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AEC RESEARCH AND DEVELOPMENT REPORT

IODINE RETENTION STUDIES

PROGRESS REPORT: JANUARY - JUNE 1969

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Savannah River Laboratory

Aiken, South Carolina

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Reactor Technology
(TID-4500, UC-80)

IODINE RETENTION STUDIES

PROGRESS REPORT: JANUARY - JUNE 1969

by

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Approved by

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November 1969

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ABSTRACT

The removal of iodine from activated carbon by flowing air was measured to evaluate various carbons by determining the effects of impurities, service, temperature, loading, and partial regeneration. Carbon service in the confinement system and increased carbon temperature significantly increase desorption of iodine from carbon. Iodine adsorption efficiency decreases slightly with service and with increased loading. Partial regeneration increases iodine adsorption efficiency, but it does not decrease desorption significantly.

A treatment was developed to make artificially aged carbon for evaluation of service-related properties.

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INTRODUCTION

The activity confinement system for the Savannah River production reactors is designed to collect radioactivity that might be released in the unlikely event of a major reactor accident. At the request of the AEC Division of Operational Safety, a continuing program is in progress at the Savannah River Laboratory to evaluate the performance of carbon, used in the confinement system, for removal of iodine under adverse operating conditions and to develop techniques to enhance its reliability and efficiency. Previous reports summarize the progress from January 1965 to December 1968.[1,2]

Activated carbon is a superior adsorber for elemental iodine that might be released from a nuclear reactor. To be effective in the activity confinement system the activated carbon must have a very high retention of iodine under a variety of adverse operating conditions and for a reasonably long service life. The carbon may be subjected to high temperature and radiation from decay of adsorbed iodine or from decay of radioactive debris.

This report summarizes the iodine retention studies at the Savannah River Laboratory from January to June 1969.

SUMMARY

The desorption of iodine from several types of activated carbon at temperatures up to 200°C, for service times up to 4 years, with and without partial regeneration, and for several iodine loadings showed that:

- One of the key impurities in the confinement system that degrades the quality of carbon is nitrogen dioxide.
- The two key factors that affect desorption of iodine are carbon service in the confinement system and carbon temperature.
- The effective surface area of carbon in the confinement system decreases linearly with service.
- The diffusion coefficients for both unused carbon and carbon with 30 months service obey Fick's first law for temperature and iodine loading.
- Partial regeneration with heated air is the best technique that has been developed to restore the iodine adsorption efficiency to acceptable levels, but this technique is inadequate for reducing desorption.

The iodine adsorption efficiency decreases slowly with service but is within specification (99.85% efficiency) following partial regeneration after 55 months service. The iodine adsorption efficiency also decreases linearly with increased loading.

A treatment to artificially age carbon was developed. Two hours exposure to 30 ppm nitrogen dioxide in air approximately equaled 5½ months service in desorption tests.

A new ^{60}Co irradiation facility with an intensity of about 10^8 rad/hr will be completed in December, 1969. This facility will be used for methyl iodide radiolysis.

DISCUSSION

HIGH TEMPERATURE DESORPTION OF IODINE FROM CARBON

Proposed Mechanism

The following mechanism for desorption of iodine from activated carbon is proposed. During continuous on-line service, activated carbon in the Savannah River confinement system adsorbs or reacts with impurities from the air in the ventilation system, consuming active sites on the micropore structure. Hence, the number of sites available for iodine is reduced.

Effects of Impurities

The impurities measured in the ventilation air were:

$\sim$0.03 ppm NO_2
$\sim$0.02 ppm O_3
$\sim$0.04 ppm SO_2

Organic impurities such as methane and ethylene were also found.

Nitrogen dioxide is produced as ventilation air flows through the intense gamma field around the reactor.

The concentration of ozone in the ventilation system was equal to that in the laboratory, or approximately the normal ozone background of the atmosphere.

Sulfur dioxide is formed from the combustion of coal in a nearby power plant. Sulfur dioxide was detected only when exhaust from the power plant was carried to the reactor building by the wind.

Nitrogen dioxide reacts to destroy active sites in the carbon. In addition, one of the reaction products, nitric oxide, is adsorbed on the carbon.[3] Nitrogen dioxide and sulfur dioxide react with impurities in the carbon such as sodium and potassium. Such reactions prevent the formation of stable, nonvolatile compounds of iodine such as sodium iodide (boiling point 1300°C) and potassium iodide (boiling point 1420°C). Adsorbed organic compounds migrate into the micropores and hamper the subsequent adsorption of iodine. After several years service, the carbon may adsorb some of the iodine as molecular iodine, which would be desorbed rapidly because of its low boiling point (184°C). Hence, the iodine adsorption efficiency of activated carbon decreases with service in the confinement system and iodine desorption increases.

Determination of Impurities

Carbons with up to 24 months service were screened by thermogravimetric analysis to determine qualitatively the temperatures at which various adsorbed species were desorbed. A sample of carbon was placed in a recording balance, and the system was evacuated. The temperature of the sample was increased at a uniform rate, usually 1°C/min, and the desorbed gases were analyzed by a mass spectrometer. Nitric oxide, the reaction product of nitrogen dioxide and carbon, was desorbed between 45 and 300°C. Hence, partial regeneration (discussed on p 16) at 65°C removes part of the nitric oxide in the carbon. Sulfur dioxide was desorbed between 200 and 300°C. Because such high temperatures were required to remove SO_2 it is unlikely that SO_2 is adsorbed as a gas, but that it reacts to form a compound in the carbon. Organic matter was desorbed between 140 and 300°C.

