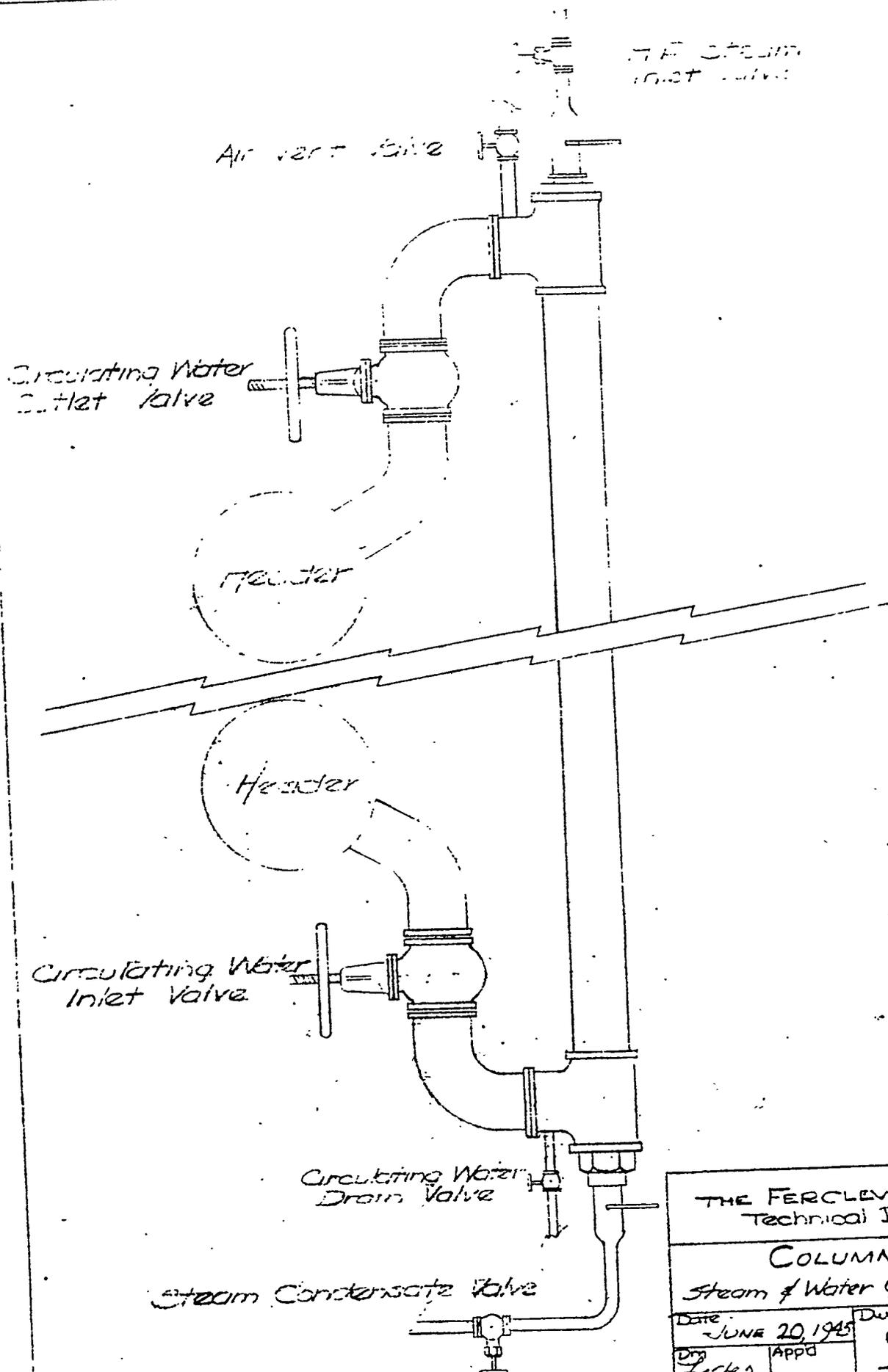
2. Rack Conditioning Procedure

Introduction

When the columns, convectors and conduit lines are furnished to the operating personnel, they are wet, dirty and greasy. Such is the great chemical activity of the process material that it will react to a certain extent with practically all substances. Especially violent and irreversible is the reaction with water and oils. Among the reaction products are non-fluid, non-volatile materials which tend to accumulate in vital spots in such a way as to impede or stop the flow of the process material. A number of the connecting link lines possess an inside diameter of only 1/16" and it is possible to plug these lines by the action of as little as a few milligrams of water. From past experience it has been determined that if the operating system is freed of moisture and reacting substances, no difficulty is experienced in setting equipment into satisfactory operation.

Principles of Conditioning

The conditions of moisture removal must be particularly rigorous since there is a strong tendency for all the water in the system to collect at a cold spot. These remarks lead also to the conclusion that it is essential that all components of the system be free of water leaks.



THE FERGUSON CORP. Technical Divn.		
COLUMN Steam & Water Connections.		
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For instance, a convecter line or conduit line immersed in low pressure steam will accumulate water within it if even the smallest leak exists. Furthermore the copper wall of the column being under a great tension under operating conditions sometimes springs a leak at a sleeve joint midway up the column, permitting circulating water to have access to the process region of the column. Another way of producing non-fluid plug material is by the action of the process material on oxides or carbonates. The oxide is introduced into the column at the time of silver soldering the copper. Carbonates can be introduced if the columns are allowed to stand wet while exposed to air.

The following procedure, accordingly, has for its objective to guarantee that all reducing substances, water and oxides shall be removed completely from the system:

a. Column Cleaning and Drying

1. Blow most of the moisture out of the columns by passing in dry air from the top for 10 minutes.

This and all other operations involving the passing of liquids and gases can be done conveniently by use of manifolds made from copper tubing. These manifolds may be made by silver soldering copper tubing into another long piece of copper. The manifolds may be made up in whatever lengths desired but are frequently made with either nine or seventeen outlets on each one. The seventeen tit manifold is fairly satisfactory since it can be moved only twice to cover one side of a rack. The outlets are soldered on eleven inches apart to conform to the distances between the center tits of the columns. In the end of the outlet, a nickel tube is soldered or brazed into the end of the copper. This nickel tube is made of pipe having an outside diameter of 1/8" to which is fastened a standard Lunkzheiner connection by which it can be fastened to the center tit of the column. One end of the manifold may be soldered shut but the other end usually has a standard 3/8" Lunkzheiner or tapered fitting to fit the outlet on the air line or on the pressure tank when forcing liquids through the columns.

2. Degrease roughly using tetra-chlor ethylene solvent or carbon tetrachloride.

Using the manifold mentioned in the previous step in conjunction with a pressure tank, this can be done rather rapidly and easily. The pressure tank is a nickel container having two openings with standard fittings in the top. One of the openings is a pipe which goes just below the surface of the container.

The other opening has attached to it a longer pipe reaching to the bottom of the tank. Compressed air is forced in one opening and when a liquid is contained in the tank will force the liquid out the second opening and up into the columns. Usually the tank is fitted with a gauge for measuring the air pressure and a release valve to let the pressure down when sufficient fluid has passed through the column. Usually liquid is passed through the column until it appears to be clear. Pass the fluid from the bottom to the top. It is usually satisfactory to pass about one-half pint through each of the 3 top tits.

When rack is degreased blow out the columns with air through a manifold from the top. Open all tits.

3. Test all columns in the rack with 100% steam.

The difference between the steam and the circulating water temperature should be about 400° F. The object of this step is to subject the columns to the stresses which they will encounter during normal operation. Initially some water will blow out the bottom tits in the form of a mixture of water and steam. However, if this moisture continues to issue forth, the column is undoubtedly bad. Usually about one out of every 100 columns has failed during the application of this test. Shut steam off.

4. Make a final and careful solvent wash using clean solvent and passing fluid through all top tits. Crack bottom tits until liquid is clean. Drain fluid back into the tank.
5. Attach manifold at top and blow out solvent with air for 10 minutes.
6. Wash the columns with dilute hydrochloric acid.

This acid is made up in the approximate ratio of six parts of water to one part of concentrated acid. This step is usually done by forcing the acid up the columns with the pressure tank and collecting the acid at the top of the rack. The usual method of collection of the acid or other liquid is to attach a short length of rubber hose to the tit and pass the fluid into a milk bottle.

This enables one to determine if the fluid is coming through clean. The clean acid may be used again but any dirty acid should be discarded.

7. Wash the columns with water until the water is clear following the same procedure as with the hydrochloric acid.
8. Blow out water with air for five to ten minutes. Open all tits to determine if they are open to the passage of air.

If for any reason conditioning procedure must be interrupted at this point, the columns should be flushed with nitrogen and capped. In general, any interruption of the conditioning procedure should be avoided particularly between the time when the column is first wet with solvent and the time it is filled with nitrogen. Do not plan the work so that any of the columns are left moist and with air in them for any appreciable length of time. If the columns are left wet for as long as eight hours, they should be reconditioned starting with the acid washing step.

9. To start the drying procedure, attach a steam traced copper manifold to each of the four circuits of the rack.

See that the duct heating steam is on and that the 1000 lb. steam is turned on the rack. Close the duct doors as far as possible. Adjust the circulating water temperature to 150° F. The steam traced manifolds are 25 outlet manifolds which have another line soldered to the main section of the manifold. This is connected to a low pressure steam line and is used to heat the manifold so that drying may be facilitated.

10. Evacuate the system for one hour with the top middle tit of each column left uncapped.

The purpose of leaving one tit uncapped is to allow air to stream through the columns, thus sweeping out the gross amount of water and water vapor left in the columns after the water washing treatment.

11. Evacuate for two or more hours with all the tits capped off at a vacuum of 27 inches or more.

[REDACTED]

If slight leaks exist in a circuit, it is unimportant at this time. No effort need to be made to locate minor leaks until the fresh air treatment.

12. Disconnect one side of each intercolumn connector and cap off the tit and link line at both bottom and top of the rack. Remove manifolds.

The purpose of this step is to isolate each column. This step is unnecessary if the intercolumn connectors have not yet been installed.

13. Attach a connection from a nitrogen gas cylinder to the middle bottom tit of the first column of the circuit or to the tit from which the intercolumn connector was removed. Attach to the top middle tit of this first column a rubber connection leading to a glass trap immersed in liquid nitrogen.

The trap must be attached before any nitrogen gas has been blown through the column as the plan here is to be able to trap water or water vapor which has accumulated in the column in order to magnify the test. The columns must have stood at least one hour after bringing them up to atmospheric pressure before making this moisture test.

14. Blow nitrogen gas at a pressure of 5 pounds on the cylinder gauge through the column for precisely one minute.
15. Inspect visually the moisture collected in the glass trap during this one minute test.
16. Record extent of frost formed or moisture collected giving a rough estimate of the amount as far as possible. Repeat the test for all the column of the circuit.

The outside surface of the glass trap must be wiped free of frost with a dry cloth before making this observation.

Occasionally, it is satisfactory to do this test using two columns in each test. If this seems to be bad the test should be run on each of the columns.

17. Isolate any suspicious columns by installing bypasses with water freeze-off line attached.
- [REDACTED]



18. In case of doubtful columns, isolate these, let stand for six hours and repeat the test.
19. Connect or re-connect intercolumn connectors.
20. Make a moisture test on the column circuit by the following nitrogen method for thirty minutes.

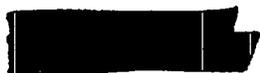
For details of this test see section on Moisture Tests.

21. While step No. 20 is in progress make a similar moisture test by the flowing nitrogen method on the convector and conduit system.
22. If the moisture tests of the circuit and of the conduit and convector loop system show less than 0.05 g. connect the two together and evacuate to 28 inches.
23. Fill the whole circuit with fresh air under pressure, test for leaks, repair any bad connections and by-pass top and bottom any columns with leaks in the welded joints.

Fresh air leaks may be tested for by one of three methods:

Testing for Leaks with Fresh Air

- (a) Allow illuminating gas to stream over the connection from an unlighted Bunsen burner. If fresh air is present in large enough quantities, the gas will be ignited.
- (b) Tie a cloth rag to one end of a metal or wooden rod, soak the cloth with concentrated ammonia and probe about the welded joints or Lunkenheimer connections with the rag. If the fresh air is leaking out, white fumes should indicate it.
- (c) Lay about the weld or connection a special chloride indicating test paper (obtained from the chemical storeroom of Laboratory No. 2) If fresh air is leaking out, the paper will turn brown or brownish blue. This test is particularly good for locating the exact spot of a leak.



[REDACTED]

In testing for leaks, be sure that all Lunk-enheimer connections are tested as well as all welded joints, the later where the tits are welded to the columns and where the inside and outside columns are welded together. Be sure to test both top and bottom of a rack as well as the conduit and convector loop connections.

b. Conduit Line Cleaning

1. Conduit lines should be rough dried by blowing air through them.
2. Lines should be degreased using carbon tetrachloride or other solvent and the solvent removed by passing dry nitrogen through the line while flaming the tits at the terminal boxes on both ends of the conduit.

It is necessary that the conduit be tested with 5 pounds steam during this procedure.

3. Conduit lines should then be capped on both ends and allowed to stand for 24 hours with steam on the line.
4. At the end of the 24 hours a test for leaks can be made by introducing dry nitrogen at the transfer room terminal box and running a moisture test qualitatively. Great care should be employed in this step since a number of conduit lines have been found to possess water leaks.

When not in use all conduit lines should be left uncapped.

c. Cleaning of Convector Lines

The procedure used in cleaning the convectors is essentially identical to that employed for the conduit lines.

d. Cleaning of Link Lines and Inter-column Connectors

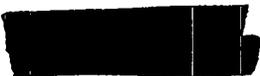
1. Lines are degreased and flow tested with grease solvent.
 2. The solvent should be blown out of the lines and convectors and placed in the hot cabinet in the materials shop. The temperature of this box should be maintained at a minimum of 120° C.
- [REDACTED]

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3. After remaining in the box for one hour the lines should be blown out with dry nitrogen and capped.

3. Operating Instructions for Rack

a. Completing the Material Circuit

At this stage the various component parts of the rack have been cleaned and dried. They should now be connected together with great care being taken that no moisture be permitted to get into the system. The connections up to the transfer room duct and including all links, intercolumn connectors and convectors should be made with the orientation being that desired for the operation of the rack.

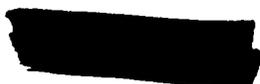
1. The conduits should be chosen from among the list found to be open in the cleaning procedure. The two end conduits on each terminal box should be used where possible.
 2. The link lines should be obtained from the conditioning shop of the correct length to reach from the terminal box to the ends of the circuit. It is customary to have 26 columns in a circuit but there are several special cases where this is not followed throughout the plant. This would make four circuits per rack.
 3. The freeze-off coils should be attached to one of the end columns of the circuit. On the other end the freeze-off coils should be attached to the 100 lb. convector.
 4. The convector loops should be placed at the end of the circuit using lines numbers 2 and 3 whenever possible. The convector loops should be set up so that the flow proceeds from the low numbered columns to the high numbered columns. For top circulation systems the flow should be from high numbered columns to low numbered columns along the top of the circuit.
 5. Every circuit should have a flow indicator equipped with thermometer wells and thermocouples wherever possible.
 6. Provisions should be made for evacuating the system and introducing fresh air. The rack should be pumped to a vacuum of 28" and fresh air passed into the system to a pressure of 10 lbs. The gas should be bled from all top center tits and at the terminal boxes both on the rack and in the transfer room.
- 

7. If any delay is to be encountered in setting the rack into process operation the equipment should be left with a positive fresh air pressure. It is highly desirable to start process fluid flowing in immediately after fresh air treatment has been completed.

b. Starting-up Procedure

1. Check all valves. Be sure that all steam, water, and air vent valves on the columns are open on all the columns which are in the circuit. It is usual for the steam to be turned off from columns out of service but not the water valves.
2. Make sure that the circulation water is flowing. This should be done by the Steam and Power Department but the operators should always check to make certain that it is flowing.
3. Have the steam turned on through the by-pass and bring circulation water temperature up to 150° F.
4. Have the main steam valve opened and bring pressure to 1000 pounds.
5. At this point columns should be clean, dry and full of fresh air.
6. Check to see that all tits of all columns have connectors or caps on them and that these are tight and not crossthreaded.
7. Check to see that the freeze-off water system is complete.
8. Check to see that hook-up is complete and that it corresponds to diagram on rack sheet.
9. Check especially to see that convectors and conduit lines are correctly connected. A common mistake is to connect to one line at one end and to some other convector or conduit line at the other.
10. Check location of thermocouples.
11. Check duct temperature to see that it is above 80° C.
12. Check convectors and conduit lines to see that they are properly hot.

The rack is then ready to be filled or ready for operation.



4. Filling the Rack

a. Filling from the Transfer Room

The original method of filling a rack with process material has been largely supplanted by a more rapid method which requires less manipulation of equipment. Both methods will be described. The first method which is that of filling the rack from the transfer room is more fully described in the Section on the Transfer Room since it is essentially transfer room operation.

