DP-1075

AEC RESEARCH AND DEVELOPMENT REPORT

HIGH TEMPERATURE ADSORBENTS FOR IODINE
PROGRESS REPORT: JANUARY 1965 - SEPTEMBER 1966

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Printed in the United States of America
Available from
Clearinghouse for Federal Scientific and Technical Information
National Bureau of Standards, U. S. Department of Commerce
Springfield, Virginia 22151
Price: Printed Copy $3.00; Microfiche $0.65

66473

DP-1075

Reactor Technology
(TID-4500)

Release Cat 1

HIGH TEMPERATURE ADSORBENTS FOR IODINE
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by

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December 1966

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CONTRACT AT(07-2)-1 WITH THE
UNITED STATES ATOMIC ENERGY COMMISSION

ABSTRACT

A new high temperature coconut-shell carbon (ignition temperature ~530°C), which was developed by an American manufacturer, has been shown in preliminary tests to meet specifications of the Savannah River Plant confinement system for removal of iodine.

A procedure was developed to measure ignition temperature, to evaluate promising new types of activated carbon, and to evaluate the effects of variables (such as air flow, bed properties, impregnants, and plant service) on ignition temperature. Results of these evaluations and a literature survey of ignition temperature are reported.

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INTRODUCTION

Removal of radioiodine from confinement systems⁽¹⁾ is of special interest,⁽²⁾ because it is potentially the most hazardous fission product to the surroundings of a nuclear reactor.⁽³⁾ At the request of the AEC Division of Operational Safety, a broad program was undertaken at the Savannah River Laboratory to seek a system that would remove fission product iodine from air at temperatures higher than the limitations of the present activated carbon systems. As a first step in this program, removal of radioiodine by a variety of materials was reviewed extensively* and a literature survey on the cleaning of reactor effluent gases⁽¹⁰⁾ was utilized. Confinement of iodine by various alternative systems was carefully considered, especially (1) by adsorption on activated carbon and on noncombustible materials such as silver, copper, and molecular sieves; and (2) by mechanical separation with filters and scrubbers.

In the unlikely event of a reactor accident, some risk of igniting activated carbon occurs because of the decay heat of adsorbed radioiodine or because of the decay heat from uncooled reactor fuel that heats air flowing through the beds to a high temperature. In either case, low flow of air would aggravate the situation. Appreciable burning of the carbon could destroy the beds and release most of the iodine. A second problem is the possibility that some of the iodine may be desorbed at high bed temperatures below the ignition temperature. This second problem will be investigated in the future.

The objectives of the work at the Savannah River Laboratory (SRL) on adsorbents for iodine were: a) to locate an adsorber that would reduce the probability of fire, b) to develop a standard procedure for the measurement of ignition temperature, c) to measure the ignition temperature of promising carbons, and d) to evaluate the effects of variables (such as air flow, bed properties, impregnants, and plant service) on ignition temperature. This progress report summarizes work from January 1965 to September 1966.

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* See References, especially items 4-9.

SUMMARY

Activated carbon is the superior adsorber of radioactive elemental iodine, despite the risk of ignition. An ignition temperature of 340°C was measured with a full-size, Type 416* carbon bed now used in the Savannah River Plant (SRP) reactor ventilation systems. The same temperature was measured in a small-scale apparatus with a standardized procedure developed at SRL. This small-scale apparatus was used to measure the ignition temperature of commercial carbons and to evaluate the effect of variables on ignition temperature.

A coconut shell carbon, Type 592**, ignited at 530°C at an air velocity of 105 ft/min, both in a full-size bed and in the small-scale apparatus. In preliminary tests, this carbon meets specifications of the SRP confinement system for removal of iodine and provides additional protection against ignition by the decay heat of adsorbed fission products. Full-size beds of this carbon are being evaluated in the SRP confinement system.

Iodine forms a chemical bond with carbon and is not significantly desorbed from coconut carbon at temperatures up to 250°C.(1&2) Measurements are planned at SRL of the desorption of iodine from unimpregnated and impregnated carbons at temperatures approaching the ignition temperature.

A survey of the literature revealed no satisfactory substitute for activated carbon in applications requiring >99% removal of radioactive iodine. Carbon has the advantages of:

- >99.9% efficiency for adsorption of iodine under a variety of adverse conditions,
- service life up to 3 years,
- essentially maintenance-free operation,
- low pressure drop, and
- low cost.

A survey of the literature also showed that the ignition temperature can be raised by using a carbon:

- with a smaller internal surface area;
- with a coarser mesh;
- impregnated with phosphates, borates, or halogens; or
- purified to remove impurities such as sodium and potassium that catalyze the oxidation of carbon.

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* Product of Barnebey-Cheney Company.

**Recently developed product of Barnebey-Cheney Co., discussed in more detail in Reference 15.

Noncombustible adsorbents of radioiodine are less satisfactory than carbon because:

- metals, such as silver, require temperatures >100°C for acceptable efficiencies,
- metals, such as copper, lose efficiency because of surface contamination, and
- nonmetals, such as molecular sieves, activated alumina, and silica gel, adsorb water preferentially.

Removal of radioiodine by liquids is unsatisfactory because:

- expensive equipment is required if a large flow must be treated,
- organic compounds and aerosols of iodine are not removed efficiently,
- liquids such as carbon tetrachloride are toxic and volatile, and
- aqueous solutions evaporate at elevated temperature.

DISCUSSION

LITERATURE REVIEW (SUMMARY)

Published data indicate that activated carbon from coconut shells has adsorption properties for iodine superior to those of activated carbon from coal or wood. The coconut shell carbon* contains fine-capillary pores, and therefore a large internal surface area; when new, it removes iodine with >99.99% efficiency under a variety of adverse conditions. Carbon from coal has a somewhat lower efficiency for adsorbing iodine. In addition, iodine desorbs at rates of only 0.002-0.02 %/hr from coconut carbon at 250°C, but at a rate of 0.7 %/hr from coal carbon under the same conditions.(152) Coal carbon is softer than coconut carbon; soft carbon forms dust that can be carried out of the bed during service. The superior adsorption characteristics of coconut carbon make it the preferred material in confinement applications, even though it may have a lower ignition temperature.

Published ignition temperatures are inconsistent; the data appear to depend on the measuring apparatus and procedures. As examples, the ignition temperature of coconut carbon ranges from 270 to 560°C, and the ignition temperature of coal carbon ranges from 345-515°C. Conditions that lower the ignition temperature

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* In this report, the word carbon refers to activated carbon.

are: impurities in the carbon that catalyze its oxidation, high ash content, fine aggregate size, thick beds, and large internal surface area. Data in the literature on the effect of air velocity are also inconsistent.

The ignition temperature of carbon is raised 1) by impregnation with halogens, phosphates, or borates; 2) by removal of impurities such as sodium and potassium that catalyze oxidation; and 3) by coating the surface with a heat-resistant porous material.

Published data indicate that noncombustible substitutes for activated carbon are unsatisfactory for most reactor confinement applications. The iodine adsorption efficiency of the noncombustible adsorbents is significantly lower than that of activated carbon. The ventilation air causes oxidation and other contamination of the surface, which interferes with the adsorption of iodine on copper or silver. Humid atmospheres also interfere with the adsorption of iodine on molecular sieves, activated alumina, and silica gel.

A detailed discussion of the literature review is in Appendix A, and a summary of carbon specifications is in Appendix B.

EXPERIMENTAL PROCEDURES

Thermobalance

The relative ignitibility of a variety of activated carbons and other carbonaceous materials was measured qualitatively with a thermobalance*, which recorded continuously the weight of a sample heated at a constant rate in flowing air.

About 250 mg of carbon was heated on a platinum disk in dry air at a rate of ~6°C/min. The linear air velocity was 0.14 ft/min. The recorded plot of the weight of carbon against temperature showed a loss of weight (moisture) up to 100°C; there was no significant loss of weight between 100°C and the ignition temperature. As combustion occurred, the sample lost weight because of the oxidation of carbon to carbon monoxide and carbon dioxide.

No additional measurements were made with the thermobalance, because the data are qualitative and the range of conditions is restricted.

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* Type RV thermobalance, Wm. Ainsworth & Sons, Inc., Denver, Colorado.

Small-Scale Apparatus

A small-scale apparatus for the quantitative measurement of the ignition temperature of carbon under a wide variety of conditions was developed by SRL (see Figures 1 and 2). Air to the apparatus is cleaned by a 1-inch-thick carbon bed and an absolute filter. Flows up to 6.5 ft^3/min (face velocity of 130 ft/min) are measured with calibrated rotameters. The temperature of the air flowing through the preheater is increased at rates of 2 to 4°C/min by adjusting the "Powerstat."* The temperature of the oven is controlled automatically (at the same temperature as the preheater) by a temperature controller connected to "Chromel-Alumel"** thermocouples 1 and 2. Stainless-steel-clad, 0.025-inch OD, thermocouples were used initially to measure the bed temperature to avoid bypassing of air. Now, 0.125-inch thermocouples are used because they give the same result and are more durable. Thermocouples 3 and 4 measure the temperature near the upstream face of the carbon bed, and 5 and 6 near the downstream face. The ignition temperature is indicated by an abrupt increase in the rate of temperature rise of the bed. The downstream air temperature, which also gives a prompt indication of ignition, is monitored by thermocouple 7. The furnace temperature is measured with thermocouple 8. Carbon beds with diameters and depths up to 3 inches can be tested in this apparatus. The 3-inch diameter was chosen, because this size gave satisfactory results in earlier tests of iodine adsorption at SRL(11-14) and because it was expected to adequately simulate a full-size bed.

The standard procedure for measurement of ignition temperature of carbon at SRL is described in Appendix C.

Full-Size Apparatus

The ignition temperatures of Type 416 and Type 592 carbon in a bed framed with stainless steel were measured at the Lawrence Radiation Laboratory (LRL). Unfiltered air and the combustion products from six gas burners entered a horizontal, uninsulated metal duct 2 feet square x 15 feet long, as shown in Figure 3. This duct contained four baffles to mix the air to uniform temperature before it entered the carbon bed. Effluent air from the carbon bed passed through a 1-foot-diameter duct, 15 feet long.

The downstream face of the bed was observed through three glass windows in the duct. An air eductor that was operated by compressed air moved air through the apparatus, and a baffle controlled air flow through the bed.

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* Trademark of Superior Electric Co., Bristol, Connecticut.

** Trademark of Hoskins Mfg. Co.

The temperature of the bed was measured where ignition was likely with 20 calibrated iron-constantan thermocouples (0.025 inch OD, stainless steel clad). Temperature from each thermocouple was recorded every 26 seconds.

A pitot tube connected to an inclined manometer measured the air flow. Air temperature was increased in ~50°C increments by adjusting the burners; the bed temperature was allowed to come to equilibrium after each increase. At ~50°C below the ignition temperature, the air temperature was increased continuously at ~10°C/min. At the ignition temperature, the thermocouples in the center of the bed suddenly indicated a rapid temperature rise.

RESULTS

Relative Ignitibility

Thermobalance measurements showed the following relative ignitibility of activated carbons: Whetlerite* (lowest temperature), coconut shell, and bituminous coal. Pyrolytic graphite foam** (an allotropic form of carbon, less reactive than activated carbon) ignited at the highest temperature.

The loss of weight at temperatures up to ~100°C, as shown in Figure 4, is due to the desorption of water. Above 100°C, the weight of the carbon does not change significantly until ignition occurs; at ignition, there is a rapid loss of weight. Whetlerite was heated in a vacuum in the thermobalance to ~400°C with no significant loss of weight. This verified that the loss of weight in air at an elevated temperature was caused by the combustion of carbon and not by decomposition of the impregnated salts.

The relative ignitibility of the carbons tested agreed with relative ignition temperatures measured in the small-scale apparatus under different conditions. Whetlerite ignited at a low temperature, because the impregnated metal salts catalyzed the oxidation of carbon.

Although pyrolytic graphite foam has the highest ignitibility, it adsorbed iodine with an efficiency of only 2% in the apparatus described in DP-778.(14)

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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.

** Product of General Electric Co.

Type 416 carbon used in SRP confinement facilities was coated with porous pyrolytic graphite.* This material was tested in the thermobalance to determine if the porous coating (~50% voids) might increase ignition temperature without decreasing iodine adsorption by the carbon.

The coatings, 80- and 200-μ thick, were not adherent and some was removed by gentle brushing. The pyrolytic coating was too soft for confinement purposes and insignificantly improved the ignition temperature, as shown in Figure 5.

Dependent Variables in Small-Scale Ignition Tests

Tests at other sites and at SRL with the small-scale apparatus showed that the ignition temperature of carbon is affected by:

- type of carbon
- internal surface area
- service life
- particle size
- bed depth
- air velocity
- adding carbon with lower ignition temperature
- iodine impregnation

Because the ignition temperature of carbon is a function of several variables, measurements were made as each variable was changed.

Type of Carbon

Three materials commonly used to prepare carbon are coconut shell, petroleum, and bituminous coal. The ignition temperatures of six commercial carbons are shown in Table I.

Normally, carbon from coconut shell has the lowest ignition temperature. However, by special processing to remove impurities that catalyze oxidation, the ignition temperature of coconut shell carbon can be increased to 530°C.

Normally, bituminous coal has a higher ignition temperature than coconut shell because it is more crystalline than coconut shell. Impregnation of bituminous carbon with ~10 wt % salts of copper, silver, and chromium (Whetlerite) lowered the ignition

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* Coated by the General Atomics Division of General Dynamics Corp.

temperature from 480 to 250°C. Most metallic salts, especially sodium and potassium, catalyze the oxidation of carbon as discussed in Appendix A.

Internal Surface Area

The effect of internal surface area was reported by Kovach.(15) He showed a rapid decrease in ignition temperature from 485 to 370°C when the internal area was increased from 750 to 1500 m^2/g. These results agreed with kinetics of oxidation of carbon, as discussed by Kovach.

Service Life

The ignition temperature of Type 416 carbon increased about 80°C after 18 to 25 months of service in the SRP reactors. This was due to the increased concentration of iodine, chlorine, phosphates, and other impurities adsorbed on the carbon, which was confirmed by mass spectroscopic analysis. These impurities (atmospheric comtaminants) inhibit the oxidation of carbon, and increase the ignition temperature, as shown in Table II.

Particle Size

Ignition temperature increases with increasing particle size. However, the effect of particle diameter on ignition temperature was not significant (~25°C) for diameters of 1 to 4 mm(15) normally used in confinement applications.

Bed Depth

The dependence of ignition temperature on bed depth from ½ to 3 inches was determined with Type 416 and Type 592 carbon. Air velocity was 70 ft/min, the normal face velocity for SRP carbon beds.

The ignition temperature of Type 416 was unaffected as the bed depth increased from ½ to 3 inches, as shown in Figure 6. Similar results were obtained for PCB* coconut shell carbon at 1- and 2-inch depths. However, the ignition temperature of Type 592 carbon decreased significantly as the bed depth increased from 1 to 3 inches. In the deeper Type 592 beds, ignition usually occurred inside the bed, as reported in the literature,(15) instead of near the face of the bed.

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* Product of Pittsburgh Activated Carbon Co.

Air Velocity

The dependence of ignition temperature on air velocity was measured with a 1-inch-thick by 3-inch-diameter bed filled with Type 416 carbon currently used at SRP, or with Type 592 high temperature carbon. The results are summarized in Figure 7.

The ignition temperature of Type 416 carbon was unaffected (within the limits of precision, ±10°C) as the velocity increased from 10 to 120 ft/min. The ignition temperature of Type 592 carbon differed between batches. The first (and highest) ignition temperatures were obtained with carbon prepared on a laboratory scale by the Barnebey-Cheney Company. Results from this carbon are labeled Batch A in Figure 7. Results from a second batch of Type 592 carbon (Batch B, Figure 7) were lower and were only slightly affected by air velocity. This batch was one of the first made on a plant scale. Results from the Type 592 carbon used in the full-size test are shown as Batch C in Figure 7. This curve shows the same velocity dependence as the carbon prepared in the laboratory, but the ignition temperature is ~55°C lower. After these measurements were made, Barnebey-Cheney installed new equipment for the manufacture of Type 592 carbon with a consistently high ignition temperature. Measurements on subsequent batches have shown uniformly high ignition temperatures.

In actual confinement applications, ignition is not probable at high air velocities, because air flow through the bed will rapidly remove heat from radioactive decay. Ignition is much more likely at low velocities (<10 ft/min, for example). More experimental measurements of ignition temperatures at low velocities are in progress at SRL to evaluate the risk of ignition.

Adding Carbon with Lower Ignition Temperature

The effect of adding carbon with a low ignition temperature in a bed packed with high-ignition-temperature carbon was tested. 5g of Whetlerite was placed around an upstream thermocouple in a bed packed with 100g of BPL* carbon; the bed ignited at 270°C. This temperature is about the same as that measured for a bed of Whetlerite only. When the Whetlerite was placed at a downstream thermocouple, the bed ignited at 260°C. The bed was cooled after ignition, and the test was repeated on the same material. The second ignition occurred at 280-300°C; the third ignition occurred at 310°C. The rise in ignition temperature with repeated ignitions was attributed to decomposition of the chemicals used to make Whetlerite. Successive ignition temperatures of 250, 280, and 290°C were measured for a bed packed with Whetlerite only.

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* Product of Pittsburgh Activated Carbon Co.

Iodine Impregnation

The effect of impregnating carbon with elemental iodine and potassium iodide was investigated because of the increased interest in impregnated carbons to adsorb methyl iodide from humid air. Methyl iodide, which may be present in small quantities in confinement applications, is not adsorbed efficiently by carbon from very humid air. However, radioactive methyl iodide exchanges rapidly with iodine-impregnated carbon; the radioactivity is retained on the carbon and inactive methyl iodide is released. SRL requested Barnebey-Cheney Co. to impregnate Type 416 and Type 592 carbon with 1, 3, and 10% iodine by weight.

Impregnation of Type 416 carbon increased the ignition temperature 20-40°C, as shown in Figure 8. Apparently there is a saturation effect at about 3% iodine, and added iodine has little effect on ignition temperature. In each case, the second ignition (350°C) always occurred at a lower temperature than the first. Apparently, the first ignition volatilized some of the iodine impregnant. The loss of iodine, an inhibitor of ignition, depressed the ignition temperature.

Measurements of the desorption of iodine from unimpregnated and impregnated carbon at temperatures up to the ignition temperature are planned at SRL.

The results obtained with impregnated Type 592 carbon were erratic, as shown in Figure 8. The ignition temperatures of the 1 and 3% iodine impregnations occurred unexpectedly below that for unimpregnated carbon. A second ignition of each of these carbons occurred unexpectedly at a higher temperature than the first ignition. Additional tests are scheduled.

The ignition temperatures of other commercial impregnated carbons, measured with the small-scale apparatus, are summarized in Table III.

