ORNL-3726
UC-80 – Reactor Technology
TID-4500 (37th ed.)

IODINE VAPOR ADSORPTION STUDIES FOR THE NS "SAVANNAH" PROJECT

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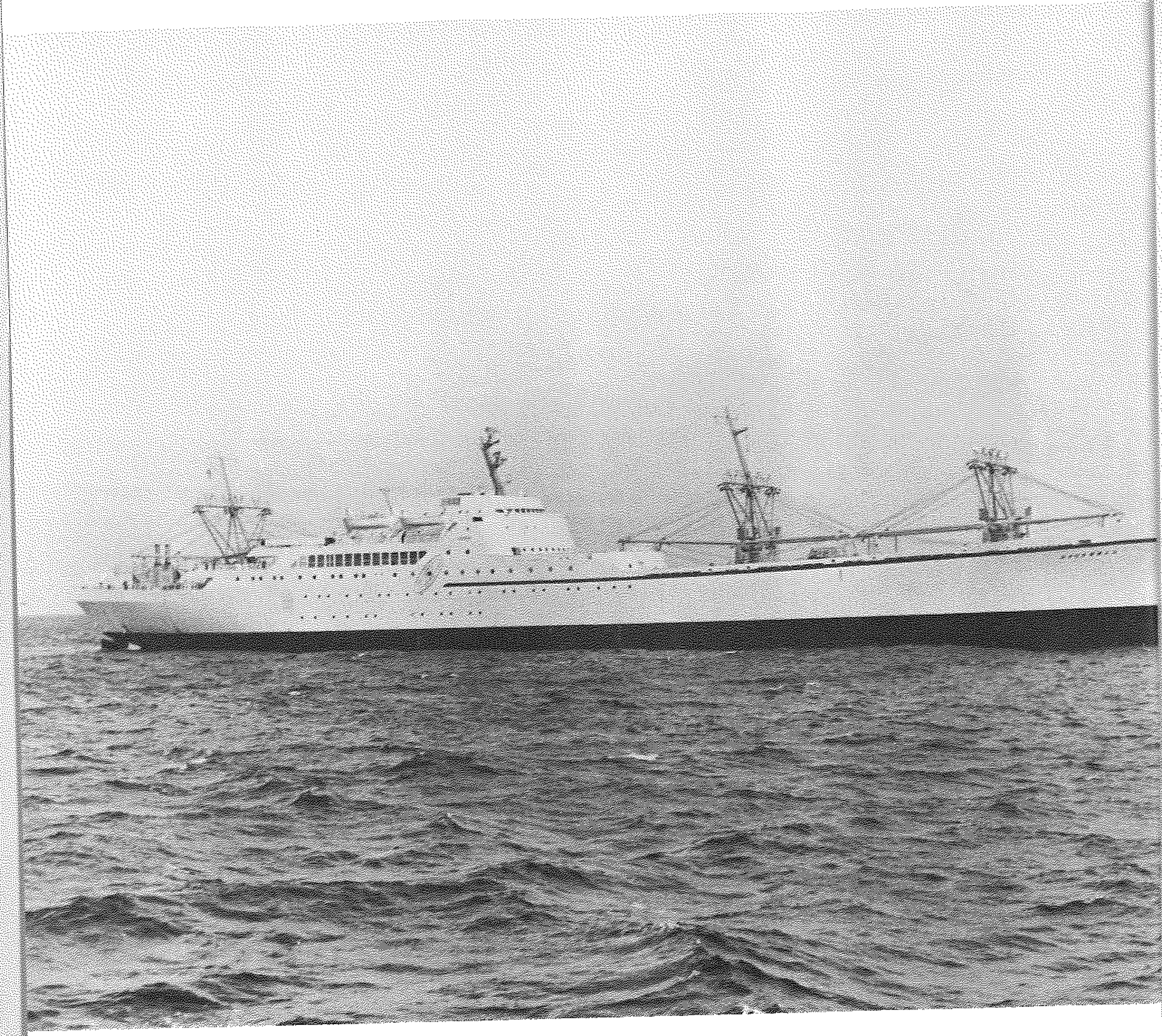

Nuclear Ship "Savannah."

