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SUBJECT OR TITLE

Graphite Development Special Agreement G-3

TO

W. K. Woods, Richland Washington

FROM

V. C. Hamister

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CLEVELAND 1, OHIO

TELEPHONE
MELROSE 1-3100

OFFICES
1280 WEST 73RD ST.

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May 13, 1953

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8. File

Dr. W. K. Woods
General Electric Company
Hanford Works
P. O. Box 100
Richland, Washington

Graphite Developments Under
Special Agreement G-3

Dear Dr. Woods:

The provisions of Special Agreement G-3 called for a final report describing the work carried out by us at the Niagara facility. Four copies of this report, Document number HWCo-13 under the title "Graphite Developments under Special Agreement G-3" are being sent with this letter.

We want to express our appreciation for the fine cooperation of the Hanford technical personnel while carrying out this development. It inspired us to the best we knew how.

Procedures developed during this work on the F process will raise the general level of quality and uniformity of graphite produced under G-5 and G-12.

Very truly yours,

VCHamister

V. C. Hamister.

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Page

Graphite Developments Under Special Agreement G-3

by

V. C. Hamister
H. A. Downey
E. Micinski

Research Laboratories
National Carbon Company
A Division of
Union Carbide and Carbon Corporation

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HWCo-13

May 1, 1953

Graphite Developments Under Special Agreement G-3

V. C. Hamister
H. A. Downey
E. Micinski

Introduction

This report describes work carried out in fulfillment of a development contract designated as Special Agreement G-3, dated November 15, 1951, between National Carbon Company, a division of Union Carbide and Carbon Corporation, and the General Electric Company, Nucleonics Division. The prime objective of this agreement was to establish the feasibility of producing pile-grade graphite from petroleum cokes not hitherto used for this purpose. Cleves coke, the principal raw material previously used for pile graphite was no longer produced by the Gulf Oil Company. A secondary objective of this agreement was improving the F process.

To fulfill the commitments of this contract and also comply with the security provisions, a special facility was installed in an isolated building located in National Carbon Company's plant N, Niagara Falls, New York. With this facility it was possible to carry out the simultaneous graphitization and purification of 4" x 4" x 18" carbon bars under conditions closely simulating large scale operations.

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Under the terms of the contract, National Carbon agreed to produce 16,667 pounds of graphite in this experimental facility at a unit cost of six dollars per pound. Actually thirty-six heats were made producing a total amounting to 19,640 pounds. The excess over the contractual quantity was produced and shipped no charge until the funds in the experimental account were exhausted.

Conclusions

The following conclusions can be drawn from DIH measurements made on graphite bars produced in the small scale facility:

1. Graphite bars made of petroleum coke from the Lockport refinery of the Texas Oil Company can be brought to the same purity level as bars made from petroleum coke from the Cleves refinery of the Gulf Oil Company.

2. Chlorine can be substituted for carbon tetrachloride in the F process.

3. Permeability control of the carbon materials surrounding the bars during purification is an important factor in process control and simplification.

[redacted]

4. A visual flame test, passing a combustible gas through the furnace charge while it is being assembled, measures uniformity of gas distribution.

5. Higher density graphite bars obtained without pitch impregnation can be made through addition of furnace blacks.

6. Increased graphite density obtained in this way results in lowering of stock permeability and DIH.

7. Graphite bars made from Texas coke may be susceptible to cracking in normal F processing. This cracking results from sudden volume increase at temperatures ranging from 1400° to 2500°C. This swelling can be prevented by the addition of small quantities of sodium carbonate (approximately 1 per cent) either as an impregnant or solid addition to the initial coke-pitch mixture.

Equipment

The production of graphite bars for moderator use in nuclear reactors is mainly carried out by conventional procedures, namely extrusion of a plastic mixture of petroleum coke flour and coal tar pitch to form rectangular bars, firing the shapes in an inert atmosphere, pitch impregnation, followed by a second firing. The final stage, however, is

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unconventional in that the graphitization of the bars is carried out in an atmosphere of halogen gases. In this step, halogen gases permeate the bars at temperatures ranging from 1200° to 2500°, and combine with undesirable elements to form chlorides and fluorides which volatilize, leaving a mass of substantially pure carbon. Also, by virtue of the refining temperatures, carbon is transformed into graphite which is amenable to machining with metal tools. Security restrictions on this final operation, called the F process, necessitated the installation of equipment in an isolated space. The first section of this report deals with a description of the equipment which had to be installed to carry out the contract commitments.

Transformer

In order to evaluate graphite bars made from new materials, a size requirement had to be met which to a large degree determined the scale of the project. No chemical methods had the requisite sensitivity for defining purity levels of graphite. The only certain method was to determine DIH of test bars in the Hanford test pile. To do this, experimental bars had to be approximately four inches square and in sufficient quantity to make forty-eight inch lengths. This meant building a small-scale Acheson type furnace with volt-ampere characteristics which would match available transformers.

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The volt-ampere characteristics of an Acheson graphitizing furnace are notoriously bad. The initial furnace resistance is high, dropping off rapidly with increasing temperature and ending up at anywhere from one-tenth to one-eighth of the starting resistance. Transformers of special design are therefore required to meet the power requirements. After a survey of all available electrical equipment, two 300 KVA twenty-five cycle transformers were located, with secondary voltages ranging from 30 to 70 volts. By interconnecting the primaries of these transformers with open disconnect knife switches, they could be operated in parallel, thereby obtaining a low voltage power source up to 600 KVA. To meet underwriters' requirements, a concrete block vault was built, and a 12,000 volt cable installed in conduit for a distance of 450 feet. The transformer vault was built adjacent to the furnace building, serving as an office with two security safes. The door leading into the transformer room was made of heavy steel plate, with locks which could not be opened with master keys.

Furnace

An Acheson graphitizing furnace consists essentially of a firebrick hearth mounted on concrete piers with U-shaped concrete structures at the ends of the hearth holding graphite conductors called head electrodes. Spacings between the bed piers serve as air ducts for cooling the firebrick

[redacted]

hearth. The small-scale Acheson furnace used for this project is shown in Figure 1. It had an overall length of 18-1/2 feet, and width 9 feet with 4" x 16" head electrodes.

Knowing the volt-ampere characteristics of the power source it was possible to calculate what the furnace dimensions might be to hold a charge of bars which could be heated to the required temperatures. By cut-and-try methods, it was found that bar length should be eighteen inches, and twenty-five bars constitute a furnace charge weighing five hundred pounds. Spacing between the bars would be one inch, thereby fixing the distance between head electrodes at twelve feet. A furnace of these dimensions connected to the aforesaid power supply would be large enough to simulate conditions in production units, and furnish bars of the required cross-section without requiring a large tonnage for experimental work.

Control of the experimental conditions for this small F process was centered largely in the control board shown in Figure 2. On this board were mounted voltmeters showing the primary and secondary voltage, an integrating wattmeter, an ammeter for current in the primary coils and an indicating wattmeter. With these meters it was possible to control rates of power input and therefore rates of temperature rise in the charge. Metering of the reagent gases was accomplished by three twelve-inch Meriam

[redacted]

Figure 1

Figure 2

[redacted]

manometers which measured differential pressures across three disc orifices mounted in flange-type couplings. A fourth manometer indicated back pressure in the furnace manifold. No pressure reducing valves were used. Gas flows were throttled with Meriam micrometering valves directly from cylinder pressure.