The total amount of gases adsorbed on carbon with up to 4 years service was determined by mass spectrometric analysis. A sample of carbon was placed in an extraction vessel and cooled with liquid nitrogen. Residual air was removed, the extraction vessel was sealed, and the apparatus was warmed to room temperature. Then the sample was evacuated and the resulting gases were analyzed by mass spectrometer. The sample was then heated to a higher temperature and desorbed gases were collected and analyzed. The mass spectrometer was designed to analyze gases up to mass 44. Heavier gases were measured and reported as unidentifiable gases. Water was removed with magnesium perchlorate and weighed.

The concentration of the gases found in unused Type 416* carbon is plotted as a function of the temperature at which they were removed from carbon by a vacuum (Figure 1). Most of these gases are impurities normally found in air. Desorption of only two gases, CO_2 and CO, was significantly affected by increasing the temperature to 300°C. The amount of nitric oxide desorbed at 300°C was very small (<0.01 mg/g C).

The analysis of a sample of carbon with 30 months service in the confinement system is shown in Figure 2. The most significant difference between Figures 1 and 2 is the large increase in the amount of nitric oxide released (2.7 mg NO/g C). As mentioned

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* Product of Barnebey Cheney Co., Columbus, Ohio.

previously, nitric oxide is the reaction product of nitrogen dioxide (found in the confinement system) and carbon. The large increase in the nitric oxide is evidence that it is one cause of increased desorption of iodine with carbon service. There is also a marked increase in the concentration of ethylene with service. The increase in the concentration of hydrogen is probably caused by the thermal decomposition of organic matter adsorbed on the carbon.

