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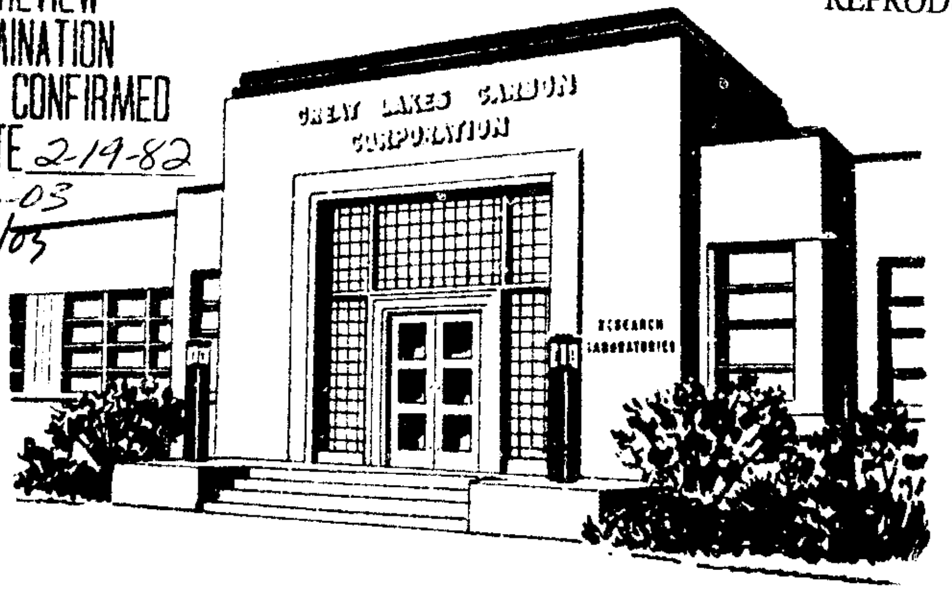

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RESEARCH AND DEVELOPMENT DIVISION
8210 AUSTIN AVENUE • MORTON GROVE, ILL.

RESEARCH REPORT

QUARTERLY REPORT

CONTRACT No. AT(11-1)-172

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GREAT LAKES CARBON CORPORATION
Research and Development Division
Morton Grove, Illinois

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SECURITY INFORMATION

Contract No. AT(11-1)-172

QUARTERLY REPORT - October, November, December, 1951

Submitted by: L. H. Juel

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Dr. B. W. Gamson
Great Lakes Carbon Corporation
8210 Austin Avenue
Morton Grove, Illinois

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SUMMARY

The experimental building belonging to A. E. C. and on the property of Great Lakes Carbon Corporation has been provided with an oil-burning space heater for supplying heat during the winter months.

The purification of gas-baked bar stock during graphitization using SF_6 as the purifying agent has been investigated. Under the most favorable conditions explored, the ash content was reduced to about 10 p.p.m. and the boron to less than 0.1 p.p.m. A residual sulfur content of about 650 p.p.m. was found in the purified bar, however. Purification of the bars under a variety of conditions failed to reduce this residual sulfur content appreciably. In addition, a serious operating problem was encountered in all purification runs employing this reagent. The SF_6 so vigorously attacked the graphite gas diffusion tube that it was almost completely reacted away at one point. A poor distribution of the SF_6 or its reaction products with graphite over the length of the bar to be purified resulted.

In contrast to the difficulty experienced with the SF_6, CF_4 exhibited no appreciable tendency to attack the graphite diffusion tube when used as a purifying agent during the graphitization of gas-baked bar stock. Under the best conditions investigated, the ash content of the purified bar was reduced to about 5 p.p.m. and the boron content to about 0.1 p.p.m. The absence of any element with a high neutron absorption cross-section in this purifying agent is a decided advantage in its use.

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When chlorine was used as a purifying agent for gas-baked bar stock during graphitization, the ash content could be reduced to less than 5 p.p.m. under a variety of conditions. However, there were indications that the boron was more resistant to removal by chlorine than by such a reagent as freon. It was possible, nevertheless, to reduce the boron content to about 0.1 p.p.m. A far more serious factor encountered was the high concentration (as high as 2000 p.p.m.) of chlorine in the purified bars. Since chlorine has a high neutron absorption cross-section, its presence in high concentration is just as objectionable as the ash itself. The elimination or removal of the residual chlorine in the graphite is under investigation.