From the control board, reagents were piped to a manifold made of two-inch extra heavy steel pipe which paralleled the furnace hearth. Risers from this manifold were made of 1/4 inch standard pipe welded in place, spaced on 5-1/2 inch centers. Each riser had a 1/4 inch gasket type pipe union serving as a holder for a steel orifice plate. Gas from the riser was carried into the furnace by a carbon tube 3/4 inch O.D., 1/4 inch I.D., 19 inches long. Each of the twenty-five tubes was connected to a 1/2 inch street elbow at the top of the riser by a flexible rubber sleeve. This simple system could be used with chlorine and Freon 12. It could not be used with carbon tetrachloride which required cemented connections and flexible stainless steel tubing. Small holes, .021 inch in diameter, in orifice discs held in the pipe unions in each of the risers gave uniform gas distribution along the furnace length. The uniformity of gas distribution can be seen in Figure 1 which was taken with propane gas being fed into the manifold. The white spots in this figure are incandescent propane flames.

Scrubber

Because of the noxious nature of gases produced by high temperature reactions in the furnace charge, a scrubber had to be installed to neutralize these before they were vented into the atmosphere. This scrubber was a cylinder fifteen feet in height and three feet in diameter, filled with 1-inch carbon Raschig rings. A two per cent caustic soda solution was fed counter-current to gas flow at a rate of six gallons per minute. Raw gases from the charge were withdrawn from the furnace hood consisting of corrugated iron laid across the side walls, by a Sturtevant exhaust fan having a discharge of one thousand cubic feet per minute against a nine-inch water head. This exhaust fan was connected to the top of the scrubber as shown in Figure 4. Hot gases from the furnace entered at the bottom.

To prevent harmful gases leaking out through cracks in the furnace side walls, 3/4 inch plywood boards were mounted at an angle against the side wall, and held in place by furnace piers as shown in Figure 3. This combination formed a convenient duct through which air was drawn by the exhaust fan. Transfer from this duct to the furnace hood was by means of U-shaped steel pipe.

Screening

Recovery of bulk carbon materials which surround the burden during heating and cooling is an important part of the operation. These

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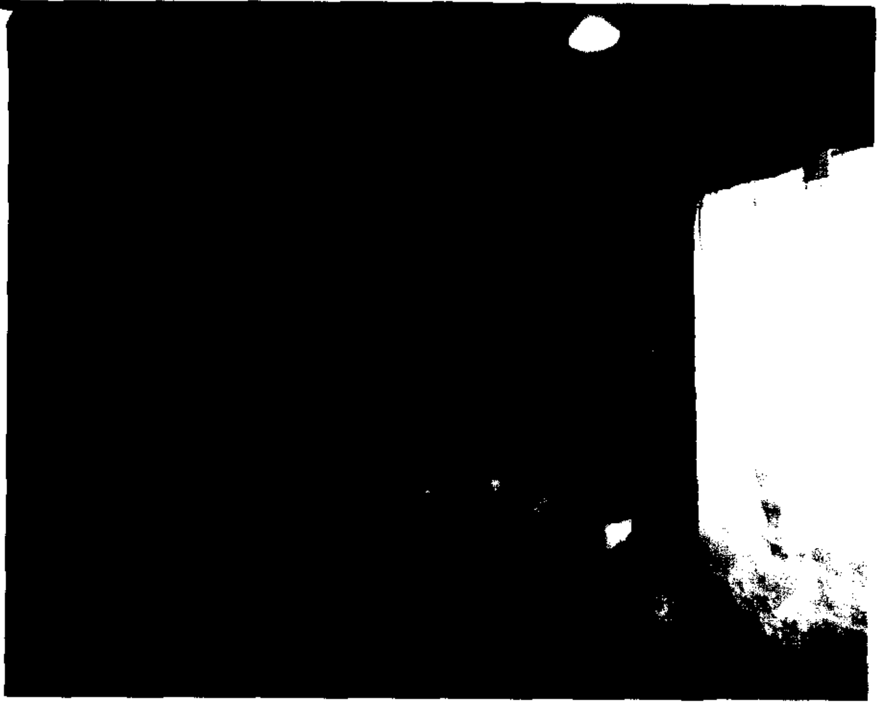

Figure 3

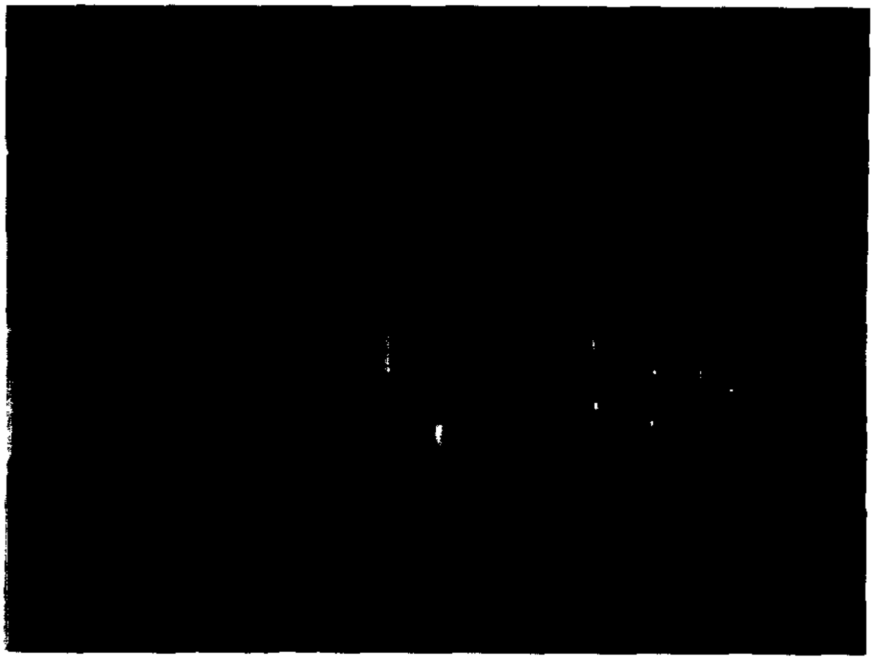

Figure 4

[redacted]

bulk materials, varying in particle size, act as heat insulation, while power is going through the burden, prevent oxidation during the cooling cycle, and serve as impermeable barriers, forcing the reactive gases through the burden. The weight of this carbon blanket or envelope amounts to four or five times the weight of the burden. By judicious selection of particle size, the components of the envelope can be separated by screening on multiple deck screen and thus become available for re-use. The Rotex screen used in this facility can be seen in the right hand side of Figure 4.

These bulk materials were moved about in a ten cubic foot hopper by means of a hand operated crane which travelled the length of the building.

In essence then, this facility was a miniature graphitizing plant in which all of the operations could be carried out in a self-contained unit. It was ideally adapted to study the F process.

Operations

In this section of the report we will describe how this facility was operated, the problems encountered and how they were solved. The general practice was to carry out one heat per week. This was usually done on Thursday, and thereby have Friday, Saturday and Sunday for cooling. On Monday, the furnace was unpacked, the individual bars measured for resistance and density, marked as to furnace location and heat number, individually wrapped with manila paper and boxed for shipment by railway express to Hanford. On Tuesday and Wednesday, the bulk materials were recovered by screening, and the furnace was packed for the next heat. A set of curves was prepared for each heat showing the time relationship for furnace resistance, temperature and cumulative kilowatt-hour input. These curves and also a sheet of individual bar properties were sent to Mr. L. P. Bupp soon after the heat was made with a letter stating the objective of each trial.