ORNL-3726
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REACTOR CHEMISTRY DIVISION

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ABSTRACT

An experimental program was undertaken at Oak Ridge National Laboratory between September 1961 and January 1964 to determine the efficiencies of the iodine sorption systems installed aboard the NS "Savannah." The function of these systems is to prevent escape of radioiodine from the reactor compartment in the event of a reactor accident. The results of this program are reported herein.

Laboratory tests conducted under accident conditions on charcoal-adsorber units, reduced in scale but otherwise identical with those in the ship, showed their efficiency (for removal of iodine vapor from air-steam mixtures) to be 99.86 ± 0.07% at the 95% confidence level.

Two methods were developed for in-place testing of adsorber systems, one using ^{131}I with radioassay and the other using ^{127}I with activation analysis. Both methods were applied on board the NS "Savannah"; the ^{131}I method was rapid and more sensitive but required precautions to avoid accidental release of ^{131}I; the ^{127}I method was slow and less sensitive but required fewer safety precautions. Fifty-six shipboard in-place tests, conducted under various circumstances, demonstrated the system efficiency to be greater than 99.9% and showed that the adsorbers had been installed so that their full efficiency could be realized.

Laboratory studies of iodine adsorption indicated that certain iodine compounds, which are more volatile than iodine and which can occur in very small amounts, are adsorbed by charcoal less efficiently than is elemental iodine, especially under moist room-temperature conditions. The probable presence of these compounds did not appear to limit seriously the efficiency of the systems in the large-scale tests under steam conditions.

A method using Freon for testing adsorbers showed little promise for shipboard application because of interference by humidity at levels expected in the ship. An adsorber was developed for environmental iodine monitoring.

It was concluded, based on results from both laboratory study and shipboard testing, that the adsorber system as installed on the ship could be expected to retain iodine with an efficiency of 99.86% in the event of an accident.

INTRODUCTION

One of the primary measures for minimizing the public hazard associated with the operation of nuclear reactors has conventionally been the concept of separation and exclusion. A substitute for this safety concept had to be provided in the case of the NS "Savannah" since, to accomplish its mission, the ship (and its reactor) must be brought into close contact with the public associated with the marine industry (see Frontispiece). Accordingly, rather elaborate precautions have been taken to ensure the containment of the reactor and its associated radioactive material, especially radioiodine, in the unlikely event of a reactor accident. The reactor is totally enclosed in a containment vessel which is in turn enclosed within a reactor compartment that is maintained at subatmospheric pressure by ventilation. The ventilation gases from the reactor compartment are processed through particulate filters and iodine sorption units to provide for control of any radioactive iodine vapor and particulate material which might be present in the compartment atmosphere through accident. The efficacy of this gas-cleaning system is proved by laboratory tests which show that the basic components are capable of functioning adequately under accident conditions, and by periodic in-place tests which show that the units are installed so that their full capabilities are indeed realized.

Initially, two systems for ventilating the reactor compartment of the NS ''Savannah'' were provided having separate filters and iodine sorbers (Fig. 1). The normal ventilation system was of 4000 cfm capacity with provisions for processing the gases through three cleaning stages: (1) a prefilter, (2) a high-efficiency filter for particulate control, and (3) a silvered-copper mesh unit for iodine control. An emergency system of 200 cfm capacity was provided with five stages of gas cleaning: (1) a prefilter, (2) a high-efficiency filter, (3) a silvered-copper mesh unit, (4) an activated-charcoal unit, and (5) a second silvered-copper mesh unit. The emergency system served as a backup to the normal system and was to have operated only in the event of a release

