1 - AB Greninger
2 - CH Gross - WP Overbeck - 700 Fi
3 - JE Maider - EP Lee - PE Lowe
4 - VC Hamister - National Carbon Co.
5 - CWJ Wende
6 - WK Woods
7 - PF Gast
8 - JN West - VL Redding
9 - CW Botsford
10 - Pink Copy
11 - Yellow Copy

May 19, 1948

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TO: File

FROM: C. W. Botsford

SUBJECT: Trip No. 2 to Morganton Plant of the National Carbon Company

Object of Trip: The Morganton Plant of the National Carbon Company was visited for the second time from April 25 to April 28 to observe a series of purification test runs which were made to determine the effect of various purification gases and scrubbing gases on the quality of purified graphite.

Description of Process: The standard purification process is essentially as described in HW-8699, though the cycle has been accelerated considerably. The following diagram shows a breakdown of the typical purification cycle described in the manner used by the National Carbon Company.

Bracket No.	1	2	3	4	5	6
Temp. Range °C	0 to 1000	1000 to 2000	2000 to 2500	2500	2500 to 2000 ±	2000 to 1000
Gas Feed	None	CCl_4 & N_2	F-12	F-12	F-12*	N_2 6 hours

* The average time of F-12 addition is about 5 hours.

The power input is maintained at a maximum rate until 10,000 KWH has been used. The time of power input is approximately 6 hours.

The experimental runs involved changes in brackets 2, 3, 4, 5 and 6 and are summarized in the following table.

Run No.	Changes
121 X	CCl_4 replaced with Cl_2 in second bracket. N_2 used only in sixth bracket.
122 X	No CCl_4 or N_2 used in second bracket. Standard run otherwise.
124 X	No F-12 used in third, fourth, or fifth brackets.

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Run No.	Changes
124 X (cont'd)	Cl_2 was used with the addition started at 2000°C in the third bracket. Three hundred pounds of Cl_2 were added over a six hour period. N_2 was used as a gas sweep in the sixth bracket. No N_2 or CCl_4 was used in the second bracket.
125 X	This run was similar to 124 X, except that He was used as a gas sweep in the sixth bracket. The sweep was maintained at 100 cu.ft./hr. for 20 hours.
129 X	This was a standard run except that a He flush of 100 cu.ft./hr. was maintained for 20 hours in the sixth bracket.
136 X	Standard run, except a six hour He flush was used in the sixth bracket.

A considerable portion of the time was spent with Mr. V. C. Hamister of the National Carbon Company in an effort to correlate all the known data and information regarding past graphite purification runs and in a discussion of graphite production in general.

The data of purification runs 28 through 70 were analyzed and checked against the results of the functional tests for each run. No positive trends could be detected which would guarantee an improvement of product quality. The data showed, however, that the amount of CCl_4 consumption had little effect on product quality. The consumption varied from 20 pounds to 150 pounds per run with little effect on quality.

There were indications that runs of short duration produced better product quality indicating that with a higher gas flow rate, better elimination of impurities was obtained. A possible relationship between the better product quality and runs of short duration might be the effect of closed gas line orifices, slowing the over-all gas feed rate and reducing the gas diffusion to the individual bars above the plugged distributor. This evidence is not conclusive but it does indicate that every effort should be made to keep the gas orifices open.

During the discussion it was discovered that when gas cylinders are switched, a positive pressure is not maintained on the furnace, and it is possible for furnace gas to flow back through the orifices and plug them due to condensation of volatile matter. The operating procedure was changed and instructions issued to cover maintaining positive gas pressure in the furnace at all times. This procedure has been followed on all runs following run No. 125.

The National Carbon people were asked to submit a list of ten bars in which it was discovered that the gas orifices in the feeding line was plugged. These bars will be tested functionally and a correlation between gas flow and product quality should be obtained. This will also give an indication of the amount of lateral gas diffusion in the furnace.

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The following are general topics which were discussed briefly and are noted for the purpose of the record.