Packing the Furnace

Figure 5 shows a cutaway isometric view of the furnace and also the space relations of the charge in the furnace. The wall on the left hand side is a single concrete casting resting permanently on the firebrick hearth and extending the full length of the hearth. On the left hand side, the side wall was formed of concrete blocks resting on a permanent wall 13 inches in height. The side blocks were removed after each heat by means of the travelling crane. A refractory bed was formed on the firebrick hearth by tamping a mixture of granulated petroleum coke, through 14 on 100 mesh, with finely divided coke powder obtained from the plant dust collectors. An 80/20 mixture of these two components could be tamped in place as if it were moist earth. Its resistance to gas flow was high, thereby preventing gas leakage through the firebrick hearth. The top of this bed was at the same level as the top of the 13-inch permanent wall. This carbon bed was never changed. Occasionally it was spaded and re-tamped to minimize the risk of forming a low electrical resistance path which would divert current from the bars.

Graphite distributor tubes are laid directly on the tamped bed. These are 20 inches long, 2-3/4 inches O.D. by 2 inches I.D. with two 3/8 inch slots 8 inches in length cut out on one side. Initially one of these tubes was laid directly below each bar with the slot at the eight o'clock position. After positioning each of these tubes opposite a riser from the gas manifold, the space between is filled with granulated graphite screened through three on eight mesh. This material is levelled off from 1/4 inch to 1 inch above the distributor tubes.

Connection between the individual graphite distributor tubes to the external gas manifold is made by a carbon sleeve 9 inches long, 1-1/2 inches O.D. by 1 inch I.D. and a carbon tube 19 inches, 7/8 inch O.D. by 1/4 inch I.D. If the 1/4 inch I.D. tube were carried directly into the graphite distributor, the resulting high temperature would bring about carbon deposition in the tube, stopping it completely. By having the small diameter tube far enough away from the hot zone, carbon deposits form in the one-inch sleeve, but do not plug it completely.

After the carbon tubing is in place, side boards with dowels are put in place as shown in Figure 6. These hold tubes in alignment, and also make it possible to tamp in side insulation made of the same 80/20 dust mixture used in the furnace bed. This material effectively seals junctions in the carbon tubing. Now the carbon bars which are to be purified and graphitized are placed in position as shown in Figure 7.

The carbon cover over the bars is made up of two components, a cone-shaped section running the full length of the furnace consisting of through three on six mesh coke or graphite particles, and side cover composed of 8/20 particle-dust mixture which covers the cone leaving the tip exposed. Corrugated steel sheets are then laid between the side walls, and the furnace is ready to be fired. This arrangement can be clearly seen in Figure 5.

Firing Operation

The carbon bars constituting the furnace burden are purified and graphitized by a heat treatment, called the F process. Heat is developed in the charge when a potential is applied to graphite electrodes located at the ends of the furnace. Less than one-tenth of the total heat developed by passage of current through the charge is within the bars. Heating takes place almost entirely in the graphite granules which fill the one-inch

Figure 6

Figure 7

[redacted]

spacing between adjacent bars. In this manner bars can be heated to 2500°C. in a gas atmosphere containing chlorine and fluorine. Given sufficient time, these gases penetrate into the bars forming compounds with bar impurities having a higher vapor pressure than the impurities by themselves. Because the reaction products must diffuse outward, while reagents are diffusing inward, the four important factors in the process are temperature, time, gas concentration around the bars, and bar permeability. The first three are controlled by the manner in which the furnace is packed, and the time sequence and flow rates of reagents during the firing operation.

In practice, the firing operation can be divided into four distinct brackets whose boundaries are defined by the kind of gas metered to the furnace at different temperatures. These brackets for standard procedure were as follows:

Bracket No. 1

Power turned on with nitrogen flowing at 25 cubic feet per hour until charge temperature reached 1000°. Power input kept below 250 KVA to prevent cracking of the bars. Bracket time was 2.5-3.0 hours.

[redacted]

Bracket No. 2

Chlorine feed started at a rate of four pounds per hour for two hours. Power input increased to bring charge to a temperature of 1700 to 1800° at the end of this bracket.

Bracket No. 3

Freon 12 feed started at a rate of twelve pounds per hour for four hours. Power conditions adjusted so as to reach a temperature of 2450-2500° in three hours after starting Freon feed. Freon flow continued for one hour after power was off and the furnace was cooling down to 2200-2300°.

Bracket No. 4

Nitrogen gas fed at 25 cubic feet per hour for 16 to 18 hours when the charge temperature had dropped to 1000-1200°.

For the most part, temperatures were sighted with an optical pyrometer pointed into a graphite distributor tube usually in the number thirteen bar position. There were a number of heats made without an optical pyrometer. Then the bracket endings were based on K.W.H. input.

Figure 8 displays three curves showing how furnace temperature, furnace resistance and total energy input changed with time of operation,

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Heat No. 40

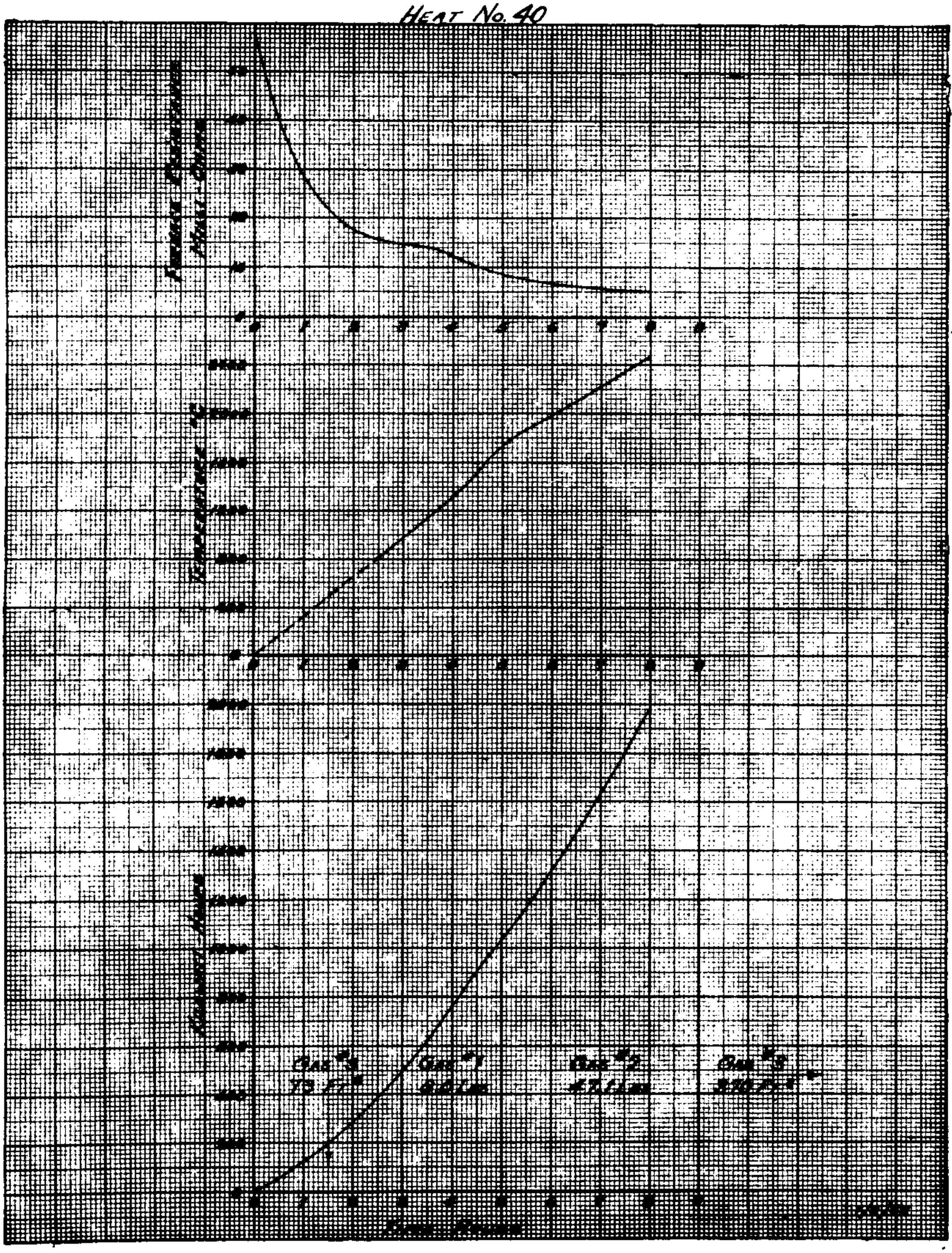

Figure 8

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while Figure 9 shows the apparent densities and resistances of individual bars in the heat, and discharge rates of the manifold orifices before and after the heat was made. These two sets of data were sent to Mr. L. P. Bupp for every heat.