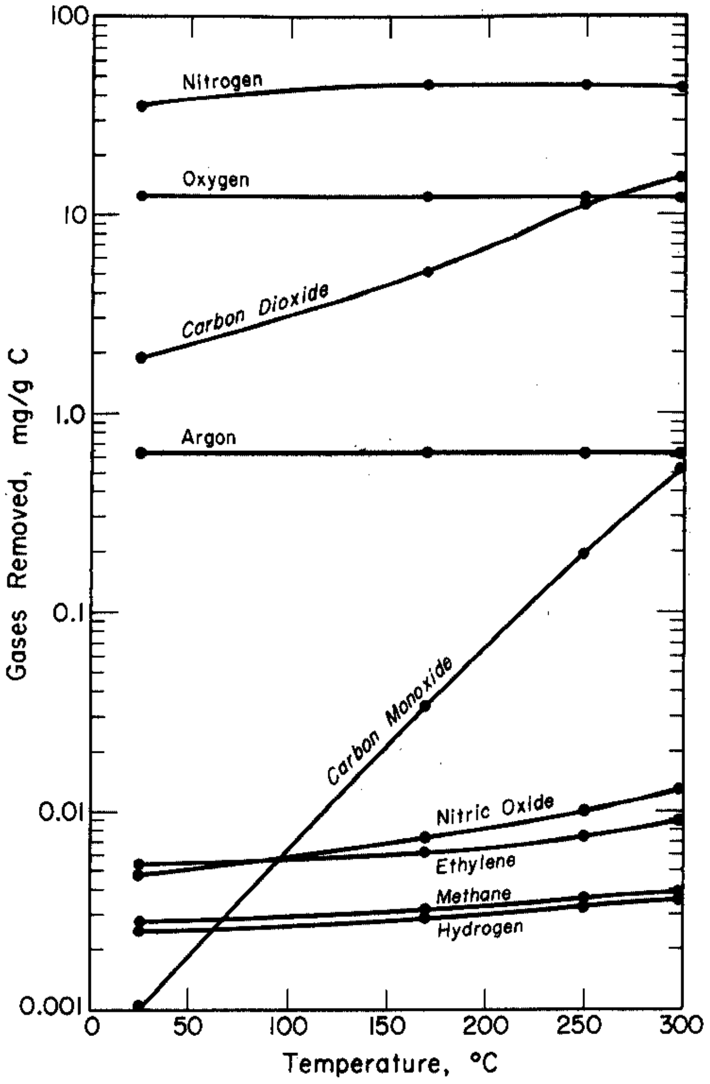

FIG. 1 REMOVAL OF GASES FROM UNUSED TYPE 416 CARBON

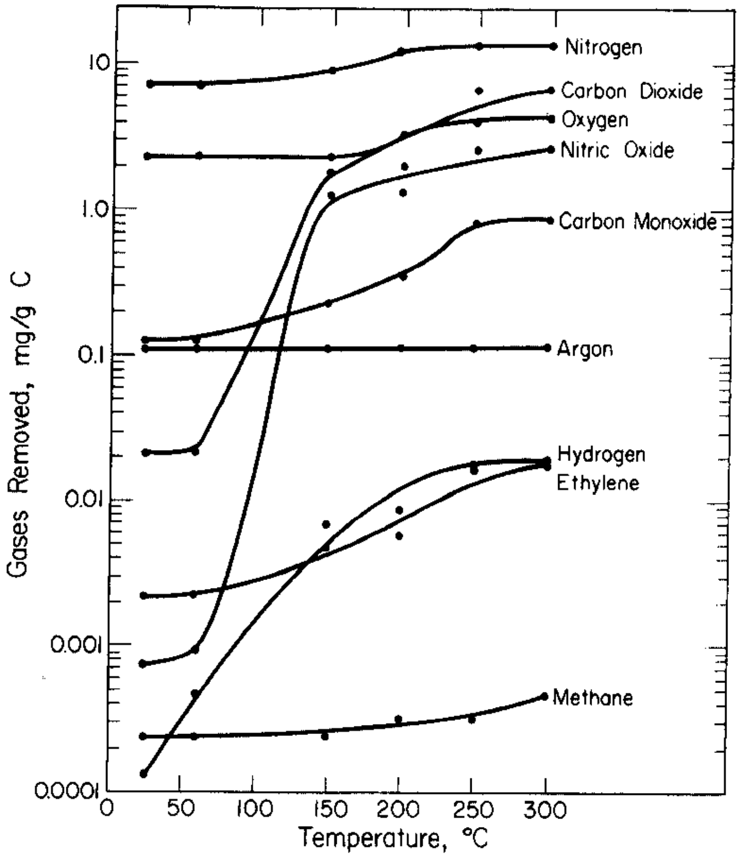

FIG. 2 REMOVAL OF GASES FROM CARBON WITH 30 MONTHS SERVICE

Increase with Service

Service in the confinement system is one of the most significant variables that affects the desorption of iodine from carbon.[4,5] The desorption of iodine increases rapidly with service in the confinement system, as shown in Figure 3, while the iodine adsorption efficiency decreases very slowly.

Desorption is measured with a test bed loaded by adsorbing elemental iodine tagged with ^{131}I from ambient air or from a steam-air mixture at 65°C. Then air, at the desired temperature and purified of dust and vapors, is passed through the test bed and any desorbed iodine is adsorbed on backup beds. The ratio of ^{131}I on the backup beds to that loaded on the test bed is the fraction of iodine desorbed. In this work, desorption temperatures up to 200°C were used to determine the maximum allowable temperature of the exhaust air in the ventilation system. In subsequent work, the standard desorption temperature will be 100°C.

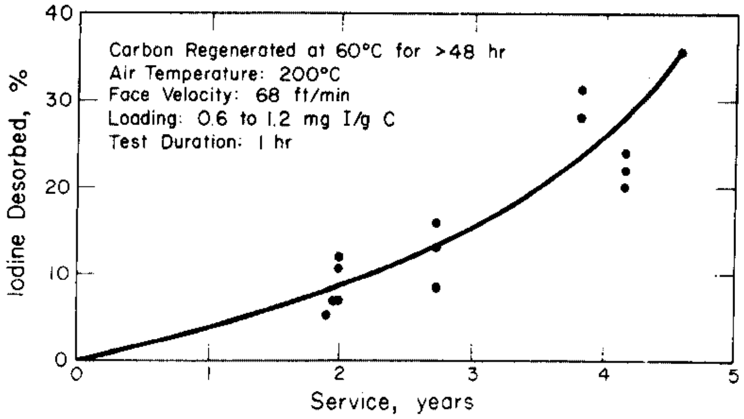

FIG. 3 EFFECT OF SERVICE ON DESORPTION

Before these measurements, each carbon was partially regenerated by passing air at 60°C through the carbon at a velocity of 7.5 ft/min for at least 48 hours to remove impurities that were not strongly adsorbed.

Carbon in the downstream and upstream halves of the bed was evaluated and the iodine desorption of each was compared. As shown in Table I, in upstream, composite, and downstream samples of carbon with 33 and 55 months service, the iodine desorption was outside specification (<0.15%). The iodine desorption decreased from upstream to downstream by over a factor of 2.

TABLE I

Desorption of Iodine as a Function of Carbon Position

Sample	Service, months	Loading, mg I/g C	Desorption,[a] %
Upstream	33	0.56	41.8
Composite	33	0.99	26.4
Downstream	33	0.63	18.4
Upstream	55	0.81	66.8
Composite	55	0.98	51.9
Downstream	55	0.57	29.4

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[a] In air at 200°C for 3 hours, face velocity 68 ft/min.

As carbon adsorbs impurities from the ventilation air during continuous on-line service in the confinement system, its surface area will decrease. The surface area was measured by nitrogen adsorption to determine the correlation between desorption and surface area. As shown in Figure 4, the effective surface area decreased linearly with service up to 56 months. Samples from the upstream and downstream halves of the beds showed that the surface area of the upstream samples was smaller than that of the downstream samples. This difference was caused by the greater concentration of impurities in the upstream half of the bed. Samples of the carbon were partially regenerated (method discussed on p 16) at 60, 100, and 150°C to determine the increase in surface area with increased temperature of regeneration. Regeneration at 60 and 100°C restored a significant fraction of the surface area. Regeneration at 150°C produced no additional improvement.

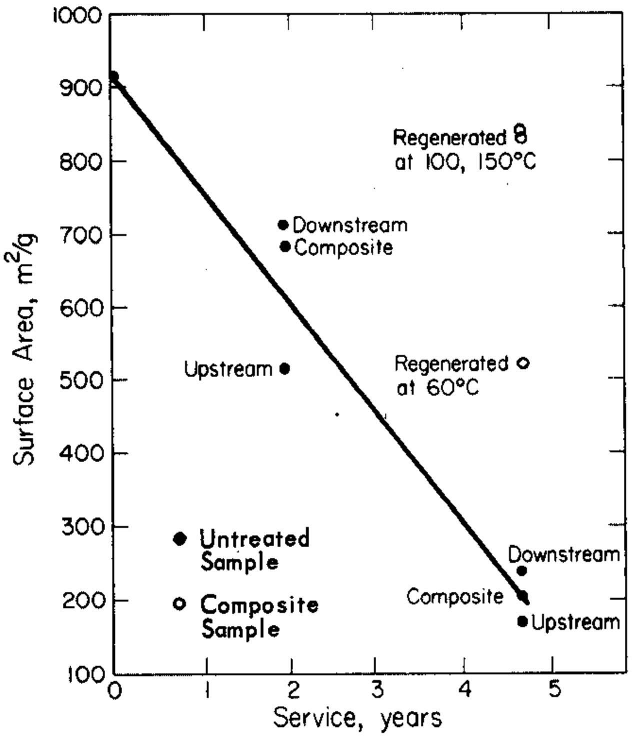

FIG. 4 EFFECT OF SERVICE ON SURFACE AREA

As shown in Figure 5, some correlation exists between desorption and surface area. However, the correlation is not sufficiently precise to predict the remaining useful life of the carbon.