Type CB* carbon, a coconut carbon with the same internal surface (1100 m^2/g) as Type 416 but a coarser mesh (6 x 10) and impregnated with 10% I_2, ignited at 400 and 415°C on the first ignition. The second ignition, as expected, occurred at lower temperatures (385 and 400°C) due to the loss of iodine during the first test. Type CK* carbon, a coconut carbon with a high internal surface area (1600 m^2/g) and impregnated with 20% I_2, ignited at a low temperature despite impregnation, because of the large internal surface.

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* Product of Barnebey-Cheney Co.

The ignition temperature was 360°C for type 727* coconut carbon impregnated with iodine and potassium iodide (surface area of 1600 m^2/g and 8 x 14 mesh). The second ignition on the same bed occurred at only 310°C because the inhibitor of oxidation, iodine, was released during the first test; the catalyst for oxidation, potassium iodide (boiling point, 1420°C), was retained on the carbon.

Independent Variables in Small-Scale Ignition Tests

The ignition temperature of carbon in the small-scale apparatus was unaffected by the following variables:

- bed diameter
- baffles
- corrosion products
- reignition
- relative humidity of supply air
- reduction of oxygen content of supply air
- heating rate
- prolonged heating below ignition temperature

Bed Diameter

The effect of the diameter of the test bed on the ignition temperature of carbon was investigated for 1- and 3-inch-diameter beds of Type 416 carbon. The ignition temperature was independent of the bed diameter, as shown in Table IV.

Baffles

Baffles minimize bypassing of air at the carbon-metal interface. Elimination of baffles from the bed had no effect on ignition temperature, as shown in Table IV.

Corrosion Products

The corrosion products of stainless steel produced during repeated tests with the same bed had no effect on ignition temperature. The same ignition temperature was observed with a new bed and with a bed that had discolored during reuse. Some materials (e.g., compounds of iron, cobalt, and nickel) catalyze the oxidation of carbon and cause low ignition temperatures.(18)

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* Product of Barnebey-Cheney Co.

Reignition

Contrary to expectations, the ignition temperature of unimpregnated carbon was not affected by igniting the bed a second and third time, as shown in Table V. Ignition temperature has been shown to be dependent upon ash content,(15) and repeated ignition should increase the ash content and depress the ignition temperature. In SRL tests, ignition may not have produced sufficient ash to cause this effect or air flow may have swept any ash out of the bed. As many as three ignitions can be made on the same bed without affecting its ignition temperature if the fire is extinguished immediately after ignition occurs.

Reignition of carbon impregnated with halogens (inhibitors of ignition) produces lower ignition temperatures because of the loss of some of the halogens. Reignition of Whetlerite (carbon impregnated with salts of copper, silver, and chromium) produces higher ignition temperatures probably because of decomposition of the salts.

Relative Humidity of Supply Air

In preliminary measurements, the humidity of the supply air to the bed had no effect. Air, saturated with moisture at 43°C, produced a normal ignition temperature.

Reduction of Oxygen Content of Supply Air

Reduction of oxygen content of the supply air to the bed from the normal 20.9% to 15.9% had no effect, as shown in Table VI. In the industrial preparation of activated carbon, ignition of carbon can occur in an atmosphere of 10% oxygen. The SRL test was made to verify that the ignition temperature of a full-size bed (discussed later) was not affected by air that contained 16.2% oxygen.

Heating Rate

The ignition temperature was not affected by changes in the rate of heating the bed from 0.5 to 4.8°C/min, within the limits of precision (±10°C). As discussed later, a heating rate of 10°C/min had no effect on the ignition temperature of full-size carbon beds. Tests at even higher heating rates are planned.

Prolonged Heating Below Ignition Temperature

The effect of prolonged heating of Type 416 carbon just below the ignition temperature was evaluated in the small-scale apparatus. A sample, heated at 325°C (about 15°C below the ignition temperature) for 6 hours with an air flow of 70 ft/min, lost 11% of its weight. A second sample, heated at 335°C for 5 hours, lost 10% of its weight. Most of the weight losses were attributed to the desorption of water.

Barnebey-Cheney Measurements

At the request of SRL, Barnebey-Cheney Co. measured the ignition temperature of Type 416 as a function of air velocity and bed depth in an apparatus described in the literature.(15) The ignition temperatures of both the 1- and 3.5-inch-thick beds increased with an increase in air velocity, as shown in Figure 9. This increase was not observed at SRL on Type 416 carbon. The Barnebey-Cheney Co. measurements were obtained with a 1-inch-diameter carbon bed in an "Inconel"* holder. The SRL measurements were made with a 1-inch-thick by 3-inch-diameter bed in a stainless steel holder. SRL measurements showed that changing the diameter of the carbon bed from 1 to 3 inches and changing the thickness from 1 to 3 inches had no effect. The differences in these results may be due to the material of construction of the bed. Additional work is in progress to resolve the difference.

Adsorption Properties of Type 592 Carbon

Efficiencies of 99.99+% were measured for the adsorption of iodine on new, Type 592 carbon at loadings to 1.5 mg I_2/g carbon. The results, shown in Table VII for steam-air mixtures,(14) are comparable to those for Type 416 carbon now used in SRP confinement facilities.

Full-Size Ignition Tests

Type 416 Carbon

The ignition temperature of Type 416 carbon was 340°C at a face velocity of 70 ft/min in a full-size, stainless steel-framed

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* Trademark of International Nickel Co.

bed that was a prototype of a bed under evaluation in the SRP confinement facilities.[1] This agrees with the temperature measured for a 1-inch-thick by 3-inch-diameter bed in the small-scale SRL apparatus (340±10°C).

The oxygen content of air flowing through the apparatus at the ignition temperature decreased from the normal 20.9 to 16.2% because of the combustion products from the burners. As expected from tests in the small-scale SRL apparatus, the decrease in oxygen had little effect on the ignition temperature. Carbon can ignite in an atmosphere of 10% oxygen.

When the Type 416 carbon ignited, glowing particles of carbon blew out of the center of the bed. The gas burners and air flow were stopped immediately, and the top of the upstream duct was removed. The upstream face was sprayed with 160 pounds of carbon dioxide that solidified on the stainless steel mesh and plugged the perforations. The CO_2 cooled the upstream face, but the thermocouples in the middle of the bed indicated that the bed was still burning. Because it appeared that carbon dioxide would not extinguish the fire quickly, water was used.

Water reacts with hot carbon to produce a mixture of hydrogen and carbon oxides that might form an explosive atmosphere. Therefore, air flow was restored and the bed was flooded with water from a 1-inch-diameter hose. The water melted the solidified carbon dioxide and steam flowed out of the apparatus.

After 10 minutes of flooding, two areas of the bed continued to burn, so two of the glass windows on the downstream face were opened and water was sprayed on the downstream face. After 5 minutes, the fire was extinguished and all thermocouples indicated normal temperature.

Type 592 Carbon

An ignition temperature of 530°C (face velocity 105 ft/min) was measured for Type 592 carbon packed in the same stainless-steel-framed bed described previously.

The fire in the Type 592 carbon was extinguished easily with a water spray* that blanketed the face of the bed. The gas burners were stopped immediately after the carbon ignited and the air flow was maintained at a velocity of 10 ft/min to avoid explosion of the effluent gases. The water spray, at a rate of

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* From Fulljet ½HH29 Square Nozzle, product of Spraying Systems, Bellwood, Illinois.

8 gpm, cooled the bed from a maximum of 850°C to ambient temperature in 5 minutes. After the fire was extinguished, the bed was dried with warm air and then removed from the apparatus for inspection. There was negligible damage to the stainless steel framing of the bed and to the rubber gaskets. The upstream face of the bed showed no corrosion, and there were only a few square inches of a white deposit on the downstream face. The ignition caused loss of less than ½ pound of carbon.

The bed was reinstalled in the apparatus and a second ignition test was begun at a face velocity of 40 ft/min. The bed ignited at 330°C; it ignited at 510°C in small-scale tests. Sodium and other impurities in the water that extinguished the fire were adsorbed by the carbon. These adsorbed impurities catalyzed the oxidation and lowered the ignition temperature. A sample of carbon from this bed was boiled in distilled water and washed to remove these adsorbed impurities. The washed carbon ignited at 465°C. Additional washing probably would restore the normal ignition temperature of ~500°C at a face velocity of 70 ft/min.

In the second ignition test, the bed was cooled from a maximum of 850°C to ambient temperature in only 2 minutes by the water spray. The fire was localized and produced a flame about 1/8-inch in diameter. Only 1 thermocouple of the 19 mounted in the bed indicated the fire.

In an endurance test, the same bed of Type 592 carbon was exposed for 35 minutes to air at 280°C flowing at a face velocity of 70 ft/min with no significant damage.

A nondestructive test(17-22) of this bed with "Freon"-112* showed that leakage flow was less than 0.01%. This low leakage rate confirmed the visual observation that there was no significant damage to the bed or gaskets as a result of the ignition test and from heating the bed at 280°C for 35 minutes.

The iodine adsorption efficiency of a carbon sample from the section of the bed in which ignition occurred was 99.98% in a steam-air test.(11) The carbon for this test contained visible amounts of ash. The adsorption efficiency of new Type 592 carbon is 99.99+%, as shown in Table VII.

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* Du Pont's registered trademark for its fluorinated hydrocarbons.

TABLE I

Ignition Temperature of Various Commercial Carbons

	Mesh[a]	Ignition Temperature,[a] °C
Coconut Shell		
Type 416[b]	10 x 14	340
Type 592[b]	10 x 14	530
Type PCB[c]	6 x 16	340
Petroleum		
Type 348[d]	6 x 16	420
Bituminous Coal		
Type BPL[c]	4 x 10	480
Whetlerite[c]	4 x 10	250

(a) The variations in mesh are not expected to affect the ignition temperature significantly. Air velocity, 70 ft/min; 1-inch-thick by 3-inch-diameter bed.
(b) Product of Barnebey-Cheney Co.
(c) Product of Pittsburgh Activated Carbon Co.
(d) Product of Witco Chemical Co.

TABLE II

Effect of Service[a] on the Ignition Temperature of Type 416 Carbon

Service, months	Ignition Temperature, °C
0	340
18	400
18	410
25	430

(a) Exposure to flowing air in the exhaust ventilation system of SRP reactors. Face velocity, 70 ft/min; 1-inch-thick by 3-inch-diameter bed.

TABLE III

Ignition Temperature of Commercial Impregnated Carbons

(Face velocity, 70 ft/min; 1-inch-thick by 3-inch-diameter bed)

Barnebey-Cheney Co. Carbon	Ignition Temperature, °C
Type CB (10% I_2)	400, 415
Type CK (20% I_2)	305
Type 727 (I_2 + KI)	360

TABLE IV

Effect of Bed Diameter and Baffles on Ignition Temperature

(Type 416 Carbon, 1 inch thick)

Bed Diameter, inch	Ignition Temperature, °C	Air Velocity, ft/min
3 (baffled)	340	20
1 (baffled)	350	22
1 (unbaffled)	360	22

TABLE V

Effect of Reignition on Ignition Temperature of Type 416 Carbon

(Face velocity, 70 ft/min; 1-inch-thick by 3-inch-diameter bed)

Ignition Number	Ignition Temperature, °C
1	340
2	350
3	360

TABLE VI

Effect of Reduced Oxygen in Supply Air on Ignition Temperature of Type 416 Carbon

(Face velocity, 60 ft/min; 1-inch-thick by 1-inch-diameter bed)

Oxygen Content, %	Ignition Temperature, °C
20.9 (normal)	340
19.3	330
16.8	330
15.9	340

TABLE VII

Adsorption Properties of Type 592 Carbon(a)

Iodine Loading, mg I_2/gC	Conditions(b)	Iodine Adsorption Efficiency, %
0.34	Steam-air	99.99+
0.84	Steam-air	99.99+
1.51	Steam-air	99.99+

(a) Product of Barnebey-Cheney Co.

(b) Conditions: Duration of test: 2½-3 hr
Face velocity: 70 ft/min
Isotopic dilution: 1.5 mCi ^{131}I/100 mg ^{127}I
Temperature: ~73°C.