For the main part, this firing method was adhered to in the majority of the heats. A few variations from this standard procedure were tried, for example longer Freon time, longer chlorine time, and lower gas rates. None of these gave any indication of quality improvement or lowered costs. Summing up, the standard F process conditions were three hours in a nitrogen atmosphere to 1000°, two hours in chlorine to 1750°, four hours in Freon 12 to 2450° including one hour during cooling, ending up with sixteen hours flushing in nitrogen.

Unpacking, Marking and Shipping

On the Monday following the firing operation, the concrete side blocks were removed from the furnace and the envelope materials shovelled into hoppers for subsequent recovery by screening. Bars were marked at the ends with rubber stamps and an aluminum ink with the designation XGBF, heat number and furnace position. Densities of individual bars were obtained from measurements of weight and volume, and resistances by potential drop using ten amperes from a storage battery. Individual bars were then

[redacted]

BAR PROPERTIES AND ORIFICE CONDITION

HEAT NO. 40 DATE 1/2/53

Position	Specific Resistance micro-cm. ohms		Apparent Density grams/cc.	Orifice Flow - cc./min.	
				Before	After
1	1093	TSX-1	1.54	505	426
2	1111	TSX-1	1.55		
3	1233	TSX-1	1.53	488	450
4	1151	TSX-1	1.54		
5	1169	TSX-1	1.55	505	426
6	919	X185F	1.59		
7	729	XCHF	1.66	513	441
8	1261	TSX-1	1.54		
9	908	X185F	1.59	473	433
10	649	XCHF	1.65		
11	1864	TSX-1	1.53	513	426
12	1006	X185F	1.57		
13	619	XCHF	1.64	513	433
14	1332	TSX-1	1.54		
15	989	X185F	1.61	505	426
16	695	XCHF	1.62		
17	1429	TSX-1	1.52	505	441
18	1035	X185F	1.60		
19	624	XCHF	1.63	505	441
20	1844	TSX-1	1.53		
21	962	X185F	1.61	497	433
22	670	XCHF	1.63		
23	1341	TSX-1	1.53	530	480
24	1418	TSX-1	1.53		
25	1183	TSX-1	1.55	490	433
	1229	TSX-1	1.55		
	1209	TSX-1	1.54		

Figure 9

[redacted]

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[redacted]

wrapped in manila paper and sealed with tape. These were packed in wooden boxes and shipped by prepaid railway express.

Carbon Bar Composition

This phase of the report deals with the different kinds of bar materials which were put through the experimental F process. In a sense, this evaluation of base materials hitherto not used in the manufacture of pile-grade graphite appeared to be the most important aspect of Special Agreement G-3. Previous trials of other materials during the Morganton operations such as bars made of Whiting or pitch cokes, always gave bars of lower DIH than those made of Cleves coke. Hence, there was a considerable element of risk in going to other base materials without knowing how they would respond to F-processing.

Then, too, there was the question of evaluating an experimental facility for carrying out the F process in comparison with the Morganton experience. One might be built for evaluation purposes and not do as good a refining job as the larger installation. Fortunately, a small quantity of 4" x 12" x 60" carbon bars made from Cleves coke in 1944 for another phase of the Manhattan Project were still available at Clarksburg. This material made a good starting point for evaluating any new facility.

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After a general review of the properties and availabilities of petroleum cokes from different oil refineries, a coke made by the Texas Oil Company at their Lockport, Illinois, refinery was chosen for the initial trial. This choice was based on sulfur and vanadium content in the coke. On both of these counts it was known that this coke was inferior to Cleves. Later on it was established that the sulfur level in Texas coke could cause cracking of bars during the F process. Cleves coke bars processed by the same temperature cycle would not crack. This gave a valuable clue in locating the source of the difficulty.

Because of the cross sectional size of experimental bars required by the test pile at Hanford and also in the F process, experimental batches had to be extruded in factory equipment. No pilot plant equipment was large enough to extrude cross sections this large. The system of bins, elevators and mixers used with plant equipment is so large that the minimum weight of a trial batch would have to be five tons.

Soon after signing of Special Agreement G-3, about 500 bars were extruded at Niagara Falls under laboratory supervision made from Texas coke and standard pitch. In addition, 400 - 4 x 4 bars were obtained from the 4 x 12 x 60 Cleves coke bars found in the Clarksburg stock pile. These two items made up the major stock pile for carrying out the experimental work.

[redacted]

[redacted]

Whiting coke bars large enough for these trials were available as standard product. Each trial with this bar composition supported the earlier Morganton experience that this kind of coke produces bars more difficult to purify than other cokes. These early trials led to the finding that with normal purifying conditions, a central impurity core persists in the bar, in which vanadium was the principal element. Later on with improvement in furnace packing conditions, Whiting coke bars were brought to DIH levels above 0.90.

Then there were three bar compositions made in a 3-3/4" x 4" cross section in which one of the raw material components was furnace black such as is used by the tire industry. It had long been known that additions of this material to petroleum coke mixture raises density levels. One of these called T-12, was made from a mixture of 88 parts Texas petroleum coke with 12 parts furnace black. A second, called T-25 contained 75 parts coke and 25 parts black. A third called T-30-S contained 70 parts coke and 30 parts black together with powdered sulfur for increasing coking value of pitch binder. All of these gave high density levels without pitch impregnation and rebaking. T-12 had a density level of 1.70, T-25 - 1.77 and T-30-S - 1.83. These bars were poor structurally, sawed sections exhibiting many laminations. Moreover, they did not respond to the purifying treatment, probably because of low permeability resulting from addition of furnace black.

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Another set of bars made from a petroleum coke from the East St. Louis refinery of Socony-Vacuum coke was obtained by extruding bars from a mixture used for eighteen inch electrode coke electrodes. This material contained about 50 per cent coarse petroleum coke particles, had an apparent density of only 1.45. With this low density, it was expected that the bars would have extremely high permeabilities which could be refined to high DIH values. This assumption, however, was incorrect. Gas permeability of this low density stock was no higher than finer grained material of higher density.

Then there was another set of experimental bars made up containing additives in the form of iron oxide and sodium carbonate. The function of these additives was to suppress rapid swelling at high temperatures resulting from the presence of carbon-sulfur complexes in Texas coke. As will be shown later on in this report, cracking of bars made from Texas coke during F processing was due entirely to this high temperature swelling phenomenon. Fortunately, it was later established that this high degree of swelling was confined to this initial Texas coke lot, and did not appear in as high a degree in bars made for Special Agreement G-5. For several months, however, the high incidence of cracked bars in the experimental furnace was a big cause of worry about meeting cracking specifications. This phase absorbed most of the developmental energies during the last three months of the contract.

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Following is a summary of all the carbon bar compositions which were evaluated in the Niagara facility. Wherever the designation NP appears after the grade designated, the bars were not pitch impregnated and rebaked.