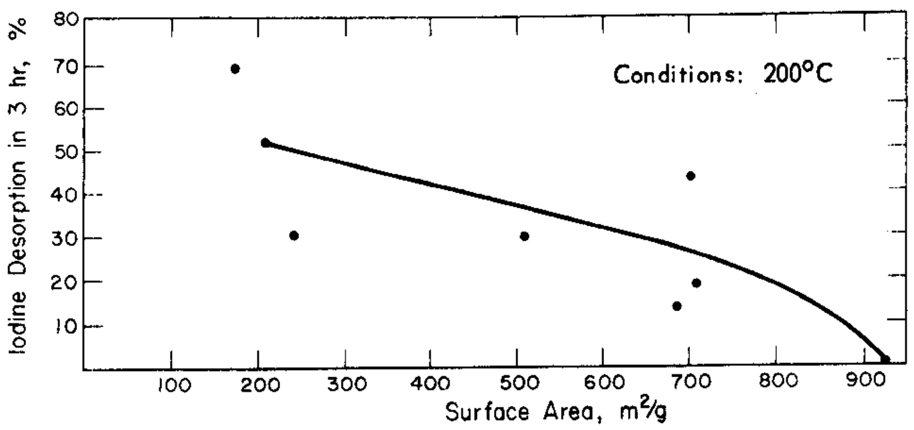

FIG. 5 CORRELATION OF DESORPTION AND SURFACE AREA

Increase with Increased Temperature

The previous report[2] showed that desorption of iodine from activated carbon increases exponentially with increasing temperature. The combined effect of temperature and service on desorption was studied. The desorption of iodine from unused carbon remained within specification (<0.15%) up to 200°C as shown in Figure 6 (shaded area is within specification). The desorption of iodine from regenerated carbon with 30 months service remained within specification only below 120°C. Regenerated carbon with 56 months service must remain below 92°C.

A supplementary cooling system for the exhaust air has been designed for the reactor building to ensure that carbon in the confinement system will not become overheated, but it has not yet been installed.

Calculation of Diffusion Coefficients

The diffusion coefficient measures the rate of iodine desorption. Calculations showed that the diffusion of iodine followed the laws of mass transfer.

The rate of mass transfer[6] for ordinary molecular diffusion in the y direction at constant molal density is given by Fick's first law

$$I_A = -D_{AB} \frac{d\rho_A}{dy}$$

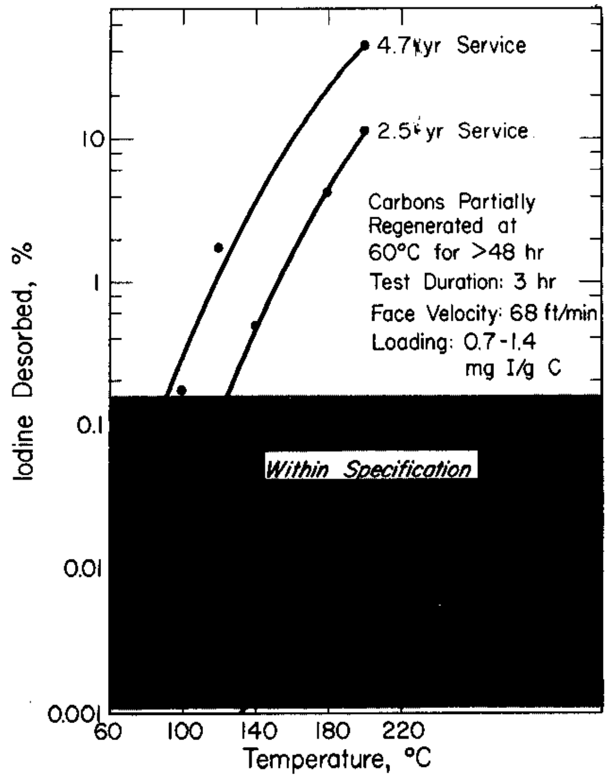

FIG. 6 EFFECT OF TEMPERATURE AND SERVICE ON DESORPTION

where I_A = flux of constituent A, (gram moles)/(sec)(cm^2)
D_{AB} = diffusion coefficient for a binary mixture of A and B, (cm^2)/sec
$\frac{d\rho_A}{dy}$ = concentration gradient, or driving force, (gram moles)/(cm^3)(sec)

In this calculation, $d\rho_A/dy$ was the difference between the iodine concentration in the pores and that in the bulk gas phase. Concentration in the pores was determined from vapor pressure data,[7] and concentration in the bulk gas phase was measured experimentally; dy was 1/4 of the average particle diameter (3.7×10^{-2} cm).

The flux of constituent A was the gram moles of iodine desorbed (experimental values) per hour per square cm. Surface areas were 1000 meters^2/g for new carbon and 600 meters^2/g for carbon with 30 months service.

The increase in the diffusion coefficient with increased temperature for unused Type 416 carbon and carbon with 30 months service is shown in Figure 7. The diffusion coefficient at temperatures below 140°C for unused carbon was too small to be measured.

The diffusion coefficient is in the range of chemisorption. In normal physical adsorption, the diffusion coefficient of gases from solids ranges from 10^{-5} to 10^{-6} cm^2/sec.[8] However, in chemisorption diffusion coefficients are much smaller. Studies of the chemisorption of oxygen on tungsten gave values of 2.5×10^{-16} cm^2/sec for a 0.5 monomolecular layer at 557°C, and 4.6×10^{-16} cm^2/sec for a 0.7 monomolecular layer.