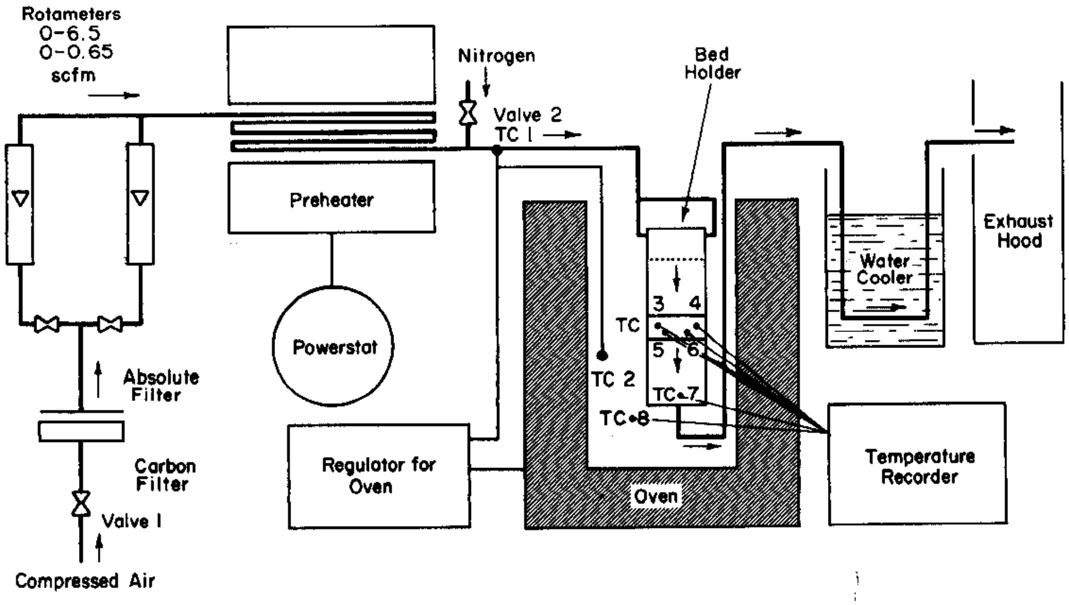

FIG. 1 SMALL-SCALE IGNITION APPARATUS

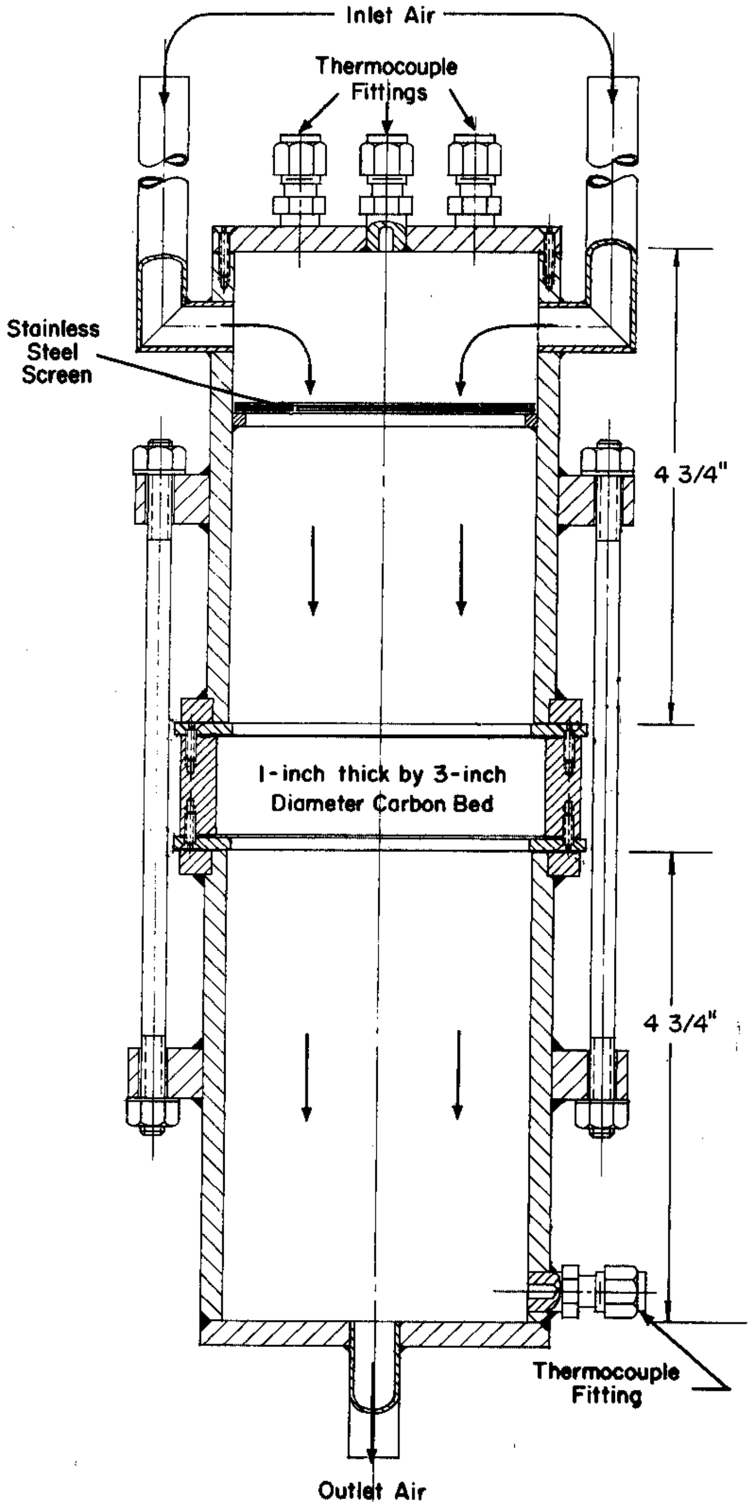

FIG. 2 DETAILED DRAWING OF SMALL-SCALE IGNITION APPARATUS

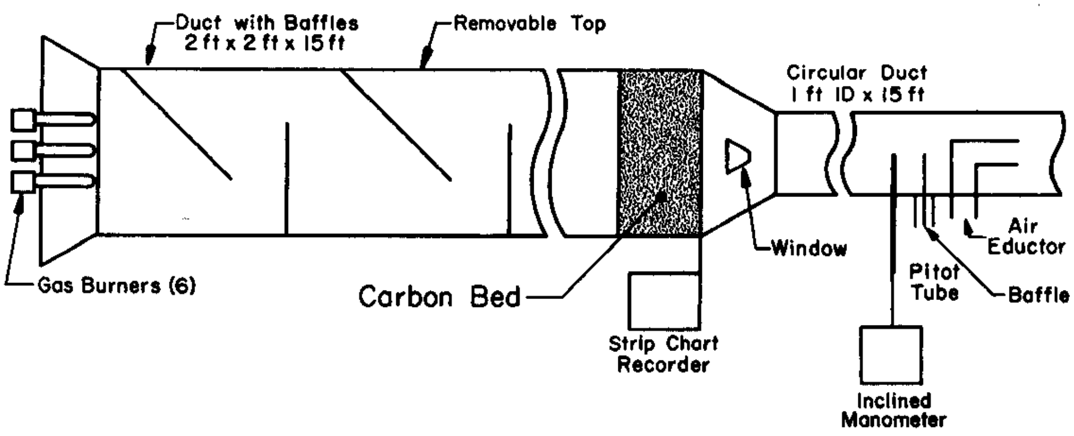

FIG. 3 FULL-SIZE IGNITION APPARATUS

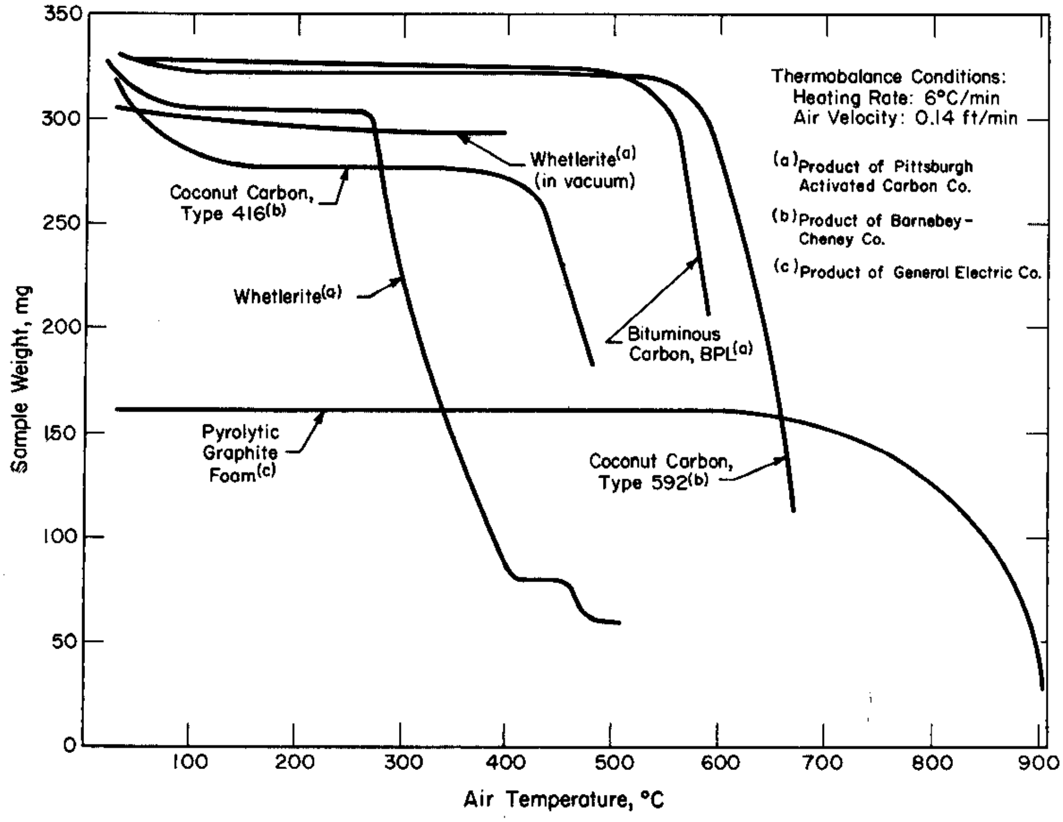

FIG. 4 RELATIVE IGNITIBILITY OF CARBON

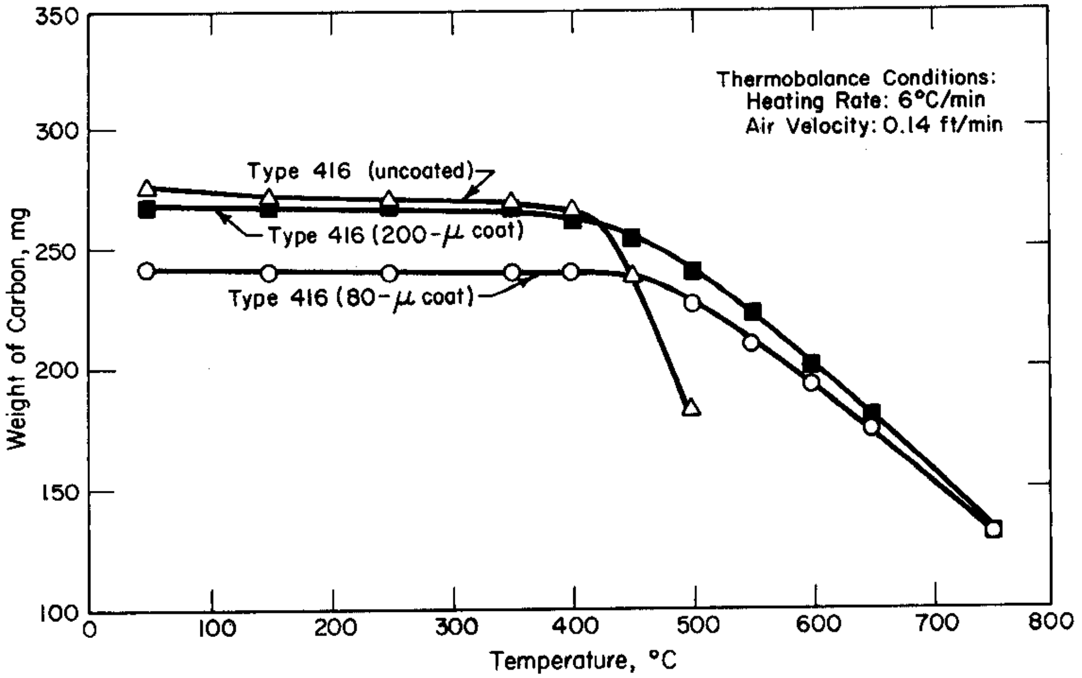

FIG. 5 RELATIVE IGNITIBILITY OF ACTIVATED CARBON COATED WITH POROUS PYROLYTIC GRAPHITE[a]
([a] Coated by General Atomics Division of General Dynamics Corp.)

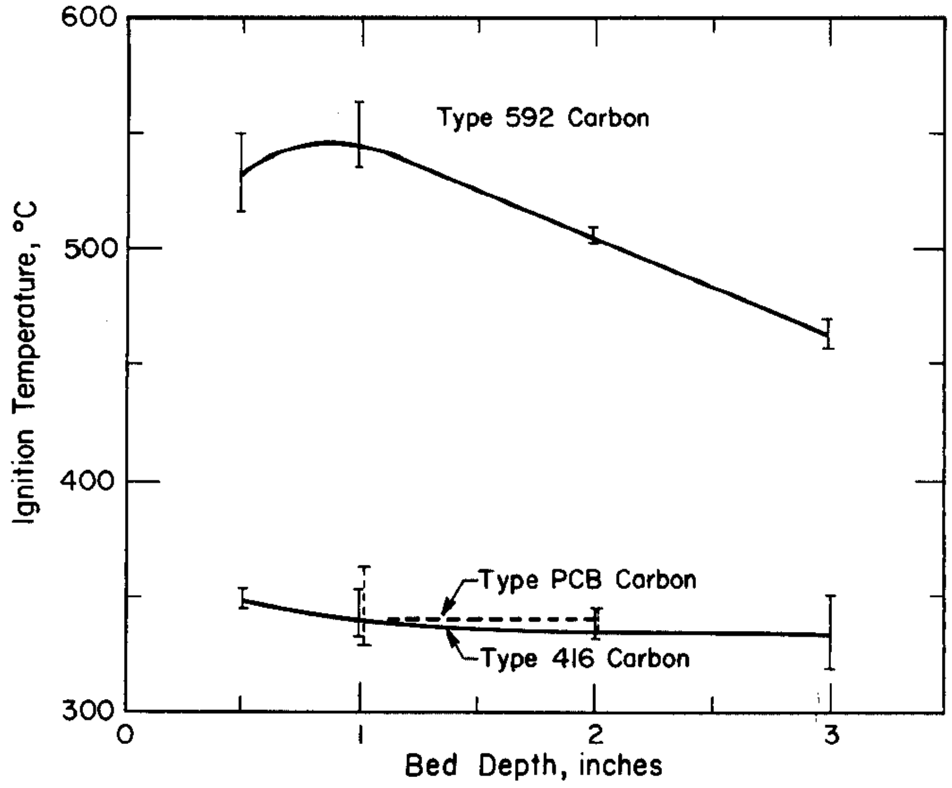

FIG. 6 IGNITION TEMPERATURE OF CARBON AS A FUNCTION OF BED DEPTH
(Air Velocity, 70 ft/min; 3-inch-Diameter Bed)

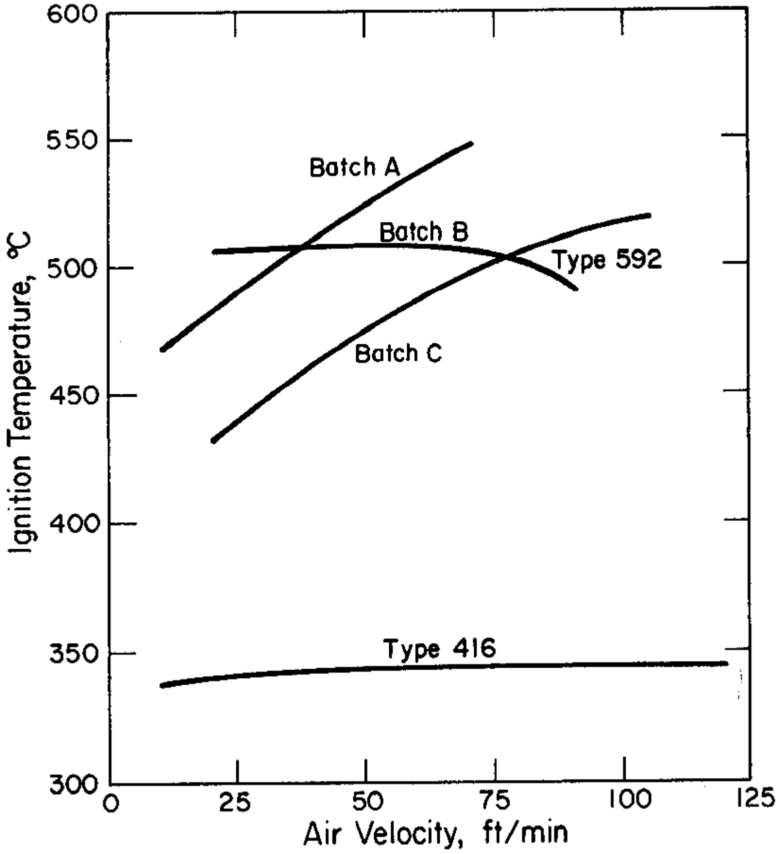

FIG. 7 IGNITION TEMPERATURE OF CARBON AS A FUNCTION OF AIR VELOCITY
(1-inch-thick by 3-inch-Diameter Bed)

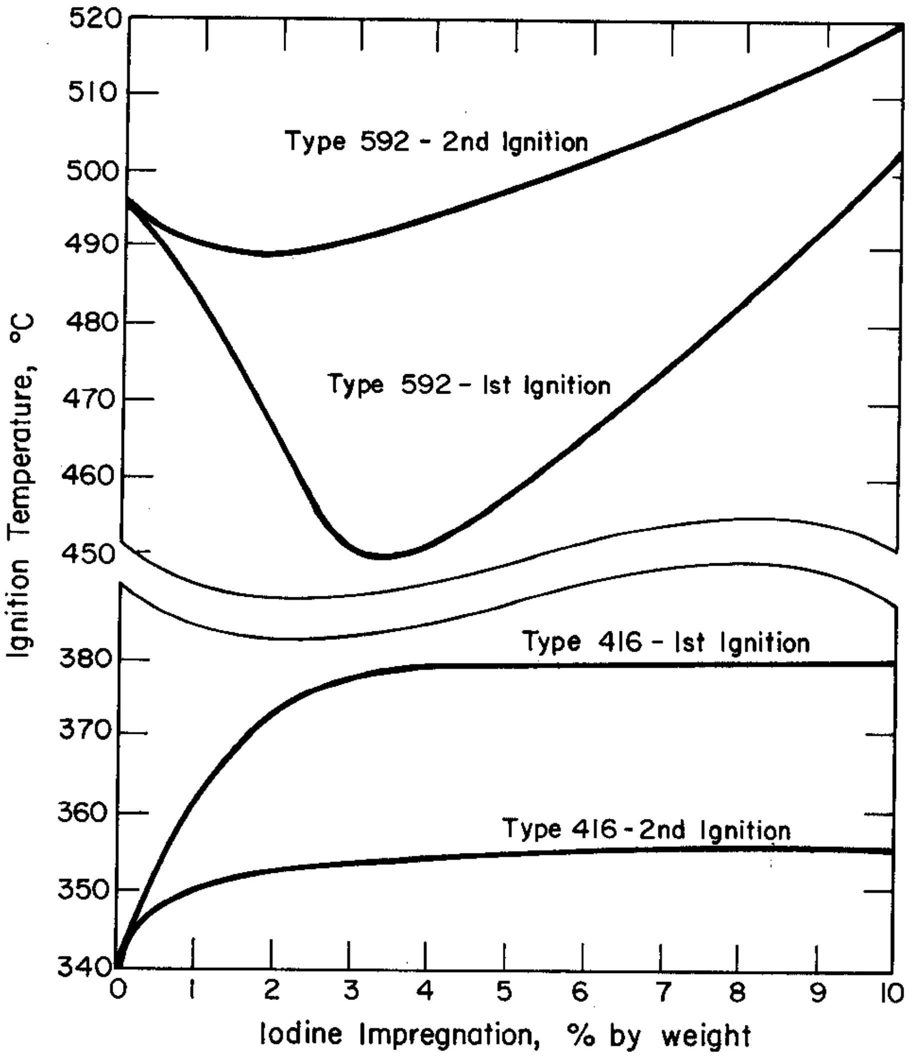

FIG. 8 IGNITION TEMPERATURE OF CARBON IMPREGNATED WITH IODINE
(Air Velocity, 70 ft/min; 1-inch - thick by 3-inch - Diameter Bed)

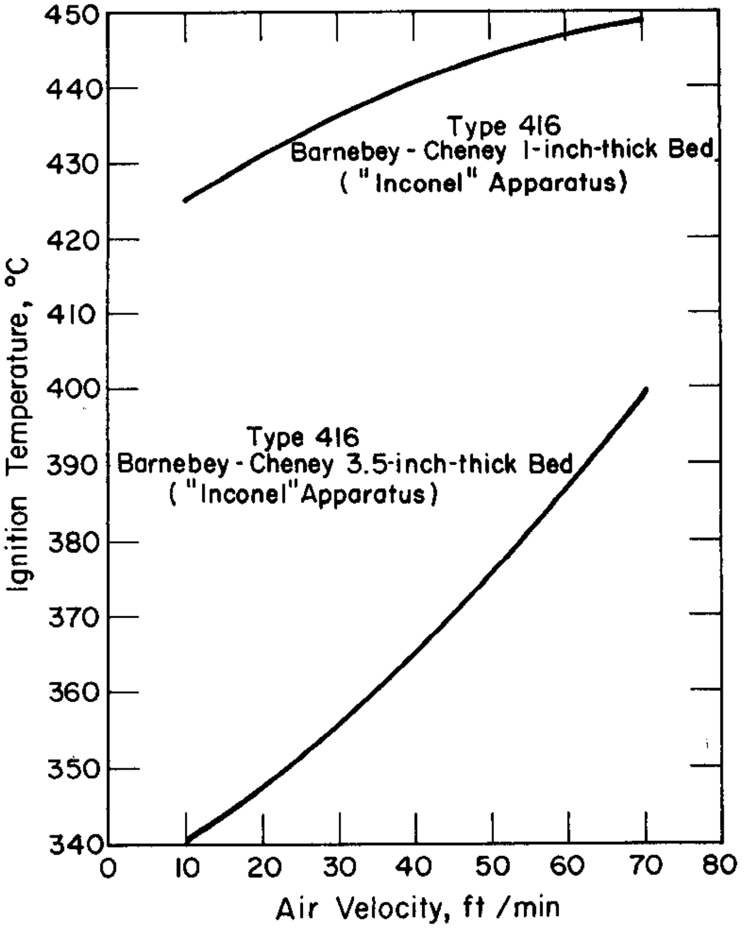

FIG. 9 IGNITION TEMPERATURE OF TYPE 416 CARBON AS A FUNCTION OF AIR VELOCITY (Measured by Barnebey-Cheney Co.)

REFERENCES

1. W. S. Durant, R. C. Milham, D. R. Muhlbaier, and A. H. Peters. Activity Confinement System of the Savannah River Plant Reactors. USAEC Report DP-1071, E. I. du Pont de Nemours and Co., Savannah River Laboratory, Aiken, S. C. (1966).

2. J. R. Beattie. "General Assessment of Possible Fission Product Releases from Reactor Accidents and the Ensuing Environmental Hazards." Food and Agricultural Organization, United Nations Atomic Energy Service No. 5, 260-9 (1964).

3. W. E. Browning, Jr. "Removal of Fission-Product Activity from Gases." Nucl. Safety 1(3), 40 (1960).

4. W. E. Browning, Jr. "Removal of Fission-Product Activity from Gases." Nucl. Safety 1(4), 47 (1960).

5. W. E. Browning, Jr. "Removal of Fission-Product Activity from Gases." Nucl. Safety 2(3), 35 (1961).

6. J. M. Holmes. "Removal of Contaminants from Gas Streams." Nucl. Safety 2(4), 39 (1961).

7. W. E. Browning, Jr. "Removal of Radioiodine from Gases." Nucl. Safety 4(2), 83 (1962).

8. G. W. Keilholtz and C. J. Barton. Behavior of Iodine in Reactor Containment Systems. USAEC Report ORNL-NSIC-4, Union Carbide and Carbon Co., Oak Ridge National Laboratory, Oak Ridge, Tenn. (1965).

9. D. Geldart. "Iodine - Its Removal from Gas Streams." Nucl. Eng. 5, 217 (1960).

10. M. N. Myers. Literature Survey: Sampling, Plate-Out and Cleaning of Gas-Cooled Reactor Effluents. USAEC Report XDC 61-4-702, General Electric Co., Aircraft Nuclear Propulsion Dept., Cincinatti, Ohio (1961) (TID-14469).

11. W. S. Durant. Performance of Activated Carbon Beds in SRP Reactor Confinement Facilities - Progress Report: September 1962 - September 1965. USAEC Report DP-1028, E. I. du Pont de Nemours and Co., Savannah River Laboratory, Aiken, S. C. (1966).