Work on the ultimate purity of graphite attainable by repeated application of the F process is continuing. Certain incongruities in the results indicate that contamination of the purified bars by back diffusion of ash from the packing material will necessitate purification of the packing material before a higher degree of purity in the bars can be achieved. A serious problem of the existence of residual chlorine in bars purified by the standard F process has been encountered and is being investigated.

Further exploratory work has been conducted in the laboratory on the preparation of "chlorocoke". It was found that the chlorine content of raw cokes after calcination to and chlorination at various temperatures between 400° and 1000° C. passed through a maximum (4.0%) at 800° C. Further calcination of

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this product to 1000° C. caused a reduction in the chlorine content to about 0.8%. Since the quantity of chlorine required to produce "chlorocoke" at 800° C. is only a fraction of that required at low temperatures, the preparation of pilot plant quantities will be conducted at the higher temperature.

INTRODUCTION

The general objectives toward which the work under the present contract No. AT(11-1)-172 is directed may be summarized as follows:

1. To develop an improved purification process through the use of such purifying agents such as CF_4, SF_6, and Cl_2;
2. To determine the ultimate purity of graphite attainable through repeated applications of the F process;
3. To develop a simplified purification process through the use of self-purifying raw materials or by purification of the raw materials prior to processing into graphite bars.

During the last quarter, the building and equipment belonging to A.E.C. and on the property of Great Lakes Carbon Corporation at Morton Grove, Illinois were repaired, reconditioned, improved, and otherwise put in first-class operating condition. In addition, a number of purification runs were made according to the standard F process as well as with CF_4 and SF_6 as purifying agents replacing freon in the F process. A quantity of purified coke was prepared for use in the direct processing of graphite bars. Finally, a laboratory investigation was initiated to define a set of conditions by which to produce a

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"chlorocoke" containing a sufficient amount of chlorine to theoretically volatilize the ash constituents present therein.

During this quarter, the purification of graphite by CF_4, SF_6, and Cl_2 has been explored under a variety of conditions. The production of "chlorocoke' has been investigated further in the laboratory. The further purification of graphite by repeated application of the F process has been investigated, and the accumulation of a quantity of the ash from a purified graphite bar sufficient for elemental analysis has been accomplished. The results are reported herein.

DISCUSSION OF RESULTS

Building and Equipment

In order to provide heat in the experimental building during the winter months, an 80,000 BTU oil-burning space heater was installed and is operating effectively. A 250 gal. drum for oil storage was also purchased and installed.

While the conversion from graphite to metal clamps on the graphite tube of the F unit greatly increased the life of the tube, the problem of oxidation of the tube just inside the clamps has not been eliminated. During the period covered by this report, one tube change was required after completing 12 runs. Examination of the tube upon removal from the furnace disclosed rather severe attack by oxygen at the points indicated above. At one place the tube had been oxidized completely through

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leaving a large hole about 2 1/2" in diameter. In replacing this tube, precautions were taken to minimize this oxidation condition, but the principal obstacle that is confronted is in effecting a tight seal around the tube that will not be destroyed when the tube expands with increasing temperature. It is recognized that this whole problem could be solved by simply redesigning the furnace on the basis of a longer tube. However, the characteristics of the available transformer are inadequate for such a load, and the longer graphite tube would have to be made to order. As a result, the only rational solution to the problem consists in preventing the access of air to the tube at any point where oxidation will occur at a rapid rate.

Improved Purification Process

A comprehensive summary of all data pertinent to purification runs appears in tabular form in the Appendix.

Graphite Purification with SF_6

A purification run in which SF_6 was used to replace the CCl_2F_2 in a regular F run was reported previously. The purified graphite had less than 5 p.p.m. of ash and less than 0.1 p.p.m. of boron. The purification of graphite with SF_6 alone as the purifying agent has been investigated during this period. Four purification runs have been made to date,

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TABLE I

PURIFICATION OF GRAPHITE WITH SF_6

Run No.	Purifying Agent 1000 - 2000° C. Bracket Reagent Wt. (oz.)	Purifying Agent 2000 - 2500° C. Bracket Reagent Wt. (oz.)	Total Wt. of Reagent Used (oz.)	Boron p.p.m.	Ash p.p.m. N End	Ash p.p.m. S End	Sulfur p.p.m.	Remarks
15	None	37.5	37.5	<0.1	15	10	650	Ash - light yellow.
17	8	31	39.0	0.2	< 5	10	590	
25	36	None	36.0	0.18	128	940	910	
28	18	19	37.0	0.12	< 5	262	620	

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the results of which are summarized in Table I.

