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MANUFACTURING INSTRUCTIONS COVERING THE "T" PROCESS

OBJECT

In this process objectionable impurities are removed from graphite bars by bringing them in contact with chlorine and fluorine at elevated temperatures. Gaseous chlorine is introduced into the furnace by partial saturation of nitrogen gas with carbon tetrachloride vapor. The latter compound is broken down into chlorine and carbon after it enters the hot furnace.

Fluorine is introduced into the furnace by means of freon (F12) (Dichloric Diflure Metham)(CCl_2F_2). Chlorine can be brought into contact with the graphite at any temperature. Fluorine, on the other hand, has a disintegrating effect on the graphite at temperatures somewhere below 2,000 degrees centigrade, it has no effect at temperatures above 2,000 degrees centigrade.

These two gases diffuse into the graphite bars at elevated temperatures converting undesirable impurities into volatile chlorides and fluorides.

DESCRIPTION OF EQUIPMENT

In general there are two pieces of equipment needed for carrying out the purification -- the furnace in which the bars are heated electrically to a minimum temperature of 2,400 degrees centigrade and a washing tower in which the reaction by-products and also uncombined chlorine and fluorine are removed from air circulating through the hood covering the furnace by bringing them intimate contact with caustic soda solution.

The graphite bars are laid crosswise in a single layer a full length of the furnace. The bed in which these bars rest consists of two layers - the bottom one a 12" layer of finespetroleum coke (through 20 mesh to dust) and an upper layer approximately 2½" of BO graphite through 3 mesh on the 8 mesh.

Distribution of the gases to the individual bars is accomplished by means of a manifold equipped with 68 equally spaced outlets. Each outlet contains a copper disc orifice with a hole .024" in diameter. Connected with the pipe union containing the copper disc orifice is a piece of Saran tubing approximately 4" in length. Both ends of the individual pieces of Saran are reinforced with lengths of flared copper tubing approximately 3/4 of an inch in length.

The other end of the Saran tubing is coupled to a one inch pipe cap cemented to a carbon tube 1½" OD x 7/8 ID and 30" in length. This length of carbon tubing will hereafter be referred

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to as the inlet tube.

This inlet tube leads directly into a graphite distributor tube 2-5/8" OD x 2" ID and 53" in length. This graphite distributor tube has 3/8" slots machined on one side with struts being left in the tube wall for mechanical strength. The function of this graphite distributor tube is to distribute the reacting gas uniformly along the full length of the graphite bar. The function of the upper disc orifice is to distribute the gases from the manifold uniformly to each individual bar.

Carbon tetrachloride is introduced into the furnace by passing nitrogen gas over carbon tetrachloride maintained at a suitable temperature in a horizontal cylindrical tank approximately 12" in diameter and 10' in length - this piece of equipment will hereafter be referred to as the evaporator.

Gaseous F12 is introduced into the furnace directly by evaporating gas in the cylinder into which it is received. This is accomplished by partially emersing the F12 cylinder in a shallow iron tub containing water in which steam is introduced by means of a rubber hose. Heat is required for evaporating the F12 otherwise this evaporation would cool the liquid F12 with a consequence that the pressure would become zero. By bubbling steam into the water in which the F12 tank is emersed at a suitable rate, the Freon is evaporated at a rate to maintain a gage pressure of 60 to 90 pounds pressure per square inch.

The rate of flow of the F12 into the furnace is accomplished by means of a reduction valve and also a needle valve. The rate of flow is measured by means of a Rotameter. When the spinning metal top in the Rotameter is elevated to the 100 millimeter lever a tank of F12 containing 145 pounds of liquid will be emptied in 2½ hours.

Nitrogen flow through the evaporator is controlled by means of a reducing valve connected to the nitrogen header having a capacity of ten 220 cubic feet nitrogen cylinders. There are two needle valves and also two reducing valves connected on this header which makes it possible to connect the nitrogen cylinder in banks of five.

In the pipe leading from the nitrogen header to the evaporator is an ordinary gate valve. The function of this valve is to maintain a back pressure on the down stream side of the pressure regulators. These nitrogen pressure regulators will not operate satisfactorily unless there is a back pressure of 25 to 90 pounds per square inch.

The rate of nitrogen flow is measured by means of a Merian Orifice flow meter containing a metal disc in which there is a 1/8" orifice. A mercury manometer scaled in inches of water measures the pressure drop across this 1/8" orifice. When the mercury level in this manometer reads fifty scale divisions, the rate of nitrogen flow in the system is 100 cubic feet per hour. At any other pressure differential the rate of nitrogen flow is directly proportional to the square root of the differential pressure; for example, at a differential pressure of 100" of water, the rate of nitrogen flow will be

$\sqrt{\frac{[illegible]}{50}}$ X 100 or $\frac{10}{7.[illegible]}$ X 100 = 141 cubic feet per hour.