Grade	Base Material	Additive	DIH
TS (NP)	Texas Coke	-	1.02 - 1.04
TS	Texas Coke	-	.77 - 1.01
T-12 (NP)	Texas Coke-furnace black	-	.83 - .84
T-25 (NP)	" " " "	-	.85 - .93
T-30-S (NP)	" " " "	-	.56
CS	Cleves Coke	-	.80 - .96
CS (NP)	Cleves Coke	-	.97
TA (NP)	Socony Coke	-	1.03
WS	Whiting	-	.62 - .97
TS-B (NP)	Texas	1% FeO	- .54
TS-C (NP)	Texas	2% FeO	-1.72
TS-D (NP)	Texas	1% Na_2CO_3	.86
TSX (NP)	Texas	.8% Na_2CO_3	1.05

The final column in the foregoing table lists a range of DIH values obtained at Hanford on bars made of these compositions refined under standard conditions. The DIH ranges for TS and CS bars are wider than they should be, because for a period of 8 weeks, Heats 21 to 28 inclusive, the process was out of control. On the other hand, DIH values on bars containing furnace blacks, and also the iron oxide additives reflect purity levels attainable with these compositions under standard F process conditions. More drastic refining conditions would have to be developed to bring these compositions to DIH levels attainable with TS bars.

In every instance where TS and CS bars were purified under the same conditions, the DIH level for TS was slightly higher than for CS. At no time was there any indication that TS bars were more difficult to purify than CS. This was indeed fortunate, because any increase in refining time would decrease graphite production by a percentage equal to the per cent increase in the power time.

Gas Test

Up until Heat 11, operations were planned to duplicate the Morganton conditions as described in Document GEH-15043 as closely as possible. The resulting DIH values proved that the small experimental facility produced purity levels matching the large scale Morganton operations. The two operations had another factor in common, namely a wide spread in DIH values among individual bars or groups of bars within a given heat. Previous

[redacted]

work at Morganton had furnished strong proof that this non-uniformity factor was not due to temperature variations. Specific resistance of the bars gave an index which would tell the temperature a bar had reached in the process. No correlation could ever be found between DIH and bar resistance. It was therefore concluded that non-uniform treatment was due to variation in reagent concentration around the bars, or stating it in another way, reagents were not flowing uniformly through the charge. Now it could be easily established by simple flow measurements that orifices in the manifold risers were dividing the total gas flow uniformly to the burden. Non-uniformity in gas distribution must be due to some factor within the charge.

The solution of the problem of how to evaluate gas distribution through the charge turned out to be a very easy one. Simply pass a combustible gas through the manifold system and the charge which had been assembled to a point where the tops of the bars were still visible, but the spacing between the bars had been filled with the conventional through 3 mesh on 8 mesh granular graphite, then ignite the gas. Figure 10 illustrates the extent to which the charge had been assembled while Figure 11 shows the flames of burning gas as after passing through the burden. It was quite apparent to the eye that gases were not flowing uniformly around the bars. A number of attempts were made to improve the distribution uniformity

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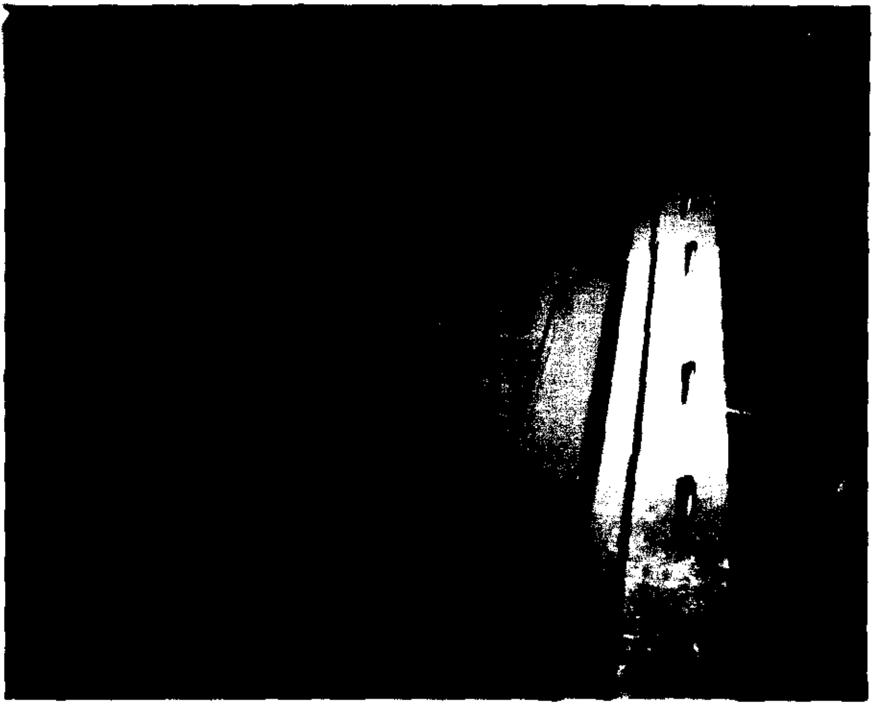

Figure 10

Figure 11

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through tamping or knifing the material between the bars, also by closer screening of the granular graphite resistor. None of these resulted in any appreciable improvement. These observations, however, led to the concept that gas distribution might be improved if the permeability of the graphite resistor between the bars were lowered to a point where the spacings might behave like orifices in pipes.

Permeability of Graphite Granules and Solids

The foregoing considerations led to a study of gas flow through granular and solid graphite masses. This was carried out at the National Carbon Research Laboratories by Mr. Erwin L. Piper.

No attempt will be made in this report to go through the theory of flow through porous media. According to Darcy's law, the rate of gas flow through a porous medium is directly proportional to the cross-section and pressure drop through the medium, inversely proportional to the length and viscosity of the gas or

$$Q = \frac{KA\Delta p}{\mu}$$

where Q is in cm.^{3}/sec., A in cm.^{2}, Δp in atmospheres/cm. and μ in centipoises, K is the permeability in terms of a unit called a darcy.

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This unit is analagous to the electrical conductivity of a conductor. These relations, however, only hold true below limiting flow rates, in what is called the viscous region. For a gas like nitrogen or air, the superficial velocity should be less than 3 cm./second.

A series of closely sized graphite granules was obtained by crushing and screening using Tyler standard screens in which each successive screen opening increased by a factor equal to the $\sqrt{2}$. The permeability of these screened fractions was then obtained by measuring the pressure drop when nitrogen was passed through a bed whose dimensions could be measured. The permeability of these screen fractions together with their average particle diameter as defined by the average of the screen openings which passed and held the particles, is shown in the following table:

Screen Series	Average Particle Diameter Inches	Permeability in Darcys
Through 4 on 6	.158	16,000
" 6 " 8	.112	9,160
" 8 " 10	.079	4,890
" 10 " 14	.056	2,640
" 14 " 20	.039	1,500
" 20 " 28	.028	752
" 28 " 35	.020	461
" 35 " 48	.014	244

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When these permeability numbers were plotted against average particle diameter in inches on logarithmic paper, the points fell closely on a straight line yielding the relation

$$K = 389{,}000\ d^{1.72}$$

where K is permeability in darcys,

d is average particle diameter in inches.

It would hardly be feasible to obtain the required tonnage of screen fractions sized as closely as the ones in the foregoing table. As a compromise, particles were obtained between screens in which the larger opening was double the smaller.