The diffusion coefficient was calculated for Type 416 carbon, both unused and with 30 months service (Figure 8). As expected, with more iodine loaded on the carbon, iodine diffused at a greater rate.

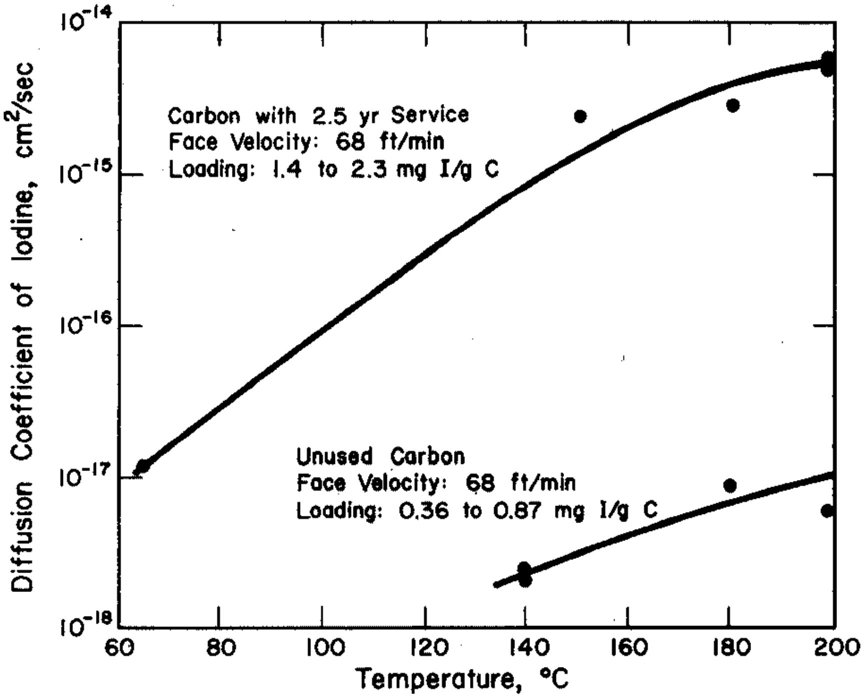

FIG. 7 EFFECT OF TEMPERATURE ON DIFFUSION COEFFICIENT

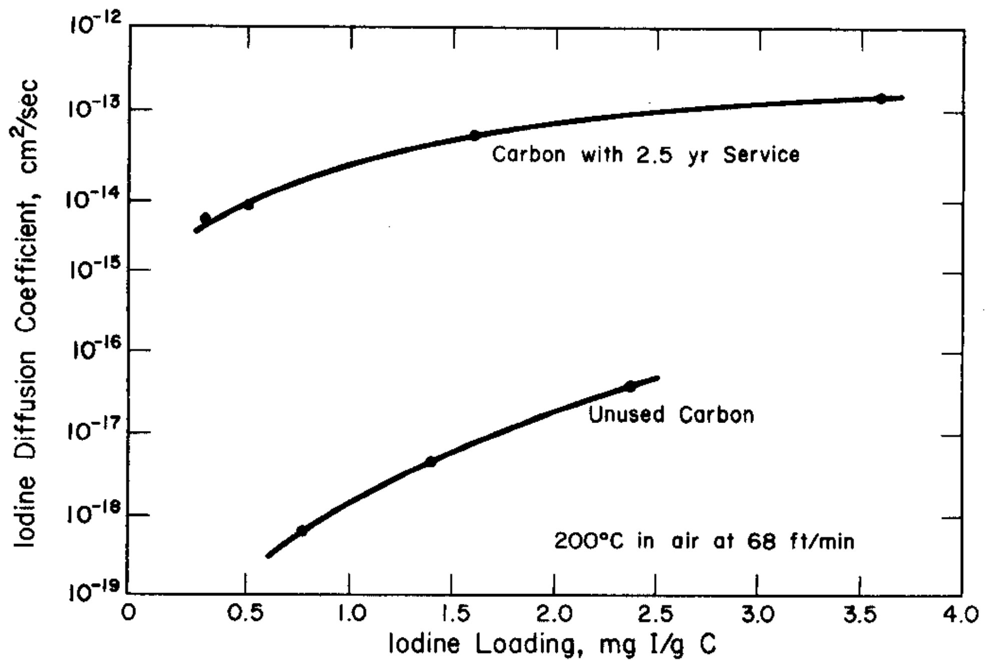

FIG. 8 EFFECT OF LOADING ON DIFFUSION COEFFICIENT

Partial Regeneration of Carbon

The iodine adsorption and desorption properties can be enhanced by partial regeneration by passing air at 60°C through the carbon with a face velocity of 7.5 ft/min for at least 48 hours. At higher regeneration temperatures, more of the adsorbed impurities are removed, and less iodine is desorbed.

The removal of gases as a function of temperature from partially regenerated carbon with 30 months service is shown in Figure 9. The release of nitric oxide, measured immediately after partial regeneration, increased significantly to 0.6 mg NO/g C. Removal of nitric oxide probably accounts for the beneficial effect of partial regeneration.

Most of the nitric oxide is released between 150 and 250°C in a vacuum (Figure 9). Although such treatment is not practical, the results indicate that high temperatures must be used to reduce NO in used carbon.

As shown in Table II, partial regeneration decreases the desorption of iodine, but the amount desorbed after partial regeneration at 200°C is still unacceptably large. Therefore, a superior method of partial regeneration is needed.