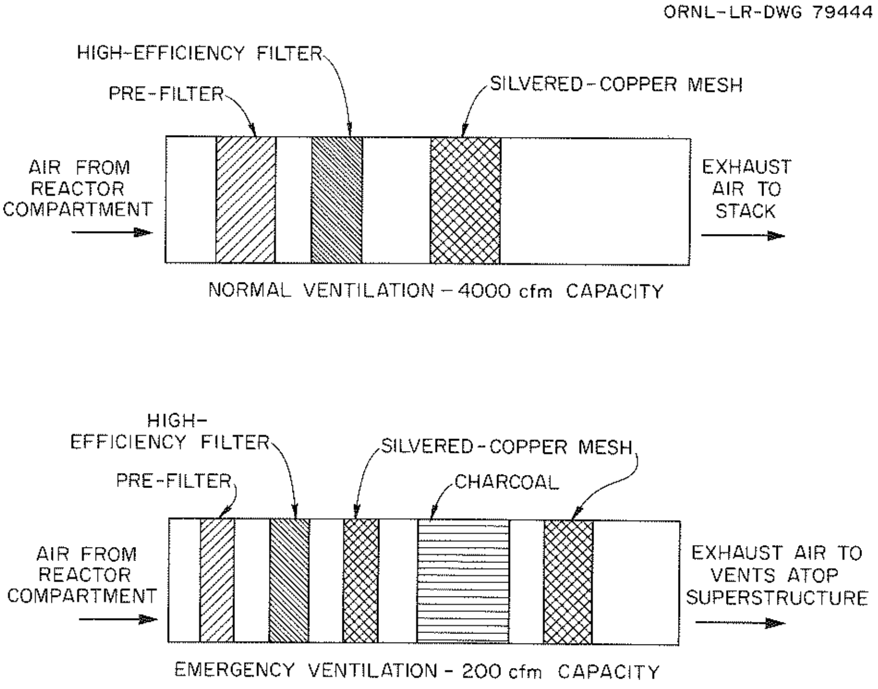

Fig. 1. Reactor Compartment Ventilation Filter Units Initially Used in the NS "Savannah."

of radioactivity. The normal system discharged to the atmosphere through an off-gas stack on the radio mast, while the emergency system discharged through two vents atop the superstructure of the ship.

Having two different ventilation systems presented some operational problems. In the event of fission product release, the main system flow would be stopped and the emergency system would be started manually. The necessity for this, and the superior iodine-removal efficiency observed in the emergency system, prompted the design and installation of an improved ventilation system for the reactor compartment. The main and emergency systems were removed, and an enlarged version of the original emergency ventilation system was installed during March and April 1963. Two parallel air-cleaning units (F-1 and F-2) were provided, with one unit to be operated continuously at a system flow rate of 1000 to 1100 cfm and the other to be held in standby.

Although the information available at the time of design of the original system was sufficient to demonstrate the feasibility of using activated charcoal for the removal of iodine vapor from moist air at elevated temperatures,[1] it was not sufficient to ensure that the full-scale activated-charcoal units designed for the NS "Savannah" would indeed provide the required iodine decontamination efficiency under conditions postulated to occur following a reactor emergency involving fission product release. For this reason, an experimental program was undertaken at ORNL to determine the iodine-removal efficiency that could be expected of the activated-charcoal units installed aboard the NS "Savannah." The program involved the development of methods for testing iodine adsorbers, the testing of adsorber units both in the laboratory and in place, and the training of ship's personnel for continued in-place testing. It also involved a laboratory investigation of iodine adsorption.

The present report describes the work on this program between September 1961 and January 1964, when active sponsorship by the NS "Savannah" project ended. After that date, in-place testing was performed by ship's personnel and shore staff, with only minor consultation from ORNL. The laboratory investigation of iodine adsorption was continued under other sponsorship and is reported in the ORNL Nuclear Safety Program Semiannual Progress Report series.[2]

LABORATORY TESTS ON REDUCED-SCALE CHARCOAL UNITS

The capability of the NS "Savannah" iodine adsorption units under accident conditions was determined in laboratory tests which were made as realistic as possible, with the intention that even unanticipated factors would be made to conform to accident conditions. In these tests steam-air mixtures carrying radioiodine were passed through reduced-scale iodine adsorption units, 11 in. square and 1.125 in. thick, produced by Flanders, Inc. These units, containing Pittsburgh BPL

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[1] R. E. Adams and W. E. Browning, Jr., *Removal of Radioiodine from Air-Steam Mixtures*, ORNL-CF-60-11-39 (Nov. 14, 1960).

[2] "Characterization and Control of Accident-Released Fission Products," *Nucl. Safety Program Semiann. Progr. Rept. June 30, 1964*, ORNL-3691, pp. 46–74.

charcoal, were manufactured using the same materials and techniques as those used in producing the full-scale units installed aboard the ship. The reduction in scale involved the face area only and not the depth of charcoal in the unit.