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By means of suitable valves, nitrogen gas is carried to the evaporator where it is partially saturated by passage over warm carbon tetrachloride. The resulting gas mixture then goes to the furnace manifold in which there is a steam tracer to prevent condensation of carbon tetrachloride from the nitrogen.

In the final stage of the "F" process nitrogen is passed into the furnace for a period of six hours without any carbon tetrachloride. In this operation, the evaporator is by passed by means of suitable valves. The purpose of this treatment is to flush out any individual chlorine remaining in the furnace charge.

PACKING THE FURNACE

In packing the furnace, all bars shall be numbered serially with a steel stamp prior to packing them in the furnace. For the present at least, the bars shall be numbered from 0 to 67 inclusive and they should be arranged in consecutive order when packed in the furnace. In packing the furnace, the graphite distributor tubes are spaced on the bed of fine coke with the slots located at the seven o'clock position. The purpose in arranging the slots in this way to prevent blocking the slots with the fine material of the coke bed.

These graphite distributor tubes are so spaced that the distance between the tube centers is 5-3/8". After these tubes have been spaced, coarse BO graphite is distributed over the tubes until the top of the distributor tube is just visible. Sufficient BO particles shall be distributed in this way so that the end of the BO flow line coincides with the ends of the distributor tubes.

After the graphite distributor tubes have been suitably spaced, graphite bars 4-5/8 x 4-5/8 x 50" in length are laid directly over the tubes so that the vertical center lines of the distributor tube coincides with the vertical center lines of the bars. When this is done there should be an open space between adjacent bars approximately 3/4 of an inch. This space between the bars is then filled with coarse BO graphite which is later rammed in place with a thin flat steel bar. It is important that this space between the bars be uniformly packed in order to obtain uniform current distribution of the length of the bar and consequently uniform temperatures.

After the bars have been uniformly spaced along the length of the furnace, a hopper filled with coarse BO graphite is suspended above the furnace in such a manner that when the crane travels along the length of the furnace a stream of coarse BO graphite is dropped on the center line of the bars through travel of the crane. In this manner a triangular mound of coarse BO graphite is deposited on the center line, either side of the base of this triangular mound shall extend to within approximately ten inches from the ends of the bars. The purpose of this triangular mound is to act as a chimney to vent gases from the furnace. The permeability of the

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coarse BO graphite to the passage of gases is roughly twenty times that of fine packing coke. The apex of this triangular mound is roughly eleven inches above the graphite bars.

After this triangular mound of coarse BO has been placed in position, fine packing coke is distributed on either side of this mound until the depth of the packing coke above the bars is nine inches.

Before the fine packing coke is poured into the furnace, the carbon inlet tube shall be placed in position with the open end extending into the graphite distributor tube for a distance of approximately 1½ inches. Before covering the carbon inlet tube with fine packing coke, they shall be inspected for cracks or clogging due to carbon deposits. The present 1½ x 7/8" tubes are unsatisfactory because carbon deposits formed in the hot end of the inlet tube for a distance of five inches break the inlet tubes. These inlet tubes will be satisfactory for further use if there is simply a longitudinal split. However, they will have to be replaced if the ends are completely clogged with carbon deposits or if there are transverse fracture. This condition, however, is a temporary one. A new design calls for a 7/8 OD x 1½" x 24" carbon inlet tube which will be connected to the graphite distributor pipe by means of a carbon sleeve 1½" OD x 1" ID 9" in length. When this combination is in use, the carbon sleeve will be regarded as an expendable unit and either scrapped or used again depending upon its condition.

Occasionally, it will be desirable to check the functioning of the copper disc orifice. During the cooling operation, furnace gases may diffuse back into these orifices and partially close the minute opening. The closure of the orifice openings will be indicated by increase in the manifold back pressure. The proper function of the orifice can best be judged by passing propane gas into the manifold and igniting the gas. If any of the orifices become clogged, this can readily be determined by absence of flame from any particular orifice. If any orifice becomes clogged, it must be cleaned out, otherwise non uniform treatment would result.

After the fine packing coke has been added to the furnace with the side blocks in place, the joints in the side block should be sealed with fire clay. Corrugated sheet iron covers are then placed on top of the side blocks with asbestos paper insulating with individual corrugated iron sheets from each other. A seal consisting of fine coke is poured along the edges of the corrugated sheets to close any openings to prevent any loss of draft.