In following this procedure the rack has the complete circuits hooked up with at least four gauges on the rack with two at the top and two on the bottom. The freeze-off water is not turned on the top or the bottom of the rack. When the transfer room storage tank reaches a pressure of 1500 pounds material pressure, the storage tank tits are unfrozen and the material is allowed to flow through one of the conduit lines into the rack. The circuit being filled usually exhibits the phenomenon of "pitching". The main problem involved in the filling of a rack is to get the material into the columns as rapidly as possible to cut down the time which the circuit pitches. This frequently takes place quite rapidly with the circuit filling in an hour or less. However, it is often the case that because of improper conditioning, a plug of hydrolyzed material is formed in an intercolumn connector which prohibits the material from flowing any farther. Weight-time readings should be taken in the transfer room and when the weight stops falling off it may be assumed that the circuit is no longer being filled. A circuit will usually take between 90 and 100 pounds before being pressured up high enough to stop pitching.

Pressuring up a Circuit

If the weight stops dropping before this much has come into the circuit, it is probable that a plug exists. The location may frequently be checked by finding out where the circuit stops pitching. If the material is being put into the circuit from a given conduit line and the circuit pitching, one for example, half way down the circuit it is probable that a plug has been formed in that neighborhood. The usual method of procedure in this case is to obtain a nickel jumper and attach it from the center tit of the column which is pitching and attach it to the center tit of another column which is farther down the circuit thus by-passing the intercolumn connector. This will frequently allow the material to flow into the other part of the circuit and complete the filling of the circuit satisfactorily.

It is common practice to place a gauge on the conduit line in the transfer room which is not being used to fill through and notice when the pressure is up to standard on that gauge.

In other words, if a circuit is being filled through one conduit and the material flows around the circuit until it pressures up the gauge on the other end of the circuit, it may be assumed that the circuit is full of material. It may be necessary to raise the pressure to 1700 p.s.i. to get enough pressure to stop the pitching but it is very desirable to have contact with the scale tank so that this may be transmitted through the circuit as soon as possible. The steam consumption of a circuit which is pitching is usually about twice the steam consumption of circuit which is not pitching. When the pitching stops, indicating that the circuit is filled, the gauge may be disconnected from the end of the conduit and the conduit may be hooked to the scale tank with a nickel jumper. The freeze-off water should be turned on the top of the rack and tests for circulation may be begun.

b. Filling From the Floor of the Process Building

This method is used extensively since it is considerably more rapid than is the other method. It consists in assembling a transfer pot containing a shipping tank on the floor of the building. It is hooked up with a line which can be connected to the 125 pound steam system on the rack. There is installed a steam pressure gauge and valve on the upstream side of the transfer pot and a line for condensate removal at the bottom of the regular pot. This is fitted with a steam trap to build up the pressure in the tank.

Transferring

It is customary to use large nickel tubing for the material to flow from the transfer pot into the rack. The tubing used for this usually has an outside diameter of 1/4". This is connected to any convenient column and the pressure turned on into the transfer pot. When beginning a transfer from a full tank it is quite inadvisable to allow a steam pressure on the tank of more than 15 pounds. This will permit the material to melt in the tank without any danger of reaching excessive pressures.

After 20 or 30 pounds have been transferred into the rack the steam pressure may be raised to any value which permits fairly rapid flow. After the removal of several pounds there is no danger of having anything but liquid material in the tank so the only expansion that takes place is that depending on the increase of vapor pressure with the increase in temperature.

Completing the Filling

Using this system it is possible to put 70 or 80 pounds of material in the circuit in this manner. It is not usually possible to fill the rack completely in this way since the temperature  is such that some

pressure is exerted in the backwards direction to the flow. Consequently, it is usually necessary to pressure the system from the transfer room in much the same way as the other method of filling was finished. In this method the conduits are not attached to the storage tank so when the material stops coming over from the transfer pot on the floor of the plant the pot may be disconnected. When the pot is disconnected, the conduits should then be attached to the scale tanks in the transfer room and the pressure raised in the same manner as was done when the original filling was done in that manner. This method is more satisfactory particularly because the number of plugs formed seems to be much less than when the other method is used.

c. Filling From the Top of the Rack

While this method has not been used, there seems to be no good reason why additional advantages should not accrue by filling the rack from the top rather than from the bottom. When the rack is nearly full after filling from the bottom there is a back pressure caused by the height of the material column in the rack which probably accounts for the fact that it is not completely filled.

It would seem that this could be eliminated by moving the installation from the floor to the top of the rack thus eliminating the pressure forcing the material back into the pot. It would be possible to make the same connections at the top of the rack as at the bottom and this method seems to have some possibility of development if a complete circuit needs to be filled.

5. Locating and Eliminating Plugs

Introduction

By the work "plug" we mean any obstruction which prevents the process fluid from flowing through its normal circulatory system. In addition to this type of solid plug there may exist "partial plugs" which hinder the flow rather than stopping it entirely. The proof that partial plugs exist is much less direct and convincing than the evidence for complete obstructions, but it is reasonable to suppose that sediment may collect in spots, slowing the rate of circulation, or that solid plugs may form gradually and constitute partial plugs before becoming complete solid plugs.

The general problem of detecting, locating and eliminating plugs in process circuits is one of the most important encountered in plant operation, for unless a circuit has many hours of good circulation every day, it is not operating at peak efficiency.



Formation of Plugs

Plugs may be formed by any of the following methods, but little can be written at the present time about the details of plug formation, as the mechanism of the forming of plugs is in many cases obscure.

(a) Chilling of Process Fluid Lines

As the process fluid solidifies at approximately 70° C. at the operating material pressures, it is obvious that if the temperature of any section of the fluid in the circulating system falls below this value, the material will solidify, thus forming a plug and stopping circulation. This type of plug is the most frequently encountered and, fortunately, the most easily remedied.

(b) Hydrolysis of Process Fluid

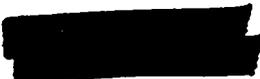
Liquid or gaseous process fluid coming in contact with liquid water or water vapor rapidly reacts with the water liberating an acid and producing a high melting solid which forms usually an immovable plug. Because of the difficulty, great difficulty, in drying completely all the columns, intercolumn connectors and other component parts of the circulating system, plugs of hydrolyzed material usually occur chiefly in the initial filling of a circuit with material. Once a circuit is in good operating condition with no internal water or steam leaks, no plugs can be formed as a result of reaction with water except when the circuit is opened to the atmosphere. Even in this case there is no danger of a plug of hydrolyzed material forming if the proper precautions are taken in preventing water from condensing in the exposed opening.

(c) Accumulation of Sediment

Details and reasons for the formation of plugs by the accumulation of sediment are little known at the present time. Inasmuch as plugs have apparently formed in certain cases for no obvious external reason, it is believed that these plugs must have been built up by the accumulation of sediment. The nature of the sediment has not been determined.

(d) Reaction of Process Fluid With Organic Matter

Sediment and plugs formed from this sediment can occur when the process fluid reacts with organic matter. In fact, any chemical reaction of the material will form plugs.



(e) Welding Shut of Joints

In the original fabrication of welded joints or in the later repair of such joints, the welder has occasionally welded such joints completely or nearly completely plugged up with metal. The only cure for such accidents is a new joint made by a good welder.

Proof of Plug Existence

(a) Pitching Test

If pitching is occurring throughout a circuit at the same time that the material pressure in the scale tank supplying the circuit, is up to the normal operating value, then it is known immediately that at least two plugs exist in the circuit, that both conduits or their associated connections are plugged; otherwise the high pressure of the material in the scale tank would be transmitted to the rack and the pitching stopped.

(b) Circulation Test

If circulation in a circuit stops, if there are no open connections, and if the convector loops, conduits and inter-column connectors are up to temperature, the circuit is plugged. As it is not always evident whether circulation has really stopped or not, the methods for testing for the presence or absence of circulation shall be discussed here in some detail.

Observe the Hoffman Flow Indicator for indications of a temperature decrease across the meter in the correct direction for flow. At the bottom of the circuit the flow in the plant is always from low to high column numbers and at the top in the reverse direction. Inasmuch as a slow surge of material into or out of one end of a plugged circuit may give rise to an apparent flow in the correct direction for approximately 15 minutes, it is necessary to watch the Flow Indicator over a longer period of time than this. If the temperature difference remains steady in the correct direction, circulation probably exists. If the temperature difference changes, even going negative in many instances, surging, not real flow, is occurring, or perhaps a surge superimposed upon a weak flow. These surges are particularly noticeable at the moment of starting up a circuit.

There may be another cause of misinterpretation of the Flow Indicator readings. In certain cases the thermometer well of the Flow Indicator on the left may not be located with respect to the tit to which it is attached in exactly the same way as the thermometer well on the right, thus leading to an asymmetry in thermal conductance and a difference in the temperature readings.



Such as ΔT is not only usually low, but the temperature itself is also low and only slightly higher than the duct temperature.

When pitching or hammering is occurring in the column to which the Flow Indicator is attached, the thermal effects of the circulating flow may be entirely masked by virtue of the rapid oscillation of hot process fluid through the Flow Indicator. In this case the material pressure in the circuit should be raised until the pitching and hammering stop. The observations for flow can then be made.

In summary the criteria for circulation can be listed as follows:

- (1) ΔT on Hoffman Flow Indicator persisting in correct direction for half hour or more.
- (2) Centigrade temperatures on lower side 150° or higher.
- (3) ΔT equal to 30 to 50° C. when lower temperature is in the neighborhood of 150° C.
- (4) ΔT equal to 20 to 30° C. when lower temperature is in the neighborhood of 180-190° C.
- (5) Hot side thermocouple temperatures 350 to 400° F.
- (6) Decrease of ΔT to nearly zero when the circuit is frozen off and the circulation purposely stopped. If no decrease occurs in the ΔT when the circuit is frozen off, the circuit is definitely plugged.

(c) Elimination of Cold Spots

After it has become certain from the above criteria that no circulation exists, the next step is to search for and to eliminate any relatively cold spots in the circuit. Possible locations are as follows:

- (1) Cold ducts, particularly at the ends which in cold weather should be shielded from drafts by tarpaulins.
- (2) Cold spots formed by drafts through holes in duct work.
- (3) Cold ducts due to water dripping on them, shield ducts from the water or stop the water leak.

- 
- (4) Cold conduits. Check conduit steam pressure, valves and condensate drains.
 - (5) Cold convector loops and cold jumpers between loops. High temperature should be 170-180° C.; cold temperature 105-110° C. Check steam pressures, valves and traps. Flame jumpers.
 - (6) Link lines touching cold duct doors, or cold copper freeze-off lines. Change position of link line to free position.
 - (7) Cold link lines due to water dripping on them or to steam blowing on them. A bad steam leak into the duct may seriously affect circulation as a result of the combination of expanding steam and wet vapor.
 - (8) Cold ducts, link lines or tits in Transfer Room. Heat link lines and tits with flams. Check steam pressure, valves and trap or duct heating line.
 - (9) Intercolumn connectors or transfer room tits cooled by freeze-off water leaking past closed valves, or by water backing up common drains.

In flaming link lines care should be taken to begin the heating at an open end and then to work slowly along the length of the line.

It is only after eliminating all of the above cold spots in a circuit that a non-fusible plug can be assumed to exist. The next series of steps in plug elimination can then be taken.

Detection and Elimination of Plugs in Conduits and Convector Loops

The first step in locating and eliminating plugs is to test the conduits, convector loops and associated connections to make sure that they are plug-free before beginning the difficult operation of trying to locate and eliminate plugs in intercolumn connectors. However, before any actual work is done on the rack the history of previous rack operations should be received inasmuch as hints as to possible plug locations might be obtained. For example, if the scale tank on a circuit had just been changed before circulation stopped, one would suspect the plug to be located in the link lines, conduit and scale tank tits involved in the transfer. Attention should, therefore, be focused first on this location and these link lines and tits proved to be clear before working on other sections of the material flow system.



(a) Pressure Variation Method

A method of varying the pressure which at the same time causes process fluid to surge through the conduits seems to be best for not only locating plugs but also possibly eliminating them by flushing them out of the line.

Steps in this process with reference to the circuit diagram may be listed as follows:

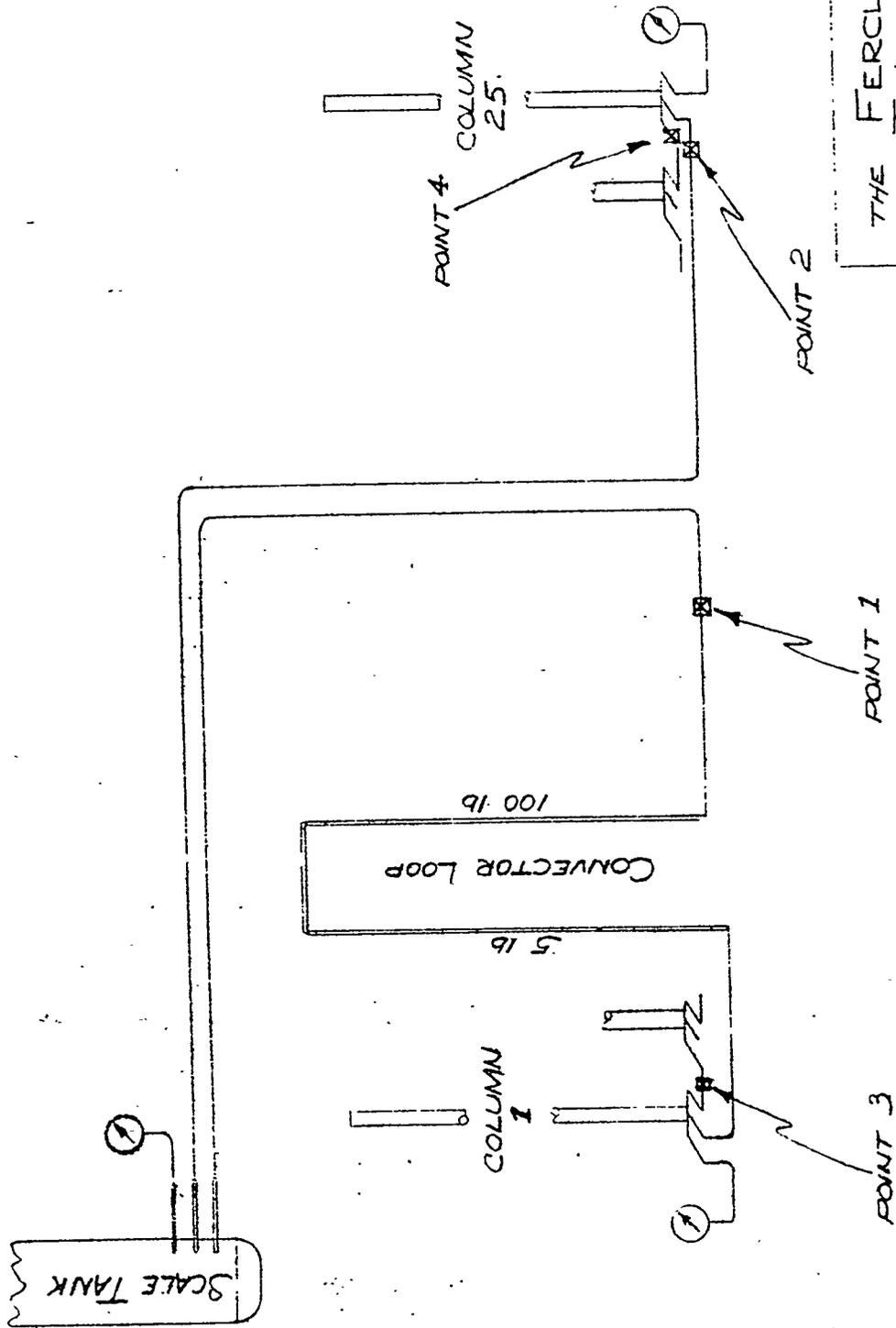
1. With the material pressure in the rack at the normal value of 1500 p.s.i. Freeze off at points 1 and 2.
2. Raise the material pressure in the scale tank to 1700 p.s.i.
3. Freeze off at points 3 and 4.
4. Thaw point 1. If the conduit and convector system leading to Column 1 is clear, the gauge reading on column 1 will immediately rise to 1700 p.s.i.
5. Freeze 1 and thaw 5. The pressure in column 1 will now drop to slightly above 1500 p.s.i. and material will flow or surge from the convector loop and column into the rest of the circuit.
6. Thaw 2, repeating at the column 25 end of the circuit the process described above.
7. Freeze 2 and thaw 4.
8. Finally check for freedom of plugs across the whole bottom of the circuit by thawing at which ever end is free observing change in gauge pressure.

The above described method has several advantages; not only does it serve to locate a plug, but the pressure change cause a surge of fluid which may possibly sweep or flush a plug out of a conduit or convector loop. Furthermore, it enables the operator to check both the conduits and convector loops system and the bottom of the circuit in a minimum number of operations.

