CLEARINGHOUSE FOR FEDERAL SCIENTIFIC AND TECHNICAL INFORMATION CFSTI
DOCUMENT MANAGEMENT BRANCH 410.11

LIMITATIONS IN REPRODUCTION QUALITY

ACCESSION # 604 426

☐ 1. WE REGRET THAT LEGIBILITY OF THIS DOCUMENT IS IN PART UNSATISFACTORY. REPRODUCTION HAS BEEN MADE FROM BEST AVAILABLE COPY.

☒ 2. A PORTION OF THE ORIGINAL DOCUMENT CONTAINS FINE DETAIL WHICH MAY MAKE READING OF PHOTOCOPY DIFFICULT.

☐ 3. THE ORIGINAL DOCUMENT CONTAINS COLOR, BUT DISTRIBUTION COPIES ARE AVAILABLE IN BLACK-AND-WHITE REPRODUCTION ONLY.

☐ 4. THE INITIAL DISTRIBUTION COPIES CONTAIN COLOR WHICH WILL BE SHOWN IN BLACK-AND-WHITE WHEN IT IS NECESSARY TO REPRINT.

☐ 5. LIMITED SUPPLY ON HAND: WHEN EXHAUSTED, DOCUMENT WILL BE AVAILABLE IN MICROFICHE ONLY.

☐ 6. LIMITED SUPPLY ON HAND: WHEN EXHAUSTED DOCUMENT WILL NOT BE AVAILABLE.

☐ 7. DOCUMENT IS AVAILABLE IN MICROFICHE ONLY.

☐ 8. DOCUMENT AVAILABLE ON LOAN FROM CFSTI (TT DOCUMENTS ONLY).

☐ 9.

PROCESSOR:

TSL-107-10/64

NRL Report 6090

The Ignition and Combustion Properties of Activated Carbon Containing Adsorbed Hydrocarbons

F. J. Woods and J. E. Johnson

Fuels Branch
Chemistry Division

July 28, 1964

U.S. NAVAL RESEARCH LABORATORY
Washington, D.C.

CONTENTS

Copies available at Office of Technical Services
Department of Commerce – $.75

ABSTRACT

Activated carbon contained in large filter beds is used in nuclear submarines for removal of odors and trace contaminants. Because organic vapors are concentrated in this way in the carbon, a study was made to get information for assessing the fire hazards involved in maintaining the carbon filter in the ventilation system of the submarines. In this study, spontaneous ignition of submarine-exposed carbons occurred at temperatures as low as 490°F (255°C) in air at one atmosphere. Exothermic oxidative reactions occurred in a flow system at temperatures as low as 390°F (200°C). Combustion of carbons containing hydrocarbon produced considerable quantities of carbon dioxide and carbon monoxide, and significant amounts of these gases were evolved even under precombustion conditions. Of a number of carbons which had been used in submarines, the lowest flash point found was 160°F.

PROBLEM STATUS

This is an interim report; work on this problem is continuing.

AUTHORIZATION

NRL Problem C08-30
Project SF 013-08-03-4092, 4093, 4094, 4095

NRL Problem C01-03
Projects RR 010-01-44-5850 and SR 001-06-02-0600

Manuscript submitted March 4, 1964.

THE IGNITION AND COMBUSTION PROPERTIES OF ACTIVATED CARBON CONTAINING ADSORBED HYDROCARBONS

INTRODUCTION

Activated carbon contained in fixed filter beds is used as part of the air purification systems of nuclear submarines for removing trace contaminants and odors. Because of its enormous effective surface area, activated carbon has a large capacity for adsorption of organic molecules. Analysis of the organic liquid desorbed from activated carbon which had been exposed in nuclear submarines showed that it consists principally of hydrocarbons (1,2). This concentration of flammable material on activated carbon raised a question concerning the hazard of maintaining these carbon beds in the nuclear submarines.

The total concentration of hydrocarbons and other related organic compounds in submarine atmospheres is normally much too low to be of consequence as a fire hazard in the form of a dispersed vapor (3,4). However, it is the function of the main filter beds, containing several hundred pounds of activated carbon, to separate these hydrocarbon vapors from the atmosphere and to concentrate them for disposal. In the early years of nuclear submarines, carbon taken from the main filter beds was often saturated, i.e., to contain 20% to 30% by weight of organic compounds, and became saturated after only a few days of operation. By employing better removal techniques and stricter control of sources of organic materials, this condition is not as prevalent today (3). However, in newly activated submarines the carbon quickly becomes loaded with hydrocarbons due to the evaporation of solvents from freshly applied paints, cements, cleaning fluids, etc.

Since hydrocarbons are flammable and carbon itself will burn under certain conditions, it was important to know the ignition characteristics of the activated carbon, especially when it contains varying amounts of hydrocarbons. It was important, for example, to establish whether or not the activated carbon used might act as a catalyst for ignition under these circumstances. Would it catalyze the spontaneous ignition of hydrocarbons adsorbed on it? Could it lower the flash point of such hydrocarbons? A 500-pound bed of carbon, when saturated, will contain 125 to 150 pounds of hydrocarbons. The consequences of an unexpectedly large temperature rise resulting in the sudden evolution of hydrocarbon vapors from such a carbon bed must be considered. The location of this large amount of potential fuel in the air stream of the ship's ventilation system emphasized the importance of this problem.

With these considerations in mind, a study was undertaken to provide information needed for assessing the fire hazards of the carbon filter system. Spontaneous ignition properties were studied as well as induced ignition, the latter being represented by the flash points of these materials.

APPARATUS, PROCEDURES, AND MATERIALS

The flash points of the liquid hydrocarbons and mixtures of hydrocarbons were obtained in a Pensky-Martens Closed Tester by ASTM Method D93 (5). The flash points of the various carbon samples were obtained by using the same apparatus and essentially the same procedure.

The spontaneous ignition temperatures (SIT) of carbon and liquid samples in air and at atmospheric pressure were determined by two methods, one in a static system and the other in a flow system. The static method for the determination of SIT's was the method of the Bureau of Mines (6), which has been proposed as a modification of the ASTM tentative method D286-58. Ignition temperatures were obtained by (a) injecting small measured quantities (ca. 0.1 ml) of the test liquid into the 200-ml flask by means of a hypodermic syringe, or in the case of carbon, dropping in the appropriate amount of carbon granules, (b) observing occurrence of ignition, and (c) recording as the SIT the lowest temperature at which spontaneous ignition occurred. A time-temperature record was obtained during a test by measuring the internal gas temperature with a fine thermocouple connected to a fast-response two-pen recording potentiometer. A typical record is given in Fig. 1. Such a record provided a means of distinguishing between ignition and nonignition; but, as other investigators have found (7,8), the distinction between cool flames and hot flames is best made by visually observing the reaction in the flask in a darkened room. The inside of the flask was observed by means of a mirror placed at a suitable angle over the flask.

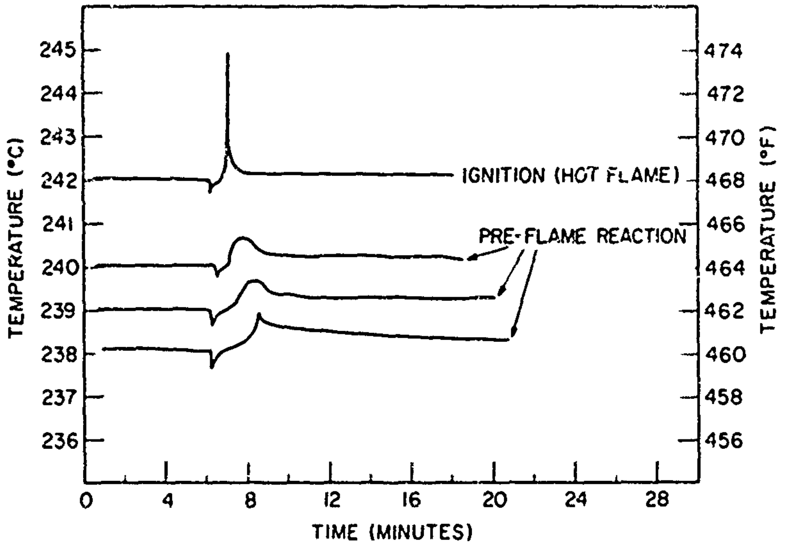

Fig. 1 - Typical ignition curves for a hydrocarbon mixture (C-214) in air (curves from four different runs sketched in for illustrative purposes only)

The apparatus used to determine SIT in a flow system at atmospheric pressure is described in Appendix A. This method consists essentially of suspending the carbon sample in a heated tube with air passing through at a controlled rate. Time-temperature records were obtained by measuring the internal temperature of the carbon bed contained in a stainless steel wire basket with a thermocouple connected to a fast-response recording potentiometer. Figure 2 shows a series of five runs made at different air-inlet temperatures.

A special method and the apparatus used to obtain SIT's at elevated pressures are described in Appendix B. The apparatus consists essentially of a cylindrical cell of copper or stainless steel which can be pressurized and heated. In these experiments oxygen was used at pressures up to 800 psi and air up to 1500 psi. After a sample was placed in the cell and pressurized with the appropriate gas, the heater which had been set at the desired temperature was drawn up around the cell. Ignition temperatures were determined from temperature-time curves such as those shown in Fig. 3.

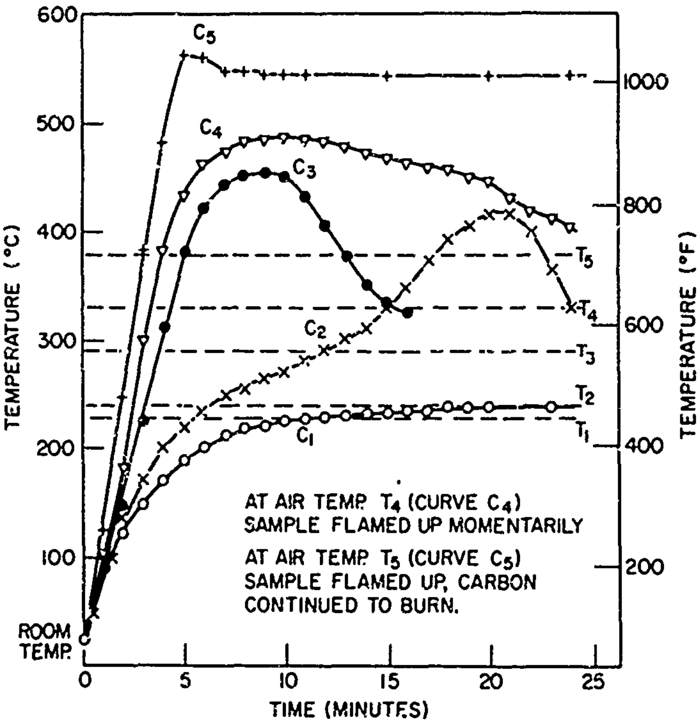

Fig. 2 - Time-temperature curves for 4 g coconut charcoal (F-1) containing 25-percent n-decane exposed to heated air stream (2 l/min) at various initial air temperatures (T_1-T_5)

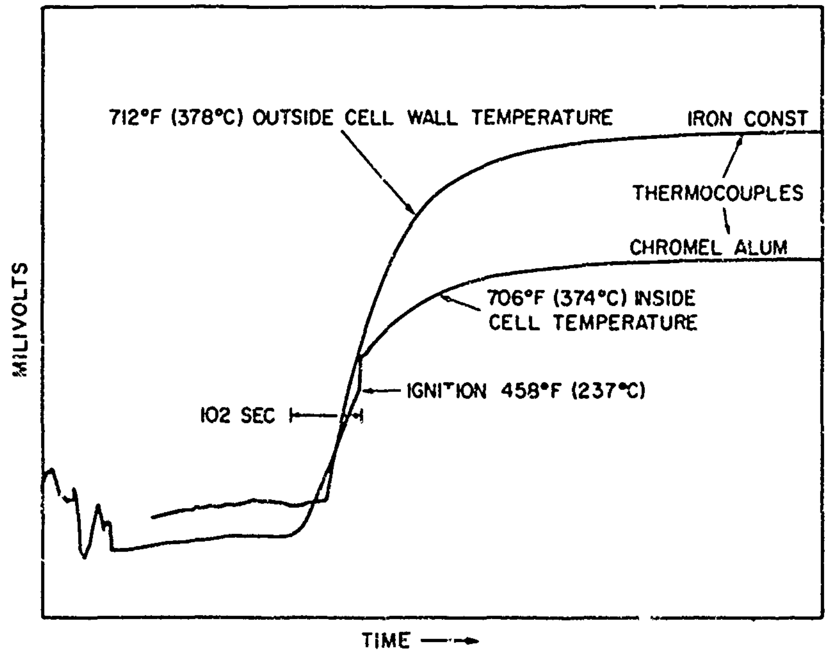

Fig. 3 - Potentiometer trace of time-temperature record showing ignition of activated carbon containing 25-percent n-hexane in pressurized system at 50 psi oxygen

The hydrocarbons used were Phillips Petroleum Co. "pure grade" and better than 99 percent pure. The fuels and lubricants used conformed with the appropriate military specifications (9).