The first run was made using an amount of SF_6 molecularly equivalent to the freon used in a normal F run. The gas was introduced only in the 2000-2500° C. bracket. As indicated in Table I, the product was highly purified with respect to ash and boron but contained a high concentration of residual sulfur. During this run the gas diffusion tube was badly eroded and eaten away, presumably the result of chemical attack by the SF_6 or its decomposition products.

Subsequent runs were made in which the quantity of SF_6 used remained constant but the temperature interval in which the gas was introduced was varied. As the results show, no significant reduction in sulfur content of the purification product could be effected. The disparity in the ash contents of the two ends of the bar in run 25 and run 28 are undoubtedly attributable to unequal distribution of the SF_6 gas, which condition arises from the chemical attack of the SF_6 on the diffusion tube. This vigorous attack of the SF_6 on graphite poses a rather serious operating problem. However, time does not permit an extended investigation of methods of circumventing the difficulty.

The low neutron absorption cross-section of sulfur (0.49 barn) would indicate that its tolerance in the graphite would be much greater than that for such an element as

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chlorine (32 barns). Thus, since the atomic weight of sulfur and chlorine are roughly equivalent, a concentration of sulfur of about 600 p.p.m. is equivalent to only about 9 p.p.m. of chlorine. In this light, the high sulfur contents of the bars purified with SF_6 are not quite so objectionable as might be thought at first glance.

Consideration is being given to modifying the procedure used in the runs with SF_6 to the extent that the gas is not employed after the top temperature of 2500°C is reached. Present practice has been to keep the SF_6 flowing during the cooling period from 2500° to 2000° C. The data of Run 25 suggest, however, that little is to be gained through the use of this modification since the sulfur content of the bar from this run was high despite the fact that no SF_6 was used above the temperature of 2000° C. A slight possibility exists, of course, that some labile sulfur is bound in the ash which was high in this instance.

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TABLE II

PURIFICATION OF GRAPHITE WITH CF_4

Run No.	Purifying Agent 1000 - 2000° C. Bracket Reagent Wt. (oz.)	Purifying Agent 2000 - 2500° C. Bracket Reagent Wt. (oz.)	Total Wt. of Reagent Used (oz.)	Product Analysis Boron p.p.m.	Product Analysis Ash p.p.m. N End	Product Analysis Ash p.p.m. S End	Remarks
13	None	16	16	<0.1	48	86	
14	None	32	32	<0.1	10	20	Ash - pale yellow.
26	None	44	44	0.14	< 5	8	
16	22.5	40.5	63	0.15	< 5	550	Ash from S end dark in color.
19	28	23	51	0.17	20	24	Ash-pale yellow in color.

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Graphite Purification with CF_4

As was the case with SF_6, a purification run in which CF_4 was used to replace the CCl_2F_2 in a regular F run was made during the previous report period. A product analyzing less than 5 p.p.m. of ash and less than 0.1 p.p.m. of boron was obtained. The effectiveness of CF_4 alone as a purifying agent for graphite has been investigated during this period. Five purification runs using various quantities of CF_4 introduced over different temperature intervals were made and the results are summarized in Table II.

In Runs 13, 14, and 26 the amount of CF_4 used was respectively about one-half,equivalent to, and 50% in excess of the molecular proportion of CCl_2F_2 used in a normal F run. The gas was introduced over the same temperature interval (2000°-2500°) as that employed with the CCl_2F_2. As indicated by the data of Table II, a progressive reduction in total ash was obtained with increasing amounts of CF_4 used. The results of Run 16 are inconclusive since the ash content of one end of the bar was inordinately high. This condition probably resulted from an unequal distribution of the CF_4 gas during the run.

The results from Run 19 suggest that the effectiveness of the CF_4 as a purifying agent at temperatures below 2000° C. is considerably less than at temperatures in excess of 2000° C. This fact might reflect the thermal stability of the CF_4. In this connection, it might be stated that the gas diffusion tube exhibited little or no evidence for corrosion or chemical

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attack by the CF_4 in contradistinction to the condition experienced with SF_6.