(b) Material Withdrawal Method

In case either of the gauges used in the above discussed tests fails to respond to the increase in pressure or does not show as rapid a change in pressure as the gauge on the scale tank, and if the plug has not been flushed from the circuit, an attempt should be made to clear the line or to locate more exactly this plug by the following procedure:





THE FERCLEVE CORP
 Technical Unit.

PRESSURE VARIATION METHOD
 OF
 PLUS HUNTING

Date July 3, 1945
 Dwg No 9-15

- (1) Break the link line connecting the conduit or convector loop to the end column of the circuit at the column end of the link line.
- (2) Attach to the link line a #20 cylinder which has previously been weighed and show to pass fresh air.
- (3) Flame the link line and bit of the cylinder until material begins to flow into the cylinder.
- (4) As soon as the temperature of the cylinder has stopped rising, freeze off and reconnect link line to column.
- (5) Weigh cylinder.

If no material has flowed into the cylinder, the indications are that not only is the conduit plugged, but also that the plug is fairly near to the rack. If up to 2 or 3 pounds of material are obtained, the plug probably exists near the scale tank. If the cylinder is filled to capacity, weight of the material in the cylinder about 1 pound, there is no plug in the circuit.

Although previous pressure tests may have demonstrated the existence of a plug, the act of withdrawing the five pounds sometimes flushes the plug out of the system. With the material in the scale tank up to pressure, there is considerably more force available for dislodging the plug than there is during normal circulation, or during the surging when making a pressure test.

If the plug still persists after the material withdrawal test, the following steps may be taken:

- (1) Repeat the material withdrawal attempts from other locations in the conduit or conduit-convector system.
- (2) Replace removable link lines with freshly conditioned link lines.
- (3) If the evidence points to a plugged bit on the scale tank, the tank should be cooled down and the bit drilled out by Maintenance.
- (4) If none of the above operations is effective in opening up the circuit, a new, freshly conditioned conduit or convector should be resorted to.

Location and Elimination of Plugs in Intercolumn Connectors

(a) Pressure Variation Method

An indication of how the pressure variation method can be helpful in locating plugs in intercolumn connectors has already been mentioned above.

The material pressure gauges should be distributed across the plugged circuit as judiciously as possible; i.e., in places such that the subsequent pressure test will show as closely as possible the actual location of the plug. Pressure gauges on columns at the top of the circuit can also be read during the test as there is, of course, only a slight difference in pressure between the bottom and top of a circuit, approximately 80-90 pounds. However, because of the numerous places plugs can occur in a circuit of 25 columns, a single pressure test cannot hope to do more than to point roughly to the plug location.

Steps in the operation of carrying out the pressure test can be listed as follows:

- (1) Attach gauges to selected columns, as many as time and availability permit.
- (2) Freeze off both ends of the circuit.
- (3) Lower or raise the material pressure in the scale tank until it is 200 p.s.i. lower or higher than the pressure on the rack.
- (4) Read and record the pressure on the rack and scale tank gauges.
- (5) Using a flame, quickly thaw the freeze-off coil at one end of the circuit.
- (6) Immediately read and record all the pressure indications.
- (7) Wait two minutes and read and record the pressures for the second time.
- (8) Thaw the freeze-off coil at the other end of the circuit and repeat the pressure measurement.

The next step is to interpret the data which should be done immediately before making any further tests. No detailed directions can be given on the data interpretation, which depends more on common sense than anything else, except to point out the obvious fact that if the pressure gauge on the rack shows a pressure change within a minute, let us say, equal to that shown by the pressure gauge in the scale tank, the circuit is open between the two gauges. If the pressure gauge changes slowly on the rack after thawing the end coil, the indications are that a partial plug or restriction exists in the line between the rack gauge and scale tank gauge.

This first pressure variation test will probably locate the plug within 6 to 10 columns; by changing the position of the gauges, narrowing the distance between them, the location of the plug can be more accurately determined. However, it takes quite a bit of time to change gauges; furthermore, each change requires the opening of the circuit with resulting danger of introducing moisture or of having a material break. Another method of determining the location of the plug is to make temperature measurements of the intercolumn connectors as described below. If thermometer wells are already attached to the intercolumn connectors, it is an easy matter to insert thermometers and make the necessary temperature measurements. However, if thermometer wells are not already on the connectors, thermometers would then have to be taped on, which might take as much time, if not more, than making one or two changes of the pressure gauge locations. The rack foreman or person directing the search for the plug will have to use his judgment in deciding which method will enable the plug to be discovered in the shortest estimated time.

The temperature method will probably be quicker if the location of the plug is not known within 3 or 4 columns.

(b) Temperature Measurement Method

This method is based on the fact that a plugged intercolumn connector will not allow material to flow through it and as a consequence cools off to a temperature near to that of the duct. In the case of a clear intercolumn connector there will be enough back and forth surging of the process fluid to maintain the temperature of the intercolumn connectors to 150-170° C., even in the absence of direct circulation. (The temperature of the fluid coming out of the columns is in the neighborhood of 220-230° C.) The temperatures of the intercolumn connectors of a whole circuit can easily be read and recorded in 10 to 20 minutes, once the thermometers have been installed and allowed to come to thermal equilibrium. Thus the locating of the plug is done quickly and easily by the temperature method.

The technique is as follows:

- (1) Install the thermometers, either one to each intercolumn connector of the whole circuit, or 6-12 thermometers covering the presumed plugged region.
 - (2) Close the duct doors.
 - (3) Wait 10 - 15 minutes.
- 

- (4) With one person reading and a second person recording, read and record the temperatures of all the installed thermometers.
- (5) Study and interpret the data.

As it is in the case of the pressure variation method, the detection of the plug location by the temperature method requires considerable personal judgment and common sense. As an example of the sort of temperatures observed and the interpretation of the results, the following observations can be given:

<u>Intercolumn Connector</u> <u>Temp. Between Cols. Numbered</u>	<u>Temperature</u> <u>°C.</u>
1 - 2	170
2 - 3	158
3 - 4	165
4 - 5	145
5 - 6	130
6 - 7	121
7 - 8	118
8 - 9	125
9 - 10	140
10 - 11	130
11 - 12	158
12 - 13	155

The temperature measurements show a definite although flat minimum between Columns 7 and 8. The plug may be in this intercolumn connector. However, before concluding that this is the case, the duct surrounding Columns 7 and 8 should be inspected carefully to make sure that the observed lower temperatures are not the result of cold spots, cold air drafts, or dripping water.

At this point it should be remarked that the "spit" test for cold intercolumn connectors often enables one to locate quickly the cold spot in the circuit; this is particularly true if pitching is occurring in the circuit, inasmuch as in this case there is a large difference in temperature between a clear and a plugged intercolumn connector. However, the "spit" test cannot show the fine differences in temperature required to locate a plug by the temperature measurement method described above.

Temperatures indicated by the thermometers in the Hoffman Flow Indicator should also be observed, because from these temperatures and the change of the temperature of the two ends of the Indicator with time one can tell whether the Indicator itself is plugged or not. If the ΔT 's change in magnitude and also in sign with time, the Flow Indicator is clearly not plugged. One can also obtain indications as to the location of a plug by noticing the period of a surge; a short period indicates a location of the plug closer to the Flow Indicator than does a long period.

(c) Use of By-Pass

Unfortunately neither the pressure variation method nor the temperature measurement method enables one to locate the plugged intercolumn connector always beyond doubt; for final proof the connector should be by-passed by a short link line running from the middle tit of the column on one side of the connector to the middle tit of the column on the other side. If circulation is started by virtue of material being able to flow through the by-pass, the by-passed intercolumn connector is probably plugged. If circulation does not start, the plug is probably in another intercolumn connector. Other possibilities are plugs in either of the two middle tits or in the temporary link line. Careful conditioning of the link line before use will prevent the latter difficulty from occurring. If a gauge had been previously installed on either of the two middle tits and had been responding properly, that particular tit was free.

The use of temporary by-passes is recommended because sometimes it saves the operator from removing an intercolumn connector which is not plugged; it is a difficult and dangerous job changing an intercolumn connector and should not be attempted unless absolutely necessary. However, there are certain precautions in the use of temporary by-passes which should be emphasized, namely:

- (1) A temporary by-pass if left on the rack should be wired to a copper freeze-off line so that it can be frozen off when the rest of the bottom circuit is frozen off; otherwise intercolumn circulation will occur when the top circuit becomes thawed.
 - (2) Before changing the intercolumn connector the temporary by-pass should be frozen off to see if circulation is stopped. If circulation continues, the intercolumn connector is not plugged and need not be changed. Sometimes plugs thought to be in intercolumn connectors either never really existed or disappear due to pitching or for other unknown reasons.
- 

- [REDACTED]
- (3) After normal operation has been established on a rack, the rack should be "cleaned up" by the removal of all temporary by-passes and the replacement of the plugged intercolumn connectors with clean ones.

In case there are several by-passes on a circuit in which circulation has not been started and if it is desired to remove some of these by-passes, test of circulation through the by-passed intercolumn connector can be made by measuring the temperature of the intercolumn connector before and after the by-pass is frozen off. If the temperature rises on freezing off the by-pass, the connector is not plugged (care should be taken to allow temperature equilibrium to be established in the duct before making the measurement.)

(d) Partial Pitching Method

Although normally pitching is to be avoided at all costs because of the disturbances it causes to good operating conditions, nevertheless, a limited amount of it may be resorted to for 10-15 minutes as a means of locating a plug quickly and reliably in a particularly baffling plugged circuit without noticeable damage to other circuits on the rack. Time saved in clearing a circuit may far outweigh disadvantages occurring from a few minutes of pitching. Technique of carrying out the pitching test for a plug may be summarized as follows:

- (1) Freeze off the end of the circuit nearest to the suspected location of the plug. The material pressure in the circuit should be between 1400-1500 p.s.i.
- (2) Between the above two points withdraw a No. 31 capsule of material.
- (3) Observe the extent of the pitching in the circuit. The plugged intercolumn connector will be between the end pitching column and the first column whose material is up to pressure.
- (4) Immediately thaw the frozen end coil so that the pitching can be stopped without delay.

Not only is the pitching method extremely reliable, but the pitching also sometimes helps to sweep out the plug because of the considerable difference of pressure existing during pitching across the plug. Furthermore, the pitching method may uncover the existence of two plugs when only one is suspected.

[REDACTED]

If the pitching region is bounded by region of non-pitching columns, at least two plugs would be indicated.

If a convector loop exists in the middle of a plugged circuit, it is a good idea to withdraw material from both ends of the top of the loop into a No. 120 cylinder. If each cylinder is filled, the entire circuit must be free. If a plug exists, the circuit will pitch from the convector loop up to the location of the plug.

(e) Material Surge Method

Sometimes a plug can be flushed out by causing the material to surge back and forth in the circuit. This is done easily by the following series of operations:

- (1) Freeze off the far end of the circuit and build up the pressure to 1700 p.s.i.
- (2) Freeze off the near end.
- (3) Lower the pressure in the scale tank to 1400 p.s.i.
- (4) Thaw the far end of the circuit.
- (5) If this treatment fails to dislodge the plug, repeat reversing the direction of the surge.

It has sometimes been suggested that rapid surges in circuits may stir up sediment, thus causing plugs to form where plugs never had previously existed. While this may be true, it is also possible that sediment may be washed out of a circuit by rapidly flowing material through it as in the filling process of the Rack 9 method of operation.

Plugs in Tits

(a) Indications of plugged Tits

A plugged tit is suggested by:

- (1) Failure of the material to circulate through a freshly conditioned intercolumn connector.
- (2) No fuming of material out of a "cracked" Lunkenheimer cap or out of a "cracked" Lunkenheimer connection to a frozen intercolumn connector.

(b) Methods of Detecting and Eliminating

Attempt to withdraw material out of the suspected tit into a previously weighed small size capsule, No. 2 and No. 4. If an ounce of material is not obtained, the tit is plugged.

If the tit cannot be cleared by the withdrawal of material, it must be drilled out by Maintenance. In the meantime another tit on the column can be used.

In concluding this section on the location and elimination of plugs, it should be reemphasized that a circuit should never be opened to the atmosphere unless absolutely necessary, that all new equipment added to a circuit such as link lines and intercolumn connectors should be carefully cleaned, dried and shown to pass fresh air before use, and that not only is circulation absolutely essential, but that the circulation should be the most rapid possible.

6. Procedure in the Event of a Planned Shut-down

a. Objective

The objective sought in the event of a shutdown is to guarantee that no damage shall result to equipment either during the shutdown or subsequent to it. Furthermore, it is desired to guarantee that start-up procedure be made as simple as possible by reason of the fact that proper steps have been taken at the time of the shut-down.

In any case of stoppage of operation it is always necessary to isolate the transfer room from the rack. It is further necessary to isolate all columns from each other. If this practice is followed start-up procedure becomes very simple and the possibility of damaging columns due to excessive hydrostatic pressures of the process fluid vanishes.

b. Steps in Shutting Down a Rack

- (1) Freeze off freeze-off coils between terminal boxes at the rack and columns. This will prohibit any transfer of material from the rack to the transfer room or vice versa.

If an extreme emergency exists, the conduit lines can be flooded but this should be done only on the specific authority of the Technical Superintendent.

- 
- (2) All columns should be isolated from each other by freezing all intercolumn connectors.

This may be done on some racks manually by opening the valves on the standby water line or on other racks by throwing the proper solenoid switches to flood the lines leading to the intercolumn connectors. It must be noted that if only one circuit is being frozen off the valves leading back from the circuit being frozen off should be closed before opening the standby water line valves. Otherwise, the water will go back through the automatic valves and flood all the lines in the rack. This, of course, need not be considered when shutting the whole rack down.

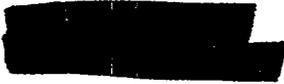
- (3) Once the isolation of all columns from each other and the transfer room has been accomplished, it is then permissible to close the main 6" steam valve supplying steam to the rack.
- (4) Circulating pumps may be turned off after steam pressure has dropped to 25 lbs.
- (5) The steam on the convectors and conduits should not be turned off unless the rack is to be down for a considerable length of time or unless asked for by the Steam Department. In this case, the steam should be turned off after the rest of the rack has cooled down so that material will not be drawn into these lines by cooling them first. It is considered better to have the material drawn out of them by cooling the columns first so there will be no probability of breaking them when starting up.
- (6) It is extremely important that the responsible person operating the rack at the time of the shut-down record in detail the procedures employed in shutting down. It is especially important that any observations regarding flow of material in or out of the rack as recorded by the transfer room scales be noted.

7. Procedure for Starting up a Rack After a Shut-down

a. Check List Before Starting Up

While a rack is shut down for either maintenance or steam difficulties the rack crew has many things to check and to repair before starting up the rack again. Some of these are listed below:



- 
- (1) Remove from each circuit all columns showing material leaks at the top of the rack. Exceptions to this are columns having leaks at Lunkenheimer connections which can, of course, be changed to better fittings.
 - (2) Inspect all intercolumn connectors to make sure that the nickel link lines are well-soldered to the copper freeze-off lines. Replace any connectors whose nickel lines have broken away from the copper. It is apparently satisfactory to wire these tubes together if necessary but the wiring must be done in such a way as to make as long a contact as possible between the nickel and the copper.
 - (3) Check conduits to make sure that steam has been kept on them during the shut-down period. If they have been allowed to cool and must be thawed out before starting up the rack, consult with the Technical Superintendent on duty before attempting to thaw out the lines.
 - (4) Check all circuits for completeness of connections and proper freeze-off coils. Also check with the diagram on the rack chart.
 - (5) If the convector loops have been allowed to cool during the shut-down period, make sure that the intercolumn connectors are hot along the bottom and that the link lines are hot before attempting to thaw out the convector loops.
 - (6) Install new gauges wherever needed and when gauges are available.
 - (7) Before attempting to start up a rack, check all the valves on the tops and bottoms of the columns, making sure that the steam and water valves are all open, except on the by-passed columns, only the water valves are opened, check the air vent valves in the water system, check all ducts, conduits and convectors for proper temperature; in other words, check the entire rack to make sure it is ready for operation. Do not assume that it has stood all right and without change during the shut-down period.
 - (8) The final step in starting up the rack again is to make sure exactly how the rack was shut-down. If possible the operator should contact the person who was in charge at the time of the shut-down. Pertinent information should be in the log book of the rack at the time of the shut-down.
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b. Procedure for Starting Up

The first step to be taken in actually starting up is to make certain that the circulating water is flowing and that the circulating pumps are functioning properly and showing the proper discharge pressure (at least 30 lbs.) The duct system should then be closed and heated.

Steam may be valved into the system through the by-pass around the main valve with the objective of heating the circulating water up to 150° F. Pressure of fluid within the rack should be noted by means of pressure gauges at the tops of the columns and under no circumstances should the pressure be allowed to exceed 2500 lbs.