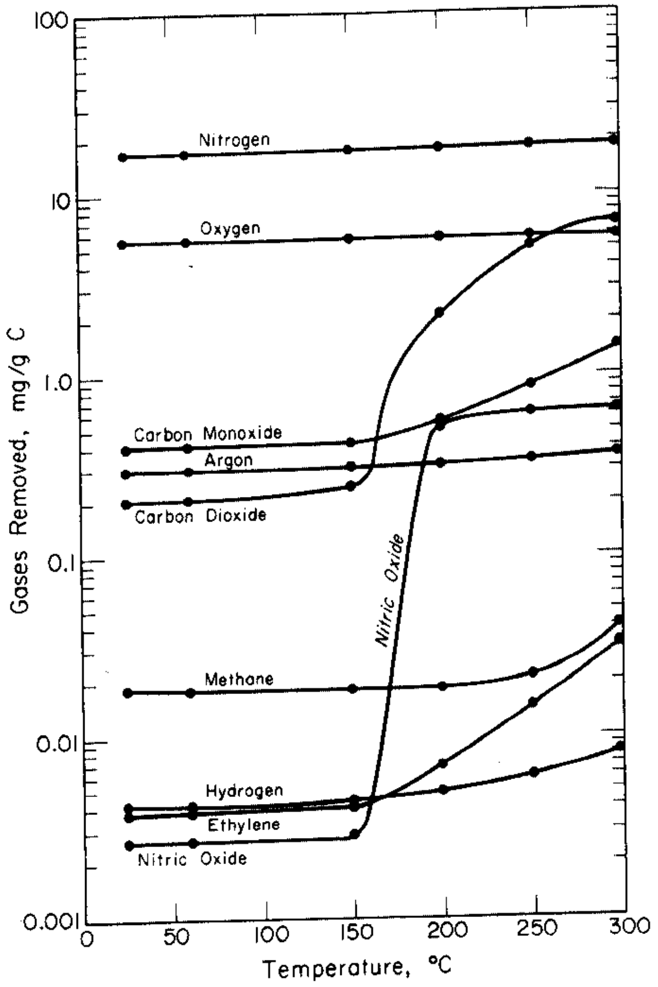

FIG. 9 REMOVAL OF GASES FROM PARTIALLY REGENERATED CARBON WITH 30 MONTHS SERVICE

TABLE II

Effect of Partial Regeneration on Iodine Desorption

30 Months Service

	Loading, mg I/g C	Desorption,[a] %
Untreated	0.76	21.0
Partially Regenerated	0.51	11.9

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[a] 125 minutes at 200°C.

Alternative methods of partial regeneration were tested to find conditions that would improve the retention properties of the carbon. Treatment of used carbon with flowing air or steam-air mixtures or steam did not reduce the desorption of iodine to an acceptably low level (Table III).

The reason for high desorption when the carbon was steamed at 100°C was investigated. Analysis of untreated and treated carbon showed that steam had removed a significant fraction of the sodium and potassium in the carbon (Table IV). The substantial loss of sodium and potassium probably interfered with the formation of temperature-stable compounds of iodine, such as sodium and potassium iodide.

TABLE III

Alternative Methods to Reduce Iodine Desorption

29 Months Total Service,
Partially Regenerated 11 Months

Treatment	Desorption,[a] %
Air at 60°C, 7.5 ft/min, for 48 hours	60.4
Air at 100°C, 8.4 ft/min, for >48 hours	44.3
Air at 150°C, 9.5 ft/min, for 48 hours	37.9
Steam-air at 70°C, 7.5 ft/min, for 48 hours	38.9
Steam at 100°C, 39 ft/min, for 1 hour	32.9
Impregnated with 1% $Na_2S_2O_3$	53.2

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[a] Percent of iodine loaded on test bed desorbed by flowing air at 200°C, 68 ft/min in 3 hours.

TABLE IV

Removal of Sodium and Potassium from Carbon by Steam Treatment

	Sodium, ppm	Potassium, ppm
Untreated carbon[a]	570	4100
Carbon steamed at 100°C for 1 hour at velocity of 39 ft/min	340	1200

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[a] Type 416 carbon with 29 months service.

In a test, the sodium content of partially regenerated carbon was replenished by impregnation with 1% sodium thiosulfate. However, as shown in Table III, desorption of iodine was not significantly reduced.

Evaluation of Alternative Carbons

Unimpregnated coconut shell carbons (Types 416* and G-600**) desorbed less iodine than any of the alternative carbons evaluated. Other carbons, impregnated coconut shell (Type 727*), and petroleum based (Type 348***), had no special properties for retaining iodine at high temperatures (Table V).

TABLE V

Desorption of Iodine from Unused Alternative Carbons

Carbon Type	Base	Mesh	Loading, mg I/g C	Desorption, %
416	Coconut	10-14	1.40	0.0105[a]
G-600	Coconut	12-16	1.32	0.0051[a]
348	Petroleum	6-16	0.90	1.02[a]
727	Coconut (Impregnated)	10-14	0.60	0.0513[b]

a In 3 hours at 200°C with a face velocity of 68 ft/min.
b In 1.8 hours at 200°C with a face velocity of 68 ft/min.

IODINE ADSORPTION EFFICIENCY

Decrease with Service

Tests showed that the iodine adsorption efficiency decreased slowly with service in the Savannah River confinement system but was within specification after 56 months service, as shown in Figure 10 (shaded area is within specification).

In these tests carbon samples were partially regenerated at 60°C for 48 hours. The test bed is equilibrated with moisture by flowing a steam-air mixture at 65°C through the bed for 10 minutes at a velocity of 68 ft/min. Iodine tagged with ^{131}I is vaporized

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*Product of Barnebey Cheney Co., Columbus, Ohio.
**Product of North American Carbon Co., Columbus, Ohio.
***Product of Whitco Chemical Corp., New York.