While it appears that the CF_4 is somewhat less efficient as a purifying agent compared to freon, it is worthwhile noting that the neutron absorption cross-section of fluorine is very low (0.01 barn) and any fluorine remaining after purification would be of little consequence. This is in sharp contrast to chlorine with its previously mentioned cross-section value of 32 barns.

The purified bars from runs 14 and 26 will be sent to Hanford for functional testing in the pile.

Graphite Purification with Cl_2

Purification of reactor graphite with chlorine alone has not been investigated,although the substitution of Cl_2 for freon or CCl_4 in the F process has been successfully applied in experiments conducted several years ago and reported summarily by J. M. West.[1] These experiments clearly indicated that purification of graphite with respect to ash could be effected but that residual chlorine was responsible for low dih values obtained when the CCl_4 was used during the cooling down period of the run. Somewhat inferior quality graphite was obtained when the Cl_2 was used in place of the freon. It must be pointed out, however, that these purification runs were made on graphite and not on baked stock.

1. J. M. West, Purification of Graphite, Document HW-12780 (1949).

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TABLE III

PURIFICATION OF GRAPHITE WITH Cl_2

Run No.	Purifying Agent 1000-2000° C. Bracket Reagent Wt.(oz.)	Purifying Agent 2000-2500° C. Bracket Reagent Wt.(oz.)	Total Wt. of Reagent Used	Boron p.p.m.	Ash p.p.m. N End	Ash p.p.m. S End	Cl p.p.m.	Remarks
18	20.6	21.6	42.2	0.15	<5	<5	1900	
20	9.7	10.8	20.5	0.15	<5	<5	1100	
22	20.2	None	20.2	0.13	<5	<5	890	
23	None	20.3	20.3	0.12	<5	<5	1100	
24	None	19.5	19.5	<0.1	32	92	300	Cl_2 off at 2500° C.

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This phase of the present investigation has been concerned with the production of purified graphite through the use of Cl_2 alone. Five purification runs on gas-baked bar stock have been made. In these runs the quantity of Cl_2 used and the temperature interval over which the Cl_2 was added were varied. The results are given in Table III.

Several observations can be made from the data. Run 18 in which the amount of Cl_2 used in the temperature range 2000-2500° C. was molecularly equivalent to the quantity of freon used in a normal F run produced a product of very low ash but with a high residual chlorine content. By reducing the quantity of Cl_2 to one-half that used in Run 18, the chlorine content of the product was reduced by almost the same amount without increasing the ash content. By introducing the same amount of Cl_2 in the temperature interval of 1000° to 2000° C. as was used over the entire temperature range 1000° to 2500° C., the chlorine content of the product could be reduced somewhat further. However, when this same amount of Cl_2 was introduced over the temperature range of 2000° to 2500° C., the residual chlorine in the product increased again. Finally, when the flow of Cl_2 was terminated at 2500° C. instead of continuing until the temperature had dropped to 2000° C. a significant reduction in the residual chlorine was effected. In this run the ash content was not reduced to the desired level, however, and so a further experiment is planned in which the amount of Cl_2 used is increased and is added over

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a longer period of time but over the same temperature range.

The persistently high boron contents of the bars purified with Cl_2 is suggestive of the lesser effectiveness of this reagent in removing boron.

The residual chlorine contents reported for the bars purified with Cl_2 are prohibitively high and would preclude the use of such bars in atomic piles. As little as 63 p.p.m. of Cl_2 will account for a loss of 1.00 in dih value.* Consequently, it becomes a matter of necessity to determine, if possible, the manner in which the chlorine is held in the graphite bar and what means may be devised for preventing its retention in the bar or for its removal after purification of the bar. It is this aspect of the problem that will be given first consideration in the immediate future.

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* Loc. cit.

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TABLE IV

PURITY OF GRAPHITE FROM F PROCESS

Run No.	Bar No.	Purifying agents 1000°-2000°C. Bracket Reagent	 Wt.(oz.)	 2000°-2500° C. Bracket Reagent	 Wt.(oz.)	Product Analysis Boron p.p.m.	 Ash p.p.m. N end	 S end	Remarks
27	1	CCl_4	20.3	CCl_2F_2	43.0	0.12	16	56	Original ash uncut end -- 3800 p.p.m.
29	1	"	20.0	"	37.0	<0.1	20	36	Second run on Bar 1.
30	2	"	19.4	"	36.0	<0.1	20	28	Prepurification analysis of bars B(ppm) Ash(ppm) Uncut end 1.24 3430 Cut end 0.57 2430
31	2	"	19.4	"	42.0	-	<5	<5	Second run on Bar 2.