If proper steps were taken when freezing the rack down there should be no difficulty in having excess pressure in the columns. If no large pressures are observed it is then possible to open the main valve gradually. This must be done fairly slowly, i.e. the pressure should be increased from 50 to 100 pounds over the course of about 20 minutes. At all times there should be operators on the top of the rack observing fluid and steam pressure and there should also be an operator at the transfer room noting any changes in the transfer room. Operators need not be alarmed if fluid pressure as recorded by at least two gauges at the top of the columns rises to 1800 lbs. However, if the rate of increase is rapid and the pressure shows signs of increasing beyond this point, the steam flow into the rack should be cut down until the pressure situation is under control. In this case, contact may be made to the scale tank by unfreezing the bottom inter-column connectors and the tits on the scale tank. This should be done very carefully with close watch being kept on the weights and pressures in the transfer room.

Unless something is radically wrong such as plugged lines, plugged gauges or leaking columns or conduit lines, it should be possible to build the steam pressure up to operating value of 1000 lbs and the gauges at the storage chamber and at the top of the columns should come to remain the same within the limits of the probable error of the gauges. This may be done in approximately 20 minutes after determining that the rack is in satisfactory operating condition with no excessive pressures being noted. If all has been done properly, the circuit should circulate very soon after the system has been opened up.

8. Special Rack Procedures

The original method of operation which consisted of allowing the circuit to circulate at all times across the bottom of the circuit except when the column was taken from the top of the column by some suitable "milking" method has given way in several racks to a modification which is a great deal easier and gives essentially as satisfactory results.

Other modifications are being tried at various times in an attempt to increase amount of product being taken off and to make the process easier in its manipulation.

a. Top Circulation or "Pineapple" Method

This procedure was first tried on Rack I.O. 9 and consists of a system designed to circulate normally through the bottom of the rack for part of the time and then, instead of taking off the product desired on the top of the columns, to circulate it into another reservoir at the top of the rack. The principle of this is that if one starts within normal material in the product container and circulates and mixes it with enriched material, it will soon reach a higher value intermediate between the material in the columns and its starting value. If then, the columns continue operation, the concentration of the product on the top of the column increases further. This is followed by another period of circulation across the top.

This intermittent circulation between the bottom and the top is continued until the reservoir reaches the concentration desired. It is then replaced by another container and the process is repeated. The advantages of the system are in the fact that individual milkings or product withdrawals are not necessary. The cylinder is changed whenever necessary saving a great deal of time in rack operation. The two methods seem to give comparable results with the top circulation method having possibly a slight advantage. Details of the procedure are given in the section on Product Withdrawal Methods.

b. Intermittent Circulation Method

Another variation on the standard procedure of rack operation is based on a possibility that the circulation process disturbs the action of the column. It is a modification of the normal method in that instead of allowing the circuit to circulate at all times except when product is being withdrawn, the circuit is frozen off approximately half the time.

The method consists of merely freezing off the rack after circulation has been established and letting the columns operate. Intermittently, then, the circuit is allowed to circulate and bring fresh material into the columns but then is frozen off again. This has been tried but no evaluation of the method has yet been obtained. There is evidence that when a rack is started up after a shut-down it is well to start the circulation and then freeze off the circuit for several hours before attempting to withdraw product.

9. General Emergency Instructions

FOR DETAILED EMERGENCY INSTRUCTIONS SEE SECTION G

Design of Equipment

In designing column tits and connecting lines, one of the principal considerations has been to limit the flow which could occur in the event of a defective connection or breakage of a line. A minimum size of orifice is required in order to permit proper circulation in the various convective loops and it is this factor which prevents decrease in the size of the connecting lines. As a result of experience to date some estimates can be made concerning the rate at which material can escape in the event of various breaks occurring on the rack.

One of the factors in the escape of the material is the tremendous compressibility of the material at the conditions of operation. At 1500 pounds pressure the process fluid is compressed about three times as much as a perfect gas would be under those conditions. This means that the tendency for the material to escape is a great deal more than even the pressure would seem to indicate. For this reason, a small break seems to be quite large.

Rate of Escape of Material

If a break occurs at the top of the rack with all intercolumn connectors frozen the loss will not exceed 4 lbs. a minute. If the freezers are open the rate of loss will not exceed 8 lbs. a minute. These figures, of course, refer to unrestricted flow through a column tit or through a 1/16" I.D. nickel line which has broken completely in two. If the tit is partially capped, flow will be considerably less and in most cases will not total more than 1/2 lb. per minute.

In the event of a break at the bottom of the rack the quantity lost will depend on the location with respect to incoming conduit lines, and also upon the diameter of the conduit lines being employed. In the worst case, namely a break or an open conduit line tit, the rate of loss will be in the neighborhood of 6 lbs. per minute. For a partially capped open the loss is probably of the order of 1/2 lb. per minute.

General Procedure

In determining the procedure to be employed in the event of a material break much depends on an estimate of the rate at which the material is escaping and its quality. During the initial period of starting up it is most important to keep equipment in operation.

Therefore, when breaks occur operators should attempt to disturb other systems as little as possible. It is possible to shut the steam off the rack but this does not usually even need to be contemplated.

With experience, operators will gain the ability to estimate the rate of loss which is occurring in a break and will recognize that a comparatively small amount of material produces a great deal of smoke. In the event of most breaks particularly those due to incomplete capping of the tube the procedure should be designed to isolate the column or part that is leaking from the rest of the system. This can be accomplished by freezing the intercolumn connectors and coils. If the break should happen to be in the conduit, then, the storage tanks should be frozen off and the break isolated. It is possible to flood the conduit but this procedure is not considered advisable in any extent as it is almost certain to break some of the lines in the conduit which have material in them. This affects the whole conduit system. Again, let it be said that this is to be done only on the express direction of the Technical Superintendent.

It might be added that personal safety is more important than the material lost. In any case of an emergency, the operator should not expose himself unduly but the Safety Department has made adequate safeguards for the safety of the operator. By following the procedures set up in this manual and adequately safeguarding himself there should be no possibility of harm coming to an operator.

10. Control Room and Control Devices

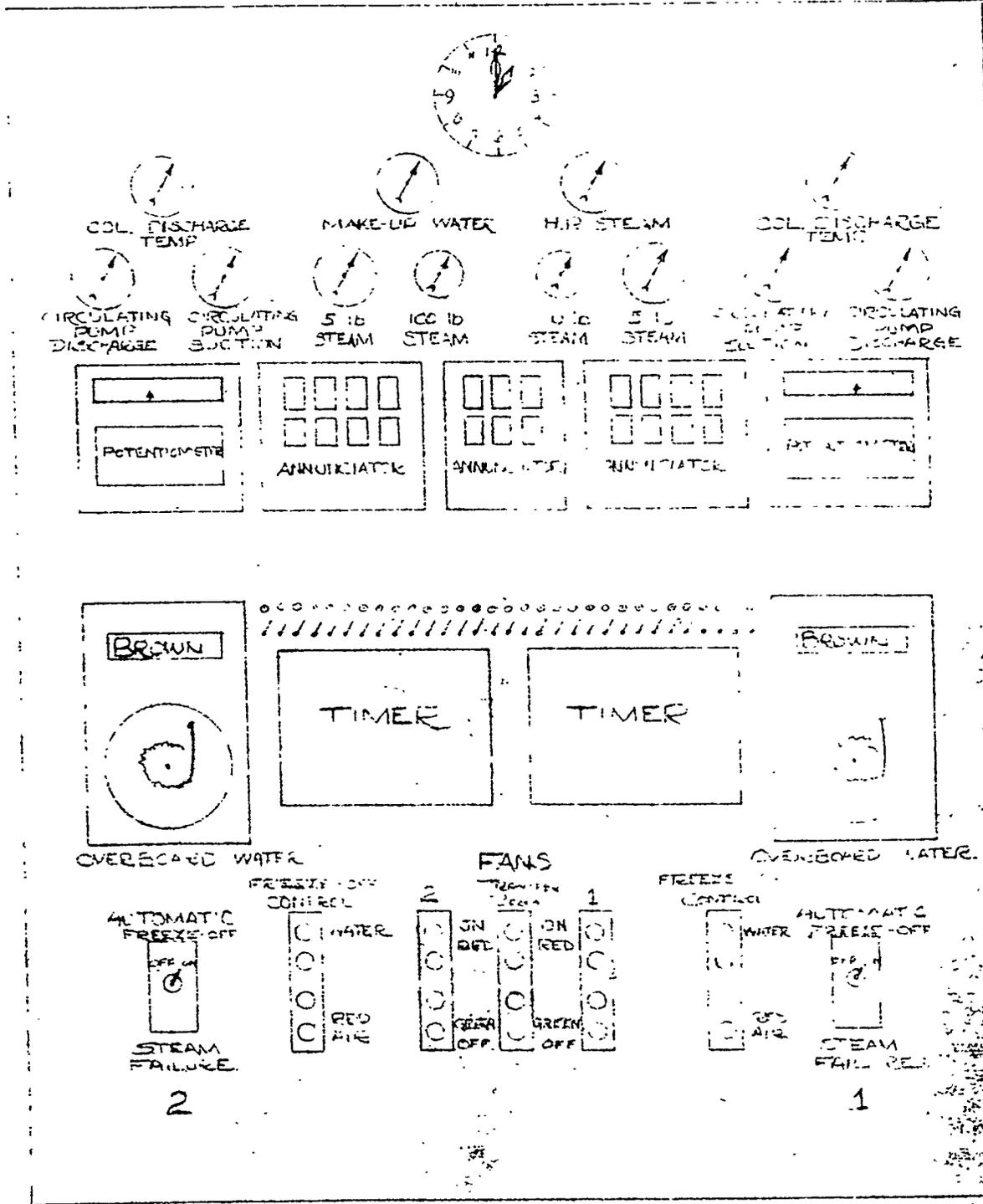
The control room is used primarily as an office or headquarters for the rack crew. It is well to know what the various devices are for control purposes, and what the limits of operation are in their operation. The following table lists the devices that are checked by the Operations Department but does not include numerous control devices around the plant which are the responsibility of the other Departments of the organization:

Control Device	Limits of Normal Operation	Whom to Notify of Abnormality
Gauges		
5 pound steam	5-9 p.s.i.	Steam Dept.
125 pound steam	90-150 p.s.i.	Steam Dept.
Make-up water	50-80 p.s.i.	Steam Dept.
Column discharge temperature	150-160° F.	Rack Foreman
1000 pound steam	950-1050 p.s.i.	Steam Dept.
Circ. water suction	10-20 p.s.i.	Steam Dept.
Circ. water discharge	15-30 p.s.i.	Steam Dept.

Table continued on next page

Control Device	Limits of Normal Operation	Whom to Notify of Abnormality
Recorders		
Thermocouples ΔT	300-450° F. 40-100° F.	Rack Foreman
Overboard Water Flow	0-150 g.p.m./10	Rack Foreman
Annunciators Drops (Buzzer signals)		
Rack Panel		
Three phase powerfailure		Tech. Supt.
Lower Manifold pressure low		Steam Dept.
Top manifold temperature high		Rack Foreman
Lower manifold temperature low		Rack Foreman
Purge air pressure low		Steam Dept.
Plant Panel		
Make-up water pressure low		Steam Dept.
Freeze-off water low		Steam Dept.
Process steam failure		Steam Dept.

See Dwg. No. 9-16



THE FERGLEVE CORP
 TECHNICAL DIVN.

CONTROL ROOM
 PANEL BOARD

Date - JUNE 20, 1945 DWG. No. 9-16
 Dpn. Luder Appd.

[REDACTED]

Section D. Sampling Procedures

Introduction. The purpose of sampling is to take a small amount of material out of the column or capsule in order that it might be sent to the Laboratory for the determination of the concentration of the product at that time. It is essential that the sample be a true representation of the conditions at the time it is taken and also a true representation of the average concentration in the part of the system being sampled. For this reason, great care must be taken to get a good sample and at the same time not interrupt the operation of the rack. Samples may be taken from the tops and bottoms of columns, either through center tits of the columns or through tees in the fluid line. They may be taken from capsules and from the scale tanks.

1. Sampling of Columns

While there is some variation in the techniques used to obtain material from the bottoms and the tops of columns, in general, the following steps should be taken when sampling columns of an operation circuit:

- a. Raise the fluid pressure on the system to 1500 pounds.
 - b. Freeze off tit to be sampled by applying dry ice as explained in Section A, Part 9.
 - c. Loosen Lunkenheimer cap one-half to one turn.
 - d. Break seal between ferrule of the cap and the tit by twisting the 1/8" solid nickel spur. If a solidly frozen plug exists only a small amount of fumes will escape and that will diminish in quantity to a point where no gas is escaping.
 - e. While still maintaining the dry ice on the tit the cap may be completely taken off and the sample tube fastened on. The operator should exercise great care to the end that the fitting is on snugly and not cross-threaded.
 - f. The tit is next unfrozen by applying a gentle Bunsen burner flame first on the end of the tit toward the column and gradually along the entire length of the tit exercising care that the flame does not linger for any period of time on one spot. The operator can determine if material has flowed into the clean-out tube by feeling an increase in the temperature of the tube or by feeling the shock as the fluid suddenly fills the tube.
- [REDACTED]

2. The tit should again be frozen off as above and the sample tube carefully removed. The cap should be replaced and warmed. The operator should be sure that the tit has warmed up before he leaves the vicinity in order that he may be certain that no leak develops at the connection. The sample tube should be examined to see if it contains material using a small wire to determine if there is material in the tube. Occasionally, it is found that either due to the presence of fresh air in the column or to improper technique on the part of the operator there is no material in the tube. This leads to complications when the sample goes to the laboratory.

1. In all cases the sample with the sample record card should immediately be taken to the Materials Section for transmission to the Laboratory.

2. Sampling through a "T".

Sampling through a "T" installed in the circuit just before or just after the convectors has for its purpose the obtaining of samples of the material in the storage tank before the process fluid has had a chance to get mixed up with the material in the columns. In these procedures the technique is essentially the same as for the columns. The steps are as follows:

- a. When a sample is to be taken, freeze off the "T" being very careful that it is adequately frozen off.
- b. Attach a sample tube to the "T" following the procedure outlined above.
- c. Heat the sample tube with the Bunsen Burner carefully to drive out some of the material into it.
- d. Cool the sample tube with dry ice.
- e. Carefully freeze off the "T".
- f. Disconnect the sample tube and cap off the "T". Again check to make sure a sample was obtained.

3. Sampling a circuit with top circulation system.

The technique of sampling this type of circuit is not particularly different from the techniques used on any circuit but there are some generalizations that can be made as to the schedule of sampling.

One top sample from each end of each circuit per twenty-four hours, beginning with the third day of operation on each run, should be sent to the Laboratory. These are in addition to the samples to be taken just after the start of the first top circulation period.



The samples taken from the top should be obtained during the time that circulation is proceeding across the top and preferably near the end of the circulation period; similarly, the bottom samples should be taken during the circulation across the bottom. In this way it will be possible to evaluate the average behavior of the column and circulating system.

The greatest precaution should be exerted to prevent a material break during the sampling period, particularly when taking top samples.

The sample would be more representative of the circulating fluid if the tit from which the samples are drawn is alternately cooled with dry ice and heated a few times before taking the sample.

4. Sampling of Capsules.

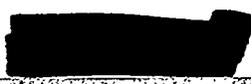
When sampling a container also the essential technique follows the general plan outlined above. However, more care must be taken to insure that material is actually passed over into the sample tube. This is necessitated by the fact that the pressure in the column is 1500 pounds per square inch and that developed by heating the capsule is much smaller than this pressure. The procedure is the same except that the container must be heated to drive over the sample. This is usually done by immersing the capsule in hot water thus liquefying the material and getting a more representative sample. The sample tubes are tested with a wire or weighed on a sensitive scales to ascertain whether or not a sample has passed over.

5. Sampling a scale tank.

In this case again the basic technique used is the same as in the other places where samples are taken. The emphasis, however, may be different when it comes to points which must be watched carefully. In the transfer room, extremely great care must be taken not to have the slightest possibility of having a material break since the quantities involved are so large.

The sampling of a scale tank is not an exceptionally common occurrence since something of the same information may be gained by the sampling through "T"s" properly placed in the circuit. It is sometimes necessary to sample a tank which is not in operation. In such cases the tank is raised to a material pressure of only a few hundred pounds and the sample tube placed on the tit. The tit is carefully heated and the sample removed.

In some cases, where the tank has stood for some time and little is known about the conditions of the tank, it might be desirable to flame out the tits before warming the tank up. This should be done carefully with the tank cold. It can be flamed back nearly to the main body of material. The tank can then be capped off and warmed up. The sample will then probably be more representative of the material in the tank.



1. Principles of Product Withdrawal

a. Purpose of the Product Control Section

To schedule periodical milking of each operating circuit in order to obtain the maximum output of product commensurate with assay specifications.

b. General Procedure.

The general procedure of the PCS is to obtain the assay reports from the laboratories and prepare a milking schedule taking into account the following factors: current assays, the general performance of that circuit in past days, circulation, depletion stage of feed material and internal disturbances such as pitching, intercolumn circulation and other interruptions. The next step is to post on the "Rack Performance Record" in each control room one hour before nominal milking time (five times a day staggered between odd and even numbered racks) an "M" informing the foreman that the circuit is eligible for milking. If the circuit is not ready the appropriate symbol is inserted in the record giving the reason for not milking. The foreman will then make preparations for milking the eligible circuits. The foreman will exercise his judgment, if for operation reasons, a delay in milking time is expedient. The actual milking time is entered on the performance record as an "M" followed by the time. If there is a large difference in the scheduled time and the actual time the discrepancy is noted.