These commercially sized particles which would be available in sufficient quantities for large scale operations have properties shown in the following table:

Screen Series	Average Particle Diameter Inches	Permeability in Darcys
Through 3 on 6	0.197	10,000
" 8 " 10	0.078	4,500
" 10 " 20	0.048	1,100
" 20 " 35	0.025	500

[redacted]

The first use of these permeability numbers was made in Heat No. 11 in which 10/20 mesh graphite granules were substituted for the 3/8 mesh previously used as resistor. As can be seen from the foregoing table, these had about one-ninth the permeability of 3/6 mesh particles around the graphite distributor tubes under the burden.

The effect of this seemingly trivial change upon gas distribution when propane was passed through the charge was startling. Instead of incandescent flame jets at isolated points above the charge as shown in Figure 11, the burning gas appeared as minute blue flame spread uniformly over each of the resistor spacings between the bars.

The improvement in gas distribution resulting from the decrease in particle size of the graphite resistor gave rise to the question of what would happen if the number of branch lines from the manifold were decreased. First alternate risers were blocked with no visible effect on flame uniformity, then alternate groups of two and still no change in flame appearance. At this point, gas was being fed into the charge from only nine branches instead of the usual twenty-five.

It was decided to stop at this point, and find out what effect the reduced particle size would have upon the furnace resistance during the firing operation. No appreciable effect could be found.

DIH results of the individual groups of bars from Heat No. 11 are of particular interest, because they showed the smallest variation within any heat made thus far and also the highest average value. These values taken from HW-25147 are listed in the following table:

Bar Nos.	DIH
1,2,3	1.038
4,5,6	1.020
7,8,9	1.024
10,11,12	1.014
13,14,15	1.038
16,17,18	1.026
19,20,21	1.020
22,23,24	1.028
23,24,25	1.024
Average	1.026

These results validated the concept that uniformity of gas distribution around the bars would be improved by increasing resistance to gas flow in the space between the bars, and at the same time have a low resistance bed below the bars. The low resistance bed would serve as a plenum chamber for equalizing gas flow in the bar spacings.

Three other implications came about through this line of approach. First, the gas test offered a means of control for large scale operations to show that the furnace had been properly assembled. This is extremely important for round-the-clock, seven-day-a-week operations. Second, particle sizing of material used within and below the burden would have to be more exacting than had hitherto been realized. Third, the number of feeders from the manifold could be reduced, thereby simplifying manufacturing operations. All of these should result in a higher and more uniform level of bar quality than had hitherto been possible.

Two other particle sizes were used as resistor material, 8/10 mesh and 20/35. The incentive to use the larger size particle resulted from difficulties in obtaining clean seperation of the 10/20 particles from the other envelope constituents. The 8/10 mesh had a permeability about one-half of the 3/6 mesh. With 8/10 mesh resistor, gas distribution as shown by the flame test was not quite as good as with the 10/20 mesh resistor. On the other hand, it was found that coke particles which had not been graphitized would contaminate the 10/20 mesh graphite resistor. This contamination increased resistance of the burden, thereby diverting power to the carbon bed below the burden. DIH results with 8/10 mesh material were not as good as the 10/20 mesh. Since the volume of resistor material required to fill spaces between the bars is small, it was decided to abandon the recovery and start with fresh resistor. The 20/35 mesh material showed no advantage over the 10/20 mesh.

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Another illustration of the importance of permeability in the F process came about unintentionally during subsequent heats which were intended to be checks on Heat No. 11. For quite a time period, DIH levels dropped below the level attained in Heat No. 11. This was puzzling, because the gas test showed uniform gas distribution. After a close inspection of all the operating conditions, it was found that the permeability of the 80-20 mixture of coke particles and dust used as packing material along the sides of the charge had risen to 35 darcys. A freshly made mixture had a permeability of only 1 darcy. Apparently, a portion of the dust from the low permeability side insulation had been lost during screening operations, so that gases could vent more readily through the sides. This situation would be aggravated by the higher resistance to gas flow between the bars. Another process variable crept into the picture during this same period. Graphite particles were substituted for coke particles in the cone, because the latter would not be available in large enough quantities for large-scale operations. Because of the higher heat conductivity the kilowatt-hour input curve derived from optical pyrometer temperature measurements gave lower end temperatures. Beginning at Heat 29, permeability of the side insulation was measured and kept below the one darcy level. After this the process appeared to be on even keel.

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Variations in Gas Conditions

A few heats were made in which flow rates of the reagents were altered from the standard conditions. For example, in Heats 17 and 19, the chlorine input was increased and Freon decreased. Both of the heats gave lower DIH values indicating Freon to be a more active or penetrating reagent than chlorine. Also in two other Heats, 10 and 14, the Freon time was double without increasing the standard fifty-pound input. This longer treating time at one-half the gas rate gave no indications of quality improvement.

Freon 114 was tried as a substitute for Freon 12. It was reasoned that the greater fluorine content might help to increase the DIH level. When this was tried in Heat No. 28 under standard conditions, the resulting product had a DIH of .92. This did not encourage further trials.

In another Heat, No. 9, no chlorine was used, and the Freon rate lowered so the fifty pounds would last for six hours. Again the resulting DIH values gave no encouragement for further work along this line.

One fact was learned from this work with variable gas rates, namely, an increase in plugging of the tubes with carbon deposits. At the lower gas velocities in the carbon branch tubes, Freon would decompose

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inside the tube and build dense deposits. When the twenty-five branch lines were reduced to nine, no carbon deposits ever formed within the tube. The same weight of gas was used per hour, so the gas velocity must have increased almost threefold. This plugging of carbon tubes in the Morganton operations was one of the major operating headaches.

Cracking of Bars in F Process

When two-thirds of the heats under Special Agreement G-3 had been completed, we awoke one day to the fact that bars made from Texas coke were showing a high incidence of visible cracks. This was a sporadic phenomenon, hard to define. Some cracks were of a hair line variety, others were wider with appreciable depth. The problem of doing something about this bar cracking suddenly assumed a very high priority when Special Agreement G-5, a production contract was signed. Among the requirements in this contract was a terse, single sentence specification that bars should contain no visible cracks. After consultation with the technical people at Hanford regarding cracks in the bars made from Texas coke in the Niagara facility it developed that the cracks were too visible and there were too many of them. That was a sad moment because production plans on G-5 were going ahead full steam, and we had no way of telling what production yields of acceptable stock might be, or how much heating rates would have to

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be decreased to prevent cracking. If the heating rate were slowed up, monthly production of the planned facility would fall off proportionally.

Fortunately, Cleves coke bars which had presented no cracking problem in the Morganton operation were still available. When these were processed in Heat 25 under the standard temperature conditions, not a single visible crack could be found in any one of the bars. This finding offered a quick clue to the nature of the problem. Cracking was due to a factor inherent in the petroleum coke from which the bars were made, and that was a sulfur complex in the coke. The presence of this complex causes rapid swelling at temperatures ranging from 1400° to 2400°C. This effect can be inhibited by metal oxide additives.

Texas coke bars were impregnated in an autoclave with a solution containing 50 grams anhydrous sodium carbonate per liter, they retained 0.7 per cent weight of this compound. When these impregnated bars were put through the F process in Heat 27, not a single bar showed a visible crack and the apparent density had risen four points, from 1.64 to 1.68.

Although this experience reveal where the seat of the trouble lay, this apparently simple solution was not amenable to the production scale called for in G-5. It was one thing to impregnate and dry twenty-five bars

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and quite another to do this to ten thousand bars per month. The sensible thing to do would be to add inhibitor to the mix before the bars were formed. One could not do this, however, on a large scale before trying it out on a small scale.