12. J. W. Walker and A. H. Peters. "Filters for Reactor Containment." Mech. Eng. 85, 46 (1963).

13. J. A. List. "The SRP Production Reactor Containment Program." Eighth AEC Air Cleaning Conference, held at Oak Ridge National Laboratory, October 22-25, 1963, TID-7677, 273 (1964).

14. G. H. Prigge. Application of Activated Carbon in Reactor Containment. USAEC Report DP-778, E. I. du Pont de Nemours and Co., Savannah River Laboratory, Aiken, S. C. (1962).

15. J. L. Kovach and J. E. Green. "Evaluation of the Ignition Temperature of Activated Charcoals in Dry Air." Nucl. Safety, 8(1), 41 (1966).

16. J. T. Gallagher and H. Harker. "Reaction of Carbon with Oxidizing Gases: Catalysis by Compounds of Iron, Cobalt, and Nickel." Carbon 2, 163 (1964).

17. D. R. Muhlbaier. Nondestructive Test of Carbon Beds for Reactor Confinement Applications - Final Progress Report: January-June 1966, USAEC Report DP-1082, E. I. du Pont de Nemours and Co., Savannah River Laboratory, Aiken, S. C. (1966).

18. D. R. Muhlbaier. Nondestructive Test of Carbon Beds for Reactor Confinement Applications - Progress Report: October 1964 - January 1966. USAEC Report DP-1053, E. I. du Pont de Nemours and Co., Savannah River Laboratory, Aiken, S. C. (1966).

19. Ibid., July - September 1964. DP-950 (1965).

20. Ibid., April - June 1964. DP-920 (1964).

21. Ibid., January - March 1964. DP-910 (1964).

22. A. H. Peters and D. R. Muhlbaier. Nondestructive Test of Carbon Beds for Reactor Containment Applications - Progress Report: June 1962 - December 1963. USAEC Report DP-870, E. I. du Pont de Nemours and Co., Savannah River Laboratory, Aiken, S. C. (1964).

23. John W. Hassler. Activated Carbon. Chemical Publishing Co., Brooklyn, N. Y. (1961).

24. John J. Schantz and Richard H. Parry. "The Activated Carbon Industry." Ind. and Eng. Chem. 54(12), 24 (1962).

25. C. L. Mantell. Industrial Carbon - Its Elemental, Absorptive, and Manufactured Forms. D. Van Nostrand Co., Inc., Second Edition, New York (1947).

26. L. C. McCabe. Air Pollution. Proc. of the U. S. Tech. Conf. on Air Polution, McGraw-Hill Co., New York (1952).

27. P. A. F. White. High Efficiency Air Filtration. London, Butterworths Ltd. (1964).

28. B. M. W. Trapnell. Chemisorption. New York Academic Press (1955).

29. M. M. Dubinin. "A Study of the Porous Structure of Active Carbons Using a Variety of Methods." Quarterly Reviews, 9, 101 (1955).

30. M. M. Dubinin. "The Porous Structure and Adsorption Properties of Active Carbons." Industrial Carbon and Graphite, Conf., London, 1957, Soc. of Chem. Ind., 219 (1958).

31. J. B. Morris, H. R. Diffey, B. Nicholls and C. H. Rumary. Removal of Low Concentrations of Iodine from Air on a Plant Scale. British Report AERE-R-3917, Atomic Energy Research Establishment, Harwell, Berkshire, England (1961).

32. R. D. Collins and A. E. J. Eggleton. "Control of Gas-Borne Activity Arising from Reactor Faults." A/CONF/.28/P/186 (1964).

33. J. W. Finnigan, R. E. Peterson, H. W. Lefevre, J. W. Culvahouse, and W. J. Friesen. Removal of Iodine Vapor from Gas Streams by Sorption on Charcoal. USAEC Report HW-26113, General Electric Co., Hanford Atomic Products Operation, Richland, Washington (1952).

34. E. C. Anderson, G. L. Wessman, and L. R. Zumwalt. Iodine Adsorption by Activated Charcoal at Low Partial Pressures. USAEC Report GA-1709, General Dynamics Corp., General Atomic Div. (1960).

35. L. M. Arnett and B. C. Rusche. "The Application of Iodine Adsorber Units to the HWCTR Containment System." Eighth Air Cleaning Conference - Oct 22-25, 1963. USAEC Report TID-7677, Division of Technical Information, Oak Ridge National Laboratory, Oak Ridge, Tenn., 248 (1964).

36. W. E. Browning and R. E. Adams. "Removal of Iodine and Volatile Iodine Compounds from Air Systems by Activated Charcoal." Nuclear Safety Program Semiannual Progress Report for Period Ending June 30, 1964. USAEC Report ORNL-3691, 70, Oak Ridge National Laboratory, Oak Ridge, Tenn. (1964).

37. J. D. McCormack. "Some Observations on Iodine Removal from Plant Streams with Charcoal." Eighth AEC Air Cleaning Conference - October 22-25, 1963. USAEC Report TID-7677, Division of Technical Information, Oak Ridge National Laboratory, Oak Ridge, Tenn., 35 (1964).

38. Prepared by Arthur D. Little, Inc. Halogen Collector Test Program. USAEC Report HW-65587, General Electric Co., Hanford Atomic Products Corp., Richland, Washington (1960).

39. R. E. Adams and W. E. Browning, Jr. Removal of Radioiodine from Air Streams by Activated Charcoal. USAEC Report ORNL-2872, Oak Ridge National Laboratory, Oak Ridge, Tenn. (1960).

40. R. E. Adams and W. E. Browning, Jr. Proposed Method for Removal of Radio-Iodine Vapor from Experiment Off-Gas System of the ORR. USAEC Report ORNL-CF-58-5-59, Oak Ridge National Laboratory, Oak Ridge, Tenn. (1958).

41. F. Hudswell, E. Furby, J. G. Simons, and K. L. Wilkinson. The Sorption and Desorption of Iodine. British Report AERE-R-3183, Atomic Energy Research Establishment, Harwell, Berkshire, England (1960).

42. R. O. Lingjaerde and L. Podo. A Study on the Trapping of Iodine at High Temperatures and on Activated Charcoal. (United Kingdom Atomic Energy Authority. Reactor Group. Atomic Energy Establishment, Winfrith, Dorset, England.) DP-Report-213, Project DRAGON (1963).

43. A. R. Bancroft, L. C. Watson, and R. Hewitt. Tests on Collectors to Remove Iodine from Ventilation Air. CRCE-981, AECL-1131 (1960).

44. F. Billard, G. Chevalier, and J. Pradel. Study of the Trapping of Iodine by Activated Charcoal. CEA-2237 (1962).

45. J. W. Walker and A. H. Peters. "The Use of Filters in Nuclear Reactor Containment." Mech. Eng. 85(9) 46 (1963) (TID-17548).

46. R. E. Adams and W. E. Browning, Jr. "Iodine Adsorption Studies. Maritime Reactor Program Annual Progress Report for Period Ending November 30, 1962. AEC Report ORNL-3416, Oak Ridge National Laboratory, Oak Ridge, Tenn. (1962).

47. R. E. Adams and W. E. Browning, Jr. Removal of Radioiodine from Air-Steam Mixtures. USAEC Report ORNL-CF-60-11-39, Oak Ridge National Laboratory, Oak Ridge, Tenn. (1960).

48. W. J. Megaw, R. C. Chadwick, A. C. Wells, and J. E. Bridges. "The Oxidation and Release of Iodine-131 from Uranium Slugs Oxidizing in Air and Carbon Dioxide." Reactor Science and Technology 15, 176 (1961).

49. A. W. Castleman, Jr. and F. J. Salzano. Current Studies of Fission Product Behavior at BNL. USAEC Report BNL-7423, Brookhaven National Laboratory, Upton, N. Y. (1964).

50. A. W. Castleman, Jr., I. N. Tang, and H. R. Munkelwitz. "Chemical State of Iodine Released from Irradiated Fuels Into Steam." International Symposium on Fission Product Release and Transport Under Accident Conditions - April 5-7, 1965. CONF-650407, 1119 (1965).

51. W. J. Megaw and F. G. May. Behavior of Iodine Released in Reactor Containers. British Report AERE-R-3781, Atomic Energy Research Establishment, Harwell, Berkshire, England (1961).

52. A. C. Chamberlain, A. E. J. Eggleton, W. J. Megaw, and J. B. Morris. "Physical Chemistry of Iodine and Removal of Iodine from Gas Streams." Reactor Science and Technology (Journal of Nuclear Energy Parts A/B) 17, 519 (1963).

53. M. E. Davis and W. E. Browning, Jr. Separation and Identification of Volatile Iodine Compounds by Fractional Sublimation and Mass Spectrometer Analysis. USAEC Report ORNL-TM-1009, Oak Ridge National Laboratory, Oak Ridge, Tenn. (1964).

54. W. E. Browning, Jr. Characterization and Control of Accident-Released Fission Products. USAEC Report ORNL-3691, Oak Ridge National Laboratory, Oak Ridge, Tenn. (1964).

55. B. F. Roberts, W. E. Browning, Jr., R. P. Shields, and C. E. Miller. "Effects of Atmosphere on Behavior of Fission Products Released by In-Pile Melting of UO_2." Transactions of American Nuclear Society 8(1) 139 (1965).

56. D. H. F. Atkins, and A. E. J. Eggleton. Iodine Compounds Formed on Release of I-131. British Report AERE-M-1211, Atomic Energy Research Establishment, Harwell, Berkshire, England (1963).

57. R. D. Collins. "Trapping Radioiodine." Nucleonics 23(9), 7 (1965) (Letter).

58. D. A. Collins, R. Taylor, and L. R. Taylor. "The Removal of Methyl Iodide from Reactor Effluent Gases." International Symposium on Fission Product Release and Transport Under Accident Conditions - April 5-7, 1965. CONF-650407, 853 (1965).

59. W. E. Browning, Jr., et al. "Retention of Radioiodine and Methyl Iodide by Activated Carbons." Reactor Chemistry Division Annual Progress Report for Period Ending December 31, 1965. USAEC Report ORNL-3913, Oak Ridge National Laboratory, Oak Ridge, Tenn., 142 (1966).

60. G. W. Parker and Alberto Ferreli. "Parametric Studies of Retention of Methyl Iodide by Coconut Charcoal." Nucl. Safety Program Semiann. Prog. Report - June 30, 1965. USAEC Report ORNL-3843, Oak Ridge National Laboratory, Oak Ridge, Tenn., 66 (1966).

61. G. W. Parker, et al. "Retention of Methyl Iodide by Charcoal Under Accident Conditions." Nuclear Safety Program Semiann. Prog. Report - December 31, 1965. USAEC Report ORNL-3913, Oak Ridge National Laboratory, Oak Ridge, Tenn., 66 (1966).

62. J. R. Beattie. "An Assessment of Environmental Hazards from Fission-Product Release." United Kingdom Atomic Energy Authority, AHSB(S)R-64, Authority Health and Safety Branch, Risley, Warrington, Lancashire, England (1961).

63. W. B. Cottrell and A. W. Savolainen. U. S. Reactor Containment Technology I. USAEC Report ORNL-NSIC-5, Oak Ridge National Laboratory, Oak Ridge, Tenn., 590 (1965).

64. W. G. Berl. "Survey of Current Fire Research Activities." Symposium on Fire Control Research, National ACS Meeting, Chicago, Ill. Sept. 3-8, 1961.

65. S. W. Churchill, R. W. Kruggel, and J. C. Brier. "Ignition of Solid Propellants by Forced Convection." A.I.Ch.E. J. 2, 568 (1956).

66. D. H. Bangham and D. T. A. Townend. "The Combustion of Carbon: An Outline of the Work of B.C.U.R.A. Laboratories 1939-49." J. chim phys. 47, 315 (1950).

67. B. Ghosh and A. A. Orning. "Igniting Pulverized Coal." Ind. Eng. Chem. 47, 117 (1955).

68. C. H. Long and R. J. Wheeler. Ignition Temperature and Reactivity of Double-Screened Coals. ASME-Paper 64-WA/FU-1 for meeting Nov. 29-Dec. 4, 1964.

69. C. H. Bamford, J. Crank, and D. H. Malon. "The Combustion of Wood." Proc. Cambridge Phil. Soc. 42, 166 (1946).

70. D. A. Frank-Kamenetskii. Diffusion and Heat Exchange in Chemical Kinetics. Translated from Russian by N. Thon. Princeton: Univ. Press (1955).

71. T. Kinbara and K. Akita. "An Approximate Solution of the Equation for Self-Ignition." Combustion and Flame 4, 173 (1960).

72. D. Gross and A. F. Robertson. "Self-Ignition Temperature of Materials from Kinetic-Reaction Data." J. Res. Nat. Bur. Std. 61, 413 (1958).

73. P. H. Thomas and P. C. Bowes. "Some Aspects of the Self-Heating and Ignition of Solid Cellulosic Material." British J. of Applied Physics 12, 222 (1961).

74. Michael G. Zabetakis. "Flammability Characteristics of Combustile Gases and Vapors." U. S. Bureau of Mines Bulletin 627 (1965).

75. N. P. Setchkin. "Self-Ignition Temperatures of Combustible Liquids." J. Res. Nat. Bur. Std. 53, 49 (1954).

76. R. E. Adams and W. E. Browning, Jr. Estimate of the Probability and Consequences of Ignition of the HRT Charcoal Beds. USAEC Report ORNL-CF-58-6-6, Oak Ridge National Laboratory, Oak Ridge, Tenn. (1958).

77. H. B. Piper. "Engineered Safeguards." Nuclear Safety 6, (2) 201 (1964).

78. S. S. Penner and B. P. Mullins. Explosions, Detonations, Flammability, and Ignition. Pergamon Press (1959).

79. W. A. Affens, J. E. Johnson, and H. W. Carhart. "Effect of Chemical Structure on Spontaneous Ignition of Hydrocarbons." J. Chem. Eng. Data 613 (1961).

80. "Tentative Method of Test for Autogenous Ignition Temperatures of Petroleum Products," ASTM Designation: D 286-58T, 1958. 1966 Book of ASTM Standards Part 17 (1966).

81. H. L. Callendar. "Dopes and Detonation." Engineering 123, 147 (1927).

82. C. Moureu and C. Dufraisse. "Autoxidation and Antioxygen Action." Compt. Rend. 185, 1545 (1927).

83. J. S. Lewis. "Low Temperature Oxidation of Hydrocarbons." J. Chem. Soc. 1555 (1927).

J. S. Lewis. "Low-Temperature Oxidation. Part II. The Ignition of Some Hydrocarbons in Oxygen." J. Chem. Soc. 58 (1930).

J. S. Lewis. "Low Temperature Oxidation. Part III. The Lag in Ignition of Some Hydrocarbons." J. Chem. Soc. 2241 (1930).

84. L. C. Gibbons, H. C. Barnett, and M. Gerstein. "Effect of Molecular Structure on Combustion Behavior." Ind. Eng. Chem. 44 (10), 2150 (1954).

85. J. E. Johnson, J. W. Crellin, and H. W. Carhart. "Autoignition Properties of Certain Diesel Fuels." Ind. Eng. Chem. 44 (7), 1612 (1952).

86. B. Lewis, and G. Von Elbe. Combustion, Flames, and Explosion of Gases, 2nd Edition, N. Y. Academic Press (1961).

87. G. J. Minkoff and C. F. H. Tipper. Chemistry of Combustion Reactions. London: Butterworth Co. (1962).

88. S. M. Grenensky. "A Problem in Self-Heating of a Spherical Body." J. Res. Nat. Bur. Std. 59, 79 (1957).

89. P. H. Thomas. "On the Thermal Conduction Equation for Self-Heating Materials with Surface Cooling." Trans. Faraday Society 54, 60 (1958).

90. P. H. Thomas. "Some Approximations in the Theory of Self-Heating and Thermal Explosions." Trans. Faraday Society 56, 833 (1960).

91. P. H. Thomas. "Effect of Reactant Consumption on the Induction Period and Condition for a Thermal Explosion." Proc. Roy. Soc. (London) A262, 192 (1961).

92. J. Griswold. Fuels, Combustion, and Furnaces, 1st Ed. New York, London, McGraw-Hill Book Company, New York (1946).

93. H. Marsh, T. E. O'Hair, and W. F. K. Wynne-Jones. "Oxidation of Carbons and Graphite by Atomic Oxygen: Kinetic Studies." Trans. Faraday Society 61, Part 2, 274 (1965).

94. X. Duval. "Reactions of Graphite with Oxygen." J. chim. phys. 58, 3 (1961).

95. E. Wicke. "Primary Processes in the Burning of Graphitic Carbon." Z. Elektrochem. 56, 414 (1952).

96. W. V. Loebenstein, and N. L. Pennington. "Fundamental Aspects of the Reaction of Oxygen with Carbon Adsorbents." National Bureau of Standards Research Paper RP 2009, 43, 87 (1949).

97. R. H. Savage. "Physically and Chemically Adsorbed Films in the Lubrication of Graphite Sliding Contacts. Ann. N. Y. Acad. Sci. 53 862 (1951).

98. F. J. Long and K. W. Sykes. "The Mechanism of the Steam-Carbon Reaction." Proc. Roy. Soc. (London) A193, 377 (1948).

99. G. R. Hennig. "Surface Reactions of Single Crystals of Graphite." J. chim. phys. 58, 12 (1961).

100. W. S. Horton. "Oxidation Kinetics of Pyrolytic Graphite." Proc. Conf. Carbon, 5th, Univ. Park, Pa., 1961, 233 (1963).

101. N. R. Laine, F. J. Vastola, and P. L. Walker, Jr. "The Importance of Active Surface Area in the Carbon-Oxygen Reaction." J. Phys. Chem. 67, 2030 (1963).

102. P. L. Walker, F. Rusinko, and L. G. Austin. "Carbon-Oxygen Reaction." Advan. Catalysis 11, 133 (1959).

103. D. Graham. "Geometric Heterogeneity in the Adsorption of Nitrogen on Graphitized Carbon Surfaces." J. Phys. Chem. 61, 1310 (1957).

104. N. R. Laine, F. J. Vastola, and P. L. Walker, Jr. "The Role of the Surface Complex in the Carbon-Oxygen Reaction." Proc. Conf. Carbon, 5th, Univ. Park, Pa., 1961 2, 211 (1963).

105. W. R. Smith and M. H. Polley. "The Oxidation of Graphitized Carbon Black." J. Phys. Chem. 60, 689 (1956).

106. F. K. Earp and M. W. Hill. "Oxidation of Carbon and Graphite." Industrial Carbon and Graphite Conf., London, 1957. Society of Chemical Industry, 326 (1958).