The Product Control Supervisors will check the records periodically to see that scheduled milkings are accomplished or that justification for not doing so was adequate. If an eligible milking was missed because of operating trouble the PCS will discuss with the Assistant Technical Superintendent and the Foreman the advisability of double milking to compensate for the omission.

Performance records are to be turned in to the PCS who will inspect them for omissions, make necessary corrections, and prepare a summary listing by racks the causes of milking omissions. This summary and a complete set of performance records are submitted to the Works Manager at 0800 each morning.

c. Procedure for the Rack 9 Method.

For these racks the PCS acts as sample expeditors by listing the outstanding pot samples from the pot performance chart and checking on their priority routing to maintain a running assay record of each pot in the operations office.

The enrichment trend of the pot is then plotted to get the over-all picture of the progress of the pot. On the basis of the trend so-established, an estimate of completion of the pot is made. The estimate is revised with each subsequent assay and a "progress rating" based on a seven day objective is determined. This estimate is based on a seven day schedule so if five days is predicted the pot is said to be two days ahead of schedule. This progress rating is used as a check on the behavior of each pot.

Another function of the PCS is to act as a liaison between the supply of capsules, the capacity of the laboratory to handle the samples and the number of circuits operating to regulate the flow of product through the withdrawal and shipping steps.

On the basis of the study of performance characteristics as evidenced in assay control charts, product removal summaries, milker yield records and product indices the PCS also makes recommendations to operation personnel as to minor adjustments in milking rates such as double milking on occasion, skipping a daily milking for "weak" circuits, installation of No. 31 capsules instead of No. 23 capsules, and any modification of the circuit systems. It is also their responsibility to follow up the effect on the assay and quantity of product resulting from such changes as are adopted by the Operations Division.

d. Summary.

The Product Control Section, in consultation with the Superintendents, is responsible for scheduling of all product removal and expediting of all the factors incident to scheduling and efficient removal of the product.

Methods of Product Withdrawal

a. Single Column Milking.

The oldest and simplest method of withdrawing product is that which is usually called "single column milking", since it is just a withdrawal of product from each of the columns that are operating. This is usually done in a No. 9 capsule since it is inadvisable to withdraw more than this amount to avoid pitching. The method was used before a satisfactory method was devised for withdrawing across the top of a circuit and is still occasionally used when there are numerous plugs that cannot be located in the top of the circuit. It has largely been supplanted by one of the other methods which are exceedingly less time-consuming than this. It is usually more advantageous to spend some time locating and clearing the circuit on top than to use this method.

b. Manual Monorail Method

This is a refinement of the previous method in that it permits the milking of the whole circuit at one time. It is necessary, of course, that the top of the circuit be free from plugs since the only method of knowing whether or not the circuit has been milked is the pressure variation obtained at the ends of the monorail or top of the circuit.

The circuit is usually raised to a pressure of 1600 pounds or higher as noted by two gauges on the ends of the circuit on top. The circuit is then frozen on the bottom and unfrozen on the top and a capsule is attached to any column in the circuit but preferably one near the center of the group. After reading and recording the pressure on the gauges on the ends of the circuit, the operator unfreezes the tit and allows the material to flow into the capsule. It is frequently advisable to let the material pass through a restrictor coil to avoid too sudden a drop in that column. This is a length of nickel tubing with fine nickel wire inserted to restrict the rate of flow into the capsules.

It probably is not necessary to use the restrictor coil when the milking is done with No. 23 capsules but it is advisable to use such a coil before attempting to use any container larger than the No. 23. The material will usually fill the container in a few minutes after which the gauges should again be read and recorded. There should be a drop in the neighborhood of 100 pounds when the No. 23 capsule is used and a drop of about 150 pounds when the No. 31 capsule is used. The drop should be equal in amount and time in both gauges. If one of the gauges does not respond, the circuit is probably plugged between the material container and that end of the circuit. The container is removed by freezing the tit. The circuit is frozen on the top and after making certain that the top is frozen the bottom is unfrozen. It is of highest importance in all milking procedures that a circuit may never be unfrozen at the top and at the bottom.

c. Monorail Method Using Freeze-off Coils.

The monorail method seems to be one of the best and reliable methods of getting the product withdrawn. It is best carried out by the use of freeze-off coils and a large container rather than by the individual draw-off method outlined above. The advantages of the method lie in the continuity of performance that can be secured. This is adapted to any of the following types of withdrawal:

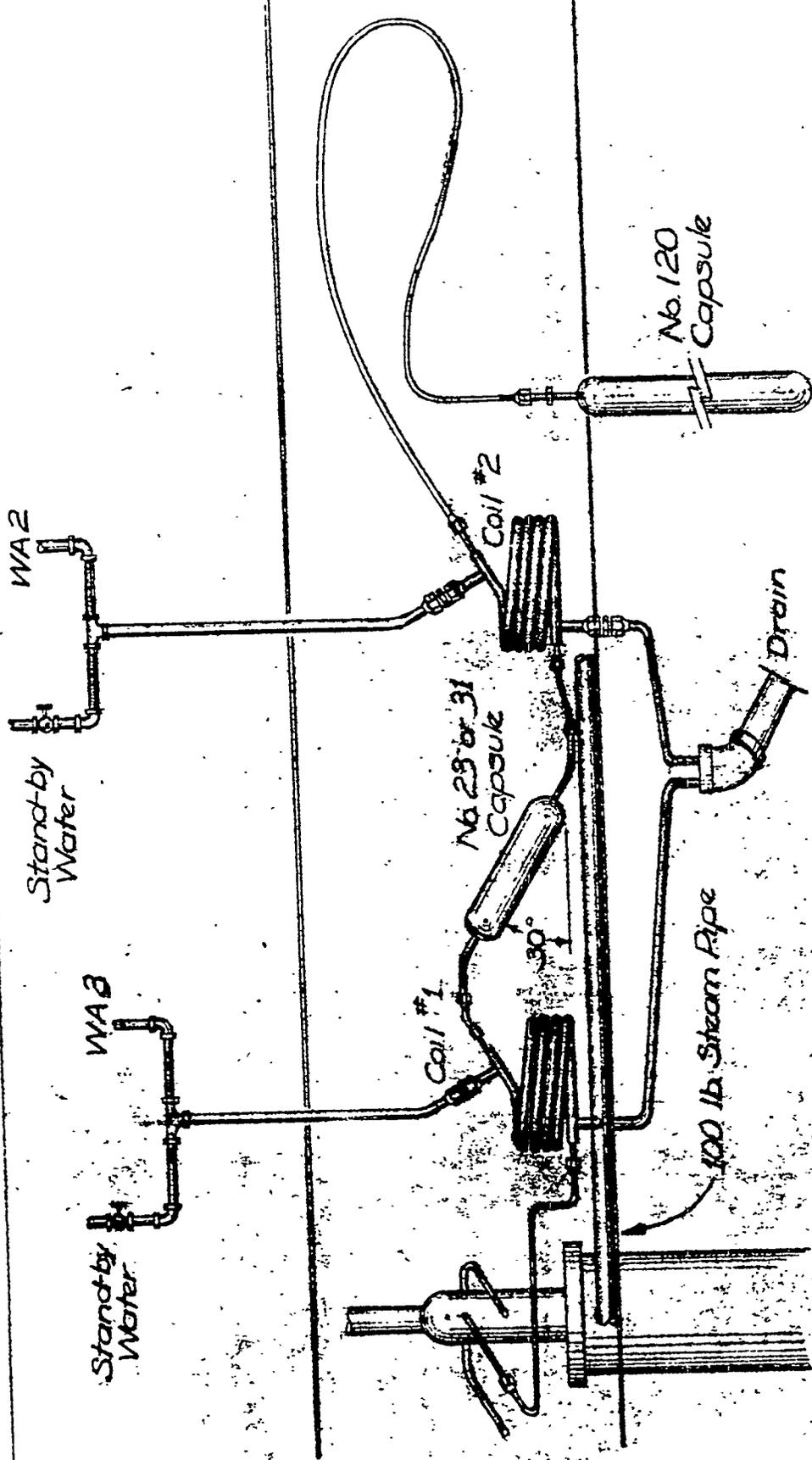
- (1) Manual method
- (2) Solenoid valve method
- (3) Can Timer method.

The installation of the equipment for this method will first be described and then its operation by the above mentioned methods will be given.

(1) Installation of Automatic Product Withdrawal Apparatus.

The steps to be followed in this are given in the following order. The parts and equipment may be seen on the accompanying diagram in Dwg. 9-17 also see Dwg. 9-13 and also their final arrangement is shown:

- (a) Prove circulation across the top of the selected circuit.
- (b) Select a column near the middle of the circuit.
- (c) Attach to a No. 23 capsule a copper freeze-off coil containing a restrictor and to the other end of the capsule another copper freeze-off coil without, however, having built into it the restrictor device. The restrictor coil is referred to as the No. 1 coil and the second coil is referred to as the No. 2 coil.
- (d) Make sure that the capsule freeze-off coil system has been well-conditioned and will pass fresh air freely.
- (e) Lay the capsule with its freeze-off coils along side one of the 125 pound steam heating pipes, near the column to be milked. The capsule should have the ends bent so that the capsule is resting on a 30° angle with the horizontal.
- (f) Attach the free end of the copper coil restrictor unit to the column selected for milking.
- (g) Drill a small hole between these two steam pipes in the bottom of the duct so that the stem of a No. 120 cylinder will protrude through it when the cylinder is placed in a vertical position inside its steam heating coil on the small scales.
- (h) Attach to the top end of the No. 120 capsule the free end of the second copper freeze-off coil.
- (i) Connect the upper end of the steam coil to the 5 pound steam line, using surgical latex tubing for the final connection between the copper steam line and the steam heating coil.
- (j) Make sure that the No. 120 capsule is free to move in a vertical direction without rubbing.
- (k) Connect the water inlet of the first freeze-off coil (coil No. 1) to the bottom of the water lines. These lines are referred to in the diagram as WA-1, WA-2 and WA-3 and the stand by water line.



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Center Column of Circuit

The WA is used since it may furnish either water or air depending on which solenoid valve is open when these valves are being used. The top line of the four which formerly was a compressed air line has been changed over a water line which is not controlled by a solenoid valve. The second from the top is labeled WA-1 and so on to the bottom line which is WA-3.

- (l) The exit end of the water line of coil No. 1 should be connected to a pipe leading to a drain. The inlet end of coil No. 1 should also be connected through a "T" to line WA-1.
- (m) The water inlet end of the water line of coil No. 2 should be connected to WA-2 which is the second pipe from the bottom of the four pipe water-air system.
- (n) By means of the "T" also connect the inlet water pipe of Coil No. 2 to the top of the air line.
- (o) The water supply for the top intercolumn connectors should be taken from the highest of the three water pipes (WA-1).
- (p) By means of a "T" also connect the inlet water line of intercolumn connectors to the top stand by water line.

(2) Operation of Manual Milking System with Freeze-off Coils.

The first capsule must be drawn off with care. Establish the monorail with pressure at 1700 pounds. Keep connection between coil No. 2 and the No. 120 capsule plugged with a fitting. Hook apparatus into monorail, unfreeze coil No. 1 leaving coil No. 2 frozen. After material has entered the system connect to the No. 120 and then freeze off coil No. 1. Unfreeze coil No. 2 and distil over. When material is in the system, normal operation may begin but be sure and do not flame coil No. 2 as it will not retain enough material to keep a plug formed the next time the circuit is milked.

The schedule for normal operation may be followed approximately as follows:

- (a) Time 0 minutes: Build pressure up to 1700 pounds, then freeze bottom.
- (b) Time 10 minutes: Shut off water on top ICC's on all of the circuit by closing water valve on WA-1. Purge with air from the air line if it is available for 30 seconds, then shut off the air. This will allow ICC's to unfreeze quickly.

(c) Time 20 minutes: Turn water off coil No. 1 by shutting valve on WA-1. Purge with air for 30 seconds if convenient and shut off air. (coil No. 2 should have water flowing through it.) Gauge will drop when the capsule fills. This may take as long as 15 minutes.

(d) Time 40 minutes: Freeze off coils No. 1 and top ICC's. Unfreeze bottom ICC's and re-establish circulation. Unfreeze coil No. 2 by shutting off water valve from WA-2. Purge with air; shut off the air.

(e) Time 60 minutes: Material will distill over in a few minutes after coil No. 2 unfreezes but 20 minutes is allowed for being certain. Freeze coil No. 2

If no sample comes over check the sensitivity of the scales. If the gauge dropped on the end of the circuit (top) the material has come over and this is the best test to ascertain that the container is full. Failure to distill over or to fill the capsule does not usually occur when adequate time is allowed.

Make very certain that the operator records the results of each draw-off on the rack sheet. Take off the No. 120 capsule each day so that there will be no possibility of attempting to milk into full capsule without knowing that it is full.

(3) Instructions for Operation of Automatic Milking Apparatus Using the Solenoid Valve System.

Most of the racks can be operated by means of the toggle switches which are in the control room. They control the solenoid valves at the top of the rack through which water or air flow. The top line of the 4 water-air system lines as was mentioned before is a standby water line.

(a) Freeze off the bottom of the rack (unless otherwise altered, the whole rack is frozen off at the bottom at the same time.) by turning the toggle switch, which will cause cold water to flow through WA-2, that is, through coil No. 2 and the bottom intercolumn connectors.

(b) Thaw the top of all circuits by turning the toggle switch on WA-1 so that air flows through the top of the intercolumn connectors.

(c) Wait 20 minutes.

(d) Turn the toggle switch which will cause air to pass through WA-3. This thaws coil No. 1

(e) Wait 20 minutes.

(f) Trip the toggle switches on WA-1 so that the air is shut off and then the water is turned on. This freezes coil No.1 and the top intercolumn connectors.

(g) Thaw the bottom of the circuit by tripping the toggle switches on WA-2 which turns off the cold water and turns on air through coil No. 2 and the bottom of the intercolumn connectors.

(h) Shut off the air flowing through WA-2

(i) Wait 15 minutes.

(j) Check to see whether the scales on which the No. 120 cylinder rests has gained in weight.

When the solenoid valves are in operation the whole rack can be frozen off by pressing the freeze-off button below the switches in the control room or the freeze-off buttons on the top and bottom ends of the rack.

In case manual operation is desired for a particular circuit, close all the valves on the lower three pipes, WA-1, 2, and 3 on that loop, both top and bottom, and control the flow of water through the intercolumn connectors freeze-off lines by means of valves on the top pipe line.

(4) Instructions for Operation of Automatic Milking Apparatus using the Cam-Timer.

This is essentially a problem which is worked out in cooperation with the Instrument Department. This is simply an arrangement of the cam-timer device which automatically switches the solenoid valves in the order given for solenoid operation. It consists of a clock device which goes through a cycle of four hours and allows a certain set of switches to be thrown for a certain part of the cycle and then automatically throws another set for the next step in the operation.

The cycle on which the timer operates is one which runs for four hours. The actual milking part of the cycle runs for 43 minutes and is divided up as given in the following table.

% of Cycle	Time	Part of Process
0	0 minutes	Water on bottom, No. 2 coil
2-1/2	6 minutes	Water off top
2-1/2 +	6 +	Air on top
3-1/4	7-3/4	Air off top

(continued on next page)

% of Cycle	Time	Part of process
8-1/2	20-1/2	Water off No. 1 coil
8-3/4	21	Air on No. 1 coil
9-1/4	22	Air off No. 1 coil
15-1/2	37-1/4	Water on No. 1 coil
15-3/4	37-3/4	Water on top
17-1/2	42	Water off Bottom No. 2 coil
17-1/2 +	42 +	Air on bottom, coil
18-1/4	43	Air off bottom, No. 2 coil

Procedure for Top Circulation Method

(1) Preparation of Rack Circuits

In the Rack 9 method of operation the product is collected in a high pressure shipping cylinder at the top of the rack by a slow process of circulation across the top of the circuit into and out of the cylinder. When the material in the milk can has been brought to the desired state, the can is removed and shipped away.

As the milk cans have but two valves each, and as only two cans per rack can be installed in the armored compartments, or "doghouses", the top circulation systems of the rack must be divided into two parts instead of four. Furthermore, convector loops, normally two per circuit, must be hooked into each circuit in order to provide the pumping pressure required to cause the process fluid to flow across the top of the columns through the milk can and back again.