Small lots of bars were made at the Fostoria plant with an improvised die giving a 3-3/4 inch by 4 inch cross section in which iron oxide and sodium carbonate were added to the mixing operation. These were labelled TS-B, TS-C and TS-D, containing 1 per cent, 2 per cent iron oxide and 0.8 per cent sodium carbonate, respectively. Another lot was extruded at Clarksburg with .8 per cent sodium carbonate. These were marked TSX-1 and TSX-2. These two differed in the way the compound was introduced into the mixing machine. In TSX-1, the carbonate was first blended with a small quantity of Texas coke flour in the ratio of one to four. In TSX-2, carbonate was added directly into the mixer.

The Fostoria made bars TS-B, TS-C and TS-D were processed in Heat 35 and TSX bars in Heat 36. None of the bars showed visible cracking. However, this trial proved that heavy metal oxides could never be used as swelling inhibitors. The bars with 1 per cent iron oxide had an average DIH level of .54 and those with 2 per cent had a DIH of -1.58. The bars with sodium carbonate, TS-D, processed in the same heat had a DIH of .86.

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The two lots made at Clarksburg processed in Heat 36 had DIH levels of 1.05 and 1.04, respectively.

While this was going on, about six hundred tons of bars had been extruded at Clarksburg for G-5, and these were in transit or storage at Columbia, Tennessee. Fortunately, a lot system was in effect. Approximately every thousand bars were stamped with a distinctive number at the time of extrusion. This made it possible to obtain samples from every lot for evaluating the swelling characteristic due to the coke. Not a single sample was found having a swelling factor large enough to cause cracking during F processing. In addition, controls were set up to evaluate incoming cars of Texas coke before it was processed. Again no shipment has been found where the swelling factor is large enough to justify carbonate addition to the mix.

It seems odd that the one lot of Texas coke bars made for the G-3 operations should have a larger degree of swelling than subsequent production bars for G-5. It was indeed fortunate it happened this way and not the opposite. With the small facility, and a few bars made from Cleves coke, it was possible to get at the bottom of the difficulty in a short time, and then find a solution for the problem. Had it occurred in large scale operations, production schedules would have been disrupted and costs gone

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sky high before bar cracking would have been brought under control. In all likelihood this difficulty will appear again because of variation in crudes going to the refinery when the coke is made. When the control tests on incoming coke shipments show that swelling has gone beyond tolerable limits, sodium carbonate will have to be added to the initial mix. The alternatives to this would be widening the cracking specifications, or lowering product yields which would mean higher costs.

Length of Diffusion Path

In a previous paragraph, it was pointed out that the removal of impurities from the graphite bars was brought about by high temperature diffusion of halogen gases throughout the bar cross section, forming volatile chlorides and fluorides. The completion of this refining must be dependent upon the length of the diffusion path.

One of the objectives in the experimental program was to find out how high the DIH level might be gotten even under conditions which might not be practical for large scale operation. One way to do this would be to shorten the diffusion path. This could be done by having the bar material in the shape of strips instead of a 4" x 4" cross section, and placing these strips at the bottom of the charge where gas concentration

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would be at a maximum. The strips could then be assembled as test bars for the test pile evaluation.

This scheme was tried in three Heats, 10, 11 and 21. In No. 10 and No. 11, the strips were 1/2" x 4" x 18" made from Whiting Coke. In both instances, DIH values were just below 1.00. Heat No. 21 contained 1" x 4" x 18" Texas coke strips. Again DIH values were below 1.00. There was no indication that purity levels under standard refining conditions were being limited by the length of the diffusion path.

On the other hand, we do have evidence that impurity levels inherent in the petroleum may mean that a larger amount of gas must diffuse into the bar to complete the refining. This is illustrated by results on bars made from Whiting coke refined in Heat No. 16. DIH of these bars average .62. We were interested in finding out something about the residual impurities in this stock and particularly the location within the bar.

Slices cut from one of these bars were ashed slowly at 700°C. on a white plate. Figure 12 shows the appearance of these residues. The chief constituent of the dark central core was vanadium. The outer framework was fluffy silica. Apparently, at the gas concentration available under the

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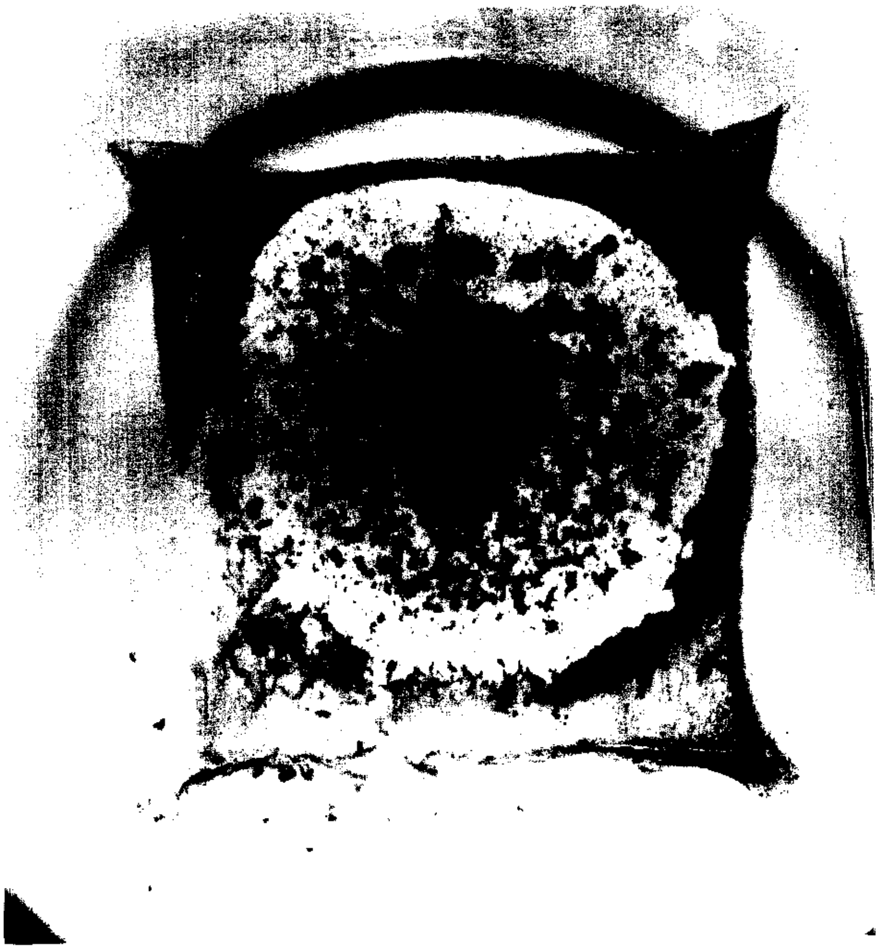

Figure 12

Full size photograph of ash remaining after a 1/2" transverse slice from Whiting coke bar No. 4, Heat No. 16 was heated 48 hours at 700°C. in the presence of air. The dark central portion is vanadium oxide and the outer frame work is stained fluffy silica.

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refining conditions when this heat was made, an inadequate quantity of reagent diffused to the center removed all of the impurity. With slices only half-inch in thickness, however, refining was complete.

This ashing of a slice cut from a bar offered some promise as a control test for large scale operations. There was some difficulty, however, in finding a surface which would be a suitable background for the impurity residue. An ordinary china dessert plate such as can be seen in Figure 12 gave the desired background. This would not be very practical for use in a control test, because the ashing furnace had to be cooled down to room temperature before the plate could be moved. Silver dishes were tried, also silica and pyrex plates. None of these was as good as a dessert plate. Ashing trials were made on slices from all of the heats, but the results were not encouraging enough to install this as a process control.