107. J. D. Watt and R. E. Franklin. "Changes in the Structure of Carbons During Gaseous Oxidation." Industrial Carbon and Graphite Conf., London, 1957. Society of Chemical Industry (1958).

108. H. Marsh, T. E. O'Hair, and R. Reed. "Oxidation of Carbons and Graphite by Atomic Oxygen: An Electron Microscope Study of Surface Changes." Trans. Faraday Society 61, 285 (1965).

109. W. F. Wolff. "The Structure of Gas-Absorbent Carbon." J. Phys. Chem. 62, 829 (1958).

110. R. E. Jones and D. T. A. Townend. "The Role of the Peroxidic Complex in the Low-Temperature Oxidation of Carbon." J. chim. phys. 47, 348 (1950).

111. A. C. Stern. Air Pollution. New York: Academic Press, (1962).

112. Walter Kwei-Yuan Tang. "Effect of Inorganic Salts on the Pyrolysis, Ignition, and Combustion of Wood, Cellulose, and Lignin." Dissertation Abstracts 25 2405, (1964-65).

113. F. J. Long and K. W. Sykes. "The Catalysis of the Oxidation of Carbon." J. chim. phys. 47, 361 (1950).

114. J. F. Rakszawski, F. Rusinko, Jr., and P. L. Walker, Jr. "Catalysis of the Carbon-Carbon Dioxide Reaction by Iron." Proc. Conf. Carbon, 5th, Univ. Park, Pa., 1961 2, 243 (1963).

115. J. Taylor and F. Pollok. "Effect of Various Chemicals, More Particularly Sodium Bicarbonate, on the Ignition of Cellulose, Coal Dust, and Activated Charcoal." Fuel 28, No. 4, 77 (1949).

116. H. G. Crone. "Modes of Burning of Solid Fuel." J. chim. phys. 47, 563 (1950).

117. Ryo Kiyama and Jiro Osugi. The Ignited Combustion of Dispersed Powders. J. Chem. Soc., Japan 72,982 (1951).

118. K. Bratzler. "Auto-Ignition of Carbon as a Process Problem. A Contribution to Operating Safety in Solvent Recovery Units." Chem.-Ing.-Tech. 28, 569 (1956).

119. N. Boden, C. F. Cullis, and A. Fish. "Spontaneous Ignition in Carbon Adsorption Beds." J. Appl. Chem. 12, 145 (1962).

120. F. J. Woods and J. E. Johnson. "Ignition and Combustion Properties of Activated Carbon Containing Adsorbed Hydrocarbons." NRL-6090 (1964).

121. G. W. Bridger and H. Appleton. "Inhibition of the Combustion of Carbon and Carbon Monoxide." J. Soc. Chem. Ind. (London) 67, 445 (1948).

122. E. Mertens. "The Primary Combustion of Carbon." J. chim. phys. 47, 353 (1950).

123. F. W. Baetke. "The Reaction Mechanism of Combustion of Carbon at Atmospheric Pressure." Z. Elektrochem. 55, 655 (1951).

124. J. R. Arthur. "Combustion of Carbon." Nature 157, 732 (1946).

125. J. R. Arthur and J. R. Bowring. "The Combustion of Carbon: Inhibition of Secondary Reactions by Volatile Reagents Added to the Oxidizing Gas." J. Chem. Soc. S1; cf. C.A. 43 (1949).

126. J. R. Bowring and H. C. Crone. "Rate of Combustion of Carbon." J. chim.phys. 47, 543 (1950).

127. J. R. Arthur. "Reactions Between Carbon and Oxygen." Trans. Faraday Soc. 47, 164 (1951).

128. A. R. G. Brown, A. R. Hall, and W. Watt. "Density of Deposited Carbon." Nature (London) 172, 1145 (1953).

129. A. B. McIntosh, W. G. O'Driscoll, and C. T. Y. Zack. "Compatibility Problems of Graphite Moderators." Industrial Carbon and Graphite Conf., London, 1957. Society of Chemical Industry, 519 (1958).

130. R. J. Day, P. L. Walker, Jr., and C. C. Wright. "The Carbon-Oxygen Reaction at High Temperatures and High Gas-Flow Rates." Ind. Carbon and Graphite, Conference, London 334, (1958).

131. F. L. Browne and W. K. Tang. "Thermogravimetric and Differential Thermal Analysis of Wood and of Wood Treated with Inorganic Salts During Pyrolysis." National ACS Meeting Chicago Sept. 3-8, 1961.

132. K. G. Falk. "Ignition Temperature of Hydrogen-Oxygen Mixtures." J. Amer. Chem. Soc. 28, 1517 (1906).

K. G. Falk. "Ignition Temperature of Gaseous Mixtures." J. Amer. Chem. Soc. 29, 1536 (1907).

133. H. F. Coward. "Ignition Temperatures of Gases. Concentric Tube Experiments of (the late) Harold Baily Dixon." J. Chem. Soc. 98, 514 (1909). J. Chem. Soc., 1382 (1934).

134. D. M. Newitt and D. T. A. Townend. "Slow Combustion and Spontaneous Ignition of Higher Hydrocarbons." Congr. chim. ind., Comt. Rend. 18me congr., Nancy, Sept.-Oct. 1938, 1023.

135. H. Holm. "Kindling Temperatures, Especially of Fuels." Z. angew. Chem. 26, 273.

136. H. Moore. "Spontaneous Ignition Temperature of Liquid Fuels for Internal-Combustion Engines." J. Soc. Chem. Ind. 36, 109 (1917).

H. Moore. "Spontaneous Ignition Temperatures of Liquid Fuels." J. Inst. Petroleum Tech. 6, 186 (1920).

137. C. W. Sortman, H. A. Beatty, and S. D. Heron. "Spontaneous Ignition of Hydrocarbons - Zones of Nonignition." Ind. Eng. Chem. 33, No. 3, 357 (1941).

138. M. G. Zabetakis, A. L. Furno, and G. W. Jones. "Minimum Spontaneous Ignition Temperature of Combustibles in Air." Ind. Eng. Chem. 46, 2173 (1954).

139. S. Martin. "Diffusion-Controlled Ignition of Cellulosic Materials by Intense Radiant Energy." Tenth Symposium (International) on Combustion, The Combustion Institute, 877 (1965).

140. C. R. Brown. The Determination of the Ignition Temperatures of Solid Materials. Doctoral Thesis, Catholic University of America (1934).

141. Fire Protection Handbook, National Fire Protection Association, 12th Edition, (1962) Boston, Massachusetts.

142. J. D. Davis and J. F. Byrne. "An Adiabatic Method for Studying Spontaneous Heating of Coal." J. Am. Ceram. Soc. 7, 809 (1924).

143. J. J. S. Sebastian and M. A. Myers. "Coke Reactivity - Determination by a Modified Ignition Point Method." Ind. Eng. Chem. 29, 1118 (1937).

144. R. A. Sherman, J. M. Pilcher, and N. H. Ostborg. "A Laboratory Test for the Ignitibility of Coal." Am. Soc. Testing Materials; Preprint No. 96 (1941).

145. A. A. Ornig. "Reactivity of Solid Fuels." Ind. Eng. Chem. 36, 813 (1944).

146. J. L. Elder, L. D. Schmidt, W. A. Steiner, and J. D. Davis. "Relative Spontaneous Heating Tendencies of Coals." U. S. Bur. Mines, Tech. Paper 681 (1945).

147. A. A. Ornig, S. Mallov, and N. Neff. "Reactivity of Solid Fuels to Air and Oxygen." Ind. Eng. Chem. 40, 429 (1948).

148. L. Coppens, W. Fassotte, and W. Duhameau. "Tendency of Oxidation of Coals: Initial Ignition Temperature." Ann. Mines Belg., 678 (1961).

149. E. V. Alekseevskii and Ya. D. Musin. "Instrument for Determination of Ignition Temperature Kinetics of Oxidation of Activated Charcoal." J. Applied Chem. (U.S.S.R.) 18, 505 (1945).

150. W. V. Loebenstein, L. F. Gleysteen, and V. R. Deitz. "Kinetic Study of the Reaction of Carbon Adsorbents with Oxygen." J. of Research of the Nat. Bur. of Stds 42, 33 (1949).

151. W. H. Raskin and A. F. Robertson. "An Adiabatic Apparatus for the Study of Self-Heating of Poorly Conducting Materials." Rev. Sci. Inst. 25, No. 61, 541 (1954).

152. F. G. May. Comparison of Charcoals in Removing Iodine from Air-Streams at High Temperature. U.K.A.E.A. Research Group. AERE-R-4145, Atomic Energy Research Establishment, Harwell, England.

153. J. E. Bigelow, M. W. Belaga, R. J. Mulvihill. The Action of Oxygen on Activated Charcoal. USAEC Report AECD-3638, Massachusetts Inst. of Tech., Oak Ridge, Tenn. (1951).

154. R. K. Hilliard, C. E. Linderoth, and A. J. Scott. "Fission-Product Release from Overheated Uranium." Health Phys. 7, 1 (1961).

155. R. F. Taylor. "Absorption of Iodine Vapor by Aqueous Solutions." Chem. Eng. Sci. 10, 68 (1959).

156. V. Griffiths. The Removal of Iodine from the Atmosphere by Sprays. AHSB-S-R-45 (1962).

157. H. R. Diffy, C. H. Rumary, M. J. S. Smith, and R. A. Stinchcombe. Clean-Up in a Steam Suppression System. British Report AERE-R-4882, Atomic Energy Research Establishment, Harwell, England (1965).

158. F. G. May and J. B. Morris. Performance of the BEPO Scrubber for Iodine Removal. British Report AERE-M-1163, Atomic Energy Research Establishment, Harwell, England (1963).

159. A. C. Chamberlain, A. E. J. Eggleton, W. J. Megaw, and J. B. Morris. Removal of Radioactive Iodine Vapour from Air. British Report AERE-R-3412, Atomic Energy Research Establishment, Harwell, England (1960).

160. D. M. Paige, P. N. Kelley, and E. S. Grimmett. "Two Gas Cleaning Problems at the ICPP Site." USAEC Report TID-7551, Fifth AEC Air Cleaning Conference held at Harvard Air Cleaning Laboratory, Boston, June 24-27, 1957 (1959).

161. L. C. Watson, A. R. Bancroft, and C. W. Hoelke. Iodine Containment by Dousing in NPD-II. AECL-1130, Atomic Energy of Canada Ltd., Chalk River, Ont. (1960).

162. A. R. Bancroft, L. C. Watson, and R. Hewitt. Tests on Collectors to Remove Iodine from Ventilation Air. AECL-1131, Atomic Energy of Canada Ltd., Chalk River, Ont. (1960).

163. V. Griffiths. "Use of Sprays as a Safeguard in Reactor Containment Structures." Nucl. Safety 6(2), 186 (1965).

164. J. R. Bower, G. K. Cederberg, D. G. Sutherland, and V. W. Irvine. Reduction of Iodine Evolution during RaLa Processing. IDO-14419 (1957).

165. R. Dennis, L. Silverman, Y. Takashima, F. L. Muller, F. Stein, J. Fitzgerald, and P. Drinker. Air Cleaning Studies, Progress Report for July 1, 1960 to June 30, 1961. NYO-4818 (1962).

166. L. Silverman. "Foam and Diffusion Board Approaches to Containment of Reactor Releases." The Third Conference on Nuclear Reactor Chemistry, Gatlinburg, Tennessee, Oct. 9 through 11, 1962. TID-7641, 169 (1963).

167. R. E. Yoder, M. H. Fontana, and L. Silverman. "Foam Suppression of Radioactive Iodine and Particulates." (Harvard Univ., Air Cleaning Lab.), Boston, Mass. Feb. 25, 1964. NYO-9324, HACL-100 (1964).

168. R. Dennis and L. Silverman. Air Cleaning Studies Progress Report, January 1, 1963 to June 30, 1964. NYO-841-1, Air Cleaning Laboratory, School of Public Health, Harvard Univ., Boston, Mass. (1964).

169. R. McNabney and A. N. Lyon. The Removal of Iodine from Gas Streams by Reaction with Silver in Packed Towers. ARSC-28 (1949).

170. R. McNabney. Absorption of Iodine Vapor. U. S. Patent 2,795,482 (1957).

171. J. F. W. Smith and R. H. A. Crawley. "Removing Radioactive Iodine from Gaseous Effluent." Nucl. Eng. 6, 428 (1961).

172. L. L. Burger. Thermodynamics of Iodine Removal with the Silver Reactor. USAEC Report HW-38311 (Rev), General Electric Co., Hanford Atomic Products Operation, Richland, Wash. (1955).

173. P. H. Harley. HRT Mockup Iodine Removal and Recombiner Tests. USAEC Report ORNL-CF-58-1-138, Oak Ridge National Laboratory, Oak Ridge, Tenn. (1958).

174. R. C. Koch and G. L. Grandy. Retention Efficiencies of Silver-Coated Filter Materials for Iodine. USAEC Report NSEC-13, Nuclear Science and Engineering Corp, Pittsburgh, Pa. (1958).

175. R. E. Adams and W. E. Browning, Jr. Removal of Radioiodine from Air Streams by Activated Charcoal. USAEC Report ORNL-2872, Oak Ridge National Laboratory, Oak Ridge, Tenn. (1960).

176. L. Silverman, R. Dennis, E. Kristal, and F. Stein. "Iodine Collection Studies." Sixth AEC Air Cleaning Conference, Idaho Falls, Idaho, July 7-9 (1959). USAEC Report TID-7593 (1960).

177. J. B. Morris, R. A. Stinchcombe, M. D. Gray, and D. H. Logsdail. "Removal of Iodine Vapour from Air by Metallic Copper." British Report AERE-R-4114, Atomic Energy Research Establishment, Harwell, England (1962).

178. A. C. Chamberlain. "Experiments on the Deposition of Iodine-131 Vapour onto Surfaces from an Airstream." The Philosophical Magazine 44, 1145 (1953).

179. F. Hudswell, E. Furby, J. G. Simons, and K. L. Wilkinson. The Sorption and Desorption of Iodine. British Report AERE-R-3183, Atomic Energy Research Establishment, Harwell, England (1960).

180. J. B. Morris and C. H. Rumary. Tests on Adsorbers for Iodine at Low Concentration. British Report AERE-R-4219, Atomic Energy Research Establishment, Harwell, England (1963).

181. R. M. Watkins, D. D. Busch, and L. R. Zumwalt. "Trapping Fission Products in the HTGR." Nuclear Engineering 6, 427 (1961); GA-1990 (1961).

182. M. A. Wahlgren and W. W. Meinke. "Molecular Sieves Absorb Iodine-131 from Air." Nucleonics 15(9), 156 (1957).

183. A. E. Symonds, Jr. Adsorption System for Radioactive Iodine. USAEC Report DP-646, E. I. du Pont de Nemours and Co., Savannah River Laboratory, Aiken, S. C. (1961).

184. Nuclear Safety Program Semiannual Progress Report for Period Ending June 30, 1963. USAEC Report ORNL-3483, Oak Ridge National Laboratory, Oak Ridge, Tenn. (1963).

185. C. J. Barton, G. E. Creek, G. W. Parker, and M. E. Davis. Reactor Chemistry Division Annual Progress Report for Period Ending Jan. 31, 1964. USAEC Report ORNL-3591, Oak Ridge National Laboratory, Oak Ridge, Tenn. (1964).

186. J. F. W. Smith and R. H. A. Crawley. The Properties of Some Iodine Adsorbers at Elevated Temperatures. AERO/CONF/.6/1960.

187. R. B. Andrews, V. J. Emerson, and F. Hudswell. Sorption of Iodine by Solids. AERO/CONF/.5/1960.

APPENDIX A

LITERATURE REVIEW

IODINE REMOVAL ON ACTIVATED CARBON

The atoms, molecules, or ions of a substance are held together in a solid by forces that exist throughout the solid and at the surface. The forces at the surface are available for binding molecules of foreign matter that come sufficiently close. Because of this binding force, a gas, vapor, or liquid tends to adhere to the surface of a solid. This behavior is called adsorption.

Activated carbon is a material that has been treated to produce a large internal surface, usually 1000 m^2/g.(23-27) The large internal surface area contains special sites where the binding forces are strong enough to bind an atom, molecule, or ion that comes sufficiently close.(28-30) In the case of iodine, a chemical bond between carbon and iodine is formed and the iodine is held securely.

The rate of adsorption of iodine is a function of the rate at which iodine molecules arrive at the active sites, the fraction of iodine molecules that adhere after arrival, and the rate at which iodine molecules escape.(14)

Activated carbon from both coconut shell and bituminous coal has been used to remove radioiodine from gas streams in the nuclear industry.(12-14,31-35) Carbon from coconut shell provides better retention of iodine and methyl iodide(36) but has a lower ignition temperature than carbon from bituminous coal.

Adsorption on carbon is affected by atmospheric conditions. The efficiency of carbon filters for adsorption of elemental radioiodine is greater than 99.9% under a wide variety of adverse conditions,(13,14,35,37-44) such as extended service(11,14,45) and steam.(46,47) Impurities in air such as NO_2 and SO_2 are also adsorbed. Water vapor is not strongly adsorbed, because it is a polar compound, and the intermolecular forces promote the attraction of water molecules for each other rather than for the carbon. In reactor confinement, sufficient carbon is used so that only a small fraction of its iodine adsorptive capacity is utilized.(14)

Desorption of iodine from unimpregnated coconut carbon is insignificant. At 250°C and an air velocity of 220 ft/min,(152) iodine desorbed from a 6-inch-deep bed at rates of 0.02-0.002 %/hr from coconut carbon, 0.3 %/hr from coconut carbon treated to raise its ignition temperature, and 0.7 %/hr from coal-based carbon. Although most of the iodine is released as elemental iodine,(48,49) some iodine may appear in other forms. Iodide (which is adsorbed efficiently), iodate, and periodate may be released.(50) Alkyl iodides (primarily methyl iodide),(51-56) which are not adsorbed efficiently by carbon, may be formed under some conditions.(1) Investigation of methods to improve the efficiency for methyl iodide led to a carbon impregnated with 5% triethylene diamine which removes methyl iodide with an efficiency of 99.99+% from humid air at 23°C.(57, 58) Methyl iodide is removed by reaction with the amine to form a methylated compound (the Menschutkin reaction).(59) Some of the amines tested were highly volatile and caused severe corrosion of the ductwork.

Commercial carbons, impregnated with several percent by weight* of iodine, will also remove methyl iodide(60,61) by a mechanism that is at least partly explained by isotopic exchange:

$$CH_3\,{}^{131}I + {}^{127}I \text{ (on carbon)} \rightarrow CH_3\,{}^{127}I + {}^{131}I \text{ (on carbon)}$$

The usefulness of impregnated carbons for trapping methyl iodide has been demonstrated at 24°C and relative humidities up to 71%.(61) These carbons probably will be satisfactory for even more severe conditions.