ABSORBER OPERATION

The function of the absorber is to prevent the exit of chlorine and fluorine compounds from the system in the outside air. Caustic soda reacts with these gases and their volatile compounds forming new compounds which are inert. For each heat there will be required 350 gallons of solution containing five hundred pounds of caustic soda flake. Not more than 350 gallons shall be introduced into the absorber tower at one time, because a larger volume of liquid than this will close the offtake pipe from the furnace to the absorber. It is partic-

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ularly important to observe this level before the pump is in operation, when the solution is being circulated in the tower the liquid level will drop. If more liquid were added to the system at this time, the off-take pipe would be closed off by liquid in the event of pump failure due to a power shutdown. Later on, it may be possible to decrease this amount of caustic soda somewhat but for the time being it is advisable to use an excess in order to prevent metal corrosion in the absorbing tower.

After a heat has been completed the final solution in the tower is to be removed by pumping it into fifty gallon drums and emptying these on the dump pile or by means of a proposed pipe line from the absorber tower to the dump.

After the power has been shut off, it is important to watch the water level in the tower, because heat coming from the furnace will evaporate water at the rate of about one gallon per minute. This loss in water will have to be made up by pumping fresh water into the system at regular intervals.

For the time being, the corrosion characteristic of the final tower solution will have to be judged by odor. If there is only a slight odor of chlorine in the solution, the chances are this will not have an adverse effect on the tower. However, to make sure of this point, the inside of the tower ought to be inspected every two weeks to determine whether the tower is being corroded.

Some insoluble salts are formed by the reaction of the furnace gases with the caustic solution and at intervals it may be necessary to remove sludge through the cleanout door located at the base of the absorber tower.

FURNACE OPERATION

In carrying out the purifying operation graphite bars are heated by a passage of electric current through the coarse BO graphite separating individual graphite bars. Each of these spaces filled with BO shall be regarded as an individual heating element. Power is started at the maximum tap with the transformer connected on low range and as the charge becomes heated reduce the secondary voltage in order to keep the primary amperes within rated limits. In other words, the bars may be heated as rapidly as the power input conditions permit.

Control of subsequent operations shall be carried out on a kilowatt input bases and these shall proceed in seven distinct brackets. These brackets are summarized in the accompanying table.

BRACKET # 1 In this brackett power shall be introduced as rapidly as possible until 2,000 kilowatt hours have been absorbed by the furnace. With this power input, the temperature of the charge will be in the range of 850 to 1,000 degrees C. No gas shall be introduced into the furnace during the first bracket.

BRACKET # 2 After 2,000 Kilowatt hours have been introduced

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into the furnace, nitrogen gas partially saturated with carbon tetrachloride shall be passed into the furnace at the rate of 100 cubic feet per hour. The temperature conditions of the evaporator shall be adjusted so that eight pounds of carbon tetrachloride are carried into the furnace per hour of operation. The conditions for this brackett shall be maintained until the total input reaches 5,400 Kilowatt hours. At this input the furnace temperature will be approximately 2,000 degrees C.

BRACKET # 3 In this bracket, nitrogen will be shut off and F12 gas introduced into the furnace at the rate of 175 cubic feet per hour. This flow rate is obtained, by maintaining the F12 gas pressure at 60 to 90 pounds per square inch and a secondary pressure of 20 to 30 pounds per square inch after passing through a reducing valve. Flow is controlled by a needle valve so as to maintain the level of the spinning top in the Rotameter at one hundred scale divisions. Power input in this bracket shall be maintained at a maximum within the rating of the transformer until the total input reaches 7,600 kilowatt hours.

BRACKET # 4 In this bracket the freon rate will be maintained the same as in bracket # 3, however, the rate of power input shall be dropped to 1,000 kilowatts. This is the power input required to maintain a furnace temperature of 2,400 degrees C. This 1,000 kilowatt power load shall be maintained until the first tank of F12 is emptied. At this juncture, power to the furnace is shut off.

BRACKET # 5 In this bracket, with no power going to the furnace and cooling having started a second tank of F12 is to be fed into the furnace at the rate of 175 cubic feet per hour. This should be accomplished in 2½ hours at which time the furnace temperature will be slightly in excess of 2,000 degrees C.