Summary of Operations

The final section of this report consists of two tables, one giving the operating conditions for every heat in detail, and the other showing the details of the packing materials used in every heat. Heats 1 and 2 are not included, because water leakage around head electrodes diverted the power

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path from the bars. These tables together with the description material making up the body of this report give a complete picture of operations carried out under Special Agreement G-3.

V.C.Hamister
H.A.Downey :GS
E.Micinski

VCHamister
Ed. Micinski
H. A Downey

[redacted]

Summary of Operating Conditions for Individual Heats

Heat No.	Pieces	Bar Compo-sition	Product Properties		Refining Treatment				KWH	Final Temperature	Finished Weight Lbs.
					Chlorine		Freon 12				
			DIH	Density	Lbs.	Hrs.	Lbs.	Hrs.			
3	25	CS	.70	1.66	9	2.0	47	4.0	1950	2430	532
4	13 12	TS (NP) CS (NP)	1.04 .99	1.52 1.53	7.5	2.1	48	4.0	1910	2440	523
5	25	CS	.96	1.62	8	2.3	48	4.0	1870	2420	536
6	25	TS	.97	1.62	11.5	2.0	48	4.0	1830	2430	587
7	25	T-30-S	.56	1.83	8.5	2.0	47	4.0	1750	2480	488
8	25	TS	.99	1.63	9.0	2.0	48	4.0	1880	2430	590
9	25	TS	.90	1.64	0	0	48	6.0	1830	2460	589
10	25 25	TS WS strip	.98 .97	1.63	9	2.0	48	8.0	2520	2480	589 45
11	25 25	TS WS strip	1.03 .98	1.64	9	2.0	48	4.0	1903	2480	586 50
12	25	TS	.80	1.64	4.5	2.0	24	4.0	1830	2450	586
13	13 12	T-12 (NP) T-25 (NP)	.84 .93	1.69 1.77	8	2.0	48	4.0	1750	-	463
14	13 12	T-12 (NP) T-25 (NP)	.83 .85	1.72 1.77	8	2.0	48	8.0	2225	2450	465
15	25	TS	.87	1.64	8	2.0	50	4.0	2040	2480	586

(Continued)

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Summary of Operating Conditions for Individual Heats

Heat No.	Pieces	Bar Compo-sition	Product Properties DIH	Product Properties Density	Refining Treatment Chlorine Lbs.	Refining Treatment Chlorine Hrs.	Refining Treatment Freon 12 Lbs.	Refining Treatment Freon 12 Hrs.	KWH	Final Temperature	Finished Weight Lbs.
16	25	WS	.62	1.62	8	2.0	50	4.0	1740	2450	522
17	25	TS	.63	1.64	26	5.0	13	1.0	2100	2450	587
18	25	TS	.96	1.65	9	2.0	50	4.0	1820	2450	585
19	25	TS	.89	1.64	45	3.0	25	2.0	1815	2480	587
20	13 12	TS (NP) TS	1.02 .98	1.54 1.64	9	2.0	50	4.0	1830	-	567
21	25 25	TS TS strips	.87 .94	1.64	8	2.0	50	4.0	1970	-	588
22	25	TA (NP)	1.03	1.45	6	1.6	50	4.0	1815	-	518
23	25	TS	.85	1.64	9	2.0	50	4.0	1820	-	589
24	25	TS	.77	1.64	8.5	2.0	60	4.0	1820	-	583
25	24	CS	.80	1.68	8.5	2.0	50	3.7	1830	-	532
26	25	TS	.95	1.64	7.5	2.0	50	3.8	1825	-	587
27	25	TS(1)	.81	1.68	7.5	2.0	50	3.8	1825	-	585
28	25	TS	.92	1.64	8.0	2.0	50	4.0	1825	-	585
29	25	TS	1.00	1.64	10.5	2.5	50	4.0	1990	2480	585

(1) These bars impregnated with sodium carbonate solution.

(Continued)

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Summary of Operating Conditions for Individual Heats

Heat No.	Pieces	Bar Composition	Product Properties DIH	Product Properties Density	Refining Treatment Chlorine Lbs.	Refining Treatment Chlorine Hrs.	Refining Treatment Freon 12 Lbs.	Refining Treatment Freon 12 Hrs.	KWH	Final Temperature	Finished Weight Lbs.
30	25	TS(1)	.87	1.67	10	2.5	50	3.9	1980	2440	582
31	25	TS(1)	.96	1.65	8.0	2.0	50	4.0	2040	2450	585
32	25	TS(1)	.84	1.68	10.2	2.0	50	3.8	1955	2440	586
33	10	WS	.97	1.61	7.2	2.0	50	3.8	1960	2490	524
	8	CS		1.68							
	8	TS		1.63							
34	25	TS-1			9.2	2.0	50	3.8	1980	2490	498
35	9	TS-B	.54	1.66	7.2	2.0	50	3.7	1930	2480	519
	10	TS-C	.58	1.66							
	10	TS-D	.86	1.63							
36	13	TSX-1	1.05	1.54	7.2	2.0	50	4.0	1980	2500	455
	14	TSX-2	1.04	1.54							

(1) These bars impregnated with sodium carbonate solution.

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Carbon and Graphite Packing Materials Used in Individual Heats

Heat No.	Resistor	Distribution Bed	Side Insulation	Cone	Cover	Number of Distribution
3	3 on 8 Graphite	3 on 8 Graphite	Coke and dust	3 on 8 Coke	Coke and Dust	25
4	" " " "	" " " "	" " "	" " " "	" " "	"
5	" " " "	" " " "	" " "	" " " "	" " "	"
6	" " " "	" " " "	" " "	" " " "	" " "	"
7	" " " "	" " " "	" " "	" " " "	" " "	"
8	" " " "	" " " "	" " "	" " " "	" " "	"
9	" " " "	" " " "	" " "	" " " "	" " "	"
10	" " " "	" " " "	" " "	" " " "	" " "	"
11	10 on 20 "	3 on 6 "	" " "	" " " "	" " "	9
12	8 on 10 "	" " " "	" " "	" " " "	" " "	"
13	" " " "	" " " "	" " "	" " " "	" " "	"
14	" " " "	" " " "	" " "	" " " "	" " "	"
15	" " " "	" " " "	" " "	" " " "	" " "	"
16	" " " "	" " " "	" " "	" " " "	" " "	"
17	" " " "	" " " "	" " "	" " " "	" " "	"
18	" " " "	" " " "	" " "	" " " "	" " "	"
19	" " " "	" " " "	" " "	" " " "	" " "	"
20	" " " "	" " " "	" " "	" " " "	" " "	"
21	" " " "	" " " "	" " "	" " " "	" " "	"
22	10 on 20 "	" " " "	" " "	3 on 8 Graphite	" " "	"
23	" " " "	" " " "	" " "	" " " "	" " "	"
24	" " " "	" " " "	" " "	" " " "	" " "	"
25	" " " "	" " " "	" " "	" " " "	" " "	"
26	" " " "	" " " "	" " "	" " " "	" " "	"
27	" " " "	" " " "	" " "	" " " "	" " "	"
28	" " " "	" " " "	" " "	" " " "	" " "	"
29	" " " "	" " " "	" " "	" " " "	" " "	"
30	" " " "	" " " "	" " "	" " " "	" " "	"
31	" " " "	" " " "	" " "	" " " "	" " "	"
32	" " " "	" " " "	" " "	" " " "	" " "	"
33	" " " "	" " " "	" " "	" " " "	" " "	"
34	" " " "	" " " "	" " "	" " " "	" " "	"
35	" " " "	" " " "	" " "	" " " "	" " "	"
36	" " " "	" " " "	" " "	" " " "	" " "	"

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