ALTERNATE FORMS OF CARBON

Carbon exists in allotropic forms, some of which may have application in confinement. One form is pyrolytic graphite foam, a product of the General Electric Company that has outstanding high-temperature properties. Porous carbon (10 to 100 pores per linear inch) is coated by passing hydrocarbon vapor over the surface at low pressure and high temperature (800 to 2800°C). Pyrolysis occurs and pyrolitic carbon is deposited on the surface. By proper control of temperature, pressure, gas flow, and starting material, a coating of 0.5 to 1.7 mils can be deposited.

IGNITION OF CARBON

Iodine that could be released in a nuclear reactor would produce a significant amount of heat by radioactive decay. The most important isotope in the production of heat for most applications is ^{131}I, which has an 8-day half-life. The equilibrium activity of ^{131}I is 25 kCi/MW.(62) The total heat generated is 7.8×10^7 Btu/g.(63)

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* Type 727, Barnebey-Cheney Co.; Mine Safety Appliances Co., No. 85851.

The temperature of carbon that has adsorbed radioiodine will increase because of the decay heat. Temperature is dependent on:

- Quantity and distribution of iodine, and
- Rate and temperature of air flow through the carbon; at very low air flows the carbon might be heated to its ignition temperature.

Ignition temperature received attention in commerce before the advent of the nuclear industry. The study of ignition temperature and the development of safe storage procedures have continued,(64) and now the technology is being applied to the ignition of propellants in the space industry.(65)

The early investigation of ignition temperature was aimed at efficient combustion of coal.(66-68) The ignition of wood(69-71) and fiberboard(72,73) has been studied to evaluate their fire hazard in buildings. The ignition of combustible vapors(74) and liquids(75) was studied to provide safe handling procedures.

In the nuclear industry, work on the ignition of carbon began in 1958,(76) and the current status of filter systems was summarized in 1964.(77) A review of ignition temperature information was published recently.(78)

DEFINITIONS OF IGNITION TEMPERATURE

Although standard definitions and methods of determination have been established for density, viscosity, and solubility there has been no general agreement on the definition or determination of ignition temperature. Consequently, determinations by different investigators have varied widely both in numerical value and significance, resulting in confusion and difficulty in the interpretation and practical application of information.(75)

Although ignition characteristics of a material are a function of some actual property, measurement of that property by any means available is greatly affected by ambient conditions. Hence, any practical definition of ignition temperature must be based on a careful definition and standardization of the test apparatus and procedure.

Ignition temperature is usually defined as the temperature at which the rate of chemical heating exceeds the rate of heat loss from a system. According to this definition, based on classical reaction rate theory, it is that temperature to which a combustible mixture must be raised so that the rate of heat evolved by the exothermic oxidation reactions of the system will just overbalance the rate at which heat is lost to the surroundings.(79)

Sometimes it is called the Spontaneous Ignition Temperature (SIT) to emphasize that it is the lowest temperature at which a substance will ignite in air without the aid of a spark of flame.(79)

Others have termed this property Autogenous Ignition Temperature,(80) Self-Ignition Temperature,(75) Temperature of Initial Combustion (TIC),(81,82) and Critical Inflection Temperature.(83)

Ignition temperature is markedly dependent on the method and apparatus employed for its determination.(79) Results obtained by one method are not easily related to results obtained by another method.(84) In early work, and even as late as 1954,(75,85) the criteria used to indicated ignition, i.e., visible or audible combustion, were quite subjective.(79)

Spontaneous ignition is subject to homogeneous catalysis.(86,87) Impurities can accelerate the rate of oxidation, which results in ignition at reduced temperature.

MECHANISM OF THE CARBON-OXYGEN REACTION

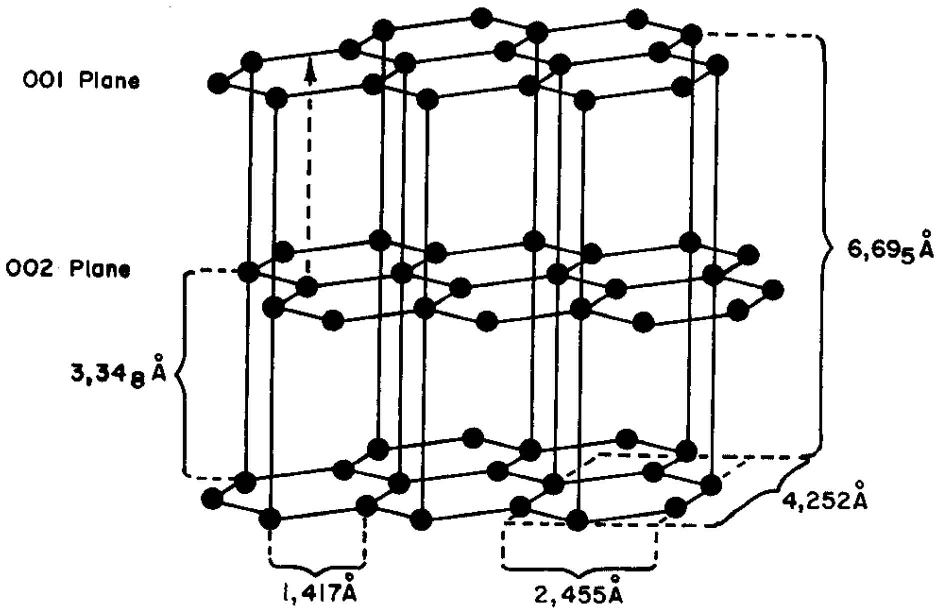
The mechanism for the reaction of carbon with oxygen begins with the adsorption of oxygen. Carbon adsorbs oxygen at room temperature. If the carbon is warmed, oxygen is released as carbon dioxide. The temperature for oxidation depends primarily on the material, its impurities, particle size, internal surface area, and the air velocity. A theoretical analysis(88-91) was made of ignition temperature.

Chemical reactions involved in the oxidation of carbon are:

$$\begin{aligned} C + O_2 &\rightarrow CO_2 \\ 2C + O_2 &\rightarrow 2CO \\ 2CO + O_2 &\rightarrow 2CO_2 \\ C + CO_2 &\rightarrow 2CO. \end{aligned}$$

The reaction between carbon and the adsorbed oxygen is heterogeneous,(92) and is first order for both atomic(93) and molecular oxygen(94) at temperatures below 1300°C. The energy of activation is 44 kcal. Below 1000°C, the reaction kinetics control the oxidation rate.

The mechanism for the oxidation of carbon is based on experimental work under a variety of conditions.(95,96) Carbon consists of a hexagonal arrangement of atoms in parallel planes, as shown in the following figure. The carbon atoms at the edges of crystallites have unpaired electrons that are available to form bonds with adsorbed oxygen.(97) These edges are more reactive toward oxygen(98) than the basal plane surfaces where electrons form chemical bonds between adjacent carbon atoms.(99,100) Impurities in carbon diffuse to, and concentrate at, crystallite edges during high temperature treatment.(101)



Small amounts of these impurities can strongly catalyze or inhibit the reaction with oxygen. The region occupied by the reactive edges is the active surface area (ASA),(102) which is a small fraction of the total surface area (TSA). The TSA is measured by the adsorption of nitrogen at 78°K(103) or by prolonged exposure to oxygen at 300°C to form a carbon-oxygen complex on the surface.(104) As oxidation occurs, the crystallites increase in size and become oriented to yield a surface composed of more basal planes.(105-110)

The effect of impurities has been studied extensively. Most impurities catalyze combustion and lower the ignition temperature.(16,111-113) Only a few impurities inhibit oxidation and raise the ignition temperature.

Measurements with the thermobalance showed that the ignition temperature decreased with an increase in cationic impurities.(102,114) Alkali metals had the strongest effect, especially potassium. The effectiveness of cations is:

$$K > Na > Cu > Ag > Zn > Ca.$$

The ignition temperature decreased linearly with the square root of the amount of impurity.(115) Arthur and Bowring showed that salts such as $CuCl_2$, $ZnCl_2$, KCl, KI, NaCl, and Na_2CO_3 increased the rate of oxidation and increased the ratio of CO_2 to CO in the exhaust.(116) The effectiveness of salts tested by Kiyama and Osugi is(117):

$$KOH > NaOH > K_2CO_3 > Na_2CO_3 > NaHCO_3 > Ca(OH)_2.$$

Removal of impurities that catalyze combustion provides a carbon with a higher ignition temperature. Such a carbon, Type 592, is available commercially.

Ignition temperatures as low as 50°C(118-120) are reported for carbon that contained adsorbed solvents and hydrocarbons. The low temperatures are attributed to the ignition of organic matter and not to any catalytic properties.

Certain compounds of chlorine, phosphorous, or boron inhibit the combustion of carbon and provide higher ignition temperatures, due to the change in the products of combustion. Certain compounds of chlorine, phosphorous, and boron promote the formation of carbon monoxide instead of carbon dioxide. These additives interrupt the chain reaction that is responsible for the oxidation of carbon monoxide to carbon dioxide. The heat of reaction for carbon monoxide is much lower than that for carbon dioxide, which leads to a lower combustion rate and higher ignition temperature. Chlorine, hydrogen chloride,(121) and carbon tetrachloride(122,123) inhibit the combustion of carbon and promote the formation of carbon monoxide. Similar behavior with carbon has been observed for phosphorous compounds, such as phosphorous oxychloride,(124-130) and with wood impregnated with dibasic ammonium phosphate or sodium tetraborate.(131) The effect of a variety of compounds on the rate of combustion of graphite is summarized in the following table.

Rate of Oxidation of Graphite Impregnated with Various Salts(106)

Impregnant	Concentration, wt %	Rate of Oxidation at 600°C, $g/(m^2)(hr)$
None	-	30
Zinc borate	1.9	7.4
Zinc borate	1.3	16
Potassium borate	1.9	72
Calcium borate	1.0	80
Cobalt sulfate	1.2	30
Cobalt sulfate	2.8	75
Sodium silicate	1.7	380
Sodium tungstate	9.4	>2000
Sodium telurate	1.2	>2000
Sodium selenate	0.24	>2000
Sodium arsenate	5.0	>2000
Barium bromide	2.1	>2000
Barium silicate	1.1	>2000
Lead silicate	3.4	>2000
Bismuth trichloride	3.0	>2000
Strontium chloride	0.5	2000
Vanadium pentoxide	9.0	2000
Arsenious oxide	4.5	2000
None	-	25
Zinc phosphate	3.1	1.4
Sodium phosphate	2.0	1.3
Sodium phosphate	4.0	1.2
Sodium phosphate	5.4	9.0
Sodium phosphate	2.6	11
Sodium phosphate	3.0	16
Sodium pyrophosphate	3.9	6.2
Sodium pyrophosphate	3.0	110
Potassium phosphate	3.4	2.7
Aluminum phosphate	4.0	7.5
Aluminum phosphate	5.4	1.3
Copper phosphate	2.1	2.2
Phosphoric acid	6.5	7.3

MEASUREMENT OF IGNITION TEMPERATURE

Ignition properties have been investigated for at least 60 years.(132) Methods used in the determination of ignition temperature may be grouped into two general types, the "rising temperature" method and the "constant temperature" method.(75) In the former, the combustible mixture is introduced into the apparatus and its temperature is continuously increased until positive evidence of ignition is observed. Self-ignition temperature is usually taken as: a) the point at which the time-temperature curve for the material shows a definite increase in slope or, b) as the point of intersection between the time-temperature curve for the material and that for the heating medium. In the constant-temperature method of determination, the apparatus is preheated to a constant temperature before the specimen is introduced; no adjustment of heat input to the apparatus is made after the specimen is introduced. The test is repeated with fresh specimens at different initial temperatures until the lowest temperature is established at which ignition is produced.

A variety of heating methods have been used: rapid adiabatic compression,(132) electric resistance furnace,(133,134) hot porcelain surface,(135) molten metal,(136) hot metal block,(137) hot Pyrex Erlenmeyer flask,(138) intense radiant heat,(139) and several other methods.(140)

Published reports state that there is an optimum air velocity for measurement of the minimum self-ignition temperature.(141) The optimum velocity varies with the type of material and the intensity of oxidation. Below optimum velocity, the products of oxidation are not removed rapidly and the combustion of the material is retarded. At low velocity, elevated ignition temperatures are usually observed. Above optimum velocity there is excessive cooling of the reacting masses, and elevated ignition temperatures are observed.

IGNITION TEMPERATURE APPARATUS

The SRL apparatus for measuring the ignition temperature of activated carbon is patterned after equipment that was designed to measure the ignition temperature of coke and coal,(142-148). A constant-temperature oven was frequently used to heat the sample. The large thermal inertia of such ovens caused difficulty in following rapid changes in the specimen temperature.

An early apparatus(149) was a vertical combustion tube in which the carbon was ignited; exhaust gases passed into an adsorption tube of barium hydroxide. The amount of carbon oxidized was determined by analysis of the adsorbent.

The first American apparatus was reported by the National Bureau of Standards in 1949.(150) Oxygen or an oxygen-nitrogen mixture at 91 ml/min was passed through a vertical 15-mm-diameter "Pyrex"* tube that contained 0.5 to 5 g of carbon. The tube fitted inside a brass block to ensure uniform distribution of heat from a vertical furnace around the block. The sample temperature was obtained from a thermocouple mounted in the block adjacent to the sample. The amount of CO_2 produced by the oxidation of carbon was determined by the amount of carbon dioxide adsorbed by an ascarite-anhydrone tube. Carbon monoxide from the oxidation was oxidized to CO_2 by "Hopcalite"** prior to adsorption.

An automatic furnace(151) was used to measure ignition temperature. The sample was brought to thermal equilibrium at a temperature below the ignition point. The furnace temperature was automatically increased and the weight changes of the sample were continuously monitored and recorded. The control system automatically stopped the operation after a preselected temperature was reached. Air was supplied at 200 ml/min, which was equal to 0.06 air change per minute. An internal fan circulated air around the sample.

Another apparatus(152) consisted of a preheater to warm the inlet air and a heated chamber that contained a carbon bed, 1.75 inches in diameter by 7 inches deep, supported by ceramic beads. The importance of loading the bed by the free fall of carbon from a funnel (to ensure dense packing and to avoid channeling) was emphasized. The bed contained 13 thermocouples to indicate a profile of the temperature prior to ignition.

Ignition temperature was measured at velocities from 94 to 360 ft/minute.(152) In this work two types of ignition temperature were reported. The actual ignition temperature is that temperature at which a runaway oxidation reaction would occur in a carbon bed of infinite area. Below this temperature, although some oxidation heating occurs, the bed temperature is stable and controllable. The temperature of the inlet air upon ignition of the seven-inch bed was always lower than the actual ignition temperature, sometimes by over 100°C, due to the heat of oxidation liberated in the bed. The lowest temperature of the inlet air at which ignition will occur in a bed of infinite area is called the effective ignition temperature.

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* Trademark of Corning Glass Works.

** Trademark of Mine Safety Appliance Co.

The ignition temperature apparatus used by the Barnebey-Cheney Company is a vertical "Inconel"* tube, 1-inch diameter by 2.5-feet long. The tube is enclosed in a vertical combustion furnace that is divided into three sections. The temperature of each section can be controlled independently.

Carbon that has been dried at 150°C is poured into the upper end of the "Inconel" tube to form a bed 3.5-inches deep. The sample is packed by the free fall of carbon onto an "Inconel" screen attached at the middle of the tube. New carbon is used for each test. An "Inconel" screen at the top of the bed prevents fluidizing by air that passes upward through the carbon.

Compressed air, dried by bubbling through concentrated sulfuric acid, passes through a rotameter to the bottom of the "Inconel" tube. A uniform air temperature is obtained by passing air over 3-mm-diameter quartz spheres that are packed in the bottom of the heated "Inconel" tube. Air temperature is increased at an average rate of 4.4°C/min by manually adjusting a controller. The temperature of the furnace adjacent to the carbon bed is regulated by a thermocouple at the top of the bed. The temperature 1/8-inch from the upstream face of the bed is measured by a thermocouple, and is recorded as a plot of temperature versus time. (This thermocouple is 0.025 inch OD to avoid air channels around the sheath and to improve sensitivity.) The ignition temperature is indicated by a sharp deviation in the nearly straight line temperature plot on the recorder. The estimated precision is about ±5°C.

EFFECT OF VARIABLES ON IGNITION TEMPERATURE

In a study of the variables that affect the ignition temperature of carbon, the Barnebey-Cheney Company, Columbus, Ohio(15) found that removal of oxidation catalysts in a proprietary manner increased the ignition temperature as much as 130°C. Both the normal carbon and the treated carbon had an adsorption efficiency of >99.9% for elemental iodine.(11)

Reduction of the ash content from 4 to 0.4% produces a linear increase of about 75°C in ignition temperature. These data were obtained by selecting carbons of different ash content and by impregnating carbon with impurities such as SiO_2 and Al_2O_3. The effect of ash content on the iodine adsorption properties of carbon has not been established.

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* Trademark of International Nickel Co.

As the internal surface area (degree of activation) is reduced, the ignition temperature increases. The effect of reduced internal surface area on the iodine adsorption efficiency of carbon has not been determined.

An increase in the particle size (from a mean particle diameter of 1 to 4 mm) produces a 30° rise in the ignition temperature because of the reduction of external surface area (for the same relative volume).

Barnebey-Cheney Co. also measured ignition temperatures in a quartz apparatus to eliminate interference from corrosion products. In such an apparatus the ignition temperature will rise because hydrogen, formed by the decomposition of moisture by hot carbon, is strongly adsorbed on carbon and inhibits the reaction of carbon with oxygen. The interaction of moisture and metal may release corrosion products that could catalyze ignition.

The effect on ignition temperature of air velocity through the carbon bed is complex. Most carbons show an increase in ignition temperature as the velocity increases. However, literature about the effect of velocity contains many inconsistencies. Some carbons show no change in ignition temperature as the velocity increases; other carbons show some maximum value for a certain velocity.

PUBLISHED IGNITION TEMPERATURE DATA

An ignition temperature of 362°C was reported for Columbia Type C* activated carbon (8 x 14 mesh)[153] in an oxygen atmosphere with essentially no flow.

An ignition temperature of 290°C was reported for Columbia Type G* activated carbon in oxygen at a face velocity of 2 and 4 ft/min[76] in a bed 0.5-inch in diameter by 5.75-inches long. The ignited carbon was completely extinguished by stopping the oxygen flow. The downstream movement of the combusion front was sufficiently slow that ignition could be detected and corrective action taken.

Ignition temperature of activated carbons manufactured in Great Britain is summarized in the following table.[152]

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* Product of Union Carbide Corporation.