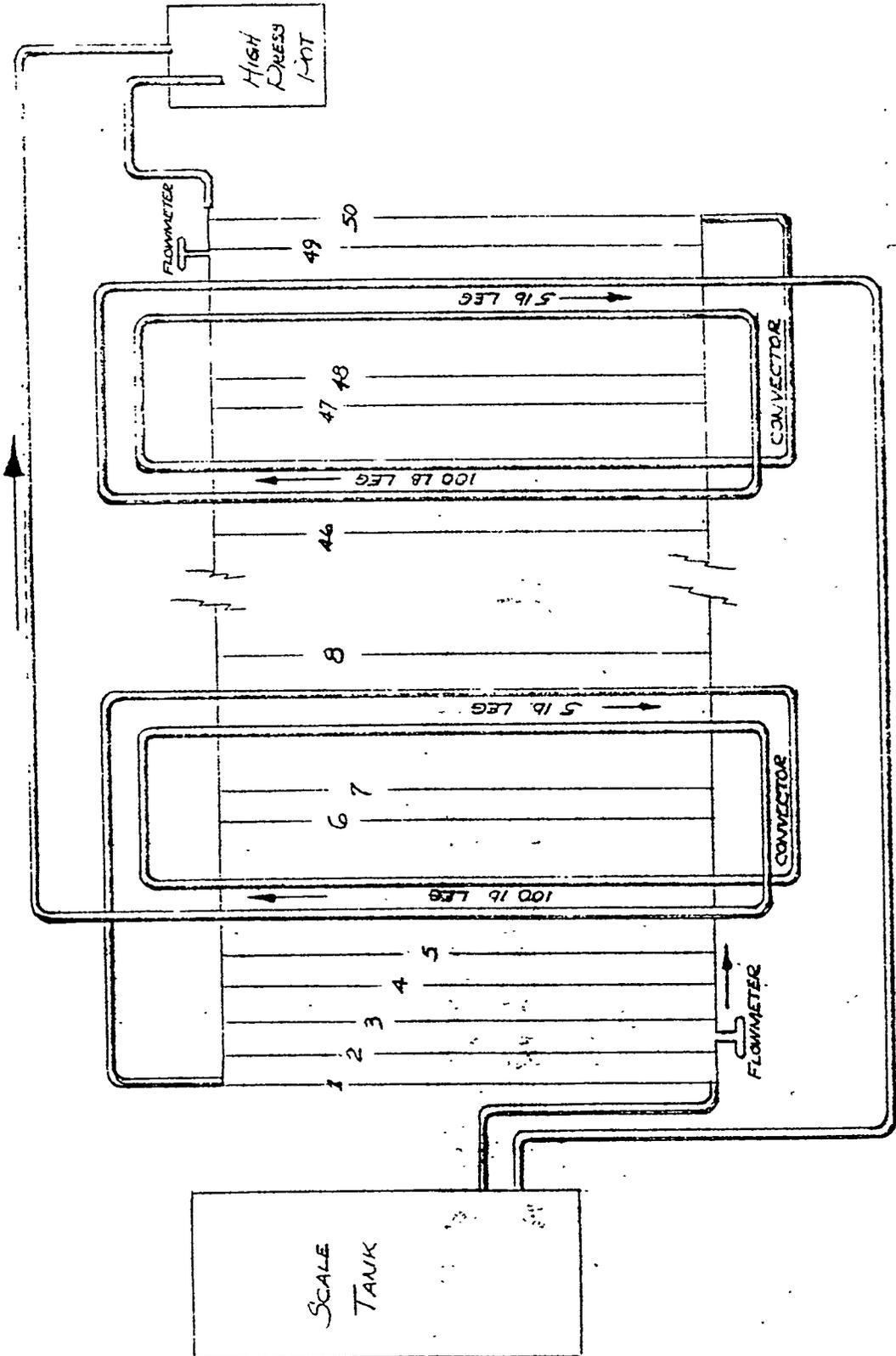
A Hoffman Flow Indicator must be also installed in each circuit and thermocouples attached to a hot and cold tit so that indication of circulation can be observed and continuously recorded.

The direction of circulation across the top of the rack should be counter-current to that at the bottom; as the flow in all of the bottom circuits is from low column numbers to high, the top circulation should be the reverse, i. e., from high to low column numbers.

An ideal circuit arrangement is illustrated in Dwg. 9-15, where it can be seen that the convector loops and flowmeters for the top and bottom circuits are installed at separate ends of the circuit. Although it may not always be possible to reproduce this circuit exactly, nevertheless the attempt should be made, because of the following advantages:

(a) The milk can can be filled from the top of the column and the material transferred from the scale tank to the bottom of the columns without the necessity of going through any convector loops. This means that the resistance to flow will be the least possible and that the time required to fill the milk can the shortest possible, something greatly to be desired.

(b) There are no convector loops in the middle of the circuit, thus assuring (theoretically) that the total resistance to flow in filling the top milk can will be the same irrespective of the column up which the material flows. Hence an equal amount of fluid should be removed from each column.



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 PRODUCT WITHDRAWAL
 TOP CIRCULATION METHOD
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 Wm. H. H. [Signature]

(c) The flowmeters are so arranged that it is easy, by freezing off at the top between Columns 2 and 3 at the bottom between Columns 49 and 50, to force all the fluid which flows into the milk can to pass through the flowmeters. By making readings of weight lost from the scale tanks every minute the rate of flow through the flowmeter can be calculated. By observing at the same time the temperatures indicated by the mercury thermometers the flowmeter can be calibrated.

However, in case circulation in either of the top or bottom circuit is insufficient (the test of inadequate circulation is described below), it may be necessary to install a third convecter loop in each circuit. The extra convecter loops for the top and bottom circuits should be selected from the same group so that the loops will be between the same columns. Otherwise the total resistance to flow in filling the milk cans will be different in different parts of the circuit, thereby taking a different amount of material out of different columns.

The outer conduits in each terminal box and convectors numbered 2 and 3 should be used as they have larger ID's than the others. The total resistance to flow should be reduced to a minimum.

(2) Installing Milk Can in Doshouses

(a) Steam Connections. As special fittings are required, the initial connections of the two steam hoses to each milk can should be made by maintenance. The milk can should first be placed on the swinging platform, the steam hoses connected and then the can swung into position.

As will be reemphasized below, the steam pressure on the can should be held constant at 5 p.s.i. The Rack Foreman should satisfy themselves that the pressure reducing valve, the steam pressure gauge and the "pop-off" valve are all in good working condition. The rubber steam hose might blow out or off if the steam pressure were allowed to rise to 150 p.s.i., but much more serious, the milk can might, and undoubtedly would, explode if it had been filled with material at the 5 p.s.i. steam pressure. The Rack Foreman should also make sure that the steam trap is not plugged and he should warn his crew members to guard continually against the backing up of cold water into the steam jacket of the milk can. A dangerous situation would be created if the milk can were allowed to cool down while in contact with the rack, thus filling up with excess material.

(b) Material Connections. On each milk can there are two valves, one marked on the can near the base of the valve with an "L" and the other with an "S", meaning long and short, respectively. The can should be hooked up to the rack so that material flows into the can through the short valve and out the long valve, although this is not a very important detail.

Each Kerotest valve has to have an adapter attached to it, and made tight using a copper washer. The adapters should be installed by Ballou's Department. The rack crew, however, should make sure that the nickel threads on the adapter are in good condition. The nickel threads on the male tits at the end of the conduits leading from the rack should also be inspected. An unnecessary amount of time of many good men in the plant has been spent struggling with poor material fittings, struggles which might be eliminated by a little preparation in advance. Before connecting the link lines to the milk can and after the milk can has become hot crack each Kerotest valve to make sure that material fumes can blow out; in other words, that the valves are not plugged. The valves should be connected to the conduits using the shortest standard length link lines. However, before connecting up the pot for the first time, circulation across the top of the circuit should be proved by attaching a jumper between the ends of the two conduits and observing the Hoffman flowmeter for indications of flow.

In freezing off the conduits between the rack and the milk can two important considerations should be kept in mind:

(1) Water flowing through one conduit might back up through the common drain into the steam jacket of the other milk can with possibly disastrous results.

(2) Moisture will condense on the conduits running down onto the nickel tits, covering the Lunkenheimer joints with water.

To avoid both of these undesirable situations, allow water to flow through a conduit only just long enough to freeze off. Then shut off the water. The material will stay frozen, but the conduit will warm up to the point where no moisture will condense; furthermore, as there is no flow of water, there will be no water pressure causing water to back up the common drain into the steam jacket of a milk can.

(c) A thermometer should be strapped by means of friction tape to one of the handles of the milk can or inserted into a thermometer well.

(3) Transfer Room Operations

The Rack 9 method operation simplifies Transfer Room procedures as it requires that a scale tank be filled with material only once before complete depletion and emptying. At the start of each run, the tank to be used on a circuit should be filled with 420 pounds of fresh material, of this approximately 70 pounds will be used in filling the milk can, leaving 350 pounds as the working weight of material. It is important that fresh process fluid of zero depletion be used.

[REDACTED]

When the material in the milk can has been brought up to par, the 350 pounds in the scale tank is nearly 100% depleted and should be rejected before starting another run.

During the filling of the milk can a record of the weight should be made every minute. As soon as the weight has become constant, the Rack Foreman should be notified without delay.

Otherwise from the above details, the operation of the Transfer Room does not differ from the usual procedure.

(4) Filling the Milk Can

- (a) Thaw the bottom of the circuit and the freeze-off coil connected to the column nearest to the scale tank, such as Column 1 of Figure 1.
- (b) Freeze the coil connected to column farthest from the scale tank.
- (c) Thaw the conduits to the milk can, but keep the Kerotest valves closed.
- (d) Thaw the top of the circuit
- (e) When the top is completely thawed, crack the Kerotest valve which is connected to the column nearest to the milk can.
- (f) By means of the valve adjust the flow of material into the can to as high a value as possible without pitching.
- (g) As soon as flow stops, quickly freeze off the top of the circuit.
- (h) Open both Kerotest valves as far as possible.
- (i) Record in the Log Book a detailed description of the filling procedure, stating whether or how long pitching has occurred; also record the length of time the top and bottom have been simultaneously thawed, whether any leaks occurred, etc.

(5) Start of Operation

When the milk can has been filled from the tops of the columns, the average concentration of the desired component in the milk can will be greater than the concentration at the tops of the column; hence, the first step should be to freeze the top and to allow circulation to proceed across the bottom just as soon as the milk can becomes full. Circulation across the bottom should continue for 24 hours before beginning the intermittent top and bottom circulation cycle.

At the end of this 24 hour period circulation should then be started across the top, but care should be taken to take samples for assay at the top ends of the circuit after about 15 minutes, of circulation so that the initial state of the fluid in the milk can will be known.

(6) Routine Operating Procedure

At the present time Rack 9 is being operated using the automatic cam-timer mechanism to give alternate periods of top and bottom circulation. The cams are set so as to give roughly 55% of 4 hours circulation at the top and 35% of 4 hours circulation at the bottom. The optimum ratio of the two has not yet been determined, but the latest results seem to indicate that this ratio is satisfactory, provided that the circulation rate is high enough.

The rate at which the desired component, call it x, is transferred to the high pressure milk can depends directly on three things:

- (a) The rate at which x is brought to the bottom of the column
- (b) The rate at which the columns transport x to the top.
- (c) The rate at which x is washed out of the top into the milk can.

Item (b) is a function of the column condition, and steam condition. Item (a) and (c) depend upon the rate of flow along the bottom and top of the columns and on the difference in concentration of the material between the first and last column in the circuit. If this difference is high, the circulation rate is low, while a more rapid flow will lower the difference in concentration between the first and last columns in the circuit. Thus, by determining the difference in concentration of material between these first and last columns an indication of the rate of flow can be obtained. On Rack 9 it was found that two convector loops in the top and bottom of each circuit were not enough to keep down the difference in concentration, so a third convector loop in each circuit was installed. This helped to reduce the difference in concentration from about 5 points to 2 or 3.

When the difference in assay between the first and last columns become greater than 2 or 3, the circulation rate should be increased.

On Rack 9 steam has been used instead of air to purge the water from the freeze-off lines in order that circulation could be established as rapidly as possible. However, the steam has a cool effect on the intercolumn connectors once circulation has been established (lowering the temperature from about 150°C. to 120°C.), thus decreasing the rate of flow.

For this reason the Rack 9 crew has been in the habit of turning off the steam by means of the toggle switches as soon as the thermocouple record indicates a good circulation. The toggle switch must be turned on again before the beginning of the next thawing period. The use of steam does not seem to offer enough advantage to make it worthwhile to install it on other racks.

Although the Rack 9 method requires less work on the part of the crew members, it places on them a greater responsibility for continually watching and maintaining the performance of the rack. If circulation stops or is decreased for any reason, or if pitching or intercolumn communication occurs, not only will the columns not "deliver the goods", but more serious, the good material which has been collected in the milk can may become lost. In this way several past days' product and performance may be ruined, in addition to the time required to rebuild the columns to a good condition.

The time required under optimum conditions to build a milk can up to par, starting from scratch is not yet known because of lack of sufficient information at this time. However, an upper limit of 10 days can be set; that is, if a milk can on a circuit cannot be brought up to par every ten days, the rack is not being managed properly. The minimum time may possibly be seven days, at least seven days is the goal we should shoot at.

The duties of the various crew members can be divided as follows:

(a) Control Room Man

- (1) Keep constant watch of gauges and continuous records.
- (2) Keep special watch of thermocouple record to make sure that circulation is proceeding satisfactorily, and that circuits are frozen on schedule.
- (3) Keep watch of thermocouple temperature of thermocouple attached to base of column. This temperature indicates the constancy of the steam conditions. If temperature is erratic, the Rack Foreman should be notified and steps taken to stabilize the condensate level.

(b) Bottom Rack Man

- (1) Keep constant watch of steam condensate level valve. If action is erratic, have the automatic mechanism readjusted by the instrument man.
 - (2) Keep watch of temperatures on flowmeters to make sure that good circulation is maintained during the period for bottom circulation.
- [REDACTED]

(b) Bottom Rack Man - Continued

- (3) Keep watch of the freeze-off water during the period for the bottom to be frozen off.
- (4) Keep watch of the material pressure gauges to make sure that the pressure, especially during the period of top circulation, is not dropping dangerously close to the pitching level.
- (5) Once per shift, during the top circulation, readings of the material pressure should be made every 30 seconds over a 5 minute period in order to check the constancy of operating conditions. These readings should be recorded in the Log Book.
- (6) A watch for material leaks should be maintained.
- (7) Keep watch of duct and convecter steam temperatures and occasionally check performance of convecter and duct steam traps.

(c) Top Rack Man

- (1) Watch the temperature of the high pressure milk c. It is most important that this does not vary.
- (2) Watch and control the steam pressure on the milk can.
- (3) Make sure that the pop-off valve is always in good functioning condition.
- (4) Make sure that the steam trap to which the milk can is attached is always hot.
- (5) Watch for material leaks (occasionally leaks should be tested by using a Bunsen flame).
- (6) Make sure that the conduits to the milk can are always hot, and when necessary to freeze them off, that no water backs up the common drain into the steam jacket of either pot.
- (7) Keep a watch of temperature on flowmeter to make sure that good circulation is maintained during the period of top circulation.
- (8) Keep watch of the freeze-off water during the period for the top to be frozen off.

(7) Sampling Schedule

(a) One top sample from each end of each circuit per 24 hours, beginning with the third day of operation on each run, sent to Laboratory No. 1. These are in addition to the samples to be taken just after the start of the first top circulation period.

(b) Any other special samples send to Laboratory No. 2.

The samples from the top should be taken during the time that circulation is proceeding across the top and preferably near the end of the circulation period; similarly the bottom samples should be taken during circulation across the bottom. In this way it will be possible to evaluate the average behavior of the column and circulating system.

The greatest precaution should be exerted to prevent a material break during the sampling period, particularly when taking top samples.

The sample would be more representative of the circulation fluid if the tit from which the samples is drawn is alternately cooled with dry ice and heated a couple of times before taking the sample.

(8) Removing Milk Can

The steps in the procedure of removing the milk can may be listed as follows:

- (a) Freeze off top of rack.
- (b) Close Kerotest valves on milk can.
- (c) Turn off 5# steam on steam jacket to the milk can.
- (d) Freeze conduits from rack to milk can by turning freeze-off water on conduits.
- (e) After freeze-off water has cooled conduits to room temperature, turn off freeze-off water.
- (f) With dry ice freeze off link lines and adapter between conduits and milk can and disconnect link lines from adapter.
- (g) Cap both link lines and adapter.
- (h) Swing out milk can and dissonnect steam lines. The milk can is now ready for delivery to Ballou's Department.

(9) Some Do's and Don'ts

(a) Unless absolutely necessary, do not change the steam pressure on the jacket of the milk can or the temperature of the milk can under any circumstances.

(b) If the temperature of the milk can must be changed, change it during the top circulation period with a constant watch on the material pressure in the column. Do not allow the material pressure to fall below 1400 or to rise above 1700 p.s.i.

(c) In filling the top milk can keep the top and bottom circuits thawed the minimum amount of time.

(d) Make sure that the tare weights of an empty milk can are known before installing the can on the rack.

(e) Crew members should not turn any freeze-off water or steam valves without notifying the Rack Foremen who should check to see that the correct valves are being turned. A few minutes spent checking an operation of this kind may save days spent trying to recover lost ground.