The activated carbon used in the submarines was of the type described in specification MIL-C-17605A. Since it had been established that the mineral content of carbon can influence ignition behavior, spectrographic analyses were made of two samples of MIL-C-17605A (coconut) charcoal. These data are compared in Table 1 with the analysis of a coal-based charcoal obtained from the Connor Engineering Company. In addition to the MIL-C-17605A carbon, another sample of coconut carbon (F-1) was obtained from Fisher Scientific Company.

Table 1
Spectrographic Analysis of Activated Carbon

Metals Found	Coconut Charcoal From HALIBUT (C-197)	Coconut Charcoal From SNOOK (C-222)	Coal-Based Carbon
Cu	vw*	vw *	w*
Ag	tr	vw	vw
Mg	m	m	m-s
Si	w-m	w-m	s-vs
Mn	vw	vw	w
Fe	w	w	s
Na	m	m	s
Al	w	w	vs
Ni	-	-	w
Ca	s	s	s
K	w	w	vw
Be	-	-	vw
Cr	-	-	w
Pb	-	-	-
Ti	-	-	m
V,Li,B	-	-	w
Co, Ba	-	-	vw

*Key (in percent)

vs - 10.0 to 100
s - 1.0 to 10
m - 0.1 to 1
w - 0.01 to 0.1
vw - 0.001 to 0.001
tr - 0.0001 to 0.001
\- not detected

The hydrocarbon-impregnated carbon samples were prepared by slowly adding the prescribed weight of pure liquid hydrocarbon or mixture to a weighed amount of the activated carbon. The weight of hydrocarbon added was adjusted carefully after allowing time for complete adsorption, and the carbon was thoroughly mixed before sampling. The samples of carbon which had been exposed in submarine atmospheres were used as received. The hydrocarbon content is expressed as grams per 100 grams of carbon; that is, percent hydrocarbon based on the weight of carbon.

FLASH POINT STUDIES

One of the standard ignition tests which was applied to this problem is the determination of the flash point in the Pensky-Martens (closed cup) apparatus. In essence this consists of heating a small amount of material in a closed cell at a constant rate with continuous stirring. At regular intervals a small pilot flame is directed into the opened cell. The flash point is the minimum temperature at which the application of the pilot flame causes the vapor above the sample to ignite, that is, the temperature at which the vapor pressure reaches the lower limit of flammability.

Although this flash point test was designed to be used primarily with liquid fuels, it is also recommended for use with viscous materials and suspensions of solids (5). At any rate it appeared that this technique would be a reasonably severe test for the relative flammabilities of carbons containing adsorbed hydrocarbons.

Studies with Pure Hydrocarbons

In order to establish baselines for interpretation of data, some preliminary experiments were made using pure hydrocarbons. Flash points were determined for several hydrocarbons at varying rates of temperature rise as shown in Table 2. It was found that the rate of heating (10° to 30°F/min) had no pronounced effect on the flash point temperatures. Also it was established that the quantity of liquid could vary considerably with no appreciable effect. These factors were explored for comparison with their effects on the studies with carbon samples. Inspection of Table 3 indicates that there is a pronounced concentration effect for n-decane adsorbed on carbon with positive flash points obtained with more than 27 percent n-decane.

Table 2
Flash Points of Pure Hydrocarbons

Hydrocarbon	Full Cup*		Half Cup†	
	Flash Point (°F)	Heat Rate (°F/min)	Flash Point (°F)	Heat Rate (°F/min)
n-Decane	126	10	116	10
	120	22	122	20
	117	26	115	30
n-Undecane	154	12	150	20
	155	12	150	20
	152	20	152	24
n-Dodecane	178	10	177	13
	178	10	182	16
	180	10	177	24

*Full cup denotes the sample size specified by ASTM (5).
†Half cup is a sample half this size.

In Table 4, additional data with three different hydrocarbons leads to the important conclusion that activated carbon with 30-percent adsorbed hydrocarbon does not tend to give a lower flash point than the liquid hydrocarbon itself. The data in Table 3 indicated that the size of sample, whether 10 or 20 grams, was not very significant. In all cases with the heavier hydrocarbons, however, 10 grams of contaminated carbon gave a lower flash point than 20 grams as seen in Table 4.

Table 3
Flash Points for Various Percentages of
n-Decane Adsorbed on Carbon

n-Decane (percent)	10-Gram Sample		20-Gram Sample	
	Flash	Flash Point or Max Temp (°F)	Flash	Flash Point or Max Temp (°F)
20	No	165	No	200
25	No	180	No	168
26	No	175	No	170
27	No	210	Yes	140
27	Yes	138	Yes	135
28	Yes	130	Yes	142
29	Yes	125	-	-
30	Yes	116	Yes	125

Table 4
Flash Points of Pure Hydrocarbons as a
Liquid and Adsorbed on Carbon

Hydrocarbon	Flash Point (°F)		
	Liquid	30% Hydrocarbon on Carbon	
		10-gram sample	20-gram sample
n-Decane	115	116	124
n-Undecane	150	153	195
n-Dodecane	177	177	230

Hydrocarbon Mixtures

In Table 5, the flash points of several jet fuels and a lubricating oil are given. Here again it is illustrated by the JP-5 fuel and the 2190 oil that the hydrocarbon flash point is not lowered by adsorption on carbon, although when the carbon has sufficient fuel adsorbed on it, the flash point approaches that of the fuel alone. The flash point of the JP-4 fuel was below room temperature. Even so, 20 percent of the fuel on carbon did not flash at temperatures as high as 150 °F. To pursue this point a bit further, the flash points of carbon samples containing several different percentages of n-hexane are given in Table 5. Although the published value (10) for the flash point of n-hexane is -15 °F, the flash point of carbon containing 25 percent of this hydrocarbon was 86 °F.

Carbons Exposed in Submarines

Table 6 lists the flash point tests made with samples of carbon taken from the main filter beds of several different nuclear submarines. Varying amounts of carbon were used to assure the lowest flash point temperatures; these were found with test samples of 10 grams in agreement with tests with carbon containing pure hydrocarbons (Table 4). It is of interest that the flash points obtained for samples C-214 and C-228 were as low as 160° -165 °F. It is reassuring that the flash points of these carbon samples containing high

Table 5
Flash Points of Hydrocarbon Mixtures Adsorbed on Carbon

Hydrocarbon Mixtures	Sample (gram)	Fuel on Carbon (percent)	Flash	Flash Point or Max Temp (°F)
JP-4	10	20	No	150
	10	25	Yes	135
	10	30	Yes	100
	10	30	Yes	97
	Liquid	-	Yes	<78
JP-5 (Spec. limit, FP > 140°F)	10	25	No	225
	10	30	No	215
	10	30	Yes	205
	20	37	Yes	165
	10	40	Yes	155
	Liquid	-	Yes	155
2190 Lube Oil	10	25	No	525
	Liquid	-	Yes	430
n-Hexane	10	20	No	100
	10	25	Yes	86
	10	30	Yes	<78

Table 6
Flash Points of Carbon from Main Filter Beds of Submarines

Submarine	Sample No.	Exposure Time (weeks)	Oil by Analysis* (percent)	Sample (gram)	Flash Point (°F)
HALIBUT	C-197	Not exposed	0.0	5	>210
				10	>200
				20	≥545
HALIBUT	C-195	3	18.3	5	>225
				10	>540
ABRAHAM LINCOLN	C-214	4	23.2	5	175
				10	165
				15	190
				20	185
SNOOK	C-222	2	21.5	5	197
				10	185
				15	218
				20	228
SNOOK	C-228	4	22.5	10	160
THOMAS JEFFERSON	C-249		18.9	10	195
ALEXANDER HAMILTON	C-251		27.8	10	175
ALEXANDER HAMILTON	C-251A		28.3	10	170

*Percent of hydrocarbon oil desorbed from the exposed carbon based on weight of dry carbon.

percentages of oil are reasonably higher than usual ambient temperatures, indicating a good margin of safety.

SPONTANEOUS IGNITION STUDIES

Another important consideration concerning main filter carbon as a fire hazard in nuclear submarines is the possibility of the occurrence of spontaneous ignition. The SIT of a given substance may be defined as the lowest temperature at which the substance will ignite in air at atmospheric pressure without a spark, flame, or other outside source of ignition.

In SIT studies, it is important to realize that a number of experimental factors have an important bearing on the minimum ignition temperature, ignition time delay, and character of ignition or resultant combustion. Several of the more important factors which must be considered are: rate of airflow, heat transfer between the sample and the ignition system, geometry of the system, composition of the atmosphere, atmospheric pressure, and both heterogeneous and homogeneous catalysis.

Three different systems, two static and one with air flowing, were used to help insure that no important variable would be overlooked in these studies.

The SIT was determined for a number of samples by a standard ASTM method (6). In addition, some data were taken at superatmospheric pressures in air and in oxygen which were helpful in obtaining a better overall understanding of the systems involved here. Finally, an important series of experiments were made in a flow system in air at atmospheric pressure.

Spontaneous Ignition at Atmospheric Pressures – Static Conditions

For the acquisition of spontaneous ignition data which could be related to the ignition data for many substances, a procedure (6) was used based on ASTM D286-30. Essentially the procedure is to introduce the sample into a heated 200-ml Pyrex flask and record evidence of ignition or nonignition.

Table 7
Spontaneous Ignition Temperatures (SIT) of Hydrocarbons on Carbon by Static Method

Material	SIT Found (°C)			Literature* SIT of Liquid (°C)
	Liquid	On Coconut Charcoal	On Coal-Based Charcoal	
n-Decane	204	227	224 (CF 222)†	208
n-Hexane	239	-	260 (CF 256)	234
1-Octene	241.5	266	275.5 (CF 254)	256
2190 Oil	369	-	410 (CF 400)	-

*W.A. Affens, J.E. Johnson, and H.W. Carhart, "Ignition Studies, Part VI – The Effect of Chemical Structure on the Spontaneous Ignition of Hydrocarbons," NRL Report 5566, Nov. 3, 1960.

†CF = Cool flame appearance, minimum temperature.

A summary is given in Table 7 of SIT's found for several hydrocarbon materials as liquids and adsorbed on carbon. Efforts were made to find the optimum conditions which would give the lowest ignition temperatures. The most important observation from Table 6 is that adsorption on carbon did not lower the SIT of these materials. Although the cool-flame-appearance temperature was lower than the SIT of the carbon samples, it was higher than the SIT of the fluids. In every case the ignition temperature was higher for the carbon samples. Additional data for SIT of these liquids from the literature (14) are included for comparison.

In addition to the data in Table 7, certain detailed observations are of interest. In Fig. 4, data are plotted which show the effect of increasing the content of n-decane on coal-based carbon. In Fig. 5, a plot for n-decane on coconut charcoal shows a very similar ignition characteristic. In Fig. 6, the time delay at a given ignition temperature for n-decane on carbon shows the typical logarithmic curve found for spontaneous ignition of hydrocarbons. It is interesting that the data for carbon containing 13 to 30 percent n-decane all fit the same time-delay curve.