Ignition Temperature of British Carbon(152)

Carbon	Description	Surface Area, m^2/g	Mesh Size	Ash, %	Effective* Ignition Temp., °C	Actual* Ignition Temp., °C	Face Velocity, ft/min
Coconut	SCII	1400	6 x 10	2.2	260	345	94
Coconut	SCII	1400	8 x 12	2.2	301	357	360
Coconut	208CAW	1200	8 x 12	1	433	515	360
Coconut	208C	1200	8 x 12	3	313	338	360
Coconut	207C	1000	12 x 25	2.5	310	355	360
Bituminous	CP	850	-	8.5	415	476	120
Bituminous	CP	850	-	8.5	442	497	360
Bituminous	CP	850	6 x 10	8.5	443	472	360
Bituminous	207B	1100	8 x 12	12	416	436	160

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* The effective ignition temperature is defined as the lowest temperature of the inlet air at which ignition will occur in a bed of infinite area. The actual ignition temperature is defined as that temperature at which a runaway oxidation would occur in a carbon bed of infinite area.

Ignition usually occurred at the center of the downstream face of the bed. In 3 out of 26 tests ignition occurred in the center of the upstream face. The depth of the bed was seven inches.

MECHANICAL SEPARATION

Although carbon beds have superior efficiency for the adsorption of iodine and are in most general use, mechanical separation has been investigated also.

Particulate Filters

Particulate filters are inefficient in removing radioactive iodine because a large fraction of the released iodine probably will exist as vapor. In laboratory tests, the efficiency of particulate filters for elemental iodine ranged from ~1(14) to 99%(154) of the iodine released.

Liquid Scrubbers and Foam Suppression

Removal of radioactive elemental iodine from gases by liquid scrubbers(155) or spray systems(156-158) is complicated by the various states in which iodine may appear.

Iodine adsorbed on particulates is not readily removed by a liquid scrubber. Carbon beds also fail to remove submicron particles efficiently. Such particulates are removed efficiently by a high efficiency particulate filter upstream of the scrubber on adsorption bed.

Iodine may react with organic matter to form alkyl iodides such as methyl iodide(1) that are not efficiently removed by a liquid scrubber.(159) Decontamination factors of only 1.3 were reported for iodine compounds.

Aqueous adsorbers are unsuited for exhaust gases at temperatures above 200°C because the heat will evaporate the water.

Liquid adsorbents are best suited to moderate air flows.(160-163) Expensive equipment is required to treat large air flows and provision must be made for safe disposal of the resulting large volume of radioactive liquid.

Carbon tetrachloride(164) is superior to the aqueous scrub solutions tested (sodium thiosulfate, sodium hydroxide, and sodium hypochlorite) but has the disadvantages of high volatility and high toxicity.

Aqueous chemical foams have been tested for confinement of iodine by the large reaction surface provided by the foam.(165-167) Iodine collection in tests was considerably less than predicted by theory. A major problem in applying the technique is the development of a foam that is compatible with chemical additives.(168) This system has the disadvantage of not being on-line and therefore must be activated rapidly during an accident.

NONCOMBUSTIBLE ADSORBENTS

Silver and silver compounds(169,170) react efficiently with iodine in air, especially at temperatures of 100-300°C,(171) to form silver iodide and silver iodate.(172) Iodine has been removed at 99.9% efficiency, and at temperatures up to 260°C. Silver iodide is stable at 350°C, but 78% is decomposed at 550°C. Silver surfaces are not completely reliable for iodine adsorption because impurities and an oxide surface film can cause unsatisfactory efficiencies.(173-176)

Clean, oxide-free copper reacts efficiently with iodine at room temperature(177,178) to form copper iodide.(168) However, copper oxidizes readily and loses its high efficiency. Oxides, grease, and inactive solids reduce the effective reaction surface.(162,179) Copper iodide is unstable at temperatures above 242°C and liberates iodine. Iodine adsorbed on copper can exchange readily with iodine vapor.(180)

Copper deposited on charcoal removes iodine from helium gas at temperatures up to 540°C.(181)

Molecular sieves can remove iodine with an efficiency greater than 99.9%.(182-183) However, molecular sieves have a strong affinity for moisture which limits the life of the bed. Molecular sieves are best suited to remove iodine from dry gas.

Activated alumina adsorbs iodine with an efficiency of 99.76% at room temperature. The efficiency decreases with increasing temperature and is not satisfactory except for dry atmospheres because of its strong affinity for water. Activated alumina containing about 0.5% platinum adsorbs iodine, but fails to retain it during continued air flow.(184,185)

Silica gel has an even lower efficiency (40%) and also has a strong preference for water. However, silica gel coated with silver had an efficiency of greater than 99.9%.(176)

Granular potassium iodide(185) was tested as a high temperature adsorber of iodine; however, the adsorber bed would probably have to be held at 250°C to ensure efficient trapping.(8) Moisture in the system would cause rapid deterioration of the bed. The possibility of applying chemical exchange with potassium iodide has been demonstrated.(184)

The adsorption of iodine has been studied for lead, bismuth, magnesium, bismuth oxide, lead oxides, uranium oxides, magnesium oxide, concrete dust and glass wool;(179) zinc, potassium hydroxide, tin, porous glass, cadmium(176) zinc granules; potassium hydroxide pellets, slag wool fiber coated with potassium oxide, and calcium hydroxide, sintered bronze;(186) and coke and magnesium carbonate.(187) None of these materials was satisfactory.

APPENDIX B
ACTIVATED CARBON SPECIFICATIONS

Property \ Carbon →	Activated, Noncoconut	Activated, High Quality	Impregnated, Type A, High Quality
Available Specifications	CWS No. 197-52-93 Approved Aug. 9, 1938	CWS No. 197-52-104 Sept. 10, 1941 CWS No. 197-52-105 Oct. 31, 1941	CWS No. 197-52-105 Oct. 31, 1941 CWS No. 197-52-104 Sept. 10, 1941
Methods of Testing	CWS Pamphlet No. 2, Part II, Section A, Chloropicrin Test Standard Sieve - Federal Specification No. RR-S-366 Pan for Hardness Test CWS No. A18-29-1	CWS Pamphlet No. 2, Part II, Section A, Chloropicrin Test Standard Sieve - Federal Specification No. RR-S-366 Pan for Hardness Test CWS No. A18-29-1	CWS Pamphlet No. 2, Part II, Sections C-F Standard Sieve - Federal Specification No. RR-S-366 Pan for Hardness Test CWS No. A18-29-1
Particle Size	Three Sizes No. 6-20 — % by wt On 6 — Not >5 Pass 6 On 8 — 10-20 Pass 8 On 12 — Not <20 Pass 12 On 16 — Not <25 Pass 16 On 20 — Not <15 Pass 20 — Not >8 Pass 30 — Not >1 No. 12-30 On 12 — Not >5 Pass 12 On 16 — 10-30 Pass 16 On 20 — Not >70 Pass 20 On 30 — 10-40 Pass 30 — Not >5 No. 12-16 On 8 — <1 Pass 8 On 12 — Not >15 Pass 12 On 16 — Not <75 Page 16 On 20 — Not >25 Pass 20 — <4 Pass 30 — <1	Four Sizes No. 6-20 — % by wt On 6 — 0-5 Pass 6 On 8 — 5-20 Pass 8 On 12 — 20-65 Pass 12 On 16 — 15-60 Pass 16 On 20 — 15-60 Pass 20 On 30 — 0-7 Pass 30 — 0-1 No. 12-16 On 8 — 0-1 Pass 8 On 12 — 0-25 Pass 12 On 16 — 65-100 Pass 16 On 20 — 0-25 Pass 20 On 30 — 0-3 Pass 30 — 0-1	No. 8-30 — % by wt On 8 — 0-5 Pass 8 On 16 — 30-60 Pass 16 On 20 — 15-45 Pass 20 On 30 — 7-15 Pass 30 — 0-3 No. 12-30 On 12 — 0-5 Pass 12 On 16 — 10-30 Pass 16 On 20 — 20-70 Pass 20 On 30 — 10-40 Pass 30 — 0-5
Material	Grown or produced in Continental U. S. Excluding: nuts, nut shells, Al, Sb, Cr, I, Mn, Ni, Hg, Sn, W, and their compounds	Three Types I - Coconut shells II - Cohune nut shells III - Carbonizable material produced in Continental U. S. in quantities of 100 tons/day	
Hardness	Not <65	Not Specified	Not <50
Water Content	Not >3%	Not >5%	Not >3%
Chloropicrin Adsorptive Activity (CCl_3NO_2)	None detectable within 35 minutes at concentration of 47 ±2.5 mg/l in dry air at 25°C	None detectable through 5-cm layer of dry 12-16 mesh in 37 minutes at concentration of 45 mg/l in dry air at a flow of 500 ml/min/cm^2	Not specified
Chlorine	Not >0.35%	Not >0.35%	Not >0.35%
Carbonization	Not specified	Heat treatment or by heat treatment in presence of $ZnCl_2$	Not specified
Activation	Steam, air, carbon dioxide, or mixture thereof	Steam, air, carbon dioxide, flue gas, or mixture thereof	Not specified
Heat of Wetting	Not specified	Not <9.0 cal/ml	Not specified
Adaptability to Impregnation (absorption activity)	Not specified	Life of not <25 minutes Gas — Concentration, mg/l HCN — 3.0 ±0.15 AsH_3 — 10.0 ±0.5 $COCl_2$ — 20.0 ±1.0 CNCl — 6.0 ±0.3	
Copper Content	Not specified	Not specified	3-10% by wt on dry basis
Ammonia	Not specified	Not specified	Not specified
Bulk Density	Not specified	Not specified	Not specified
Fines	Not specified	Not specified	Not specified
Iodine Number*	Not specified	Not specified	Not specified
Activity for Carbon Tetrachloride*	Not specified	Not specified	Not specified
Retentivity for Carbon Tetrachloride	Not specified	Not specified	Not specified
Ash Content*	Not specified	Not specified	Not specified
Efficiency for Removal of Iodine	Not specified	Not specified	Not specified
Leaks**	Not applicable	Not applicable	Not applicable
Ignition Temperature	Not specified	Not specified	Not specified
Internal Surface Area	Not specified	Not specified	Not specified

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* For definition of tests, see Adsorption, C. L. Mantell, McGraw-Hill Book Co., New York, N. Y., 2nd Ed., 1951.

** For definition of test, see AEC Research and Development Report, DP-870, Nondestructive Test of Carbon Beds for Reactor Containment Application, Progress Report, June 1962 - December 1963.

APPENDIX B (contd)

<table>
<tr><th>Property \ Carbon →</th><th>Activated, Technical</th><th>Activated, Impregnated, ASC</th><th>Activated, Technical, Unimpregnated</th></tr>
<tr><td>Available Specifications</td><td>MIL-C-10202A
June 30, 1959
MIL-C-13724A
May 4, 1960</td><td>MIL-C-13724A
May 4, 1960</td><td>MIL-C-17605B(SHIPS)
10 September 1964</td></tr>
<tr><td>Methods of Testing</td><td>Chemical Corps Directive 610
February 24, 1956</td><td>Standard Sieve - Federal Specification No. RR-S-366</td><td>Standard Sieve - Federal Specification No. RR-S-366
Hardness Testing Pan CWS No. A-18-29-1</td></tr>
<tr><td>Particle Size</td><td colspan="2">
<table>
<tr><th colspan="4">Two Sizes</th></tr>
<tr><th></th><th></th><th colspan="2">% by wt</th></tr>
<tr><th></th><th></th><th>Grade I</th><th>Grade II</th></tr>
<tr><td>Pass 8</td><td>On 12</td><td>0-2</td><td>...</td></tr>
<tr><td></td><td>On 10</td><td>...</td><td>0</td></tr>
<tr><td>Pass 10</td><td>On 12</td><td>...</td><td>0-0.5</td></tr>
<tr><td>Pass 12</td><td>On 16</td><td>10-30</td><td>10-30</td></tr>
<tr><td>Pass 16</td><td>On 20</td><td>40-65</td><td>40-65</td></tr>
<tr><td>Pass 20</td><td>On 30</td><td>10-35</td><td>10-35</td></tr>
<tr><td>Pass 30</td><td></td><td>0-2.5</td><td>0-2.5</td></tr>
</table>
</td><td>
<table>
<tr><th colspan="3">U. S. Standard Size</th></tr>
<tr><th></th><th></th><th>% by wt</th></tr>
<tr><td></td><td>On 4</td><td>0</td></tr>
<tr><td>Pass 4</td><td>On 6</td><td>0-5</td></tr>
<tr><td>Pass 6</td><td>On 8</td><td>40-60</td></tr>
<tr><td>Pass 8</td><td>On 12</td><td>40-60</td></tr>
<tr><td>Pass 12</td><td>On 16</td><td>0-5</td></tr>
<tr><td>Pass 16</td><td>On 50</td><td>0-0.4</td></tr>
<tr><td>Pass 50</td><td></td><td>0-0.01</td></tr>
</table>
</td></tr>
<tr><td>Material</td><td>Not specified</td><td>Not specified</td><td>Not specified</td></tr>
<tr><td>Hardness</td><td>Not specified</td><td>80 minimum</td><td>Not <90.0</td></tr>
<tr><td>Water Content</td><td>Not >5%</td><td>Grade I - 2.0% max
Grade II - 0.5% max</td><td>Not >5.0%</td></tr>
<tr><td>Chloropicrin Adsorptive Activity (CCl_3NO_2)</td><td>Not specified</td><td>None detectable within 20 minutes at concentration of 50 ±2.5 mg/l in 50% Relative Humidity, air at 32 l/min at 70°F</td><td>50 minutes minimum standard accelerated chloropicrin test (MIL-C-17605B (Ships))</td></tr>
<tr><td>Chlorine</td><td>Not >0.35%</td><td>Not specified</td><td>Not specified</td></tr>
<tr><td>Carbonization</td><td>Not specified</td><td>Not specified</td><td>Not specified</td></tr>
<tr><td>Activation</td><td>Not specified</td><td>Not specified</td><td>Not specified</td></tr>
<tr><td>Heat of Wetting</td><td>Not specified</td><td>Not specified</td><td>Not specified</td></tr>
<tr><td>Adaptability to Impregnation (absorption activity)</td><td colspan="2">
<table>
<tr><th colspan="7">Life in Minutes with M11 Canister (minimum)</th></tr>
<tr><th></th><th>Unaged</th><th>Aged</th><th>Rough Handled</th><th>Gas R.H.</th><th>Conc Gas, mg/l</th><th>Flow Rate, l/min</th></tr>
<tr><td>CCl_3NO_2</td><td>20</td><td>...</td><td>18</td><td>50</td><td>50 ±2.5</td><td>32</td></tr>
<tr><td>HCN</td><td>25</td><td>...</td><td>...</td><td>50</td><td>10 ±0.5</td><td>32</td></tr>
<tr><td>$COCl_2$</td><td>25</td><td>...</td><td>...</td><td>50</td><td>20 ±1.0</td><td>32</td></tr>
<tr><td>CNCl</td><td>30</td><td>40% of Unaged</td><td>...</td><td>80†</td><td>4 ±0.2</td><td>50</td></tr>
<tr><td>AsH_3</td><td>45</td><td>...</td><td>...</td><td>80†</td><td>10 ±0.5</td><td>50</td></tr>
</table>
† Charcoal humidified at 80 ±3% Relative Humidity
</td><td>Not specified</td></tr>
<tr><td>Copper Content</td><td>Not specified</td><td>Not specified</td><td>Not specified</td></tr>
<tr><td>Ammonia</td><td>Not specified</td><td>Max 0.005 mg NH_3/l air/100 ml charcoal</td><td>Not specified</td></tr>
<tr><td>Bulk Density - Packing Density</td><td>Not specified</td><td>Max 0.57 g/ml</td><td>Not >0.57 g/ml
Not <0.42 g/ml</td></tr>
<tr><td>Fines</td><td>Not specified</td><td>Not specified</td><td>†</td></tr>
<tr><td>Iodine Number*</td><td>Not specified</td><td>Not specified</td><td>Not specified</td></tr>
<tr><td>Activity for Carbon Tetrachloride*</td><td>Not specified</td><td>Not specified</td><td>50% by wt min
65% by wt max</td></tr>
<tr><td>Retentivity for Carbon Tetrachloride</td><td>Not specified</td><td>Not specified</td><td>Not specified</td></tr>
<tr><td>Ash Content*</td><td>Not specified</td><td>Not specified</td><td>Not specified</td></tr>
<tr><td>Efficiency for Removal of Iodine</td><td>Not specified</td><td>Not specified</td><td>Not specified</td></tr>
<tr><td>Leaks**</td><td>Not applicable</td><td>Not applicable</td><td>Not applicable</td></tr>
<tr><td>Ignition Temperature</td><td>Not specified</td><td>Not specified</td><td>Not specified</td></tr>
<tr><td>Internal Surface Area</td><td>Not specified</td><td>Not specified</td><td>Not specified</td></tr>
</table>

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† <0.01% through size 50.