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Ultimate Purity of Graphite from F Process

In determining the ultimate purity of graphite attainable by repeated application of the F process, the problem of following the progress of purification by some analytical technique arose. In discussing the problem with personnel at the Argonne Laboratories, the only immediate solution that could be found was to ash larger quantities of the graphite. This was considered impractical since the quantities required to provide enough ash for analysis would be so great that a considerable part of the specimen bar would be required for each analysis. Several successive runs and analyses on a bar 4" x 4" x 26" in size would leave an insufficient amount of the bar for testing in the pile. Therefore, it has been arbitrarily decided to make two additional F runs on a bar once it has been purified to the extent of <0.1 p.p.m. of boron and <5 p.p.m. of ash. This program has been initiated and the results to date are summarized in Table IV.

After the first purification run, neither bar 1 nor bar 2 was purified to the degree usually obtainable. In the second run on bar 2 the ash was reduced to the desired level of <5 p.p.m. However, the second purification run on bar 1 was without significant effect in reducing the ash content. The explanation for this behavior is not immediately apparent although it is possible that back diffusion of ash constituents vaporized in the packing media adjacent to the bar is responsible. In order to minimize any such contamination, the packing media used in the purification of the bars previously purified to the desired level of <0.1 p.p.m. boron and <5 p.p.m. ash will be purified itself.

The contamination of standard sized bars in processing and handling is revealed by the ash contents of the cut and uncut ends of a bar prior to purification. The specimen bars used in this work are obtained by cutting in half, standard sized bars. The ash of the uncut end is about 40% higher than that of the cut end. Similarly, the boron content of the former is considerably higher than that of the latter.

As an outgrowth of discussions with Argonne personnel regarding the analyses of impurities remaining in purified graphite, it was decided that sufficient ash would be accumulated by repeated ashings to make spectrographic and radioactivity analyses. These ashings have been completed and the accumulated ash forwarded to Argonne Laboratories for analyses. The results of these analyses will be reported as soon as they become available. As stated in the previous report, it is important to determine whether the constituents identified in the ash of purified graphite are present in amounts sufficient to account for the experimental values obtained for the neutron absorption cross-section of the graphite.

In addition, two special samples of graphite were purified and forwarded to Dr. Muehlhause of the Argonne Laboratories for special investigations on cross-section measurements by the pile oscillator technique.

The apparent tenacity with which the graphite held chlorine in the bars purified with elemental chlorine alone inspired the

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chlorine analysis of a bar purified by the F process itself. A most unexpected and disconcerting result was obtained -- the bar was found to contain about 490 p.p.m. of chlorine. Such a chlorine content would give a dih value well below 0.0 according to estimates based upon available data. As in the case of the graphite purified by chlorine, the manner in which the chlorine is held is not known but will be ascertained, if possible. Since standard F runs conducted under the previous contract produced satisfactory products with no apparent chlorine content (this was not analyzed for) the origin of the chlorine in this instance is difficult to determine. One possibility exists, however. Subsequent to the F run in which the bar in question was purified, two runs were made in the F unit wherein raw coke was chlorinated. During these runs the evolution of HCl and unreacted Cl_2 was so great that the ventilating system could not adequately handle the off gases. As a result, some gases escaped into the room and possibly penetrated the wrappings around the bars stored nearby. This possibility is currently being checked.

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Simplified Purification Process

Purifying Agents Liberated in Situ

The purification process having a great deal of potential benefit is the one in which the purifying agent is liberated in situ during the processing of the materials to graphite. Such a process possesses the inherent advantage of a simplified graphitization step and does not suffer the disadvantage of possible contamination during processing. The approach initiated during the previous period was to attempt to prepare "chlorocoke" by chlorination of raw coke. A number of laboratory chlorination experiments were run in the temperature range, room temperature - 300° C. The greatest chlorine pick-up was found at 300° C. but when the chlorination products were heated rapidly to a temperature about 600° C., the chlorine content dropped from an initial value of about 14% to one of about 0.1%. Strangely enough, the chlorine content after rapid heating of this same product to 900° C. was about 0.3% suggesting that the mechanism of chlorine release probably altered somewhat with a more rapid rate of temperature rise.

On the basis of the laboratory results, two chlorination runs were made in the pilot plant on 15% volatile raw coke at around 300° C. The pertinent data are summarized in Table V.