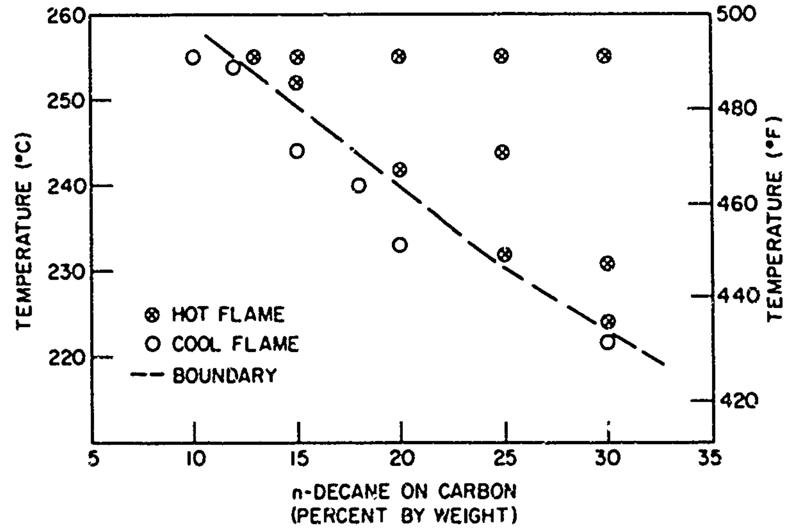

Fig. 4 - Effect of amount of adsorbed n-decane on spontaneous ignition of coal-based carbon

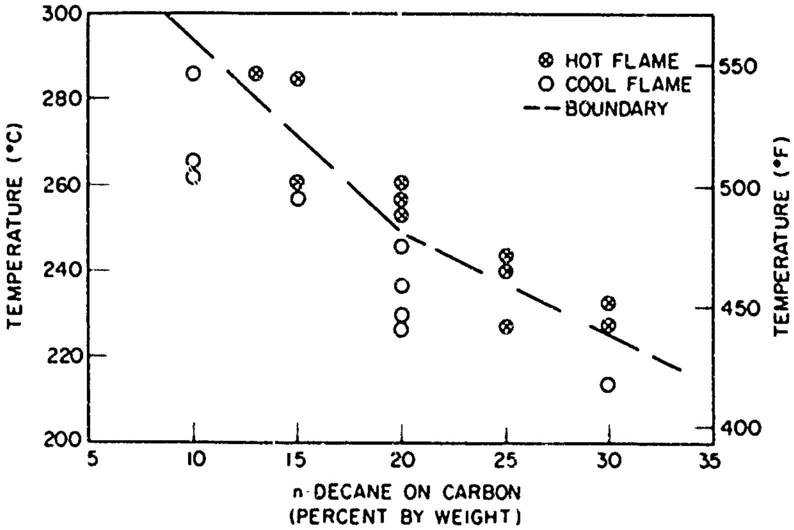

Fig. 5 - Effect of amount of adsorbed n-decane on spontaneous ignition of coconut carbon

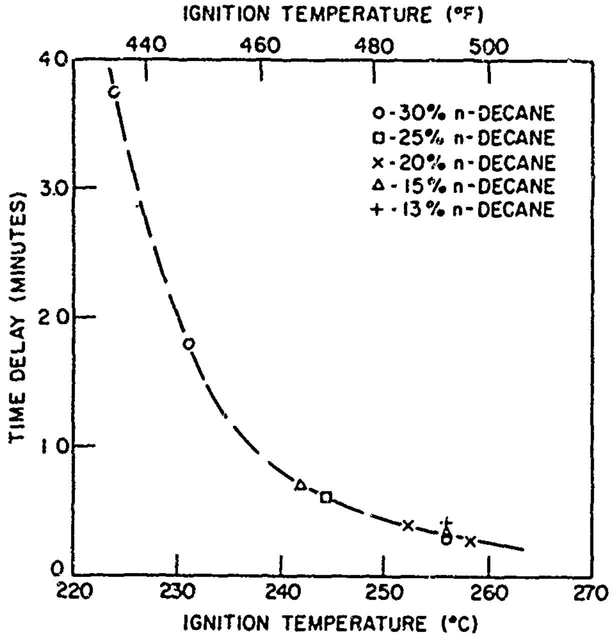

Fig. 6 - Effect of temperature on ignition delay of carbon with adsorbed n-decane

It should be noted for the 2190 lube oil (Table 7) that fog-like aerosols appeared in the ignition flask as much as 325°F (180°C) below the SIT. A heavy brown, tarry residue was left in the bottom of the flask when ignitions did occur. The white smoke (aerosol) made visual detection of ignitions difficult. Hot flames were indicated by a definite popping noise and ejection of a glass wool stopper, and by the sharp break in the time-temperature curve being recorded. This aerosol formation was not noted with samples of carbon from submarines except C-195 (Table 8). This sample had a high SIT, 770°F (410°C), similar to the high value for 2190 oil on carbon (770°F).

Table 8
Spontaneous Ignition of Activated Carbon Exposed to Submarine Atmospheres

Sample No.	Submarine	Submarine Exposure Time (weeks)	Hydrocarbon Content* (percent)	SIT† (°C)
C-195	HALIBUT	4	18.3	410
C-214	ABRAHAM LINCOLN	4	23.2	282‡
C-222	SNOOK	2	21.5	268
C-225	PATRICK HENRY	6	2.7	>427
C-228	SNOOK	4	22.5	255 (CF 245)¶
C-237	ETHAN ALLEN	20	17.7	264 (CF 255)
C-238	SEAWOLF	28	0.7	>427

*Percent of hydrocarbon oil desorbed from the exposed carbon based on weight of dry carbon.

†SIT = Minimum spontaneous ignition temperature in air at atmospheric pressure determined by the static method.

‡SIT of desorbed oil was 242°C (CF 235°C).

¶CF denotes "cool flame ignition." In this case cool flames occurred as low as 245°C although the SIT (hot flame) was 255°C.

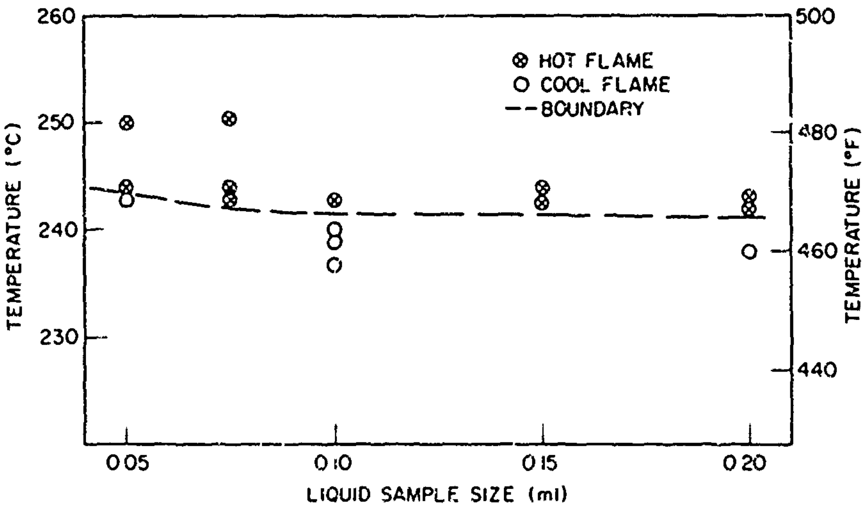

Fig. 7 - Effect of sample size on minimum ignition temperature of an oil desorbed from exposed activated carbon (C-214)

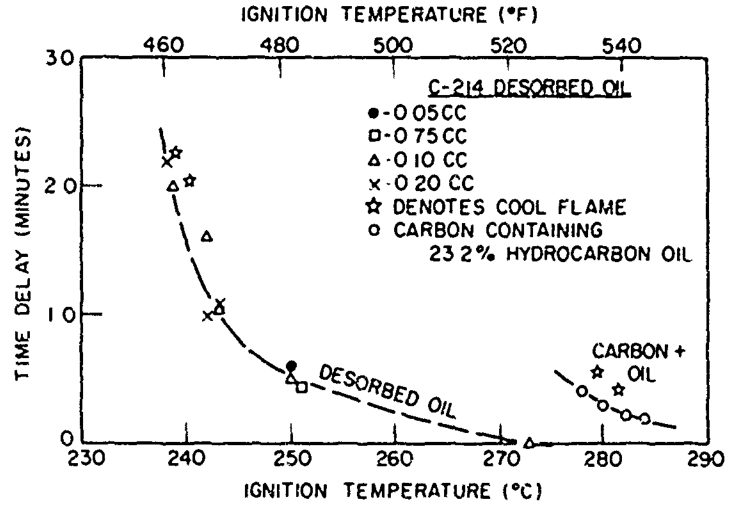

Fig. 8 - Effect of temperature on ignition delay of a submarine exposed carbon (C-214) and the hydrocarbon oil desorbed from this carbon

Spontaneous ignition data for a number of samples of carbon which had been taken from the main filters in submarines are summarized in Table 8. The hydrocarbon content of these carbon samples was determined by the steam desorption method (2). Sufficient desorbed hydrocarbon oil was obtained in the case of one sample (C-214) to permit the determination of the SIT of the oil 468°F (242°C) for comparison with the SIT of the carbon sample 540°F (282°C). The complete data for this oil sample are plotted in Fig. 7 to show the consistency of the results. The data for the ignition delay of carbon sample C-214 and the liquid are given in Fig. 8 for comparison. This shows clearly the higher temperature required to ignite the carbon sample than the liquid. Also note the lack of any change in ignition delay due to liquid sample size.

Spontaneous Ignition in a Flow System at Atmospheric Pressures

An important aspect of the possible ignition hazards associated with the carbon beds in the air purification system of nuclear submarines which had to be taken into account was the effect of airflow. In order to study this factor, a special bench-scale apparatus was used to simulate a carbon bed exposed to a stream of heated air. It consisted basically of a heated tube in which a given amount of carbon could be suspended (Appendix A). In most of this experimental work, four grams of carbon were exposed at an airflow of two liters per minute. These conditions correspond approximately to a 225-pound bed of carbon at an airflow of 2000 cfm. Some work was done with a similar apparatus in which the carbon was suspended in the heated tube in a cylindrical container with a stainless steel wire bottom. This configuration was used to insure that all the heated air passed through the carbon bed.

Ignition of Unused Carbon in Air - A series of experiments in which activated coconut carbon samples were exposed to heated air streams at successfully higher initial tube temperatures is plotted in Fig. 9. These data will serve to indicate the type of curves obtained when the temperature of the carbon bed versus time is plotted. When the stainless steel wire basket containing the carbon was placed in the center of the heated tube at a relatively low temperature (T_1 = 446°F (230°C)), the temperature of the charcoal rose quickly as shown by curve C_1, and after a few minutes it slightly exceeded the initial temperature of the tube, showing very little reaction exothermicity. As the initial tube temperature was increased successively to T_2, T_3, etc., the curves C_2, C_3, etc. were obtained and the exothermic temperature increase became much more pronounced. At T_5 (770°F (410°C)), the SIT of the carbon was reached and the sample burned to an ash.

Ignition of Unused Carbon in Air vs Nitrogen - In order to see the role of oxygen in these exothermic processes, the experiment depicted in Fig. 10 was conducted. In the first curve, C_1, the tube was heated to a constant temperature of 770°F (410°C) (at which charcoal burned spontaneously in air) with 100% nitrogen flowing through the tube at 2.0 l/min. The temperature of the charcoal bed reached the temperature of the tube in 5 minutes and after 20 minutes exceeded the tube temperature only slightly. In a second run, air was substituted for the nitrogen flow after 25 minutes. The temperature of the carbon increased very rapidly as shown by curve C_2 and reached a maximum rise above the tube temperature of 280°F (155°C). The carbon continued to burn for 90 minutes.

Ignition of Carbon Contaminated with n-Decane - In the series of runs shown in Fig. 2, the coconut charcoal was impregnated with 25 percent of its own weight of n-decane. As with the coconut charcoal alone (Fig. 9) when at the relatively low temperature (T_1 = 446°F (230°C)), the temperature of the carbon increased rapidly until it finally exceeded the air temperature slightly as shown by curve C_1. But when the air temperature of the tube was increased by 20°F (10°C) (T_2 = 464°F (240°C)), the carbon attained a ΔT of 352°F (178°C) as in curve C_2. In curve C_3, with an air temperature of 554°F (290°C), the time delay for the exothermic reaction was much reduced. At T_4 (curve C_4), flames were noted about the basket of carbon containing n-decane. These flames lasted only a few seconds although the charcoal glowed for a little longer. At T_5, the flaming occurred and died out but the carbon continued to burn as a glow. This ignition temperature is 54°F below the ignition temperature of the carbon alone (Fig. 9).

Figure 11 is a graphical presentation of the maximum temperature rises, ΔT, obtained from the carbon alone compared with carbon plus 25% n-decane (see Figs. 2 and 9). The ΔT for carbon alone begins to increase noticeably at a tube temperature of about 500°F (260°C) and increases to a ΔT of about 270°F (150°C) at the initial tube temperature of 770°F (410°C) where it ignites and continues to burn. The carbon containing 25-percent adsorbed n-decane showed a sharp rise in ΔT between 446°F (230°C) and 464°F (240°C).