Section F. Operation Records System

1. Rack Circuit Recordsa. Master Circuit Diagrams(1) Purpose of Diagrams

The purpose of the diagrams is to provide all information pertinent to the condition of the columns, convectors and conduits, the arrangement of circuits and the disposition of instruments on the rack for the use of the rack crew in an intelligent operation of the rack; and to provide a permanent record of rack circuits and condition for management and the Technical Division.

(2) Procedure in Use of Diagrams

A new master circuit diagram should be drawn on the 0-8 shift on Monday and it will be used as a guide for the rack crew during the following week. At the end of the week the charts will be collected and filed as a permanent record.

The master circuit diagrams should contain the following information:

- (a) Condition of all columns in the rack indicated by the proper symbols.
- (b) Condition of all convectors in the rack including information as to those that have been conditioned, those known to be bad, those that have been used and subsequently taken out of service and those in use.
- (c) Condition of all conduits, indicated by the proper symbols.
- (d) Correct and complete circuit diagram, showing location of all instruments, convectors, conduits and columns in use and hook-ups between them. The location and type of product removal equipment should also be shown.

These diagrams should be kept constantly correct by changing the charts whenever changes occur on the rack. Whenever a change is made on the diagram, a note of the change should also be made in writing in the "notes" section of the chart, stating briefly the change made, together with the date and time.

Changes of rack circuits from one storage tank to another should also be noted in the same place.

This information, and nothing else, should be contained on the master circuit diagrams.

b. Rack Performance Record (Automatic Milker Method of Operation)

(1) Purpose of Record

To provide rack crews, management, and the Technical Division with information concerning operation of the rack used in determining the efficiency of rack operation, rate at which product may be removed, and all other information of interest in daily control of operation on the rack.

(2) Procedure in Use of Record

This record when completely filled out provides an adequate daily record of the rack performance. On the left edge of the record a time scale is provided, and all entries on the record should be related to this time scale. For each circuit on the rack a section of four columns is provided and in these columns the following information should be recorded:

(a) Milkings

In the first column all actual milkings from the circuit in question should be entered. If the milkings are made with the regular automatic milking arrangement, the milking is indicated by the letter "M" with an exponential number to indicate the number of milkings that have been transferred into the No. 120 capsule. For example, M^3 indicates that three milkings through the small No. 23 or No. 31 capsule have transferred into the larger container. If the milkings are made manually into a small capsule, they should be indicated by the letter "M" also but with no exponential number and with a note as to the capsule number accompanying the "M". When scheduled milkings are missed for any reason this should also be indicated by the proper symbol -- "P" for pitching, "O" for operational difficulties, "C" for lack of circulation, "I" for intercolumn circulation, or "X" for low assay.



The time at which the large No. 120 capsule is removed from the circuit, and the number of the capsule should be noted in this column and when rack samples are taken they should also be entered here, with the sample number and column number included.

(b) Product Control Supervisors

The second column is for the product control supervisors. Here they will enter their instructions concerning milkings. This is the only column on the record which is not the responsibility of the rack records man.

(c) Hours of Circulation

In the fourth column space is provided for the reporting of rack difficulties, such as pitching, intercolumn circulation, or losses. When pitching or intercolumn circulation occur, their duration should be indicated, and in the case of pitching, the violence as shown by the maximum overboard water flow during the peak of the pitching, and the columns involved, should also be shown. When losses occur, the weight lost and the location of the loss should be shown. All salvage drawn off in capsules, and all product other than that obtained by regular milkings, such as capsules removed from the top of the rack in plug-hunting, should be entered in this column. Finally, all storage chamber changes should be shown here.

At the top of the page, space is provided for the reporting of the storage chamber in use and the number of columns in operation in each circuit.

Across the bottom of the report, space is provided for the signatures of the rack foremen on each of the three shifts, indicating that they have checked the record and have found it to be correct.

This report is made in duplicate. The original is sent to the plant records office for use in daily calculations for control purposes. The copy is kept by the product control supervisors for their use in making decisions on product removal, and for the reference of the Technical Superintendents.



g. Operating Records (Rack 9 System)

(1) Purpose of Record

To provide a daily report of progress and performance on racks operating by the Rack 9 system of top circulation to be used by management, the Technical Division and Operations.

Each daily form will provide a record of the operation of one rack. It is divided into two sections headed "front" and "back". "Front" refers to the low numbered side of the rack or columns 1-51 while the "back" refers to columns 52-102. Therefore the record will provide data on two high pressure pots.

At the top of each column headed "front" or "back" is a section for data on storage chamber in use, the high pressure pot number, circuit numbers and total number of columns in service on the side of the rack in question. This information should be filled in daily so that any changes will be apparent.

The same arrangement concerning the circulation bar that is now in use on the cycle chart has been utilized in this record. However, instead of marking top and bottom circulation in colors to differentiate between them, it is asked that top circulation be marked by shading with diagonal lines, and bottom circulation be shown by filling in with solid black.

On each side of the circulation bar is provided space for recording incidents of rack operation. All samples should be reported, giving the sample number and the location from which the sample was taken. Losses should be indicated showing the weight lost and the place from which the loss occurred. Pitching and intercolumn circulation, when they occur, should be shown, together with the length of time involved, and, in the case of pitching, the violence should be indicated by the note "light", "moderate", or "violent" or any other note which the records man may prefer.

At the bottom of the page is provided space for a daily total of circulation hours. Top circulation hours should be recorded on the top line and bottom circulation on the bottom line. The cycle total of circulating hours should also be carried in the space provided.

The "percent completion" space need not be filled in by the rack crews, but will be filled in by the product control supervisors on the copies they receive. The "days since pot change" should be filled in however, by the records man, indicating the total days elapsed--whether operating or not--since the last product was removed.

Space is provided at the bottom of the page for the signatures of the foremen on the three shifts.

The record should be made with two carbon copies. The original will be kept by the rack crew as their record of the progress of the cycle. The two carbons will be delivered to the plant office at 2400 each day, as are the other milking records. One copy will be kept there for the information of the Technical Superintendents. The other will be sent to the plant records office, after the "percent completion" item has been entered by the product control supervisors.

2. Transfer Room Records

a. Daily Operating Summary

(1) Purpose of Summary

To provide information concerning storage chamber weight-pressure-temperature relationships, and material transfers in and out of storage chambers used in keeping material balances on the plant.

(2) Procedure

(a) Operating Summary

Required in the "operating summary" section of the report is a set of readings, taken at the beginning and end of each eight hour shift, of steam pressure, material pressure and storage chamber weights. A fourth column is provided in which the difference between the initial weight and final weight is recorded, with the proper sign to show an increase or a decrease. To the right of this column a space is provided in which the reason for the change in storage chamber weight should be noted. If it is "normal" in view of product removal and operating conditions on the rack this can be so indicated. If weight differences exist which are not explainable in this way the operator should inform his foreman of the difference and try to account for it in some way.



In addition, on the right edge of the sheet a column is provided for readings every four hours on condensate temperatures on each of the drain lines from the 1000 pound steam traps on the storage chambers. These temperature readings provide a check against bad traps or open by-pass valves which can result in serious loss of steam. High temperature readings should be the signal for a check-up on traps and by-passes.

(b) Transfer Summary

In the transfer summary section of this report all material transfer should be reported. This included all transfers of new material into storage chambers, and all transfers of depleted material out of them. Exact information as to weights involved and serial numbers and storage chamber numbers of the cylinders used is required in every case. The operator who completes a transfer is responsible for seeing that it is entered on the sheet.

b. Transfer Room Charts

These large charts are used as a half-hourly check of operating conditions in the transfer rooms. They are primarily for the use of the transfer room crew, but are collected and inspected periodically. It is particularly important that transfer room men realize that the steam pressure-material pressure-scale weight relationship provides a cross check on any one of the factors involved. For instance, it is possible to obtain a fairly accurate weight if the steam pressure and material pressure are known-- likewise only one material pressure corresponds to a given storage chamber weight and steam pressure. By using this relationship the accuracy of all the measuring instruments in the transfer room can be checked, and inaccurate gauges or faulty scales may be discovered and corrected.

c. Material Transfer Work Sheets

These forms provide a way of keeping working information on material transfers, and are for the use of the transfer room crews. Readings should be taken every fifteen minutes during the progress of the transfer in order to keep a careful check of material passing over. After transfer has been completed and the sheets are used they should be filed in manila folders in the control room files for use as a check by foremen and transfer room men.



3. Log Books

a. Rack Log Books

These log books are intended for use as supplements to the other rack records. Any essential data for operations should be included on the performance records but all supplemental information as to techniques employed or suggestions for further work should be included in detail in this book. Some of the books also include column records, maintenance records, material balance on the transfer tanks and any other information which the chief rack foreman desires. These books are essential for information to be passed from one shift to another and are not considered as having any formal value except in the coordination of the work being done on a given rack. Make certain that all statements made in the book are signed by the one making the statement or the person giving the order.

b. Transfer Room Log Books

These log books are also primarily intended for the information and correlation of work being done in the transfer rooms. They usually contain information regarding the weights of transferred material including starting and final weights by shifts. They may contain information about maintenance work which is needed or done or other pertinent facts about the operation of the transfer room. Here, as in the other log books, should likewise be a signed statement as to what has been done on each shift. Proper headings, including the date, shift, crew, rack foreman, etc., should be used for each shift's entry in the book.

c. Superintendents and Assistants Log Books

These are summarizing books of the operation on a given shift. They include data and information on the plant as a whole as, for example, the state of the steam supply, or the order of racks starting up and shutting down. This more or less summarizes the information carried on the wall chart for a given shift concerning circulation, milking and general plant or section performance.

4. Summary

In general, the system for keeping operational records may be summarized as falling into four main parts.



Circuit and rack condition information is kept on the master rack charts. Daily performance information is obtained from two sources, the performance record referring to rack operation, and the transfer room operating summary referring to transfer room information. Detailed information for the transmittal of specific information from one crew to the next is provided by the log books. All are necessary to obtain a complete picture of plant performance, and every effort should be made to make all necessary entries, and make them complete and correct.





Section G. Plant Emergency Instructions

1. Control Devices.

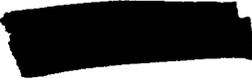
a. Temperature Controller.

The various gauges and control devices located in the control room are summarized in Section C, Part 10. Another very important device which is essential to the operation of the rack and which is very indicative of the way in which the rack is operating is the water temperature recorder located on the mezzanine level outside the transfer room. This is a dual purpose instrument in that it not only records the temperature of the water going into the rack and the temperature of the water going out of the rack but also is fitted with a controller device which operates the make-up water valve to allow more water to enter if the temperature rises and shuts off the water if the temperatures lowers. This gives important information as regards the conditions in the rack. There should be about 10 degrees F. difference between the in going and outgoing water. If there is appreciably more than that then there is probably pitching in the columns. The device is also a good indicator as to other conditions that may be existing on the rack. The exact use that can be made of the instrument is given in the next part of this section.

b. Condensate level control.

This is a device located on the bottom of the rack which while not in the province of the Operations Department to adjust or to control should be made familiar to every operator. Primarily consisting of a sight glass and air controlled valve the device maintains the proper level of condensate in the rack in order to keep constant steam conditions in the rack. The quality of the steam and the nature of the steam conditions in the rack markedly affect the temperature of the hot wall in the column so it is very important to have the proper control on this factor. It is not particularly significant exactly where the condensate level is kept but the level should remain as constant as it is possible for it to be.

Particular care should be exercised if the condensate level control should cease to operate properly and allow the condensate to build up in the rack. This in itself is of no particular importance but it is very important that the water level be lowered very slowly. When the water level rose, the columns cooled off to some extent and pulled material over from the tank. When the water level drops the material must of course be returned to the tank. This must be done through the conduits so if the lines are not large enough to accomodate the efflux of material, the pressure will rise quite abnormally. The operator should watch the material pressure gauges closely when the condensate level is being controlled by hand or when the Instrument Department is adjusting the controller.



c. Steam Flowmeter.

This device has been installed on several of the racks and probably will be installed throughout the plant. It is a triple marking recorder keeping a record of steam flow in pounds per hour, steam temperature and steam pressure. This is located on top of the rack and the operators should be instructed what the values should be and to report any abnormalities.

2. Classified According to Type of Failure

a. Material Blow-off- Stress finding correct location of leak.

(1) Plug Blown Out of Column Tit

- (a) Attempt to isolate column by turning on intercolumn connector freeze-off water on both top and bottom of rack (see Note 1).
- (b) Turn on Duct Fan (see Note 2).
- (c) If the flowing water has not succeeded in freezing off the intercolumn connectors adjacent to the columns with the break (see Note 3), attempt to freeze off the intercolumn connectors with dry ice.
- (d) Try to stop or retard flow using Roberts' Wooden Block, Tilp emergency plug or dry ice block.
- (e) Try to freeze off tit with dry ice. (see note 4)
- (f) Try to cap off with Lunkenheimer cap.
- (g) For a blowout of an intercolumn connector, try to retard flow by pinching the link line immediately back of the Lunkenheimer with a pair of pliers (see Note 5). After flow retarded, attempt to freeze off with dry ice and cap off.
- (h) For a bad leak around a Lunkenheimer connection which cannot be stopped by tightening the connection or by freezing off with dry ice; a wet rag placed around the Lunkenheimer may sometimes stop the flow by formation of solid hydrolyzed material.
- (i) If the above methods have not stopped the flow within a minute or two, turn the steam off the column closing both top steam valve and bottom condensate valve on the columns.

Every effort should be made to avoid an accident like a material break by always having at least one person as a helper to hold the dry ice and by freezing off according to the approved methods described in Note 4.

(2) Link Line Break

- (a) Turn on water on intercolumn connectors, freeze off coils, and storage tank tits.
- (b) Turn on duct fan.
- (c) Pinch off link line with pliers so as to retard flow.
- (d) Attempt to freeze off link line.

(3) Transfer Room Break

- (a) Close duct door.
- (b) Turn on duct fan and open damper (pull chain).
- (c) Turn on scale tank tit freeze-off water.
- (d) Turn on wall fan.
- (e) Turn off high pressure steam.
- (f) Flood tank jacket with water.
- (g) Freeze off rack.

b. Low Pressure Air Failure

(1) Symptoms

- (a) Drop in circulating water temperature.
- (b) Increase in overboard water flow as indicated on chart in Control Room.
- (c) Flow of water over roof.

(2) Emergency Procedure

- (a) If the air failure is noted before the water temperature has fallen below 130°F., operate the gate valve by hand as directed by overboard water chart in Control Room until air pressure is restored and automatic flow regulation is established.

- (b) If the temperature has dropped below 130°F., the columns in the rack have probably cooled off to the point where they have gained material. In this case the temperature of the circulating water must be brought back slowly and carefully to its normal operating value. This should be done by lowering the temperature control as far as it will go and then gradually raising it.

c. High Pressure Air Failure

(1) Symptoms

- (a) Fall in air pressure gauges on steam board in Transfer Room.
- (b) Fall in steam pressure on Scale Tanks.
- (c) Fall in material pressure in Scale Tanks and on racks.
- (d) itching on racks.
- (e) Gain in weights of Scale Tanks.

(2) Emergency Procedure

- (a) Freeze-off racks at freeze-off coils.
- (b) Freeze-off Scale Tank tits.

(3) Alternative Procedure for Operation (See Note 6)

- (a) Close valves on both sides of Foster Reducing Valve.
- (b) Close valve leading from by-pass to Foster valve bonnet so that one side of the Foster reducing valve diaphragm will not have a thousand pound steam pressure on one side of it and no air pressure on the other side.
- (c) Open valve leading to steam pressure gauge.
- (d) Cautiously open by-pass and adjust until steam pressure at correct value.

d. High Pressure Steam Failure

(1) Symptoms

- (a) Loss in weight of storage tanks.
- (b) Drop in material pressure on racks.
- (c) Drop in temperature of circulating water
- (d) Drop in reading of steam pressure gauges at top

of rack in case these gauges exist there.

- (e) High pressure steam gauges in transfer and control rooms may also drop in case of a general steam failure.

2. Emergency Procedure

- (a) Freeze off racks all along bottom and top.
- (b) Freeze off end coils.
- (c) Freeze off tits on storage tank.

e. Circulating Water Failure

1. Symptoms

- (a) Stoppage of circulating water pumps.
- (b) Rise in temperature of circulating water.
- (c) Slight rise in material pressure.

2. Emergency Procedure

- (a) Freeze off bottom and top of rack and at end coils.
- (b) Turn off high pressure steam.
- (c) Another remedy is to connect in another water circulating pump.

Notes:

1. During correct operation of a rack the freeze-off water should always be flowing either across the top or bottom of a rack. Hence, in case of a blowoff it should be necessary to turn on only the water circuits on either the top or the bottom which are not already on.
2. If it is inadvisable to leave the scene of the break, or if the break can immediately be stopped using some of the special plugging gadgets, it would be better to have another person, if any are available, turn on the duct fan. Usually when a break occurs, the cloud of smoke quickly attracts to the scene of the accident a number of operators who are anxious to help.

