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HW-26205

1. AEC-HDO, Attn: DC Sturges
2. LP Bupp
3. JJ Cadwell
4. RL Dickeman
5. AB Greninger
6. GE McCullough
7. [redacted] JF Music - DH Curtiss
8. WC Riley
9. HL Sterling
10. WK Woods
11. 300 File
12. 700 File
13. Yellow Copy

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W. K. Woods
Assistant Manager
Technical Divisions

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NATIONAL CARBON G-3 GRAPHITE PURITY RESULTS

Attached to this letter are neutron cross sectional purity results for XCEF graphite heats 18 through 31 inclusive. Dih results for these heats were compiled by H. A. Fowler of the Pile Physics group. The values for operational variables on each of these heats have been taken from various letters by V. C. Hamister of the National Carbon Company. This letter summarizes and brings the record up to date on experimental heats produced since my last letter to you dated August 8, 1952. (HW-25303)

A "standard run" in the XCEF experimental facilities at Niagara Falls, New York, remains the same as that reported previously and is reproduced in the following table.

TABLE I

STANDARD GRAPHITE PURIFICATION CYCLE

Operational Phase	Approx. Maximum Temperature	Time of Phase, hrs.	Gas	Quantity of Gas
1	1000°C	3	Nitrogen	75 ft.^{3}
2	1700°C	2	Cl_2	9 to 10 lbs.
3	2450°C	3	Freon 12	36 lbs.
4	2200°C	1	Freon 12	12 lbs.
5	1000°C	-	Nitrogen	370 ft.^{3}

A summary of materials studied during the period covered by this report is shown in Table II.

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TABLE II

SUMMARY OF EXPERIMENTAL GRAPHITES
Bars Designated "XGBF", ca. 4½ x 4½ x 18 in.
Twenty-five bars per run

Average Graphite Bar Properties

Heat No.	Coke	Pitch	Impreg.	Apper. density	Ave. dih	Remarks
18	Texas	Standard	Yes	1.65	0.961	Repeat of heat 11 except 8/10 mesh resistor carbon.
19	Texas	Standard	Yes	1.64	0.888	Rerun of heat 17 except more Cl_2 added beginning at a higher temperature.
20	Texas (odd)	Standard	No	1.54	1.022	Temperatures not measured. Cycles measured by KWH input. A standard run.
	Texas (even)	Standard	Yes	1.64	0.983	
21	Texas	Standard	Yes	1.64	0.967	1 x 4 x 18 in. strips of Texas coke pitch impregnated rebaked bars placed below each 4½ x 4½ x 18 in. Texas coke bars. Average dih of 1 x 4 x 18 in. slabs = 0.994. Temperature not measured. Coke packing changed to graphite.
22	Socony Vacuum St. Louis Coke	?	No	1.45	1.032	Electrode stock with ¼ in. coke particles. Bar permeability measured low, 10/20 mesh resistor. No temperatures taken.
23	Texas	Standard	Yes	1.64	0.848	Rerun of heat 11 - 3/6 mesh graphite in chimney. No temperatures taken. Later found temperatures too low.
24	Texas	Standard	Yes	1.65	0.774	Standard heat except 20/35 mesh resistor. Faulty freon gas control - 25% excess. No temperatures taken.
25	Texas	Standard	Yes	1.68	0.798	Normal run. No temperatures taken.

TABLE II (Con't)

Heat No.	Coke	Pitch	Impreg.	Appar. Density	Ave. dlh	Remarks
26	Texas	Standard	Yes	1.64	0.946	3/6 graphite mesh chimney had 2 in. sandwich of 10/20 graphite 2 in. over bars. No temperatures taken. (Later check of resistence showed temperatures hot enough)
27	Texas	Standard	Yes	1.68	0.815	Aqueous 15% Na_2CO_3 impregnated bars. Furnace unpacked at phase 1 with bars hot. Improved density. No cracks. No temperatures taken.
28	Texas	Standard	Yes	1.64	0.925	Freon 114 (45% F) in place of 12 (31% F). No temperatures taken.
29	Texas	Standard	Yes	1.62	1.004	Packing permeability adjusted to 0.5 darcy from measured 35 darcys. Temperatures measured. 200 KWH added in addition to previous maximum.
30	Texas	Standard	Yes	1.67	0.873	Repeat of heat 27 except bars not removed during process.
31	Texas	Standard	Yes	1.69	0.958	Repeat of heat 27. Bars soaked in Na_2CO_3 and 0.01% Tergitol, then dried. Standard run.