In Fig. 12, similar data for a coal-based carbon alone, and the same carbon containing 25-percent n-decane, and containing 25-percent lubricating oil (military Symbol 2190) are given. It is noteworthy that the n-decane and the lube oil yielded very similar values for ΔT at a given tube temperature. The charcoal did not continue to burn until the set

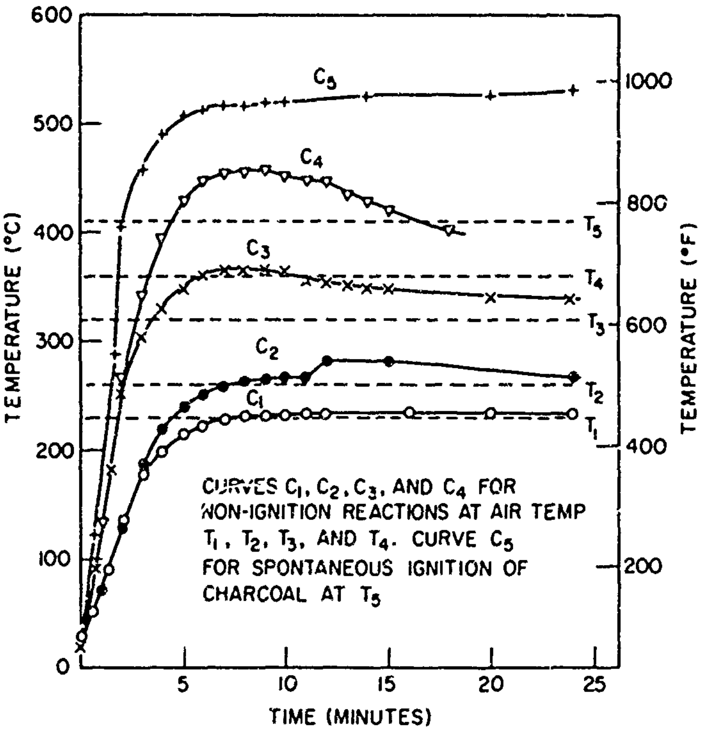

Fig. 9 - Time-temperature curves for 4 g coconut charcoal (F-1) exposed to heated air (2 l/min) in combustion tube at various initial air temperatures

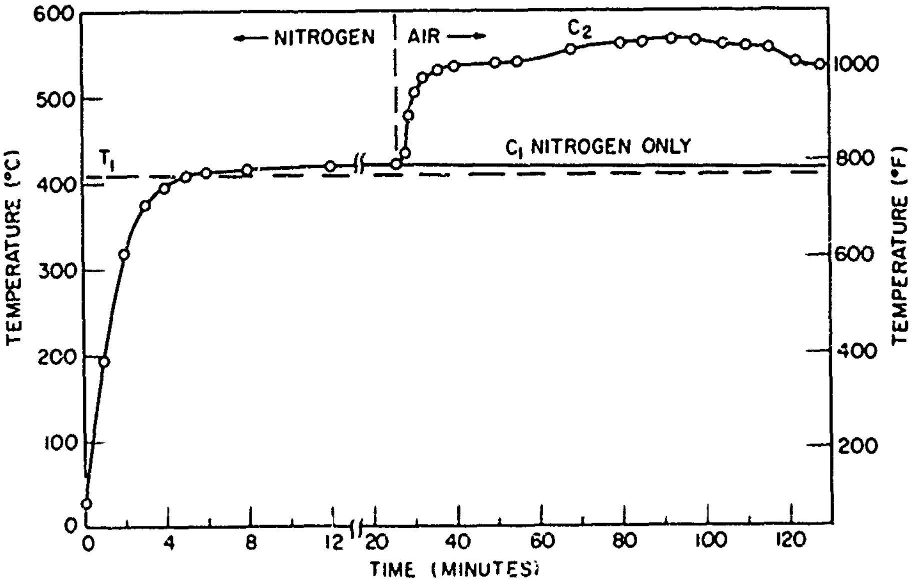

Fig. 10 - In curve C, coconut charcoal exposed to heated N_2 stream at temperature T_1 = 770°F (410°C). Curve C_2 shows spontaneous ignition of charcoal when N_2 replaced by air after 26 minutes.

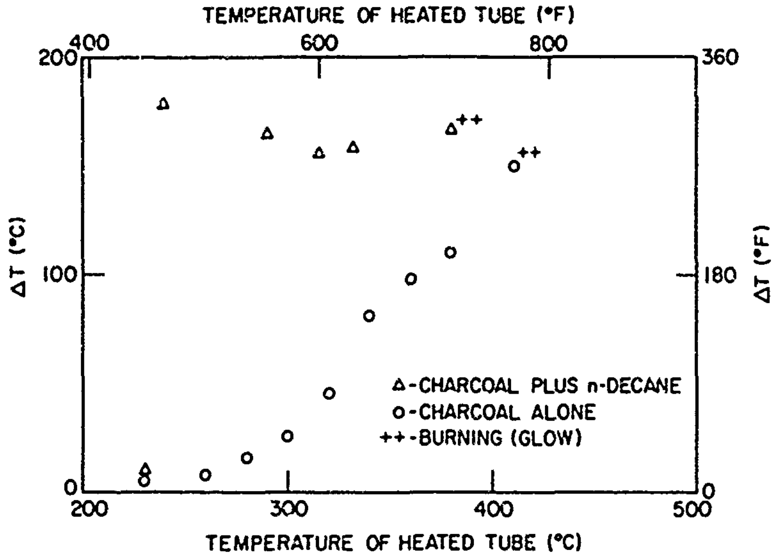

Fig. 11 - The effect of initial air temperature on ΔT for coconut charcoal (F-1) with and without adsorbed n-decane

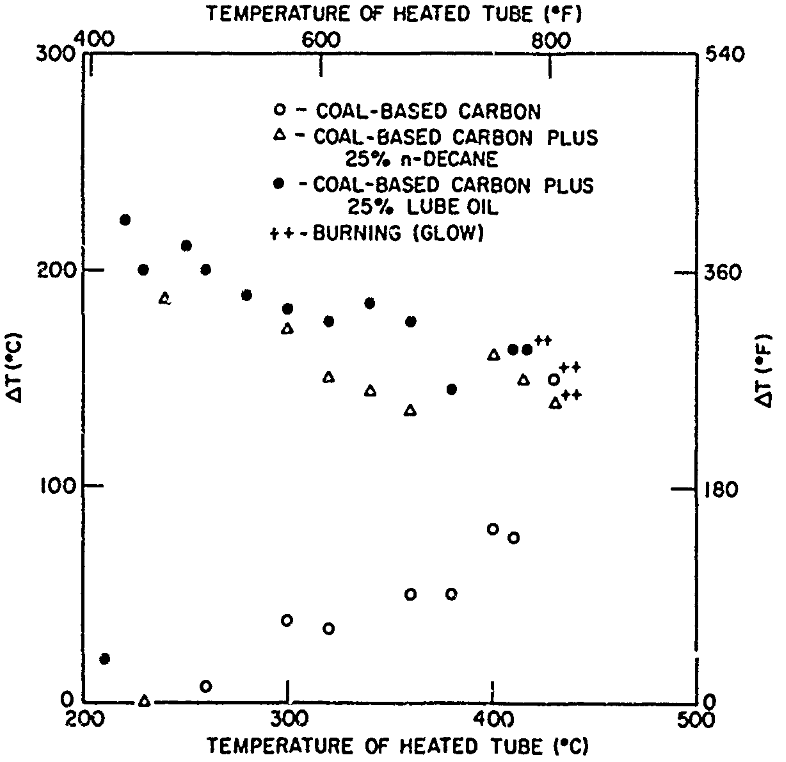

Fig. 12 - The effect of initial air temperature on ΔT for coal-based carbon (Dorex) as noted

temperature of the tube was at the burning temperature of the charcoal alone, even though with n-decane or 2190 oil the exothermic temperature rise exceeded the burning temperature of the carbon alone.

Ignition of Carbon Which Had Been Exposed in Submarine Air - Several samples of coconut charcoal which had been exposed in the main filter beds of nuclear submarines

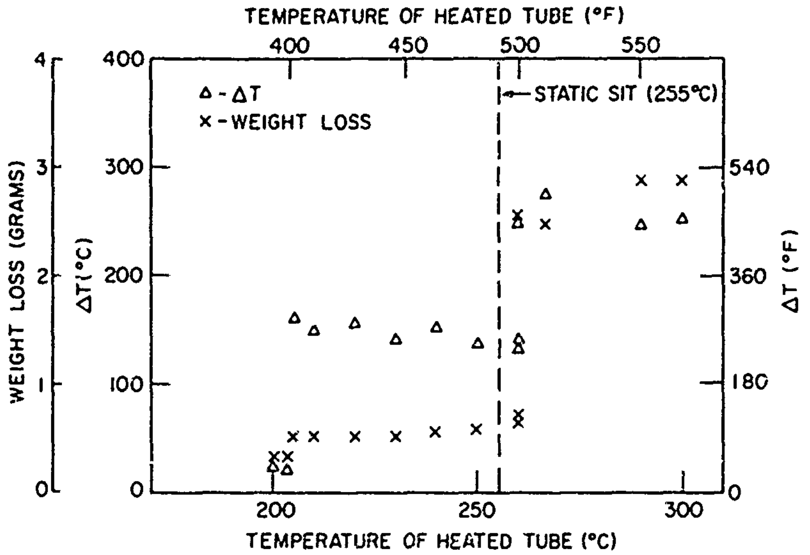

Fig. 13 - The effect of initial air temperature on ΔT and weight loss of coconut carbon (C-228) which had been exposed in submarine air

were tested in the heated airflow system. In Fig. 13 is the exothermic temperature rise ΔT as the initial tube air temperature is increased for carbon sample C-228 which came from the USS SNOOK. At an initial tube temperature of 400 °F (205 °C), the ΔT reached about 290 °F (160° C). This ΔT held steady until at an initial temperature of 500° F (260° C). This ΔT held steady until at an initial temperature of 500 °F (260° C) the ΔT abruptly increased to >450°F (250° C). The loss in weight was quite constant from 401° F (205° C) to 500°F (260°C), where spontaneous ignition occurred. At higher temperatures the carbon burned to an ash with a large weight loss.

The graphical data for the other submarine-exposed charcoal samples are given in Figs. 14a, 14b. and 14c. The results are similar to those obtained with sample C-228. In addition, data given in Fig. 14a at flow rates between 1.0 and 4.0 l/min indicate that the maximum reactivity is obtained at 2 l/min. This flow rate was used in most of the experiments.

A sample of unused coconut charcoal such as is used in the submarine filters was also studied with and without n-decane. The data were similar to that given in Fig. 11.

Spontaneous Ignition at Elevated Pressures

Although the atmospheres in submarines are not ordinarily enriched with oxygen much beyond the 21 percent found in terrestrial atmospheres, and atmospheric pressures in submarines are not usually much greater than about 760 mm of Hg, abnormal conditions in a nuclear submarine can occur on some occasions. Consequently, to learn what the trend would be in such instances, some ignition data were obtained at higher pressures in both air and oxygen. The pressures used were greatly exaggerated to establish a more complete ignition picture.

The data at elevated pressures is presented most clearly in a graphic form. In Fig. 15, ignition data for n-hexane on charcoal as well as on asbestos are given. These data represent minimum ignition temperatures in air up to 400 psi. Because asbestos had been shown in earlier experiments to have no catalytic effect on the spontaneous ignition of hydrocarbons, n-hexane was exposed on both carbon and asbestos. The asbestos was heated before use in a flame to remove any organic debris. There was a slight lowering

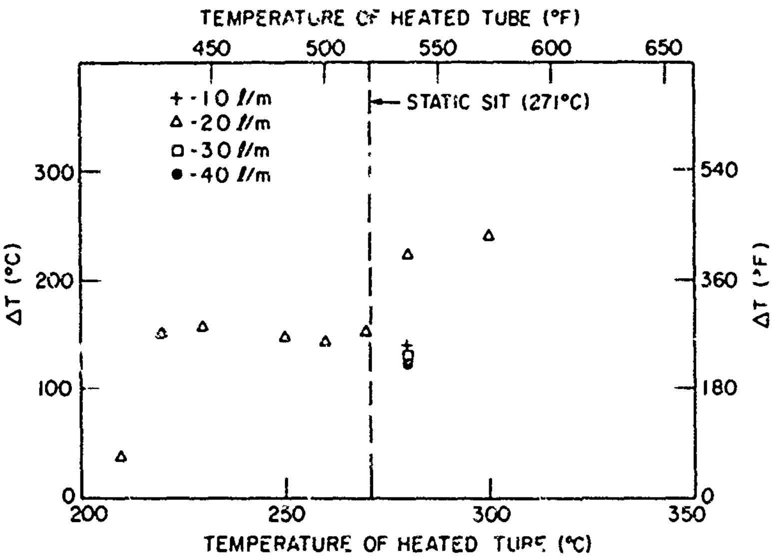

Fig. 14a - The effect of initial air temperature on ΔT of coconut carbon C-237 at various flow rates

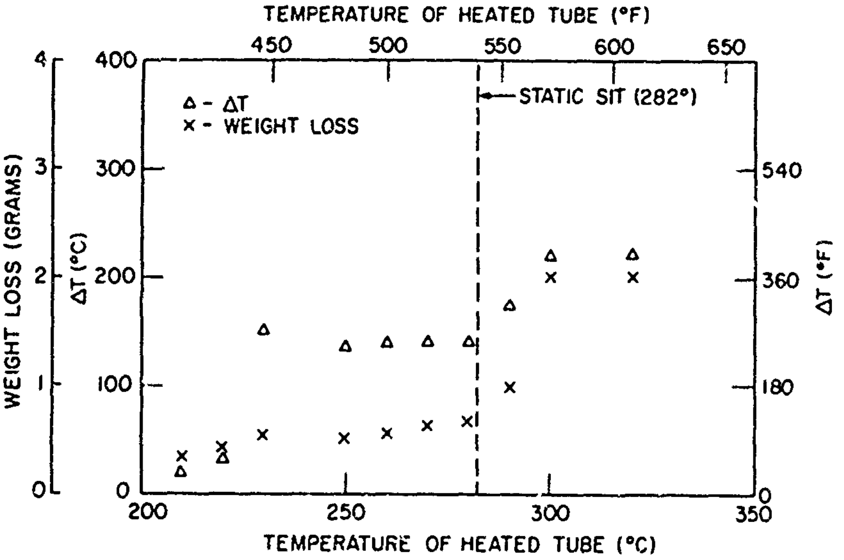

Fig. 14b - The effect of initial air temperature on ΔT and loss in weight of coconut carbon C-214

of ignition temperature in going to 100 psi, but no trend to lower temperatures as the pressure was increased further to 400 psi. The ignition temperature was always somewhat higher on carbon than on asbestos with this and other samples.