3. Sometimes the flow of the escaping process fluid is so rapid that it cannot be cooled down quickly enough to solidify. The intercolumn connectors should be felt with the fingers in order to see whether the process fluid has been frozen off or not. In fact it is always advisable to feel with the finger any link line, intercolumn connectors or tit that is thought to be frozen off before assuming that the process fluid really has solidified.
4. There is a special technique for freezing off tits with dry ice, both in the case of normal operation with no flow and in the case of rapid flow resulting from a material break. For a normal freeze-off operation, hold with gloves on a piece of dry ice firmly against the tit about two inches from the Lankenheimer connection. Feel the end of the tit with the finger and as the nickel becomes cool slowly move the dry ice toward the column until its far end rests against the column. In this way a material plug of the longest possible length can be prepared, a plug which will never blow out if the dry ice is always held firmly on the tit against the column.
5. Many operators carry with them large pliers with part of the wire cutting edges purposely drilled so that the edges will pinch a link line together, but not cut through the link line.
6. It is not recommended that Scale Tanks be operated on the by-pass except in cases where the valve system is such that the steam connection to the Foster valve bonnet can be closed while at the same time kept open to the steam pressure gauge on the Transfer Room control board.

3. Emergencies Classified According to Symptoms

<u>Symptoms</u>	<u>Possible Cause</u>	<u>What to Check to Find Cause of Difficulty</u>
Drop in Circulating Water Temperature (Control Room Buzzer).	Low Pressure Air Failure	Check air supply gauge & circulating water meter.
	High pressure steam failure.	Check steam gauge on control room board.
Rise in circulating water temperature (Control Room Buzzer).	Hitching on rack.	Check racks for pitching. Stop as soon as possible.
	Pumps stopped	Check lights on pump control boards.
	General Power Failure	Check other pumps
Drop in high pressure air gauge.	Compressor failure	Check compressor
	Broken diaphragm on Foster Valve.	Check all Foster Valve bowls for leaks.
Drop in Transfer Room steam gauge	Transfer Room header off	Check in other Transfer Rooms and Control Room high pressure steam gauge to see whether steam failure is general or limited.
Drop in Control Room high pressure steam gauges	General Steam Failure.	Check other gauges and with steam department.
Overboard water increase (water over roof).	Hitching on rack.	Check for pitching.
	Low pressure air failure.	Check air supply gauge on circulating water meter.
Overboard water decreases	Pump failure	Check circulating water temperature and pumps.
Drop in low pressure air (Control room buzzer).	Compressor air failure	Check other rack air gauges.
	Stoppage of air supply to one rack	Check valves to corresponding rack.

[REDACTED] (continued on next page).

Symptoms	Possible Cause	What to Check to Find Cause of Difficulty
Gain in weight on Scale Tank.	High pressure air failure	Check high pressure air gauge on Transfer Room board.
Loss in weight on Scale Tank.	Leak on rack.	Check for external leaks. Check internal leaks by freezing off circuits and determining if loss continues.
Loss in weight on Scale Tank	Failure of Foster valve.	Check gauges on air and steam lines.
Increase in material pressure.	"Creeping" of Foster valves.	Check gauges
	Failure of Foster valves.	Check air and steam gauges
Decrease in material pressure.	Leaks in system.	Freeze off and check for leaks.
	Steam failure	Check steam gauges.
	High pressure air failure	Check air gauges.

* * * * *

Control Room buzzer signal (the Technical Superintendent or his Assistant must be notified after silencing the buzzer, but before the drop is replaced.)

Rack buzzer panel

- | | |
|--------------------------------------|---------------------------------------|
| 1. Three phase power failure. | Check solenoids for freeze off motor. |
| 2. Lower manifold pressure low. | Check pumps. |
| 3. Top manifold above 175°F. | Check circulating water controller. |
| 4. Lower manifold below temperature. | Check circulating water controller. |
| 5. Furnace air pressure below 70%. | Check for low pressure air failure. |

<u>Symptoms</u>	<u>What to Check to Find Cause of Difficulty</u>
Center buzzer panel	
1. Make-up water pressure low	Check with steam department.
2. Freeze off water low	Check top header for supply.
3. Process Steam failure.	Check with steam department.

4. Instructions for Employees exposed to Material

Material can irritate the eyes, the bronchial tubes and lungs and to less extent the skin. However, if the instructions given below are followed, you will not be injured by contact with the material.

a. General

(1) If you have been working in an area in which material has leaked but have no pain and do not feel ill, no treatment is required.

(2) If you get enough material on your clothing that your clothing looks dusty, you will take a shower immediately and change to a clean suit of coveralls.

b. Eye Injuries

(1) If material gets in your eyes and your eyes pain, go to the nearest eye basin, immerse your face, and blink your eyes repeatedly under water. Continue washing your eyes in this way for one minute.

(2) After you finish washing the eyes report to the nurse at once.

(3) If the pain is relieved by the washing, report to the nurse when convenient, in no instance later than the end of the shift.

(4) If the pain is relieved by washing, but comes back later, report to the nurse when convenient.

(5) THE MOST IMPORTANT SINGLE FACTOR IN PREVENTING INJURY TO THE EYES IS TO FLUSH THEM OUT IMMEDIATELY WITH WATER.

c. Burns of the skin

(1) If the material gets on your skin, but no pain is felt wash your skin with plain cool water (do not use soap) at your convenience. Washing is to be continued for one minute. Reporting to the nurse is not required.

(2) If material gets on the skin and the skin hurts, either at the time the material got on or later, flush or soak the painful area in cool water for five minutes and report to the nurse.

(continued on next page)

(3) If material gets on the skin, and pain develops after you leave work, soak the painful area in cool water for two hours. If the pain has been relieved by soaking, report to the nurse at the start of your next shift.

d. Inhaled Material

(1) If you happen to breathe material but have no pain afterward report to the nurse when convenient, but in no case later than the end of the shift.

(2) If you happen to breathe material and afterward have pain in the nose and throat, pain in the chest, coughing, shortness of breath, retching or vomiting, report to the nurse at once.

~~SECTION H. MATERIALS SECTION PROCEDURE~~

1. Conditioning Procedures

As stated in the section on physical properties one of the most important aspects of the plant operation is that of preparing equipment for use in the plant. It must be entirely free from all water, organic materials, dust and anything with which the material might react. The Materials Section is primarily interested in two phases of this problem. One is to condition the equipment for use in the plant and the other is to empty the product or depleted material and recondition the equipment so it can be used again.

a. Conditioning Nickel Tubing

- (1) Tubing is first degreased by running carbon tetrachloride through it.
- (2) The tubing is then blown dry with compressed dry nitrogen.
- (3) The next step is to heat the line while evacuating to drive off all the moisture. The tubing is capped off at one end and the other is attached to the conditioning manifold. The evacuation is then started making certain that all of the joints are tight. The tubing is then heated from the capped end toward the manifold until the entire tube has been heated and is dry.
- (4) Shut off the valve to the vacuum pump.
- (5) Allow fresh air to pass into the tube while still under vacuum. Make certain, however, that none of the fresh air is allowed to pass into the vacuum pump line as it is very reactive with the oil in the pump and will easily ruin a pump if the slightest trace is allowed to enter the pump.
- (6) Turn the fresh air off and bleed the conditioned manifold down to zero gauge pressure.
- (7) Detach the tubing from manifold and cap the end being careful not to screw the cap any tighter than necessary to obtain a good seal. This avoids misshaping the ferrule on the tubing.

The tubing has then been degreased, blown dry, evacuated, heated dry and charged with fresh air and is then ready for use with process material.

b. Reconditioning Equipment

Used tubing and equipment containing process material can be reconditioned in two ways:

~~SECTION H. MATERIALS SECTION PROCEDURE~~

[REDACTED]

(1) Rapid Reconditioning Method

For immediate reconditioning small equipment is flamed out in a hood. This is done by starting at one open end and heating with a burner and driving out the process material. This is always done by heating from the open end to the closed end gradually.

After the tube is empty, blow out with dry nitrogen. Connect tubing to a source of fresh air and run the fresh air through the tube. The tube is then capped off on both ends and the tube is ready for use. The method is only used in an emergency when tubing is needed for immediate use.

(2) Standard Reconditioning Procedure

The method used wherever time will permit is to uncap both ends and immerse the whole tubing in the hot water cleaning bath. The heat of the bath will drive off all material which will be dissolved in the water. The tubing will be washed clean without any trace of material being left. Circulating water in the bath has been found to give the best results. The method is very rapid and tubing should not be kept in the bath any longer than necessary. The tubing is then treated as new and is conditioned in the same manner as other new tubing.

c. Conditioning Gauges

The procedures for conditioning gauges has been somewhat inadequate in the past. A great deal of shoddy work has been carried on because of lack of material and lack of time to do proper work. It cannot be emphasized too greatly that time saved by improper conditioning is a false economy. The gauges particularly must be conditioned exactly as outlined below since it is essential that the exact conditions of pressure on the rack and in the transfer tanks must be known at all times.

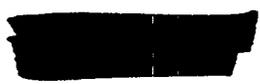
(1) Conditioning New Gauges

- (a) Remove the cap nut on the end of the Bourdon tube. It is a soft solder and can be done satisfactorily without harming the gauge.
- [REDACTED]

- (b) Fill a pressure tank with carbon tetrachloride and force a stream of solvent through the Bourdon tube allowing the solvent to pass through until the solvent is clear.
- (c) Dry by passing compressed air through the tube.
- (d) Pass a stream of fresh air through the tube.
- (e) Replace the nut on the end of the tube.
- (f) Attach the gauge to the vacuum pump, while heating gently and pass in fresh air again.
- (g) Reassemble the gauge and calibrate with nitrogen; leave capped with nitrogen.

Reconditioning Gauges

- (a) Dismantle gauge keeping indicator mechanism with the tube. If the gauge is not numbered, it should be numbered and a file card should be made out for it in order to keep an accurate record of each gauge.
 - (b) Place tube in oil bath and drive off all of the material.
 - (c) If the material is driven out completely the gauge may be fresh aired assembled and put back into service. If the material is not driven out completely then the gauge must be dismantled still further by taking the Bourdon tube out of the socket.
 - (d) The plugged material should then be drilled out if possible. This treatment is necessary particularly when organic material has been left in the tube. In very difficult cases soaking for some time in ammonium oxalate solution will help to loosen up the material. If the material does not come out after rigorous treatment the plugged part of the gauge must be discarded. After the tube has been cleaned out the cap nut should be removed if it has not already been detached and water passed through until the water is clear.
 - (e) Put the tube inside the gauge case and attach a 10" jumper which has been degreased.
 - (f) Have the tube and jumper heated while passing nitrogen through the tube. Be careful to heat the tube only in the center and not a vigorous heating at any time. Do not apply heat to the ends of the tube where they have been soldered.
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- (g) Assemble the gauge and calibrate with nitrogen.
 - (h) After the gauge has been calibrated record on its filing card the date, and process used in conditioning and the name of the person handling the gauge.
 - (i) Evacuate the gauge and charge with either nitrogen or fresh air. Cap off and the gauge is conditioned ready for operation to use.

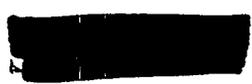
3. Plugged Gauges

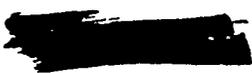
(a) Plugged tit

1. Place a length of steel welding rod into the electric drill and bore out the material.
2. If a hand plug is not removed by the above operation or if one forms at the weld, the tit must be cut off back of the weld and drilled out with the welding rod as drill.
3. If the gauge has a 1/8" nickel tubing inside the tit joining it to the gauge, this should be removed by melting the silver solder.
4. Counterbore the stem with a 5/16" drill before silver soldering.

(b) Plugged tube

A tube which cannot be cleaned of material is usually plugged where it joins the stem.

1. Heat the nut on the end of the tube with a flame. Do not apply the burner to the tube but only to the nut. Care is essential in this operation.
 2. Heat the stem and fuse the solder at that seal and unscrew the tube. Do not apply heat to the tube. Tapping the tube is more effective in breaking it loose than is applying pressure.
 3. Clean the tube in hot water removing all traces of material.
 4. Clean the stem in hot water making sure all material is cleaned from passages.
 5. Buff all surfaces which are to be soldered with a wire brush.
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6. Assemble the tube into the stem. The tube must fit tightly; if not the tube is probably not in the exact position.
 7. Screw the cap nut on following the same instructions as for the socket.
 8. The tube should then be soft soldered and tested under nitrogen pressure.
 9. The gauge is then assembled, calibrated and conditioned for use in the plant with evacuation, gentle heating, and fresh air.

2. Material Transfer

General Instructions

Material comes in from the plant in quantities from 1 ounce to 70 pounds. All this material must be transferred into suitable containers for shipment or storage. The container into which material is being transferred will always be kept cold with either circulating water or dry ice. The container from which the material is being transferred will be preheated until it reaches the liquid stage. In all cases, material transfer will be carried out by pouring the liquid material which means that the container being emptied will always be held above the receiving container.

A. Emptying Capsules

After making sure that all fittings are tight, place the capsule in a hot water bath for preheating. Indication that the liquid stage has been reached will be found by 'tipping the capsule from one side to the other and feeling the liquid moving inside the capsule. Freezing off the stem at least two inches in back of the fitting, remove the right angle cap and attach to the short female jumper on the receiving container.

Shipping containers are kept in a pot of dry ice slush which is a mixture of dry ice and carbon tetrachloride.

Larger containers cooled with circulating water. Warm the valve on the container with a burner and then open it. Proceeding from the valve, heat the material lines to the end of the capsule. After the initial heating it should not be necessary to continue heating and precautions should be taken not to heat the valve or jumper too hot.



[REDACTED]

Material will flow as a liquid and once the flow has started, all that needs to be added is enough to make up for the loss by radiation and loss by the material flow through the system. This heat loss due to material flow lowers the temperature and is the indication that material is being transferred.

The time required for transfer varies, of course, with the weight and amount of preheating. Once material flow is started, an average of a pound a minute will be approximately the transfer rate. An experienced transfer man will be able to tell by the sound of the capsule when it is rapped approximately how much material has flowed out and how much is left behind. When the capsule is empty, the valve is closed and the capsule stem is frozen off. Disconnect the capsule and replace the plug after the usual precautions have been taken.

B. Emptying Large Containers

Containers containing from 40 to 70 pounds of material are preheated in a water bath or if they have steam jackets are placed on steam for two hours or more. Connections is made between valves of two containers with a large diameter line usually ranging in diameter from 1/4" to 3/8". Hoisting the preheated container over the large cooled container, connect the valves. Warm both valves and the connecting line with a burner and then open the valves. The receiving container should be placed on scales so that the amount of material flowing over may be measured and so that it will be evident when the container is empty. The rate of material transfer will be very large and in most cases will approach 10 pounds per minute.

3. Records Systems

Several types of records are kept in a routine fashion in the Materials Sections. These records are extremely important both from a production standpoint to tell the status of material which is being taken from the rack or being shipped, and from a control standpoint to tell what the condition of the plant equipment is. Great care should be taken to be extremely accurate and punctual in the keeping of all records in this Section.

[REDACTED]


a. Sample Records1. Material Sample Record

All samples taken from capsules in the shop or taken from the plant will be recorded on this form. The assay column is for the foreman's use only. All data on this form will be recorded on the sample card. All samples on this form will be sent to Laboratory No. 1.

2. Shipment Sample Records

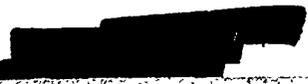
All samples taken from shipping containers will be recorded on this form. All data on this form will be recorded on the sample card with the word Shipment written in. Depleted material samples, will also be recorded on this form with the serial number of the depleted tank recorded under the Container No. heading.

3. Rack Sample Records

All samples taken in the plant from the racks will be recorded on this form. All special samples will be recorded here also.

Each shift clerk will hand the sample record forms into the office along with the material removal record. If at the end of the shift all samples ordered are not returned, the foreman should leave the sample record for the next shift to complete. This should apply only to rack sample records.

b. Material Removal Record

- (1) Write clearly the net weight of material from each capsule for rack and circuit. Do Not write the letters designating pounds and ounces but merely separate pounds from ounces by a dash. For example, 75 pounds and 15 ounces is written as, 75-15
 - (2) Below the net weight write in the capsule number.
 - (3) Opposite the net weight, indicate the number of milkings that went into the No. 120 capsule. This information will be obtained from the tag on the capsule put there by the capsule changer.
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- (4) Enter net weights of salvage material under salvage heading.
- (5) Obtain from the foreman the net weights, and rack and circuit numbers, for below-par material and enter on a regular material removal record. This record will be kept for twenty-four hours of each day and handed in at the end of each day.

c. Equipment Records

(1) Rack Equipment

Operations personnel will sign out for all rack equipment in the log book provided.

(2) Capsules

Operations personnel will sign out capsules by number in the rack equipment log book.

(3) Gauges

Records clerk will sign gauges out to operations personnel in the gauge log book. Instructions in the front of the book will be followed.

