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TITLE
TRIP TO MORGANTON PLANT OF THE NATIONAL CARBON COMPANY

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February 17, 1948

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FROM: C. W. Botsford

SUBJECT: Trip to Morganton Plant of the National Carbon Company

Object of Trip: The Morganton Plant of the National Carbon Company was visited on January 26 through January 29 to observe the graphite purification process. The object of the trip was to study the purification operation to obtain as much general information as possible regarding the process, and to follow up the sampling and sample shipments of the initial purification runs.

Description of Process: The purification process involves the conversion of graphite impurities, probably present in solid solution in the graphite, into volatile halides which are swept from the graphite by means of nitrogen. The impurities present are Si, Al, K, Ca, V, B, and others, and are probably present in the graphite due to their accumulation in the still bottoms and producer gas furnace tars from which the coke and pitch in the graphite are made.

The purification is accomplished in a converted Acheson graphitizing furnace in which sixty-eight bars of graphite are placed on a thin bed of pure carbon coke. Individual graphite gas distributor tubes are placed in the coke bed beneath the graphite bars. The bars and the small space between each bar are covered lightly with pure coke. Three carbon electrodes at each end of the furnace are in contact with the resistance coke and carry the heating current. A current in excess of 20,000 amps heats the bed to 2400° C. The gas distributor tubes are connected to a common distributor pipe which runs alongside the furnace. The entire furnace is sealed from the atmosphere by means of a ceramic sheet cover under which a slight vacuum is maintained to prevent contamination of the room with noxious gases formed in the furnace.

The purifying gas used is CCl_4 at temperatures below 2000° C. and CCl_2F_2 at temperatures above 2000° C. Nitrogen gas is fed to the furnace at all times. The heating cycle requires from 4 to 5 hours while the cooling cycle is approximately 34 hours. The furnace is above 2000° C. approximately 5 hours during which time CCl_2F_2 is fed. The CCl_4 is fed as a gas on the heating cycle below 2000° C. and on the cooling cycle from 2000° C. to 1000° C. Nitrogen flow is maintained for 6 hours after the CCl_4 flow has been cut off to insure sweeping the noxious gases from the bed. The gases are scrubbed by a caustic solution in a counter current column packed with Raschig rings before being vented to the atmosphere.

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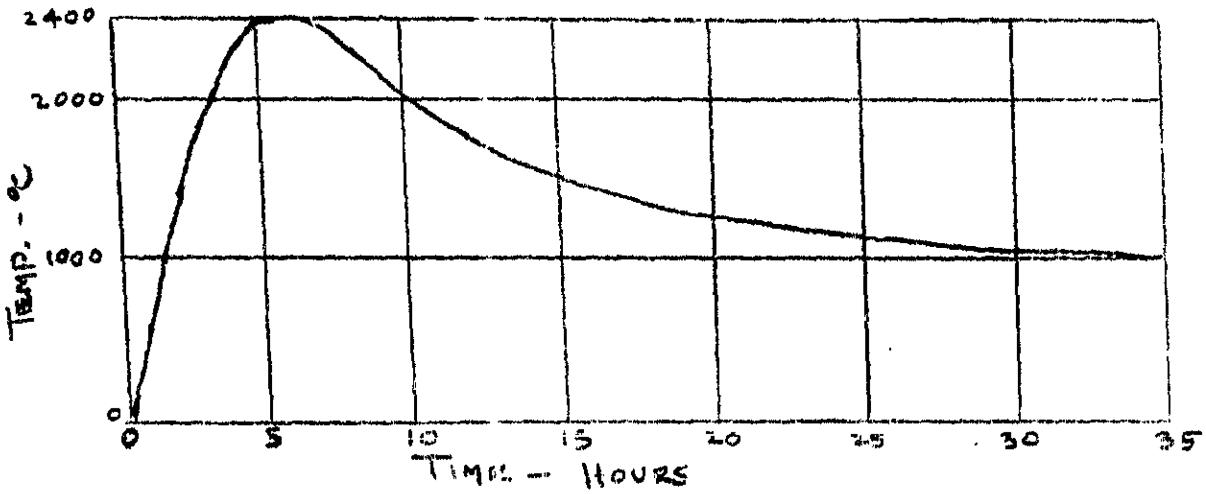
The curve below shows the approximate conditions of a standard purification run.



Nitrogen gas feed:	100 cu. ft./hr.
F_{12} feed:	50 lbs/hr
CCl_4 approximately	25 cu. ft./hr.

The CCl_4 is introduced by passing the N_2 through a long tube partially filled with liquid CCl_4 and heated to maintain a partial pressure of 140 mm.

The CCl_2F_2 must be introduced at elevated temperatures because at low temperatures fluorine formed by pyrolysis of CCl_2F_2 reacts with the graphite causing structural failure of the graphite. It is the belief of the United Carbon Company and the National Carbon Company people that all of the impurities of the graphite are removed on the heating cycle and that the CCl_4 feed on the cooling cycle is superfluous.

One Acheson furnace was converted for purification on February 1 and four more furnaces are being converted. All five units should be in operation by February 28. The complete furnace cycle requires about six days which should result in one complete heat per day when all furnaces are in operation.

Recommendations Made: The original cooling cycle used by United Carbon called for cooling down to 800° C. before stopping the CCl_4 feed. It was recommended that on the second run the CCl_4 feed be stopped at 1000° C. thus saving about eight hours on the cooling cycle. It was further recommended that the CCl_4 feed be stopped still earlier if the test results indicate no decrease in reactivity.

The writer conferred with Mr. R. M. Kennedy of the Construction Department on the graphite bar specifications and found that in view of the reduced size bars used to support the process tubes most of the bars currently being culled could be used, thereby effecting a considerable saving.

There appear to be several avenues open toward further process improvements. These may be summarized as follows:

1. Control of temperature cycle. At present, the temperature cycle is governed primarily by the power cycle. Pyrometer readings were taken at only one location. It is now known that considerable variations in quality occur

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through the heat, and these may be related to the peak temperature and the time for which it is held. Bars from the ends of the heat, which are thought to run cooler than the remainder, have shown substantially higher quality than the average. It is understood that National Carbon is planning tests to establish this point.

2. Modification of gas addition cycle: It is thought that the addition of carbon tetrachloride on the cooling cycle has questionable value. This is also being investigated in several trial heats.

3. Flushing with inert gases: It is known that nitrogen can be adsorbed strongly on graphite. Nitrogen, if present in the finished product, would be a moderately strong absorber of neutrons and would be difficult to detect by ordinary chemical means. Test runs in which carbon monoxide or helium is used as the flushing gas would help to establish this point.

4. Examination of graphite raw material supplies with respect to elimination of impurities at the source: Coke with a high impurities content could be pre-purified by an adaptation of the purification process. The possibility of using a pitch made from thermal cracking of overhead still products should be considered. Metal impurities should not be present in such a pitch.

C. W. Botsford

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