As an example of a relatively nonvolatile hydrocarbon mixture, Navy Symbol 2190 lubricating oil is compared with n-hexane in Fig. 16. These data show that the ignition temperature of 2190 oil on carbon is much higher at atmospheric pressure, but approaches that of n-hexane at 200 psi or more. At pressures of 100 psi or higher, the ignition temperature is not significantly influenced by increasing pressures in either case.

The data in Fig. 17 show the effect of using oxygen instead of air on the ignition temperatures of 2190 oil on carbon. There is a general lowering of approximately 90°F (50°C) due to using oxygen.

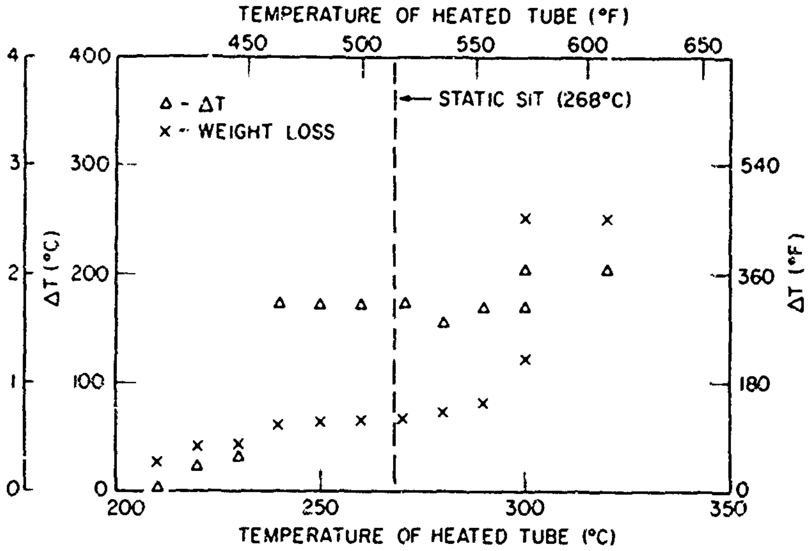

Fig. 14c - The effect of initial air temperature on ΔT and loss in weight of coconut carbon C-222

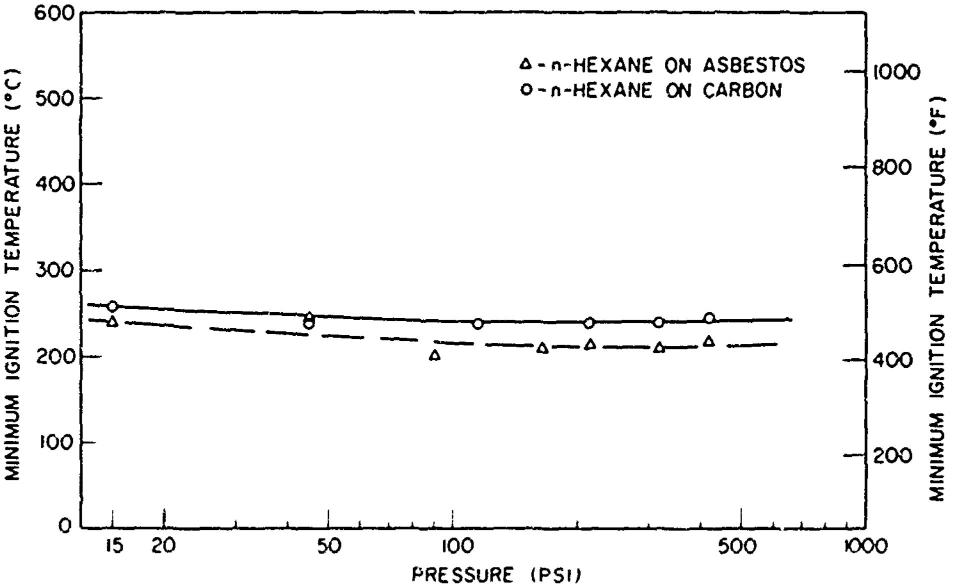

Fig. 15 - The effect of air pressure on the spontaneous ignition temperature of 25-percent adsorbed n-hexane on asbestos and coal-based carbon (Dorex)

The data obtained for several submarine carbon samples at different air pressures are given in Fig. 18. Samples C-214 and C-222 show very little effect of increased pressure, the same as n-hexane on carbon (Fig. 15). C-195, which had a much higher SIT at atmospheric pressure, shows a pronounced drop in ignition temperature in going to 200 psi air, although the final ignition temperature is not as low as the other two carbons. The C-195 sample, therefore, acted very much like the sample of carbon containing 2190 oil (Fig. 16). It is of interest that C-197, an unused carbon, had ignition temperatures of about 570 °F (300°C) at air pressures of 500 psi or greater, but no ignition was observed at air pressures less than 400 psi.

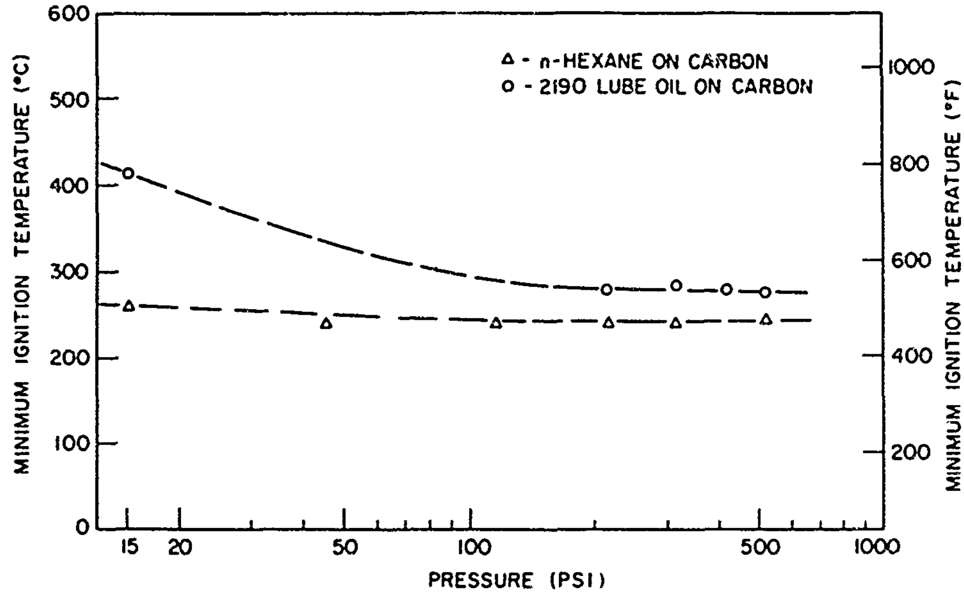

Fig. 16 - The effect of air pressure on the spontaneous ignition temperatures of 25-percent 2190 lube oil compared with 25-percent n-hexane adsorbed on coal-based carbon (Dorex)

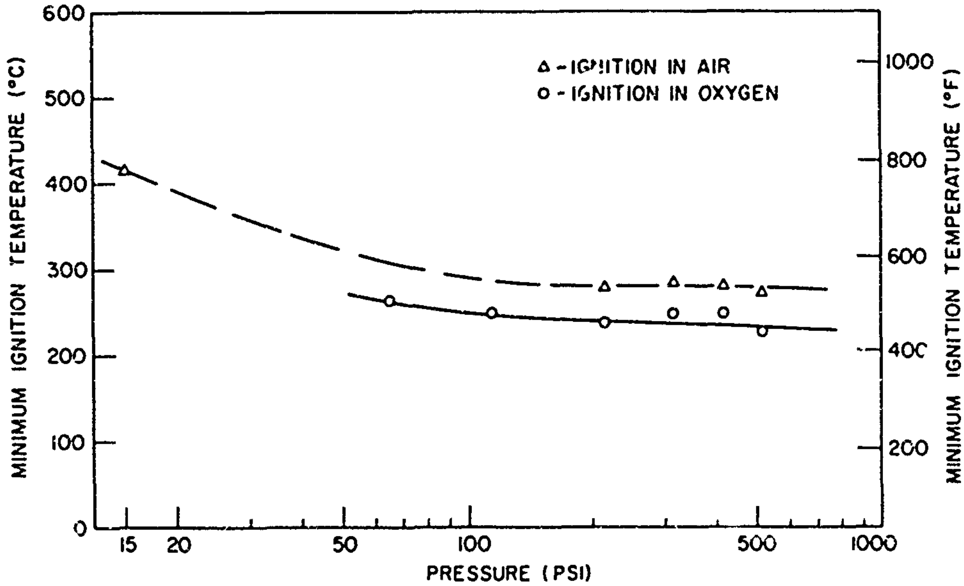

Fig. 17 - The effect of oxygen versus air pressure on the spontaneous ignition temperatures of 2190 lube oil on coal-based carbon (Dorex)

A typical effect of substituting oxygen for air in studying ignitions at higher pressures is shown in Fig. 19. Carbon sample C-222 has a decidedly lower ignition temperature in oxygen than in air. This is similar to the result cited earlier for 2190 oil on carbon (see Fig. 17).

Gaseous Products of Oxidation and Combustion

Some exploratory measurements were made of the chemical components of the effluent gas stream during burning of carbon and hydrocarbon-impregnated carbon. The

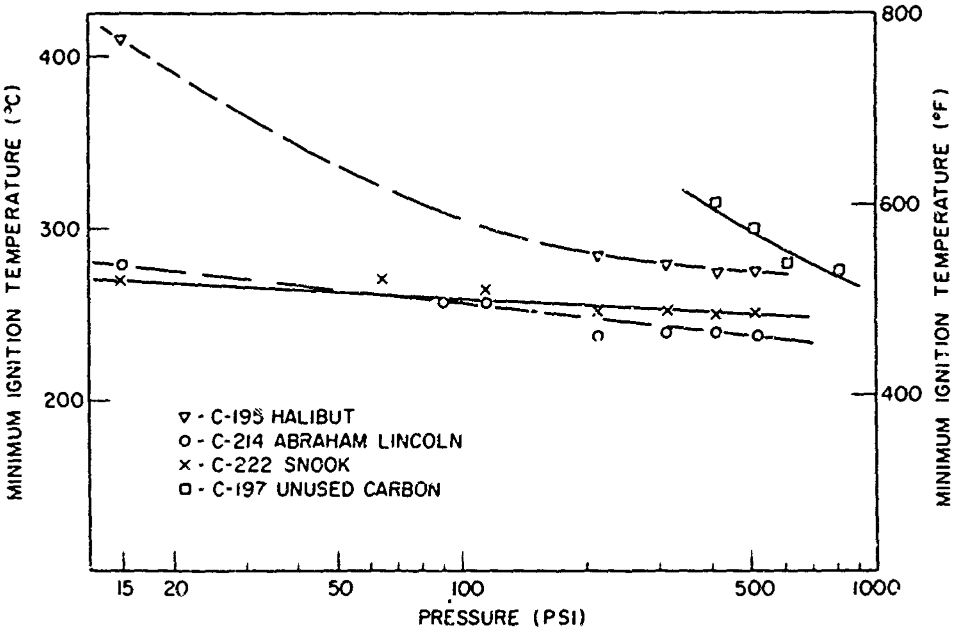

Fig. 18 - The effect of air pressure on the spontaneous ignition temperatures of carbon which had been used in main filter beds of nuclear submarines. See Table 8 for details of exposure and hydrocarbon loading of these carbon samples.