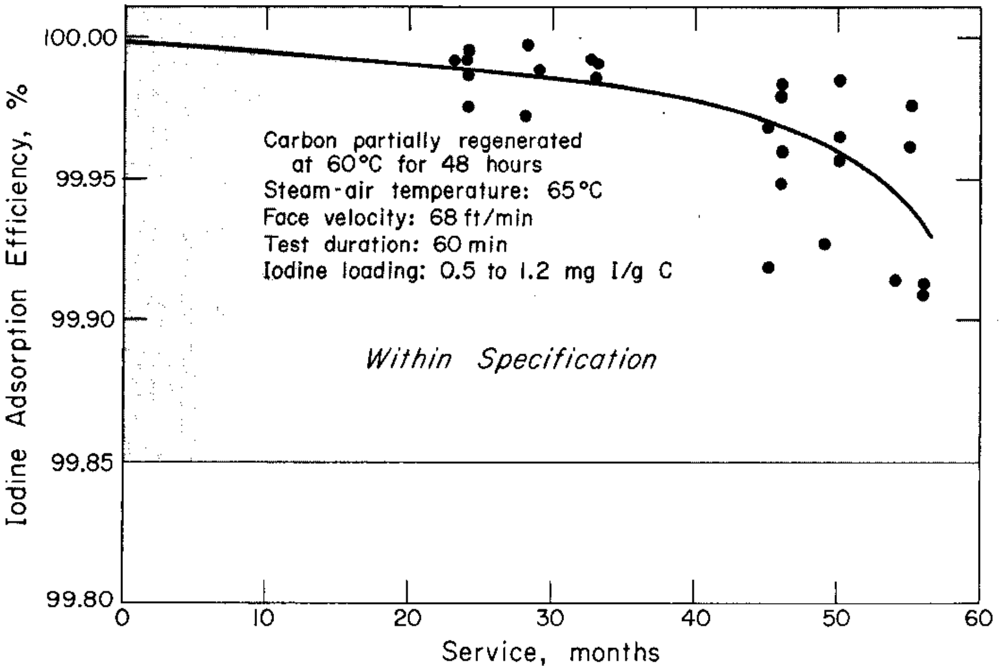

FIG. 10 EFFECT OF SERVICE ON IODINE ADSORPTION EFFICIENCY

from a glass frit, passed through a particulate filter, and adsorbed on the test bed over an interval of 60 minutes. Any iodine that penetrates the test bed is collected on backup beds, and the accumulation of iodine on the backup beds is monitored continually with a gamma monitor. The iodine adsorption efficiency is calculated from the ratio of iodine on the test bed to the sum of the iodine on the test bed and the iodine on the backup beds.

The slow decrease in adsorption efficiency is attributed to the accumulation of airborne impurities (page 7) during on-line service. Partial regeneration removes impurities that are not strongly adsorbed.

Iodine adsorption efficiency was compared in the upstream half and downstream half of carbon beds. As shown in Table VI, upstream, composite, and downstream samples of carbon with 33 months service had iodine adsorption efficiencies within specification.

The upstream sample of a carbon with 55 months service was outside specification; however downstream and composite samples of the same bed were within specification.

TABLE VI

Iodine Adsorption Efficiency of Upstream,
Composite, and Downstream Regenerated Carbons

Sample	Service, months	Loading, mg I/g C	Iodine Adsorption Efficiency, %
Upstream	33	0.56	99.977
Composite	33	0.99	99.987
Downstream	33	0.63	99.994
Upstream	55	0.81	99.639
Composite	55	0.98	99.962
Downstream	55	0.53	99.990

Decrease with Increased Loading

The iodine adsorption efficiency of carbon with 30 months service in the Savannah River confinement system decreased slightly with loading up to 9 mg I/g C (which is 13 times greater than expected in a reactor accident). The iodine adsorption efficiency was within specification at this high loading, as shown in Figure 11 (shaded area is within specification). These results show the safety factor built into the confinement system for protection against low iodine adsorption efficiencies that might be caused by heavy iodine loadings.