TABLE V

CHLORINATION OF RAW COKE

Mesh Size: 6/20

Run No.	Charge Wt. (lbs.)	Cl_2 Used (lbs.)	Time (hrs.)	Temp. ° C.	Wt. Gain %
1	25	6.5	2.5	300	4.0
2	28	8.25	3.2	300	-

Difficulty was experienced in achieving good temperature control. Also it was found that the rate of evolution of HCl was so great that the exit gases could not be entirely accommodated by the ventilating system. As a result, a considerable amount of HCl gas and unreacted Cl_2 escaped into the room. It was considered inadvisable to make any further runs under these conditions.

Calcination of the product from chlorination Run 2 to a final temperature of 1175° C. was conducted to determine the amount of residual chlorine and ash in the product. Only 0.047% chlorine remained under these conditions. The ash content was 0.062%. It was concluded that the chlorine content was insufficient to completely volatilize the remaining ash during graphitization.

In view of the operating difficulties and excessive chlorine requirements involved in the chlorination of high volatile raw coke it was decided to extend the laboratory investigation of the chlorination step to higher temperatures. Several runs were made

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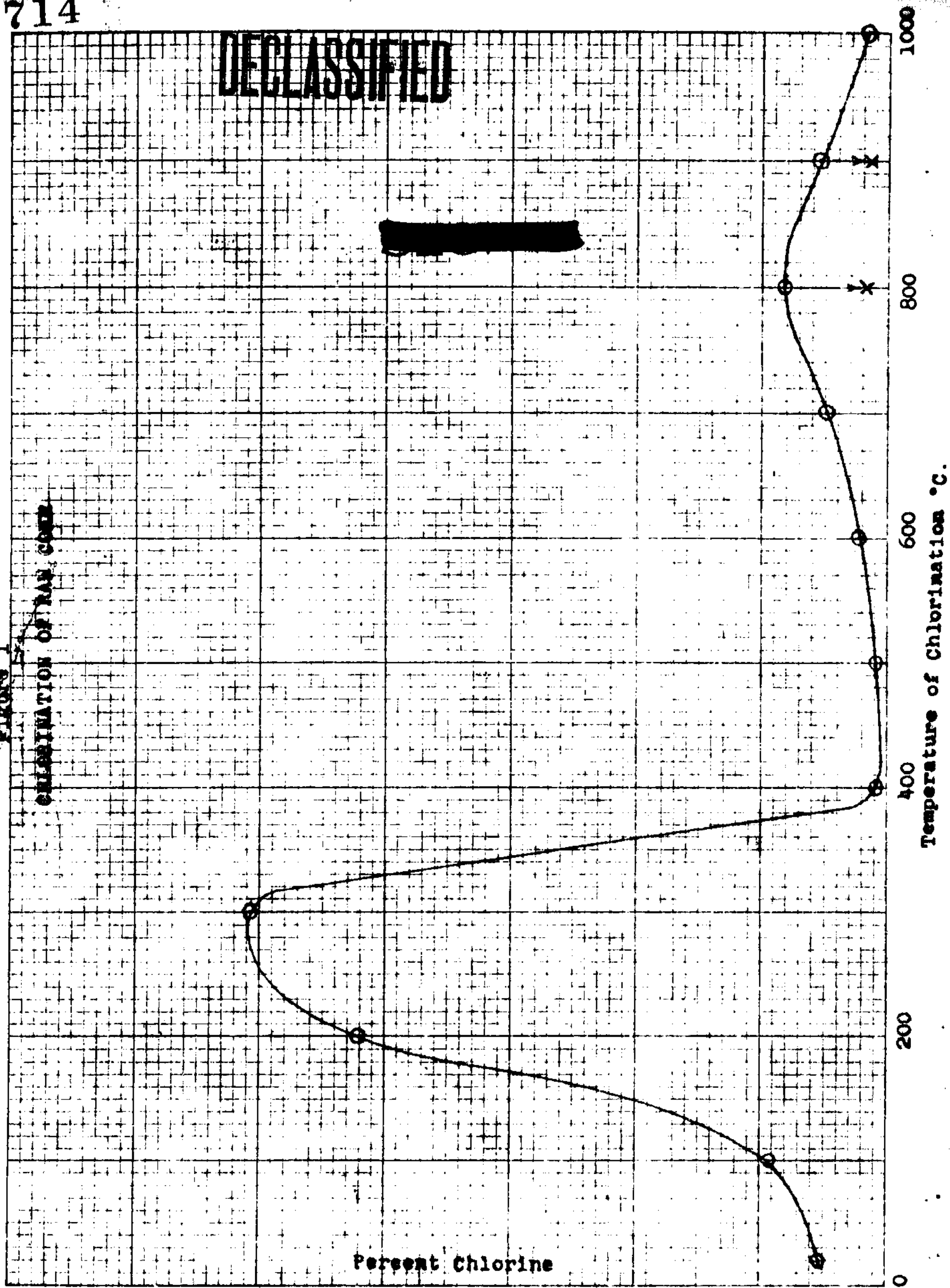