The technical factors investigated during the interval covered by this report fall into six categories:

1. Variation of the quantity and type of purification gas used.
2. Variation of the time cycle of the standard runs.
3. The investigation of diffusional effects on the purity of the product.
4. The investigation of packing carbon and resistor carbon size and types on the effectiveness of the process.
5. A standardization of results obtained on a Texas coke graphite using a standard purification cycle.
6. The investigation of a swelling inhibitor to improve density and decrease cracking in the Texas coke bars.

The relative cost of chlorine and freon gas is sufficiently different to explore the possibility of using a greater amount of chlorine in an effort to obtain the same degree of purification. In heat 19 the chlorine cycle, phase 2, was altered to introduce chlorine at a higher temperature. The average dih for this run was poorer than usual. This heat, in addition to heats 12, 14, 15 and 17, indicate that purity gains cannot be obtained by extending the purification cycle time or altering the quantity of chlorine and freon added to the furnace. In heat 28 the purifying gas used was freon 114 in place of freon 12. The objective of this experiment was to note the purifying efficiency of a gas containing a greater atomic percent fluorine. Freon 114 has 45 percent fluorine, whereas freon 12 has 31 percent fluorine. The average dih was .925. It is not clear from this experiment whether a significant improvement in purity was obtained by this substitution because other furnace variables were different. In general, the experiments to explore alternate methods of purification have not yielded any significant results.

In heat 21, 1 x 4 x 18 in. slabs of gas baked carbon, identical in composition to the full-sized bars, were placed directly underneath the bars in the furnace. A standard purification cycle was carried out. The average dih of the slabs was 0.894 compared to an average dih of 0.867 for the large bars. Temperatures were not measured during this heat and there is reason to believe they were low -- of the order of 2200°C maximum. Notwithstanding this, the higher purity of the 1 inch thick specimens indicates that bar size is an important variable in the purification process. This was likewise borne out by experiments conducted subsequent to heat 16. A cross sectional slab of Whiting coke graphite removed from one bar of heat 16 was ashed. The pattern of ash clearly showed that the non-volatile impurities in the bar were greatest in the center and decreased in concentration toward the edges of the bar where the purifying gas had easier access to the impurities. Analysis of portions of a similar cross sectional piece showed that the bar had 192 ppm vanadium in the center, although the edge of the bars had only 28 ppm vanadium. Partial oxidation of slabs from this bar also clearly showed that the vanadium oxide impurity acts as a catalyst for the oxidation of graphite by air.

Heats 18, 23, 24, 26 and 29 were attempts to duplicate the excellent results obtained in heat 11. A standard purity cycle was employed in each of these cases. The only variable altered was the size of graphite resistor carbon particles used between the bars and the size and permeability characteristics of graphite and carbon materials surrounding the bars and thermally insulating the furnace. Some appreciation of a more optimum resistor carbon has been obtained. The relative permeability of regions of the packing is better understood, although considerable difficulty was experienced in heats 21 through 28 in obtaining comparable purity results to that obtained earlier. The reason for this was determined to be principally temperature control but was also contributed to by high gas permeability through outside regions of the furnace bed. During heats 21 through 28 the method of measuring temperature previously used was abandoned and the control of the process was regulated by kilowatt hour input. This would be satisfactory and has been used in past production efforts; however, alterations in the type of packing carbon used in the furnace was later found to have altered the kilowatt hour vs. temperature relationship. It is believed that heats 21, 22, 23, 24, 26, 27 and 28 may have attained only about 2200°C maximum temperature, and because of this the effectiveness of other variables changed during this time is not clearly apparent.

Following heat 28, the permeability of the outside packing carbon was found to be about seven times too great. When this factor was corrected and the temperature adjusted to the normal 2450°C, heat 30 yielded satisfactory results. This sequence of heats, although admittedly off-standard, offers a variety of conditions which should alert National Carbon to possible operational difficulties in their production efforts. Although no temperature was taken during heat 25, the size and weight of the Cleves coke bars used in this heat was such that the furnace temperature should have reached the normal maximum with the amount of power expended. The low dip for heat 25 was thought to result from non-optimum resistor carbon size which apparently affects the diffusion of purifying gas into the carbon bars.

Heats 27, 30 and 31 investigated the use of an aqueous solution of sodium carbonate added to the green bars prior to purification to reduce the swelling and cracking of the furnace charge. This series of three heats has demonstrated that it is necessary to dry the bars prior to their being placed in the purifying furnace; however, when this is done, the density improves, there is noticeably less cracking, and the purity of the bars as judged by heat 31 remains comparable to that obtained without the use of a swelling inhibitor.

Very truly yours,

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Assistant Head, Pile Materials
PILE TECHNOLOGY UNIT
ENGINEERING DEPARTMENT

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