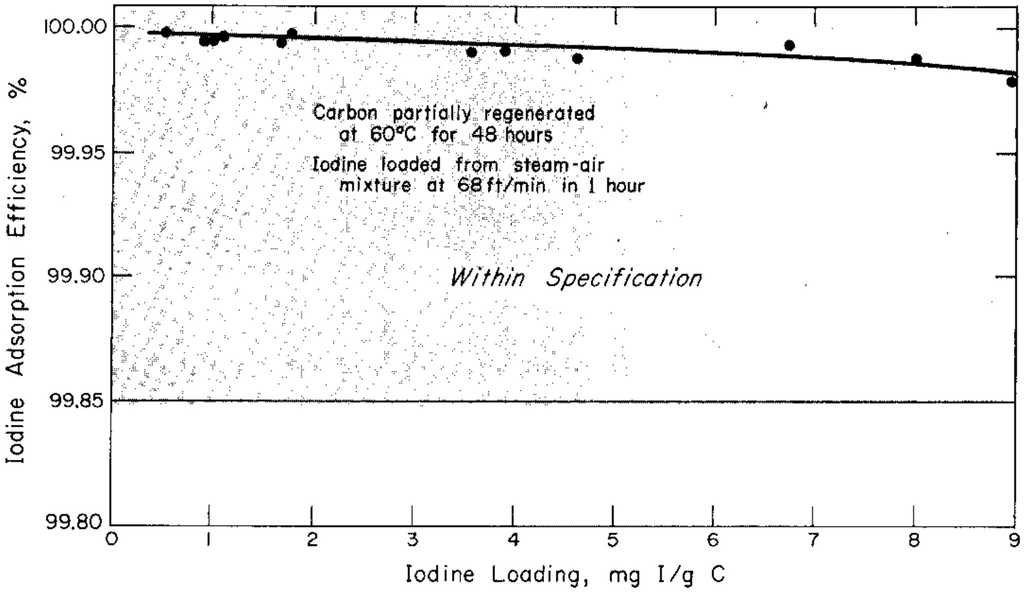

FIG. 11 EFFECT OF LOADING ON IODINE ADSORPTION EFFICIENCY OF CARBON WITH 30 MONTHS SERVICE

Increase with Partial Regeneration

As mentioned in previous reports,[1,2,3] partial regeneration of carbon from the confinement system increases the adsorption efficiency of carbon. Partial regeneration removes some of the impurities adsorbed on the carbon that interfere with the adsorption of iodine. Data tabulated in Table VII show the improvement in iodine adsorption efficiency obtained by partial regeneration.

TABLE VII

Effect of Partial Regeneration on Iodine Adsorption Efficiency

	Service, months	Loading, mg I/g C	Iodine Adsorption Efficiency, %
Untreated	23	0.66	99.931
Partially Regenerated	23	0.65	99.992
Untreated	30	0.76	99.968
Partially Regenerated	30	0.51	99.999
Untreated	46	0.68	99.727
Partially Regenerated	46	0.66	99.983
Untreated	55	0.63	99.402
Partially Regenerated	55	0.73	99.978
Untreated	56	0.59	99.345
Partially Regenerated	56	0.69	99.910

ACCELERATED AGING

Development of an accelerated aging treatment for carbon was begun to produce artificially aged carbon samples for evaluation of service-related properties of new carbons. In the accelerated aging treatment, a test bed of unused carbon was exposed to a mixture of nitrogen dioxide and air. This treatment reduced the number of active sites available to adsorb and desorb iodine. A standard treatment is sought such that a few hours exposure to the proper nitrogen dioxide concentration will produce carbon equivalent to that with months or years service in the confinement system.

Of the impurities in the confinement system, nitrogen dioxide had the most significant effect on iodine desorption. Nitrogen dioxide, present in the ventilating system during reactor operation, is a very reactive gas that consumes active sites. Nitric oxide, the reaction product of nitrogen dioxide and carbon, is partially removed by partial regeneration.

In the first test, unused Type 416 carbon was exposed to 30 ppm nitrogen dioxide (1000 times greater than in the ventilation system) in 50% relative humidity air for 2 hours at a velocity of 106 ft/min (about double normal velocity). Such an exposure is intended to be equal to 5½ months of service in the confinement system. In the second test, under the same conditions, carbon was exposed for 4 hours (11 months service). The test bed was then partially regenerated in the standard manner and the desorption measured.

The results of the first two accelerated aging treatments, summarized in Table VIII, show that iodine desorption increases with exposure to dilute nitrogen dioxide-air mixtures. The amount of desorption is slightly less than would be predicted from data obtained for samples of carbon taken from the confinement system (Figure 3). Additional tests with longer exposures to nitrogen dioxide (equivalent to 20-50 months service) are scheduled to correlate the desorption of carbons that are aged in the confinement system with those aged by the accelerated method.

TABLE VIII

Desorption of Iodine from Artificially Aged Carbon

30 ppm NO_2

Exposure, hr	Equivalent Confinement System Service, months	Iodine Loading, mg I/g C	Iodine Desorption,[a] %
2	5½	0.91	0.059
4	11	0.80	0.082

---

[a] Percent of iodine loaded on test bed desorbed by flowing air at 200°C, 68 ft/min in 3 hours.

CARBON IGNITION

Although studies related to developing a standard procedure for measuring carbon ignition temperature were concluded,[2] carbon ignition has been investigated further because of the anomalous behavior of two carbon beds while the beds were being partially regenerated for desorption tests.

One bed, which had 30 months service in a confinement system, ignited while being heated with hot air at a velocity of 9.5 ft/min. The bed ignited in three successive tests when the downstream temperature was between 100 and 150°C. In a fourth test, the bed did not ignite when it was heated to a downstream temperature of 100°C for 16 hours and then heated to 150°C for 2 hours.

The anomalous behavior of the carbon was not resolved in additional tests. A layer of carbon on a watch glass did not ignite when placed in a furnace at 400°C. However, Whetlerite* (nominal ignition temperature: 250°C) ignited at 300°C.

The second carbon bed, which had 29 months service, was being heated at 65°C with air at a velocity of 7.5 ft/min. At about 45°C, a small fraction of the carbon granules audibly disintegrated to a fine black powder that was carried out of the test bed by the air flow. Apparently, some of the granules contain an unstable material that decomposed near 45°C.

This behavior is not expected to cause problems because it was observed in only one of more than 30 beds that received the same treatment. Although this behavior could cause minor settling of a full-sized bed, baffles in the bed would prevent channeling.

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* Whetlerite, a product of Pittsburgh Activated Carbon Co., is bituminous carbon impregnated with ~10 wt % salts of copper, silver, and chromium to enhance adsorption of toxic gases.

METHYL IODIDE RADIOLYSIS TESTS

Completion of a new ^{60}Co irradiation source for use in methyl iodide radiolysis tests is expected in December. Tests will be resumed with the following equipment added:

- A cooling water jacket to maintain a constant temperature around the test carbon bed in the gamma field
- An in-place scintillation counter to monitor the amount of methyl iodide penetrating the test carbon bed as a function of time during the test.

The cooling water jacket will permit better control of the test bed temperature and relative humidity gradient. The in-place counter will provide data on methyl iodide residence time in the test carbon bed and absorbed gamma dose. With this information, decomposition characteristics for methyl iodide may be calculated and compared with published data.[9]

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