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in the temperature interval 400° to 1000° C. The pertinent data are given in Table VI and plotted in Figure 1.

TABLE VI

LABORATORY CHLORINATION OF RAW COKE

Temperature of Chlorination ° C.	Cl_2 at Chlorination Temperature %	Cl_2 at 1000° C. %
400	0.42	-
500	0.30	-
600	1.12	-
700	2.45	-
800	4.13	-
800	--	0.82
900	2.75	-
900	--	0.68
1000	0.77	-

Included in the plot in Figure 1 are the data for chlorination of the same coke below 400° C. It is interesting to note the two maxima that appear in curve. Apparently, two different types of chlorination process are taking place at the lower and higher temperature ranges. In the lower range, the coke contains a large amount of volatile and the chlorine may be held in aliphatic side chains whereas at the high temperature range, the chlorine atoms may be attached to the peripheral carbon atoms of highly condensed aromatic structures. The sudden drop in chlorine content in the temperature range 300 - 400° C. is possibly indicative of an instability in the carbon structure in which the spatial arrangement of atoms is such that adjacent carbon atoms each carry a chlorine atom or one carries a chlorine atom and the other a hydrogen atom. As the coke structure becomes more highly condensed with increasing temperature, the activation energy required to bring about substitution of chlorine atoms for peripheral hydrogen atoms increases and a high temperature is necessary to supply this increased energy requirement.

The 1000° C. calcination of the products chlorinated at 800° and 900° C. respectively reduced the chlorine contents of the cokes to about the same value (about 0.8%) as that obtained by direct chlorination at 1000° C. In view of this result, it is considered preferable to run large scale chlorinations at the lower temperature (800° C.) merely from a practical standpoint. The amount of chlorine required to produce a "chlorocoke" with a chlorine content at 1000° C. of about 0.8% will be greatly reduced over that previously employed in conducting the chlorinations at 300° C. The preparation of a sufficient amount of "chlorocoke" of this type for producing two specimen bars for functional testing is currently underway.

Purification of Raw Materials

Work on this phase of the investigation has been deferred until the extrusion and processing of these materials can be integrated with similar work scheduled for the "chlorocoke".

Experimental

Laboratory Chlorination of Raw Coke

The procedure used for all chlorination runs was as follows:

Fifty grams of raw coke (6/20 mesh, 15.1% volatile Texas Lockport coke) are placed in a graphite bottle which is in turn placed in a tube furnace. A gas delivery tube extending outside the furnace is inserted into one end of the bottle. The temperature of the furnace is raised to the desired level at a rate of about 10° C./min. To avoid any excessive deposition of volatile matter in the gas tube, a slow stream of N_2 is introduced during this period. When the desired temperature is reached, the sample is held at that temperature for 15 minutes to allow the material to come to equilibrium with the surroundings. Chlorine is then introduced through the gas tube at a rate of about 10 l/hr. for one hour. The furnace is then swept with N_2 to displace any excess chlorine not combined with the coke. The sample is removed and placed in a desiccator to cool. After cooling, a portion of the sample is submitted for chlorine analysis and the remainder calcined to 1000° C.

In those instances where the chlorinations were carried out at temperatures in excess of 700° C., the raw coke was first calcined to about 600° C. at a rate of 10 to 20° C./min. and then treated according to the preceding procedure.