BRACKET # 6 In this bracket there will be a cooling period and the temperature will drop from approximately 2,000 to 1,000 degrees. Nitrogen gas partially saturated with carbon tetrachloride shall be fed into the furnace at a rate of 100 cubic feet per hour for the nitrogen and 6 to 8 pounds per hour carbon tetrachloride. This shall be continued for a period of 30 hours.

BRACKET # 7 In this bracket nitrogen with no added carbon tetrachloride shall be fed into the furnace at the rate of 100 cubic feet per hour for six hours. The purpose of this treatment is to purge residual gases remaining in the charge.

After this six hour purging treatment with nitrogen, the sheet iron covers may be removed from the furnace and skimming of the top insulation started. This should be done by scraping the material adjacent to the side blocks with an improvised hoe to a line running through the middle of the furnace. This initial skimming will concentrate the ash which can then be shoveled into a scrap bucket and disposed of on the dump.

The remainder of the cover material will then be removed as

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rapidly as temperature conditions permit. This cover material shall be placed in a separate bucket for subsequent screening to remove the coarse from the fine material. Either of these materials may be used repeatedly in the "P" process.

UNPACKING

After the furnace has cooled sufficiently so that the bars can be handled with gloves, they shall be removed from the furnace and placed across aluminum angle bars. Warm graphite bars shall never be placed in contact with any wooden or paper surface because temperature may build up to a point where decomposition of the surface may occur and cause serious contamination of the bars.

Twelve bars are selected as samples which are distributed along the length of the furnace. For the time being, these shall be bars number 1, 7, 13, 19, 25, 31, 37, 43, 49, 55, 60 and 66. These sample bars are to be stamped with rubber stamps with the word "SAMPLE", grade designation "CSF" and heat number, finally the sample number. The bars shall be individually wrapped in rosin sized paper and packed three in a box for shipment to Richland, Washington. The remainder of the bars from any given heat shall be stamped with the grade designation "CSF" and the accompanying heat number. These bars are also to be individually wrapped with rosin sized paper and packed ten, later six to a box for shipment to Richland, Washington.

PRECAUTIONS

When either carbon tetrachloride or F12 are being introduced into the furnace some of the gases coming from the furnace are highly poisonous and should be carefully avoided. Removal of these gases from beneath the furnace hood is accomplished by ventilating fan connected to the absorber tower which pulls air through the space below the sheet iron cover at the rate of 2,000 cubic feet per minute. These poisonous gases are completely neutralized by contact with caustic soda. It is extremely important whenever a furnace is placed in operation that both the ventilating fan and the circulating pump shall be put in operation before a heat is started. The operation of these two pieces of equipment is indicated by two white signal lights located directly above the power control boxes. It would be well to inspect fan and pump from time to time to see that they are functioning properly.

In the event of a power failure, it is important that the flow of either carbon tetrachloride or F12 into the furnace shall be stopped immediately. This can be done by either closing the main valve on the evaporator, or the needle valve on the F12 control board. Auxiliary ventilation of the furnace can then be obtained by means of a steam jet flowing in the venturi throat located on the furnace stack and opening a slide valve in the off-take pipe directly above the exhaust fan. This steam jet will provide adequate ventilation with no gas flow into the furnace until power is again available.

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When carbon tetrachloride or F12 are being fed into the furnace it will be a good precaution to avoid walking between furnaces unless absolutely necessary. In current operations, there is no necessity for anyone entering between furnaces and this practice should be avoided as far as it is possible.

/s/ V. C. Hamister

C: Dr. L. M. Currie
Mr. H. T. Reid
Mr. C. E. Margeson
Mr. G. H. Fancher
Mr. V. C. Hamister

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SCHEDULE OF STEPS IN THE "F" PROCESS

Bracket	Temperature Range	K.W.	Total KWH at End of Bracket	Gas	Approx. Time
1	Room to 850° -1000°C	Full	2,000	None	1½ to 2 hours
2	1000° to 2000°C	Full	5,400	100 cu. ft. per hr. Nitrogen with 6 to 8 lbs. carbon tetrachloride per hour.	2 hours
3	2000° to 2400° C	Full	7,600	175 cu.ft.per hr.F12	1½ hrs.
4	2400° to 2400° C	1000	9,000	175 cu. ft.per hr.F12 until first tank F12 is empty. Power shut off.	1 hr.
5	2400° to 2000° C	None	None	Second tank F12 at 175 cu.Ft. per hr.	2½hrs.
6	2000° to 1000° C	None	None	100 cu.ft.per hr., nitrogen, with 6 to 8 lbs. per hr.carbon tetrachloride.	30 [redacted]
7	1000°	None	None	100 cu.ft. per hr. nitrogen.	6 hrs.