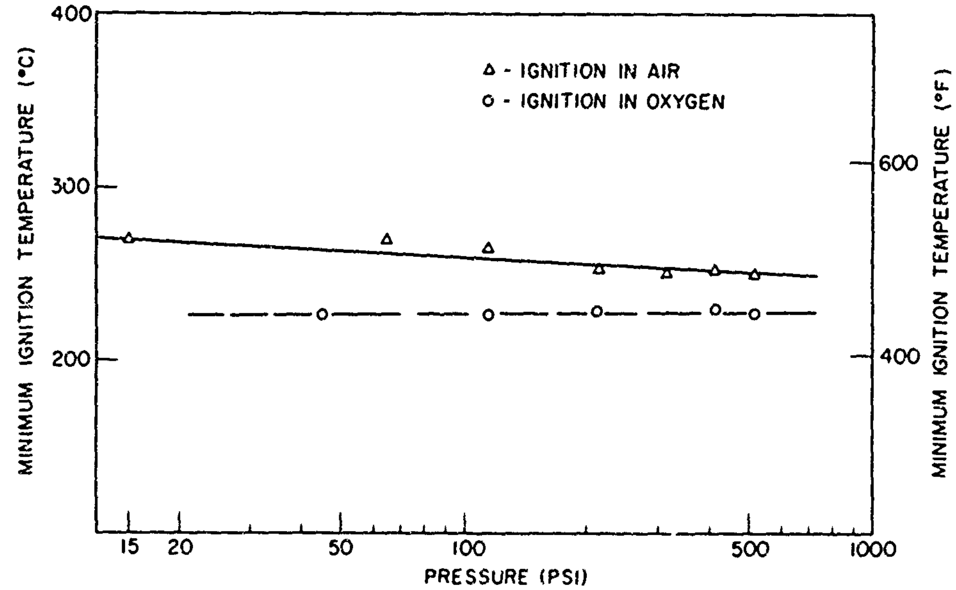

Fig. 19 - The effect of oxygen versus air pressure on the spontaneous ignition temperature of an exposed carbon C-222

effluent gas was sampled continuously and analyzed for CO, CO_2, and hydrocarbons by direct reading Beckman nondispersive infrared analyzers. The O_2 concentration was followed by means of a Beckman Model F-3 oxygen analyzer. The hydrocarbon detector used is not truly specific for hydrocarbons alone but will respond to many organic compounds, especially those containing aliphatic groups, because the detector is sensitized to n-hexane. Nevertheless, it was felt that this method would give a good indication of organic compounds in the effluent gas mixture.

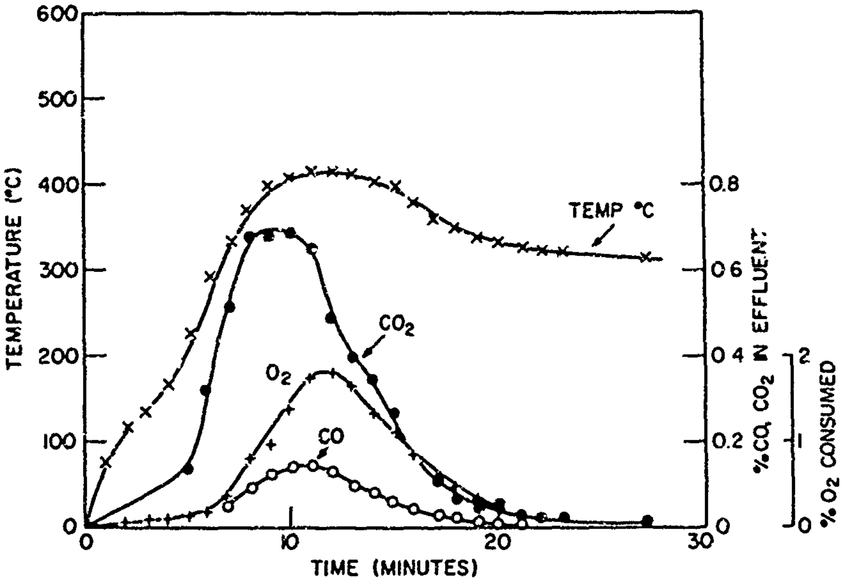

Fig. 20 - Composition of effluent gases from carbon C-197 suspended in heated air stream

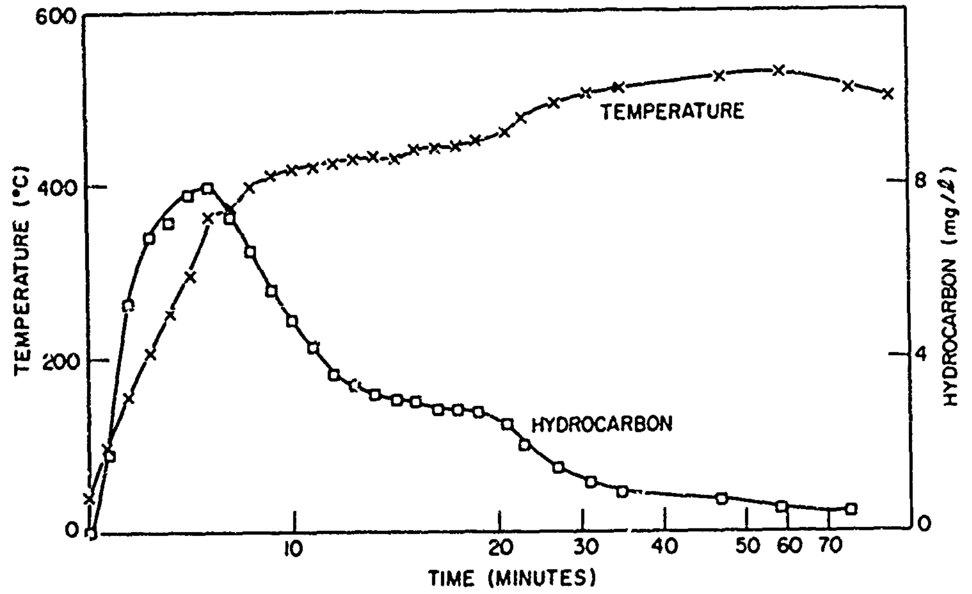

Fig. 21 - Temperature of carbon bed exposed to heated air stream 535 °F (280°C), and hydrocarbon content of the effluent air

A sample of unused coconut carbon C-197 was placed in an air stream at 535°F (280°C), which was well below its ignition temperature of 645°F (340°C). The combustion products as measured are shown in Fig. 20. Peaks in the curves for temperature and the concentrations of CO_2, CO, hydrocarbon content, as well as oxygen consumption, occurred after about 10 minutes had elapsed. The CO_2 reached a high of about 0.7% which was not unexpected. CO reached about 0.15%, and a reading of 0.5 mg/l of hydrocarbon was indicated. After 20 minutes the activity had subsided.

A sample of carbon, C-228 which had been used in a submarine, was similarly exposed to air at 535°F (280°C). The data are plotted in Figs. 21 and 22. In this case, the temperature rose to a level of about 800-820°F (425-440°C) for 10 minutes or longer, followed

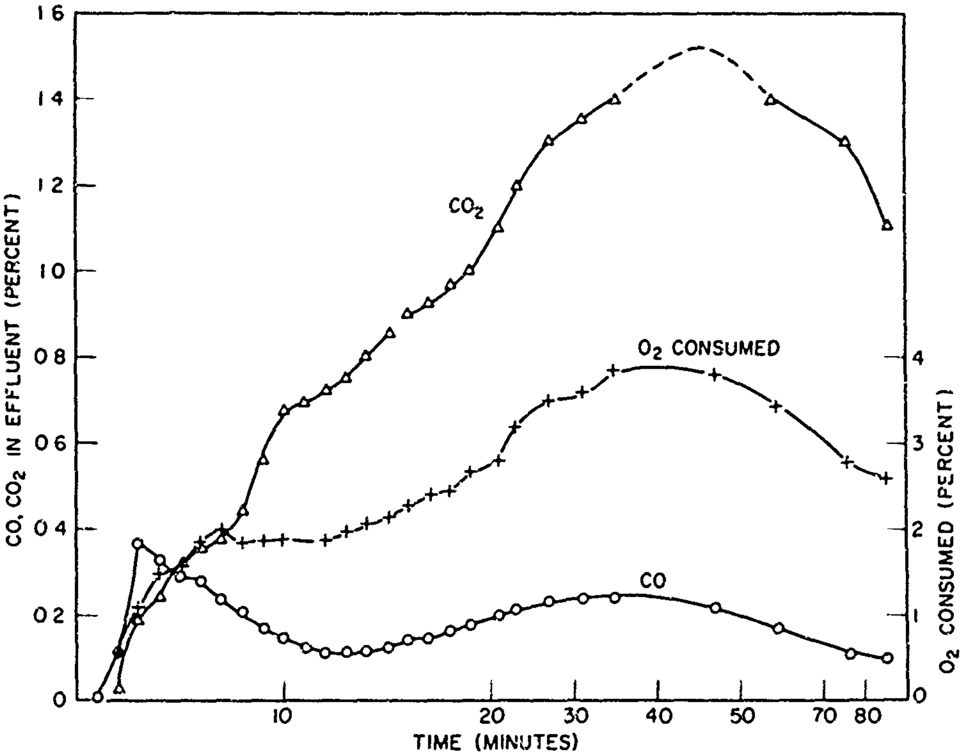

Fig. 22 - Composition of effluent gases from carbon C-228 exposed to heated air stream 535°F (280°C)

by a further climb to a maximum of 985°F (530 °C). The oxygen consumption curve in Fig. 21 shows this two-stage effect even more markedly.

The hydrocarbon detector showed a maximum at about 6 minutes after which it fell off fairly rapidly. The carbon monoxide reached almost 0.4% in a first peak, with a second peak of 0.25%. CO_2 reached more than 1.4% after a fairly steady climb of about 35 minutes.

These data show a complex pattern in the composition of the effluent from these systems. The relative response of the hydrocarbon detector, showing a maximum equivalent to 9.0 mg/l of n-hexane, shows that a considerable amount of organic materials is evolved without complete combustion. The breaks in the temperature, and oxygen consumption curves, when compared to the evolved gases, indicate a strong possibility that the organic material must be desorbed for the most part before actual combustion of the carbon gets underway.

Experimentation is underway which is designed to study the effects of airflow, sample size, configuration of apparatus, and effluent composition, other factors on ignition temperatures. Preliminary results have been obtained with a full flow carbon bed design in which all the air passes through the carbon. Significant oxidation occurs at lower temperatures than with the earlier apparatus described in Appendix A. For example, 8 grams of carbon sample C-228 exposed to two liters per minute of air at 392°F (200°C) in a one-inch tube burned actively with the CO_2 in the effluent reaching concentrations of more than 10%, and the CO approaching 10%.

It will be of interest from several standpoints to obtain more data concerning the combustion products of these carbon/hydrocarbon systems. For example, Boden, Cullis, and Fish (11) made somewhat similar studies with carbon, carbon/ethanol, and carbon/benzene. In the combustion of a coconut shell carbon at 482°F (250°C), no carbon monoxide was detected. However, with ethanol present up to 0.2% CO was found. With benzene, only CO_2 and benzene were found in the effluent, no CO being reported.

Lambert (12) has reported that some CO is formed from coconut carbon during oxidation from 480°-930°F (250°-500°C). Activated anthracite coal gave much more CO under these conditions, which Lambert attributed to the presence of iron.

We have found that the carbon which contained adsorbed hydrocarbons evolved large quantities of CO under the experimental conditions used in the present study. This may be attributed to incomplete combustion of the carbon which probably occurs at a lower temperature because of the presence of the hydrocarbon. Further study of this process is in progress.

DISCUSSION OF RESULTS

For the purposes of this discussion it is convenient to consider the ignition of hydrocarbon-impregnated carbons from two standpoints: namely, induced ignition and spontaneous ignition. In the case of induced ignition, when the local concentration of a hydrocarbon in air reaches a certain minimum value in terms of mg per liter, it will ignite if exposed to a spark, flame, hot body, or other source of ignition. It has been established by Zabetakis, Scott, and Jones (4) and confirmed at this Laboratory that if a local concentration of about 48 mg/l (STP) of most hydrocarbons in air is exceeded, it can ignite. This concentration value is lower at higher temperatures. The structure of the hydrocarbon molecules is relatively unimportant. The important factor is volatility.

Spontaneous ignition, on the other hand, is markedly influenced by molecular structure (13-15). The ignition is dependent on the initiation and acceleration of oxidation reactions which lead to an accumulation of reactive species, free radicals or molecules. When these reach a critical concentration, ignition ensues. Spontaneous ignition is very subject to catalysis, both homogeneous (16) and heterogeneous (17). For this reason, the possible catalysis by activated carbon was studied.

Induced Ignition

A convenient method for studying induced ignition of carbon samples containing hydrocarbons is the flash point method described earlier. In the case of a liquid hydrocarbon, the flash point temperature is that at which the concentration of hydrocarbon in the air has exceeded the lower limit of flammability.