APPENDIX B (contd)

Property \ Carbon →	Activated, SRP Confinement	Barnebey-Cheney-Type 416	Barnebey-Cheney-Type 592
Available Specifications	DP-778, Sept. 1962 DP-1028, Jan. 1966 DP-1071, Aug. 1966 Barnebey-Cheney-Type 416, or equal	-	-
Methods of Testing	Barnebey-Cheney Standard Methods or Methods established by the Savannah River Laboratory	Barnebey-Cheney Standard Methods or Methods established by the Savannah River Laboratory	Barnebey-Cheney Standard Methods or Methods established by the Savannah River Laboratory
Particle Size	Tyler Size — % by wt On 8 — 0.1 max On 10 — 10.0 max On 14 — 88.9 min Through 14 — 1.0 max	Tyler Size — % by wt On 8 — 0.1 max On 10 — 10. max Pass 10 On 14 — 89.4 min Pass 14 — 0.5 max	Tyler Size — % by wt On 8 — 0.1 max On 10 — 10. max Pass 10 On 14 — 89.4 min Pass 14 — 0.5 max
Material	New coconut shell	Coconut shell	Coconut shell
Hardness	97% minimum	97% minimum	97% minimum
Water Content	Not >2%	2% max as packed	2% max as packed
Chloropicrin Adsorptive Activity (CCl_3NO_2)	50 minutes minimum, Standard Accelerated Chloropicrin Test*	50 minutes, min	50 minutes, min
Chlorine	Not specified	Not specified	Not specified
Carbonization	Not specified	Thermal	Thermal
Activation	Not specified	Steam	Steam
Heat of Wetting	Not specified	Not specified	Not specified
Adaptability to Impregnation (absorption activity)	Not specified	Not specified	Not specified
Copper Content	Not specified	Not specified	Not specified
Ammonia	Not specified	Not specified	Not specified
Bulk Density - Packing Density	34 lb/ft^3	0.51-0.58 g/ml	0.51-0.58 g/ml
Fines	Fines blown out with pure air at 5000 fpm from fishtail shaped orifice 0.8-in.^{2} min	0.5% max through 14 mesh	0.5% max through 14 mesh
Iodine Number*	Minimum 1050 mg I/g carbon	1050 to 1250 mg I/g carbon	1050 to 1250 mg I/g carbon
Activity for Carbon Tetrachloride*	50% by wt minimum	50% by wt min	50% by wt min
Retentivity for Carbon Tetrachloride	Not specified	30% by wt min	30% by wt min
Ash Content*	4% by wt maximum	4% by wt max	3% by wt max
Efficiency for Removal of Iodine	99.99% minimum from steam air mixture at 160°F, face velocity 70 ft/min and loading of 1 mg I/g carbon	99.99% min	99.99% min
Leaks**	0.1% of total bed flow, max	0.1% of total bed flow max	0.1% of total bed flow max
Ignition Temperature	>330°C	320°C min	575°C min
Internal Surface Area	Not specified	1000-1100 m^2/g (BET)	1000-1100 m^2/g (BET)

APPENDIX B (contd)

Carbon → Property	Pittsburg-Activated Carbon PCB	Pittsburg-Activated Carbon BPL
Available Specifications	Pittsburg-Activated Carbon Company Data Sheet	Pittsburg-Activated Carbon Company Data Sheet
Methods of Testing	Pittsburg-Activated Carbon Company Control Laboratory Test Methods	Pittsburg-Activated Carbon Company Control Laboratory Test Methods
Particle Size	Three Sizes (U. S. Sieve Series) 4 x 10 — % by wt On 4 — 0-5 Pass 4 On 6 — 45-65 Pass 6 On 8 — 25-45 Pass 8 On 10 — 2-12 Pass 10 — 0-5 6 x 16 — % by wt On 6 — 0-5 Pass 6 On 8 — 15-45 Pass 8 On 12 — 35-55 Pass 12 On 16 — 15-55 Pass 16 — 0-5 12 x 30 — % by wt On 12 — 0-5 Pass 12 On 16 — 20-40 Pass 16 On 20 — 40-85 Pass 20 On 30 — 10-20 Pass 30 — 0-5	Four Sizes (U. S. Sieve Series) 4 x 6 — % by wt On 4 — 0-15 Pass 4 On 6 — 80-100 Pass 6 — 0-10 6 x 8 — % by wt On 6 — 0-15 Pass 6 On 8 — 80-100 Pass 8 — 0-10 4 x 8 — % by wt On 4 — 0-10 Pass 4 On 6 — 40-60 Pass 6 On 8 — 30-50 Pass 8 — 0-5 8 x 14 — % by wt On 8 — 0-5 Pass 8 On 12 — 60-80 Pass 12 On 14 — 20-40 Pass 14 — 0-5
Material	Coconut shell	Coal
Hardness	92% min	90% min
Water Content	3.0% max as packed	2.0% max as packed
Chloropicrin Adsorptive Activity (CCl_3NO_2)	43 minutes min	46 minutes min
Chlorine	Not specified	Not specified
Carbonization	Not specified	Not specified
Activation	Not specified	Not specified
Heat of Wetting	Not specified	Not specified
Adaptability to Impregnation (absorption activity)	Not specified	Not specified
Copper Content	Not specified	Not specified
Ammonia	Not specified	Not specified
Bulk Density	0.44 g/ml, 27.5 lb/ft^3	0.48 g/ml, 30 lb/ft^3
Fines	Not specified	Not specified
Iodine Number*	1200 mg I/g carbon, min	1050-1200 mg I/g carbon, min
Activity for Carbon Tetrachloride*	60% by wt min	60% by wt min
Retentivity for Carbon Tetrachloride	Not specified	Not specified
Ash Content*	4% by wt max	8% by wt max
Efficiency for Removal of Iodine	Not specified	Not specified
Leaks**	Not applicable	Not applicable
Ignition Temperature	300°C	400°C
Internal Surface Area	1200 m^2/g (BET)	1050-1150 m^2/g (BET)

APPENDIX B (contd)

Carbon → Property	Union Carbide Corp. NXC	Union Carbide Corp. JXC	Union Carbide Corp. 3LXC
Available Specifications	See Footnote (1)	See Footnote (1)	See Footnote (1)
Methods of Testing	See Footnote (2)	See Footnote (2)	See Footnote (2)
Particle Size	4/6, 6/8, 8/10 (Pellet)	4/6, 6/8 (Pellet)	4/6, 6/8, 8/10 (Pellet)
Material	Petroleum residue	Petroleum residue	Petroleum residue
Hardness	95% min	95% min	95% min
Water Content	2.0% max	2.0% max	2.0% max
Chloropicrin Adsorptive Activity (CCl_3NO_2)	Not specified	Not specified	Not specified
Chlorine	0.02%	0.02%	0.02%
Carbonization	Fluidized bed	Fluidized bed	Fluidized bed
Activation	Thermal	Thermal	Thermal
Heat of Wetting	Not specified	Not specified	Not specified
Adaptability to Impregnation (absorption activity)	Not specified	Not specified	Not specified
Copper Content	20 ppm	20 ppm	20 ppm
Ammonia	0	0	0
Bulk Density	0.48 g/ml min	0.50 g/ml min	0.55 g/ml min
Fines	1% max	0.5% max	1% max
Iodine Number*	1230 mg I/g carbon	1230 mg I/g carbon	1210 mg I/g carbon
Activity for Carbon Tetrachloride*	60% min	60% min	50% min
Retentivity for Carbon Tetrachloride	35% min	35% min	30% min
Ash Content*	2.8% max	2.0% max	2.0% max
Efficiency for Removal of Iodine	Not specified	Not specified	Not specified
Leaks**	Not applicable	Not applicable	Not applicable
Ignition Temperature	350°C min	350°C min	350°C min
Internal Surface Area	980 m^2/g (4/6 mesh)	980 m^2/g	880 m^2/g

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Footnote (1)

References to Available Specifications

Grade	Properties	Data Sheet
All	Typical	12
NXC	Typical	9
ACC	Typical	3
SXC	Typical	2
Pressure Drop	Typical	1
JXC	Typical	f
CXC	Typical	8

Footnote (2)

References to Methods of Testing

UCC Test	Catalog #	UCC Test	Catalog #
Activity	AC 5-48	Moisture on New Activated Carbon	AC 15-48
Air Retentivity	AC 6-48		
CCl_4 Minute Service Test	AC 7-50	Ash Content	AC 20-48
Iodine Activity	AC 9-48	Chloride	AC 21-48
Apparent Density	AC 10-51	Sulfur	AC 23-48
Bulk Density	AC 11-50	Chemical Examination of Activated Carbon Ash	AC 28-48
Screen Analysis	AC 12-48		
Strength	AC 13-51		
Accelerated Attrition	AC 14-51	Kindling Point	AC 29-51

APPENDIX B (contd)

Carbon → Property	Union Carbide Corp. ACC	Union Carbide Corp. SXC
Available Specifications	See Footnote (1), page 62	See Footnote (1), page 62
Methods of Testing	See Footnote (2), page 62	See Footnote (2), page 62
Particle Size	6/14 (Granular)	2/4, 4/6, 6/8 (Pellet)
Material	Petroleum residue	Petroleum residue
Hardness	96% min	96% min
Water Content	2.0% max	2.0% min
Chloropicrin Adsorptive Activity (CCl_3NO_2)	Not specified	Not specified
Chlorine	0.02%	0.02%
Carbonization	Fluidized bed	Fluidized bed
Activation	Thermal	Thermal
Heat of Wetting	Not specified	Not specified
Adaptability to Impregnation (absorption activity)	Not specified	Not specified
Copper Content	20 ppm	20 ppm
Ammonia	0	0
Bulk Density	0.49 g/ml min	0.45 g/ml min
Fines	1% max	1% max
Iodine Number*	1250 mg I/g carbon	1250 mg I/g carbon
Activity for Carbon Tetrachloride*	65% min	65% min
Retentivity for Carbon Tetrachloride	35% min	35% min
Ash Content*	2.0% max	3.0% max
Efficiency for Removal of Iodine	Not specified	Not specified
Leaks**	Not applicable	Not applicable
Ignition Temperature	350°C	350°C
Internal Surface Area	1040 m^2/g	1035 (6/8 mesh)

APPENDIX B (contd)

Carbon → Property	Witco Chemical Co. Grade 517	West Virginia Pulp and Paper Type WA	West Virginia Pulp and Paper Type C-190
Available Specifications	Witco Chemical Co. Technical Data Sheet	1961 Report No. K 4005.01	1961 Report No. K 4005.01
Methods of Testing	Not specified	1961 Report No. K 4005.01	1961 Report No. K 4005.01
Particle size	U. S. Sieves 18 x 40	4 x 30	4 x 30
Material	Liquid hydrocarbon	Wood pulp residue	Wood pulp residue
Hardness	Not specified	Not specified	Not specified
Water Content	Not specified	3%	3%
Chloropicrin Adsorptive Activity (CCl_3NO_2)	Not specified	Not specified	Not specified
Chlorine	Not specified	Not specified	Not specified
Carbonization	Not specified	Not specified	Not specified
Activation	Not specified	Not specified	Not specified
Heat of Wetting	Not specified	Not specified	Not specified
Adaptability to Impregnation (absorption activity)	Not specified	Not specified	Not specified
Copper Content	Not specified	Not specified	Not specified
Ammonia	Not specified	Not specified	Not specified
Bulk Density	0.50 g/ml, 31.5 lb/ft^3	9-11 lb/ft^3	7-9 lb/ft^3
Fines	Not specified	Not specified	Not specified
Iodine Number*	Not specified	Not specified	Not specified
Activity for Carbon Tetrachloride*	55% by wt	Not specified	Not specified
Retentivity for Carbon Tetrachloride	Not specified	Not specified	Not specified
Ash Content*	0.65% by wt	6% max	6% max
Efficiency for Removal of Iodine	Not specified	Not specified	Not specified
Leaks**	Not applicable	Not applicable	Not applicable
Ignition Temperature	Not specified	Not specified	Not specified
Internal Surface Area	1150 m^2/g (BET)	550-650 m^2/g	700-900 m^2/g

APPENDIX B (contd)

Property \ Carbon →	American Norite Co., Inc., Grade RFZI	American Norite Co., Inc., Grade RFZII	American Norite Co., Inc., Grade RFZIII
Available Specifications	Bulletin G-109-2	Bulletin G-109-2	Bulletin G-109-2
Methods of Testing	Not specified	Not specified	Not specified
Particle Size	Pellets, 1 mm diameter by 2-4 mm long	Pellets, 2 mm diameter by 4-8 mm long	Pellets, 3 mm diameter by 6-12 mm long
Material	Vegetable	Vegetable	Vegetable
Hardness	98	98	98
Water Content	Not specified	Not specified	Not specified
Chloropicrin Adsorptive Activity (CCl_3NO_2)	Not specified	Not specified	Not specified
Chlorine	Not specified	Not specified	Not specified
Carbonization	Not specified	Not specified	Not specified
Activation	Not specified	Not specified	Not specified
Heat of Wetting	Not specified	Not specified	Not specified
Adaptability to Impregnation (absorption activity)	Not specified	Not specified	Not specified
Copper Content	Not specified	Not specified	Not specified
Ammonia	Not specified	Not specified	Not specified
Bulk Density - Packing Density	0.48 g/cc	0.46 g/cc	0.44 g/cc
Fines	Not specified	Not specified	Not specified
Iodine Number*	Not specified	Not specified	Not specified
Activity for Carbon Tetrachloride*	Not specified	Not specified	Not specified
Retentivity for Carbon Tetrachloride	Not specified	Not specified	Not specified
Ash Content*	Not specified	Not specified	Not specified
Efficiency for Removal of Iodine	Not specified	Not specified	Not specified
Leaks**	Not applicable	Not applicable	Not applicable
Ignition Temperature	Not specified	Not specified	Not specified
Internal Surface Area	600-1000 m^2/g (BET)	600-1000 m^2/g (BET)	600-1000 m^2/g (BET)

APPENDIX B (contd)

Property \ Carbon →	Cliffs Dow Chemical Co., CliffChar	Atlas Chemical Industries, Inc., Granular Darco
Available Specifications	Letter dated 2/27/64	Bulletin D-80
Methods of Testing	Not specified	Not specified
Particle Size	Three Sizes, Tyler Screen Grade 2 x 4 On 2 0% Pass 2 On 4 85% Pass 4 15% Grade 4 x 10 On 4 10% Pass 4 On 14 87% Pass 14 3% Grade 10 x 20 On 10 2% Pass 10 On 28 93% Pass 28 5%	Four Sizes, U. S. Sieve Series 4 x 12 Mesh Size % by wt On 4 Not >2 Pass 4 On 12 96-100 Pass 12 Not >2 12 x 40 Mesh Size % by wt On 12 Not >2 Pass 12 On 40 96-100 Pass 40 Not >2 12 x 20 Mesh Size % by wt On 12 Not >2 Pass 12 On 20 96-100 Pass 20 Not >2 20 x 40 Mesh Size % by wt On 20 Not >2 Pass 20 On 40 96-100 Pass 40 Not >2
Material	Not specified	Lignite base
Hardness	Not specified	Not specified
Water Content	3.5%	6%
Chloropicrin Adsorptive Activity (CCl_3NO_2)	Not specified	Not specified
Chlorine	Not specified	Not specified
Carbonization	Not specified	Not specified
Activation	Not specified	Not specified
Heat of Wetting	Not specified	Not specified
Adaptability to Impregnation (absorption activity)	Not specified	Not specified
Copper Content	Not specified	Not specified
Ammonia	Not specified	Not specified
Bulk Density - Packing Density	16 lb/ft^3, 0.22 g/cc	0.39 g/ml, 24.5 lb/ft^3
Fines	Not specified	Not specified
Iodine Number*	Not specified	Not specified
Activity for Carbon Tetrachloride*	Not specified	Not specified
Retentivity for Carbon Tetrachloride	Not specified	Not specified
Ash Content*	3.8%	Not specified
Efficiency for Removal of Iodine	Not specified	Not specified
Leaks**	Not applicable	Not applicable
Ignition Temperature	Not specified	Not specified
Internal Surface Area	600-800 m^2/g	700 m^2/g

APPENDIX C

SRL IGNITION TEST PROCEDURE

APPARATUS AND REAGENTS

Air filter:	1-inch thick by 3-inches diameter stainless steel, filled with activated carbon. Barnebey-Cheney Type 416, or equal.
Absolute filter:	3-inches diameter, flat medium. Cambridge Type C-30-B, or equal. Cambridge Filter Corp., Syracuse, New York.
Rotameter:	0-6.5 scfm air. 0-0.65 scfm air.
Preheater:	Type M2018 Electric Furnace, 1870 watts, Hevi Duty Electric Co., Milwaukee, Wis., or equal.
Powerstat:	Variable Transformer, Type 3PF136 Superior Electric Co., Bristol, Conn., or equal.
Temperature Regulator:	Brown Potentiometer, Model Y153C (II) PS-(36) K1W7-(IV)N4, Minneapolis Honeywell Reg. Co., Philadelphia, Pa., or equal.
Recorder:	Brown Potentiometer, Model Y153X64(W)16-X-(71)N4 Minneapolis Honeywell Reg. Co., Philadelphia, Pa., or equal, calibrated to cover the temperature range 200 to 600°C.
Oven:	Lindberg Box Type B-6W2 Furnace, 4000 watts, Basic Products Corporation, Watertown, Wis., or equal.
Ignition Apparatus:	Thermocouples, Chromel-Alumel, Type 400-K-(A)-18-12-W4 with 1/8-inch diameter oversheath extending to 3.5-inches from hot junction. Thermocouple Products Co., Villa Park, Illinois, or equal.
	Bed Holder (See Figure 2).
	Bed, 1-inch thick by 3-inch diameter stainless steel with mesh.
	Aluminum foil gaskets, double thickness of 0.0007-inch foil.

Water Cooler:	5 gallon pail, or equal.
Exhaust Hood:	-
Compressed Air:	60 psig
Compressed Nitrogen:	20 psig
Tubing:	1/2-inch diameter stainless steel, Swagelock fittings, or equal.
Valves:	1/2-inch globe, brass, or equal.
Insulation:	1/8-inch thick asbestos cloth, wrapped 6 layers deep.

ASSEMBLY OF APPARATUS

1. Assemble apparatus, as shown in Figures 1 and 2.
2. Install preheater with about 1 ft^3 of tubing to heat air to the desired temperature.
3. Test all connections to ensure absence of leaks. Insulate tubing.
4. Connect thermocouples 1 and 2 to the temperature regulator so that the oven is maintained at the same temperature as the air leaving the preheater.

LOADING THE BED

1. Place sample of carbon to be tested in a stainless steel beaker. Weigh the beaker and its contents; record the weight.
2. Attach stainless steel mesh bottom to the retaining ring that forms the bed, as shown in Figure 2. Use aluminum foil gaskets to avoid air leaks at flanged joints.
3. Place the bed on the holder while the holder is inverted. Allow the thermocouples to penetrate the mesh to a depth of 1/8-inch from the upstream and the downstream face.
4. Pour the carbon into the bed until the bed is filled and there is an excess that forms a mound about 1/4-inch high.

5. Tap the bed with a 6-inch length of 1/2-inch diameter stainless steel tubing to pack the carbon.

6. Scrape off the remaining excess carbon with a straight edge and return the excess carbon to the stainless steel beaker.

7. Weigh and record the weight of the beaker and its contents.

8. Attach the top screen to the bed.

9. Attach the lower part of the bed holder, invert and place in oven.

10. Connect the holder to the apparatus.

TEST PROCEDURE

1. Adjust air to the desired flow. A face velocity of 70 ft/min has been used as a standard velocity for carbons whose ignition temperature is a function of velocity.

2. Apply power to the preheater by setting the Powerstat to 60%. This will heat the flowing air to about 200°C, at a face velocity of 70 ft/min.

3. Increase the temperature of the preheater by adjusting the Powerstat in 1% increments.

4. Adjust the rate of heating to 2 to 4°C/min when the temperature is ~50°C below the ignition temperature by increasing the Powerstat about 1% every 10 minutes.

5. Record the ignition temperature when a sudden rise occurs in the temperature of one of the thermocouples in the carbon bed (TC 3, 4, 5, 6) or the thermocouple at the exit of the holder (TC 7).

6. Stop the airflow by closing valve 1; start the nitrogen flow by opening valve 2; and turn off power to the preheater and oven.

7. Remove the carbon bed after it is cooled, and empty the carbon into the stainless steel beaker containing excess carbon.

8. Weigh the beaker and record the weight.

9. Calculate the approximate weight of the carbon sample and the weight of carbon burned.

10. The precision of the ignition temperature measurement is about ±10°C.