Low Temperature Graphitization

A general discussion was held in an effort to determine the possible cause of the poor product quality of the CS graphite obtained when production of this type graphite was started after a lapse of several months. The difficulty appeared to be the quality of the raw material in the green bars rather than in the process at Morganton. There had been no changes in manufacturing procedure, raw material storage, or in any other known factor at Morganton. The chemical analysis of the coke, pitch, and green bars for 20 lots covering periods when both good and poor quality CS grade graphite was produced will be analyzed in an effort to determine if there is any correlation between product quality and raw material quality.

A low temperature graphitization which might result in an incomplete graphitization of the green bar is impractical under the present manufacturing method. The carborundum envelope which normally forms around the furnace would not form with a low graphitization temperature. This carborundum layer provides a good heat radiating surface and permits removal of the heat insulating blocks from the furnace after the graphitization has taken place. This results in relatively rapid cooling of the furnace. The thermal insulation blocks which are necessary to permit reaching graphitization temperature would keep the furnace hot indefinitely if they were not removed.

Low Temperature Graphitization Agents

No agents are known which will reduce the graphitization temperature. It would be very desirable from National Carbon Company's standpoint if they could use a low graphitization temperature because most of their graphite production is used in furnace electrodes, wherein the chief requirement is electrical conductivity and any impurities could be tolerated.

Increased Production of Purified Bars

Factors which influence the possibility of using two rows of bars in the purification furnace instead of the single row now being used are:

1. Location of head electrodes would have to be raised.

2. More resistance coke would be required between bars; thus the location of distributors on the present furnaces is inadequate.

3. The maximum available power of 2000 KWH is now being used on a 6-hour cycle. This cycle would probably have to be increased to 12 hours, and would make power scheduling difficult. The shorter period of high power requirement in the present purification cycle is similar to sandwich in with the low power period requirement of the normal graphitization operation.

The experimental furnace which is being converted will be constructed so that it can perform this type of experiment. It should be nearing completion soon.

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Molded Natural Flake Graphite

A very definite controlled orientation of natural graphite can be obtained, in fact cannot be eliminated, in a process involving molding of natural graphite bars. Densities of 1.60 to 2.0 can be achieved but there is a 5% ash content in natural graphite, which when reduced by purification would reduce the density to a figure approaching the present values. Natural graphite does not extrude too well and probably could not be used in such a process.

The impurities in natural graphite are foreign particles clinging to the pure graphite crystals which might be removed by leaching with the proper solutions.

The present price of natural graphite is in excess of $200.00 a ton, but current industrial requirements do not justify any large scale development of graphite deposits.

Purification of Basic Raw Materials

It is not possible to make a gaseous purification of coke grains [illegible] the high temperatures required would graphitize the coke. Graphite particles cannot be extruded due to compressibility of the graphite. The graphite expands after passing through the die resulting in crumbling of the extruded bar.

Most impurities occur in the nature of slag inclusions and possibly could be removed by leaching. They are in the form of complex silicates and are very insoluble.

Recommendations for Possible Process Changes:

1. In the future, experimental purification runs should be made on test bars of known purity. It is suggested that a number of bars of known functional test result be sent back to Morganton to be used in future experimental runs.

2. If orifice plugging is a factor in product quality, the use of anhydrous HF should be considered for it should eliminate plugging caused by pyrolysis and cracking of F-12, resulting in carbon deposition in the orifice hole.

3. Some basic research should be done in an effort to purify raw materials. This could be done on a small scale in a laboratory.

4. More complete chemical analysis of purified and unpurified bars should be made so that a more complete picture is available on removal of various compounds. Possibly bars of known high quality should be "salted" with known amounts of various possible impurities and the extent of their removal determined. Such tests could be made in standard runs by simply introducing several bars into the furnace.

5. It is recommended that the fluorine carrier chemicals made by the Pennsalt Company be investigated as a possible purification gas. In particular it is recommended that ClF_3 (Chlorine Trifluoride) be used. This is a gas which condenses at 113°C at atmospheric pressure and can be kept in 25 lb/sq.in. cylinders. It contains 62% fluorine, all of which is available.

It would have to be introduced at elevated temperatures as does F-12, but would supply much more fluorine and would have no residual hydrocarbons formed by thermal cracking.

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C. W. Botsford