It is to be expected that when the hydrocarbon is contained on carbon, the strong adsorption will tend to prevent escape of the hydrocarbon molecules to the air and have a flash point higher than the liquid hydrocarbon. In fact, the data in Table 4 show that a carbon saturated with hydrocarbon generally had a flash point about the same as the liquid hydrocarbon, but not lower. If the carbon was not quite saturated with hydrocarbon, a flash point somewhat higher than that of the liquid was obtained (see Tables 3 and 5). These observations are compatible with simple adsorptive behavior. The partial pressure of a hydrocarbon over carbon can never exceed the limiting value of the pure hydrocarbon, in the vapor phase and the flash point can approach, but not be lower than that for the pure hydrocarbon.

Samples of carbons containing hydrocarbon taken from the filter beds of operating submarines had flash points as low as 160°F (Table 6). It is certainly possible that the composition of the adsorbed mixture of hydrocarbons on a given carbon might allow flash points somewhat lower than 160°F. In any event, it is advisable to prevent the exposure of the carbon bed to heat which would tend to desorb hydrocarbon vapors. Also, it appears advisable to avoid allowing the carbon to become overly saturated with hydrocarbons.

Spontaneous Ignition

The spontaneous ignition (SIT) studies lead to the important conclusion that the activated carbon (coconut or coal-based) used for submarines appear to have no tendency to catalyze the ignition of the hydrocarbons retained on the carbon. None of the four pure hydrocarbons studied had a lower SIT on the carbon than in the liquid form (Table 7).

To check a phenomenon reported by Lewis (18), 1-octene was used. Lewis found that the SIT of the paraffin hydrocarbons were higher in the presence of carbon, but with amylene, an olefin, the SIT was lowered considerably. Lewis concluded that the metal oxides in the carbon were the actual catalysts for the olefin oxidation and ignition. The data obtained in the present work show no catalytic effect by the coconut carbon used in the main filter beds of submarines, nor by the coal-based carbon used in the samplers for analysis of hydrocarbons in submarine atmospheres. This is in spite of the high mineral contents shown in Table 1.

It is of interest that studies of ignition at higher pressures of air confirmed the non-catalytic character of spontaneous ignition of hydrocarbons in the presence of carbon (Fig. 15). The hydrocarbons adsorbed on carbon had a higher ignition temperature than the hydrocarbons on asbestos. This effect is due to the greater adsorptive effect of carbon to retain hydrocarbons and keep the vapor concentration lower at a given temperature.

It is also encouraging that for carbons having a relatively low SIT, there is only a slight lowering of minimum ignition temperatures by increased pressure of air (Fig. 18). These data discount the possibility that operating pressures in submarines of much more than 760 mm Hg or of air enriched with oxygen somewhat above 21 percent might lead to serious hazards due to spontaneous ignition of the carbon.

Studies by Boden, Cullis, and Fish (11) of the spontaneous ignition in carbon adsorption beds contain some items of interest to this discussion. These investigators were primarily concerned with instances of ignition after admission of air to activated charcoal beds on which solvent vapors had been adsorbed. For example, when heated carbon was exposed to a stream of gas containing benzene (10 percent), oxygen (20 percent), and nitrogen (70 percent), an exothermic reaction ensued yielding carbon dioxide only. They concluded that there is no appreciable catalysis of the oxidation of organic compounds by elementary carbon. The oxidation which has been observed is regarded as due to the presence of metal oxide impurities. It was found that the reactivities of all the carbons used are related to their ash contents. Analysis of the data from the studies of Boden, Cullis, and Fish indicates that a system such as is used in submarines, in which air is passed continuously through carbon beds, should not result in any significant temperature rise due to oxidation reactions which might lead to spontaneous ignition except at high temperatures ((ca 200°C) 392°F). This has been confirmed by the present studies.

Certain findings by Russian investigators (19) are of interest in regard to the self-ignition of carbon. Eight different types of carbon black from various sources were found to ignite in a current of dry heated air at temperatures of 687°-794°F (364°-423°C). The presence of moisture in the air had little effect on the ignition temperatures found. They found that admixture of iron and copper oxides did not have any appreciable effect on the temperature of self-ignition of carbon black. In the present work, the SIT of the coconut carbon used in the main filter beds of submarines was found to be >800°F (425°C) in air in the static system. In the flow system it was as low as 645°F (340°C).

The experimental data with the airflow system presented in this report confirm in general the findings of other investigators. As in all ignition experiments, the conditions of the test such as sample size, geometry of the system, and airflow rate must be defined. An important physical aspect of such systems is undoubtedly the rate of heat transfer and the heat capacity of the sample as well as that of the adjacent components of the apparatus.

A summary of the flow ignition data is given in Table 9. It is difficult to present relative ignition behavior of different samples obtained by different methods in a simple way. However, the data are given here in terms of ignition temperatures, i.e., the lowest initial air temperatures at which the carbon samples were exposed and continued to burn. The carbon samples taken from submarine filter beds all continued to burn under these conditions when exposed to flowing air of temperatures from 500°-610°F (260°-320°C). These ignition temperatures are comparable to that for sample C-197 of the same type of coconut carbon, which when impregnated with 25% n-decane had an ignition temperature of 555°F (290°C).

Table 9
Minimum SIT of Carbon and Carbons Containing Hydrocarbons Under Static and Dynamic Conditions at Atmospheric Pressure

	Static (Flask)			Dynamic (Heated Tube with Airflow)		
Sample	Carbon plus n-Decane (°C)	Carbon (°C)	Carbons Exposed to Submarine Atmospheres (°C)	Carbon plus n-Decane (°C)	Carbon (°C)	Carbons Exposed to Submarine Atmospheres (°C)
C-197	230*	>518‡		No (290)†	340†	
F-1	-	-		330* (380)†	410†	
DOREX	231*	>518‡		320* (430)†	430†	
C-214			282*			300†
C-222			268*			320†
C-228			255*			260†
C-237			265*			280†

*Flame up.
†Glowing (Burning).
‡No ignition.

The sustained glow which was characteristic of the combustion of carbon under these conditions is similar to the glow noted by Setchkin (20) for many solid materials. He observed that actual ignition of a solid material may result from the exothermic reaction of vaporized or gaseous components of the material with oxygen to yield a flame or explosion or through self-heating within the solid material itself which will yield a sustained glow. The rate of desorption of gaseous components, as well as the rate of oxidation of the atoms forming the surface of the solid are no doubt important. In all cases a self-perpetuating exothermic reaction must be attained. In some instances in the present study where carbon had been impregnated with n-decane, a transitory flame was seen which was probably due to the reaction of oxygen with n-decane vapor. The carbon itself did not necessarily burn on these occasions.

A specific instance is the data given in Fig. 11. At an air temperature of about 465°F (240°C), the ΔT of activated coconut carbon containing 25 percent n-decane rose sharply from <20°F (10°C) to 320°F (178°C). The same sharp temperature rise occurred when a coal-based carbon was used containing n-decane. A pronounced rise of about 335°F (185°C) resulted at an initial air temperature of about 445°-465°F (230°-240°C). Since the carbon did not continue to burn in either case, it indicates that some rapid oxidation reaction of n-decane occurred.

The phenomenon occurring when sample C-228 of a submarine-exposed carbon was given the same treatment is shown in Fig. 13. A sharp rise in ΔT occurred at about 400°F (205°C). This temperature rise was accompanied by a momentary bright glow that could be seen clearly in a darkened room. The carbon did not continue to burn. A second sharp increase in ΔT occurred at an initial temperature of 500°F (260°C), very close to the SIT found for this sample in the static test.

The second sharp rise in ΔT occurred when the charcoal began to glow and it continued to burn until only an ash was left. Under these conditions the ignition of sample C-228 occurred about 145°F (80°C) lower than the unexposed carbon sample, C-197. The loss in weight of the sample C-228 corresponded to the changes in ΔT as is shown in Fig. 13.

SUMMARY

1. Spontaneous ignition temperatures of submarine-exposed carbons were found to be as low as 490°F (255°C) in air at atmospheric pressure. It was observed that exothermic oxidative reactions occurred in the flow system at temperatures as low as 390°F (200°C).

2. Increased air pressure or oxygen pressure did not lower the spontaneous ignition of hydrocarbon-impregnated carbon below 390°F (200°C).

3. There appeared to be no catalytic action by the activated carbon in regard to the spontaneous ignition of hydrocarbons. In every case studied thus far the spontaneous ignition temperature was not lowered by the presence of the carbon.

4. The flash points of hydrocarbons contained on carbon approached but were not lower than the flash points of the hydrocarbons themselves. In the case of carbons which had been used in the main filter beds of submarines, the lowest flash point temperature found was 160°F.

5. Under flow conditions, combustion of carbon containing hydrocarbons evolved considerable quantities of carbon dioxide and carbon monoxide. Even under precombustion conditions, significant amounts of carbon monoxide were evolved.

REFERENCES

1. Nestler, F.H.M., and Smith, W.D., "Submarine Habitability, Atmosphere Sampling and Analysis," NRL Memo Report 866, Oct. 1958
2. Smith, W.D., and Johnson, J.E., "Concentration of Hydrocarbon Vapors in Submarine Atmospheres by Carbon Sampling," Chapter 10 in "Annual Progress Report, The Present Status of Chemical Research in Atmosphere Purification and Control on Nuclear-Powered Submarines," edited by Piatt, V.R., and Ramskill, E.A., NRL Report 5630, July 14, 1961
3. Johnson, J.E., "Nuclear Submarine Atmospheres, Analysis and Removal of Organic Contaminants," NRL Report 5800, Sept. 10, 1962
4. Zabetakis, M.G., Scott, G.S., and Jones, G.W., Ind. Eng. Chem. 43:2120 (1951)
5. American Society for Testing Materials, "Flash Point by Pensky-Martens Closed Tester," ASTM Method D93-58T, Oct. 1960
6. Zabetakis, M.G., Furno, A.L., and Jones, G.W., Ind. Eng. Chem. 46:2173 (1954)
7. Setchkin, N.P., J. Research Nat. Bur. Standards 53:49 (1954)
8. Johnson, J.E., Crellin, J.W., and Carhart, H.W., Ind. Eng. Chem. 44:1612 (1952)
9. Military specification, "Lubricating Oil, Steam Turbine (Noncorrosive)," MIL-L-17331B(SHIPS), Sept. 21, 1959; "Jet Fuel, Grades JP-4 and JP-5," MIL-J-5624F, Sept. 25, 1962
10. Dreisbach, R.R., "Physical Properties of Chemical Compounds – II," Advances in Chemistry Series No. 22, Washington, D.C.:American Chemical Society, 1959
11. Boden, N., Cullis, C.F., and Fish, A., J. Appl. Chem. 12:145 (1962)
12. Lambert, J.D., Trans. Faraday Soc. 32:452-62 (1936)
13. Johnson, J.E., Crellin, J.W., and Carhart, H.W., Ind. Eng. Chem. 46:1512 (1954)
14. Affens, W.A., Johnson, J.E., and Carhart, H.W., "Ignition Studies, Part VI – The Effect of Chemical Structure on the Spontaneous Ignition of Hydrocarbons," NRL Report 5566, Nov. 3, 1960
15. Affens, W.A., Johnson, J.E., and Carhart, H.W., J. Chem. Eng. Data 6:613 (1961)
16. Lewis, B., and von Elbe, G., "Combustion, Flames, and Explosions of Gases," 2nd ed., New York:Academic Press, 1961
17. Minkoff, G.J., and Tipper, C.F.H., "Chemistry of Combustion Reactions," p. 224, London:Butterworths, 1962
18. Lewis, J.S., J. Chem. Soc. 58 (1930)
19. Ermilov, P.I., Polyakov, Z.N., and Syshchikov, L.I., "On the Temperature of Self-Ignition of Carbon Black," Khimicheskaya Promyshlennost, No. 7, pp. 51 (435) to 52 (436) 1954, NRL Translation 879, 1962
20. Setchkin, N.P., "A Method and Apparatus for Determining the Ignition Characteristics of Plastics," Nat. Bur. Standards Research Paper RP2052, Dec. 1959

APPENDIX A

METHOD FOR STUDYING SPONTANEOUS IGNITION UNDER FLOW CONDITIONS

The apparatus for studying spontaneous ignition phenomena of materials under flow conditions is sketched in Fig. A1. It consists of a vertical combustion tube which is coated on the outside with a very thin conducting layer of tin oxide, which allows the tube to be heated electrically without obscuring visual observation of phenomena occurring in the combustion chamber. The tube is two inches in diameter, 29 inches long, contains a bed of glass beads at the bottom end, and is open at the top. Air or other desired gases are metered and fed into the tube below the glass beads which impart an essentially laminar flow to the air stream passing through the combustion tube. Two thermocouples measured the temperature of the charcoal bed in a stainless steel basket suspended in the tube and the air in the tube near the basket.