L. H. Juel

L. H. Juel

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APPENDIX

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Run No.	HEATING SCHEDULE Time (hrs.) Room Temp. to 1000°C.	1000 - 2000°C.	2000 - 2500° C.	[redacted] PURIFYING AGENTS 1000-2000°C Bracket Reagent	Wgt. (oz.)	2000-2500°C Bracket Reagent	Wgt. (oz.)	PRODUCT ANALYSIS Boron p.p.m.	Ash p.p.m.	Sulfur %	Cl %	Remarks
12*	2.75	6.0	6.25	CCl_4	20.0	CCl_2F_2	43.0	<.1	<5	-	-	24# 12 oz. calcined coke flour 851 MG 135.
13	2.75	4.0	3.0	--	--	CF_4	16.0	<.1	67			
14	2.25	4.25	3.5	--	--	CF_4	32.0	<.1	15			Ash light yellow in color.
15	2.5	4.0	4.0	--	--	SF_6	37.5	<.1	13	0.065		Ash light yellow in color. Gas tube oxidized in two pieces at point approximately 10" from bar.
16	2.33	4.5	3.5	CF_4	→	CF_4	Total 63.0	.15	North- <5 South- 550			Ash from south end dark in color.
17	2.5	4.25	4.0	SF_6	→	SF_6	Total 39.0	.20	North- <5 South 10	0.008		Ash light yellow in color. Gas tube oxidized in 2 pieces at point approximately 10" from bar.
18	2.25	5.0	3.75	Cl_2	→	Cl_2	Total 42.2	.15	<5		0.19	
19	2.5	4.0	3.5	CF_4	→	CF_4	Total 51.0	.17	22			Ash pale yellow in color.
20	2.25	4.25	3.5	Cl_2	→	Cl_2	Total 20.5	.15	<5		0.11	
21	2.5	4.75	3.5	CCl_4	20.6	CCl_2F_2	37.0	<.1	North- 60 South- 16			Rod cut off bar from Run 5 to be sent to Argonne included in this run.
22	2.5	4.75	4.0	Cl_2	20.2	--	--	.13	<5		0.089	
23	2.33	4.5	4.5	--	--	Cl_2	20.3	.12	<5		0.11	
24	2.5	4.5	4.5	--	--	Cl_2	19.5	<.1	North- 32 South 92		0.03	Cl_2 off at 2500° C.

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	HEATING SCHEDULE Time (hrs.)			PURIFYING AGENTS				PRODUCT ANALYSIS				
				1000-2000° C. Bracket		2000-2500°C. Bracket						
Run No.	Room Temp. to 1000°C.	1000 - 2000°C.	2000 - 2500° C.	Reagent	Wgt. (oz.)	Reagent	Wgt. (oz.)	Boron p.p.m.	Ash p.p.m.	Sulfur %	Cl %	REMARKS
25	2.33	4.5	3.67	SF$_6$	36.0	--	--	.18	North- 128 South- 940	0.091		SF$_6$ off at 2120° C. Gas tube oxidized in two pieces at point approximately 10" from bar.
26	2.25	4.5	4.0	--	--	CF$_4$	44.0	.14	North- <5 South- 8			Duplication of Run 14.
27	2.25	4.75	3.75	CCl$_4$	20.3	CCl$_2$F$_2$	43.0	.12	North- 16 South- 56			Prepurification ash analysis of bar at uncut end -- 3824 ppm. This bar to be rerun two times after the ash content becomes <5 ppm.
28	2.25	4.0	3.67	SF$_6$	⟶	SF$_6$	37.0	.11	North- <5 South- 262	0.062		Duplication of Run 17. Prepurification ash analysis of bar at cut end-1900 ppm. Gas tube oxidized in 2 pieces at point app.10" from bar.
29	2.25	5.0	3.75	CCl$_4$	20.0	CCl$_2$F$_2$	37.0	<.1	North- 20 South- 36			Rerun of Run 27 with position of bar reversed in tube. Gas tube modified with 50% reduction in number of holes.
30	2.5	4.5	3.33	CCl$_4$	19.4	CCl$_2$F$_2$	36.0	<.1	North- 20 South- 28			Prepurification ash analysis of bar at uncut end-3444 ppm; at cut end-2428 ppm. This bar to be rerun two times after the ash content becomes <5 ppm.
31	2.5	5.0	4.33	CCl$_4$	19.4	CCl$_2$F$_2$	42.0		<5			Rerun of Run 30. This bar to be processed two more times. This run also contained 2 machined rods of bar from Run 5 which are to be sent to Argonne.

* Final F Run for purification of calcined coke particles and flour before actual formation of the bar. All other runs contain baked, impregnated and rebaked bars from Morganton.

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