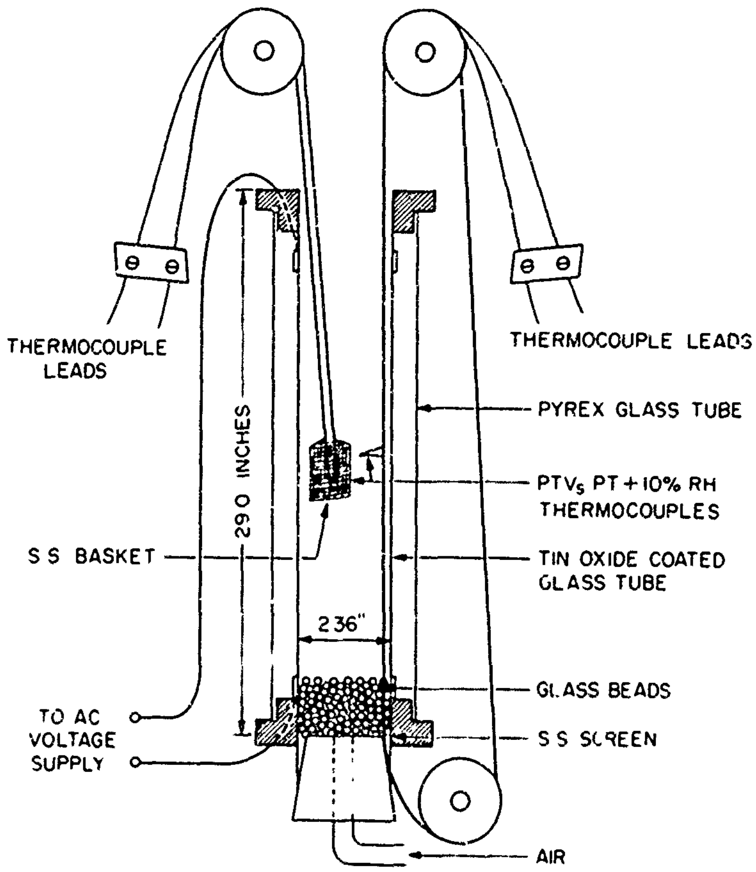

Fig. A1 - Apparatus for determining SIT under flow conditions

For a given experiment, the combustion tube is preheated to the desired temperature with the desired flow of gas. When these conditions are achieved, the basket containing the measured amount of carbon is lowered to the center of the tube. A time-temperature record is made, and analyses made on the effluent gas as desired.

APPENDIX B

METHOD FOR STUDYING SPONTANEOUS IGNITION AT ELEVATED PRESSURES

The apparatus for studying spontaneous ignition of materials at elevated pressures is shown in Fig. B1. The apparatus consists of a cylindrical cell of an appropriate metal (copper and stainless steel were used) with facilities for pressurizing with the desired gas and heating the cell. Observations were based primarily on temperature measurements.

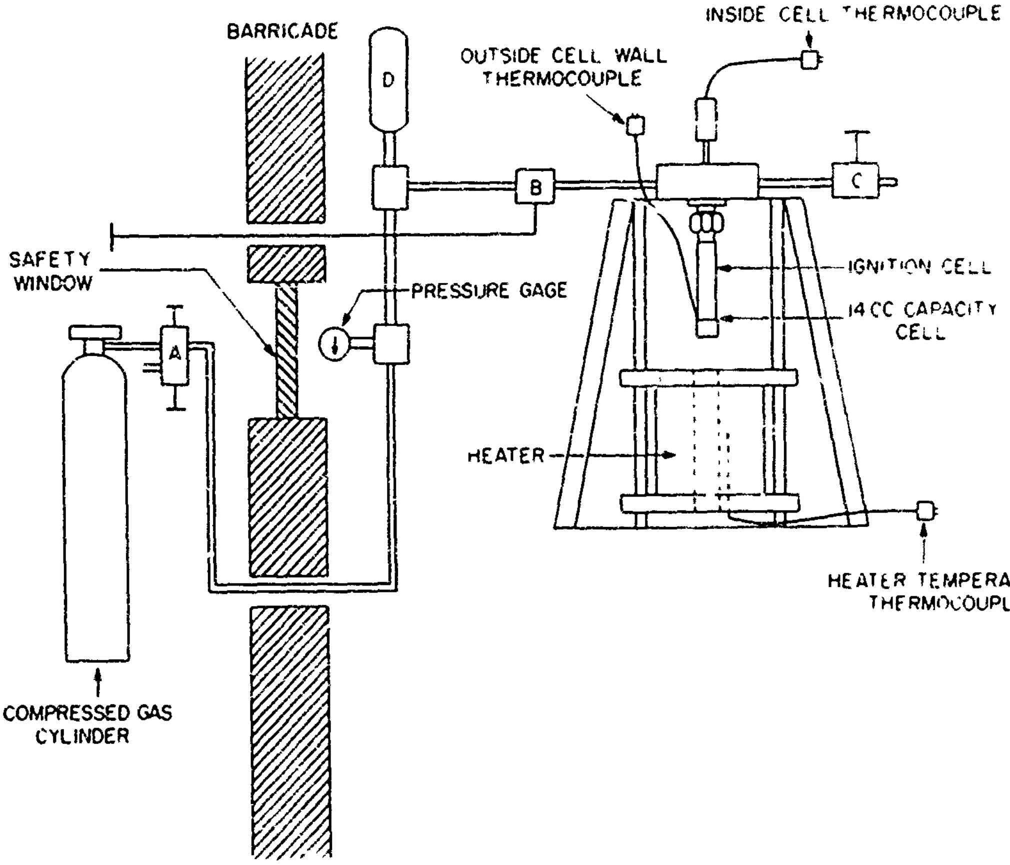

Fig. B1 - Apparatus for determining SIT at elevated pressure

The copper ignition cells were constructed from 6-inch lengths of hard-drawn copper tubing of 1/2-inch diameter and 1/16-inch wall. A copper cap was silver soldered to the tubing to close off one end, and the other end was connected to the manifold with a flare tubing nut. This provided an ignition cell of 14-cc capacity. Stainless steel ignition cells were prepared by welding a stainless steel disk to one end of a 6-inch length of 1/2-inch diameter stainless steel tubing, and welding a stainless steel flare sleeve to the other end to provide for mounting the cell.

All connections to the cell manifold and gas-handling system were made with high pressure tubing and fittings. The gas temperature inside the cell was measured with a thermocouple mounted by means of a high pressure fitting. The outside temperature of the cell was measured with an iron-constantan thermocouple which was silver soldered to the cell wall 1/2 inch from the bottom.

The ignition cell was heated by means of a furnace which consisted of a jacketed cylindrical aluminum block, 3 inches in diameter, with a center hole to accommodate the ignition cell. Heating was provided by four cartridge heaters spaced around the center hole in the block and by a heating jacket around the aluminum block. All heaters were subject to control by auto-transformers.

To make a run, the sample is placed in the cell, which is then mounted in the gas manifold, and the outside temperature thermocouple connected. After it is certain that valves B and C are closed, the appropriate compressed gas is admitted to tank D at the desired pressure by opening valve A. Valve A is then closed and valve B is opened to pressurize the cell. Valve B is then closed and the heater, preset at the desired temperature, is drawn up around the cell by remote control from behind the barricade. Temperature-time curves such as illustrated in Fig. 2 are obtained, and time and extent of ignition determined from these curves.

This system was planned and designed so that if an explosion occurred in the cell, the force would blow out the bottom, the weakest part of the cell. This occurred on several occasions with copper cells starting with air at 1500 psi. There was no damage to the furnace or gas handling system.

UNCLASSIFIED

Naval Research Laboratory. Report 6090.
THE IGNITION AND COMBUSTION PROPERTIES OF ACTIVATED CARBON CONTAINING ADSORBED HYDROCARBONS, by F.J. Woods and J.E. Johnson.
29 pp. and figs., July 28, 1964.

Activated carbon contained in large filter beds is used in nuclear submarines for removal of odors and trace contaminants. Because organic vapors are concentrated in this way in the carbon, a study was made to get information for assessing the fire hazards involved in maintaining the carbon filter in the ventilation system of the submarines. In this study, spontaneous ignition of submarine-exposed carbons occurred at temperatures as low as 490°F (255°C) in air at one

UNCLASSIFIED (over)

1. Activated carbon - Combustion
2. Activated carbon - Ignition
3. Nuclear submarines - Habitability
I. Woods, F.J.
II. Johnson, J.E.

UNCLASSIFIED

Naval Research Laboratory. Report 6090.
THE IGNITION AND COMBUSTION PROPERTIES OF ACTIVATED CARBON CONTAINING ADSORBED HYDROCARBONS, by F.J. Woods and J.E. Johnson.
29 pp. and figs., July 28, 1964.

Activated carbon contained in large filter beds is used in nuclear submarines for removal of odors and trace contaminants. Because organic vapors are concentrated in this way in the carbon, a study was made to get information for assessing the fire hazards involved in maintaining the carbon filter in the ventilation system of the submarines. In this study, spontaneous ignition of submarine-exposed carbons occurred at temperatures as low as 490°F (255°C) in air at one

UNCLASSIFIED (over)

1. Activated carbon - Combustion
2. Activated carbon - Ignition
3. Nuclear submarines - Habitability
I. Woods, F.J.
II. Johnson, J.E.

UNCLASSIFIED

Naval Research Laboratory. Report 6090.
THE IGNITION AND COMBUSTION PROPERTIES OF ACTIVATED CARBON CONTAINING ADSORBED HYDROCARBONS, by F.J. Woods and J.E. Johnson.
29 pp. and figs., July 28, 1964.

Activated carbon contained in large filter beds is used in nuclear submarines for removal of odors and trace contaminants. Because organic vapors are concentrated in this way in the carbon, a study was made to get information for assessing the fire hazards involved in maintaining the carbon filter in the ventilation system of the submarines. In this study, spontaneous ignition of submarine-exposed carbons occurred at temperatures as low as 490°F (255°C) in air at one

UNCLASSIFIED (over)

1. Activated carbon - Combustion
2. Activated carbon - Ignition
3. Nuclear submarines - Habitability
I. Woods, F.J.
II. Johnson, J.E.

UNCLASSIFIED

Naval Research Laboratory. Report 6090.
THE IGNITION AND COMBUSTION PROPERTIES OF ACTIVATED CARBON CONTAINING ADSORBED HYDROCARBONS, by F.J. Woods and J.E. Johnson.
29 pp. and figs., July 28, 1964.

Activated carbon contained in large filter beds is used in nuclear submarines for removal of odors and trace contaminants. Because organic vapors are concentrated in this way in the carbon, a study was made to get information for assessing the fire hazards involved in maintaining the carbon filter in the ventilation system of the submarines. In this study, spontaneous ignition of submarine-exposed carbons occurred at temperatures as low as 490°F (255°C) in air at one

UNCLASSIFIED (over)

1. Activated carbon - Combustion
2. Activated carbon - Ignition
3. Nuclear submarines - Habitability
I. Woods, F.J.
II. Johnson, J.E.

UNCLASSIFIED

atmosphere. Exothermic oxidative reactions occurred in a flow system at temperatures as low as 390°F (200°C). Combustion of carbons containing hydrocarbon produced considerable quantities of carbon dioxide and carbon monoxide, and significant amounts of these gases were evolved even under precombustion conditions. Of a number of carbons which had been used in submarines, the lowest flash point found was 160°F.

UNCLASSIFIED

UNCLASSIFIED

atmosphere. Exothermic oxidative reactions occurred in a flow system at temperatures as low as 390°F (200°C). Combustion of carbons containing hydrocarbon produced considerable quantities of carbon dioxide and carbon monoxide, and significant amounts of these gases were evolved even under precombustion conditions. Of a number of carbons which had been used in submarines, the lowest flash point found was 160°F.

UNCLASSIFIED

UNCLASSIFIED

atmosphere. Exothermic oxidative reactions occurred in a flow system at temperatures as low as 390°F (200°C). Combustion of carbons containing hydrocarbon produced considerable quantities of carbon dioxide and carbon monoxide, and significant amounts of these gases were evolved even under precombustion conditions. Of a number of carbons which had been used in submarines, the lowest flash point found was 160°F.

UNCLASSIFIED

UNCLASSIFIED

atmosphere. Exothermic oxidative reactions occurred in a flow system at temperatures as low as 390°F (200°C). Combustion of carbons containing hydrocarbon produced considerable quantities of carbon dioxide and carbon monoxide, and significant amounts of these gases were evolved even under precombustion conditions. Of a number of carbons which had been used in submarines, the lowest flash point found was 160°F.

UNCLASSIFIED