The test system developed for this study is diagrammed in Fig. 2. The system was constructed from 4-in.-diam Pyrex glass pipe, with stainless steel metal pieces making the transition from the round glass pipe to the 11- by 11-in. square charcoal unit. The test system was heated externally by flexible heating tapes to prevent premature condensation of the steam on the walls of the system. This heated zone terminated at point *B*, as illustrated in Fig. 2. Downstream from this point, efforts were made to condense the steam for recovery.

Construction details may be noted by referring to Figs. 3 to 10. The charcoal unit and the metal transition pieces are shown in Fig. 3. The charcoal unit is composed of a perforated aluminum box filled with granular charcoal (Pittsburgh BPL). The two halves of the aluminum box are compressed against the charcoal by the drawbolt which can be seen in the middle of the

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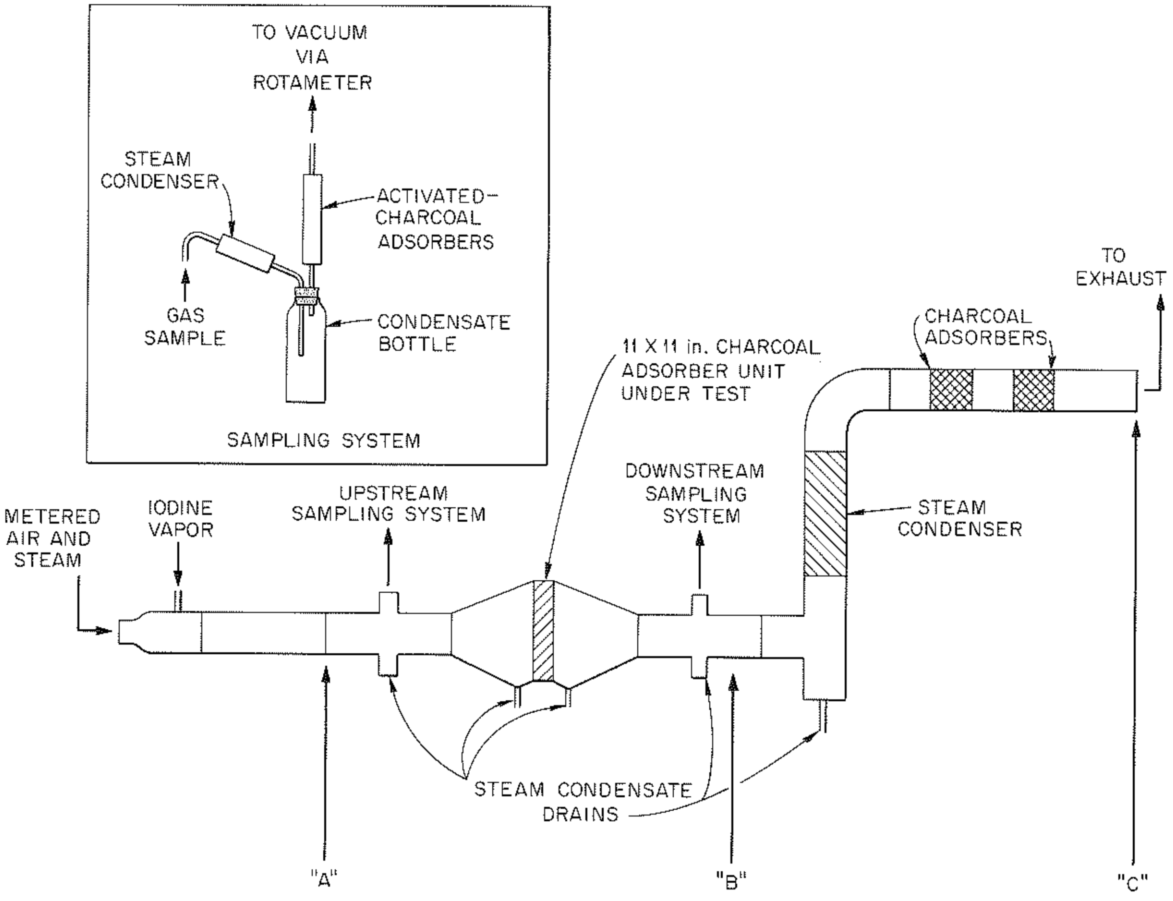

Fig. 2. Schematic of Iodine Adsorption System for Testing of 11- by 11-in. Reduced-Scale Adsorbers.

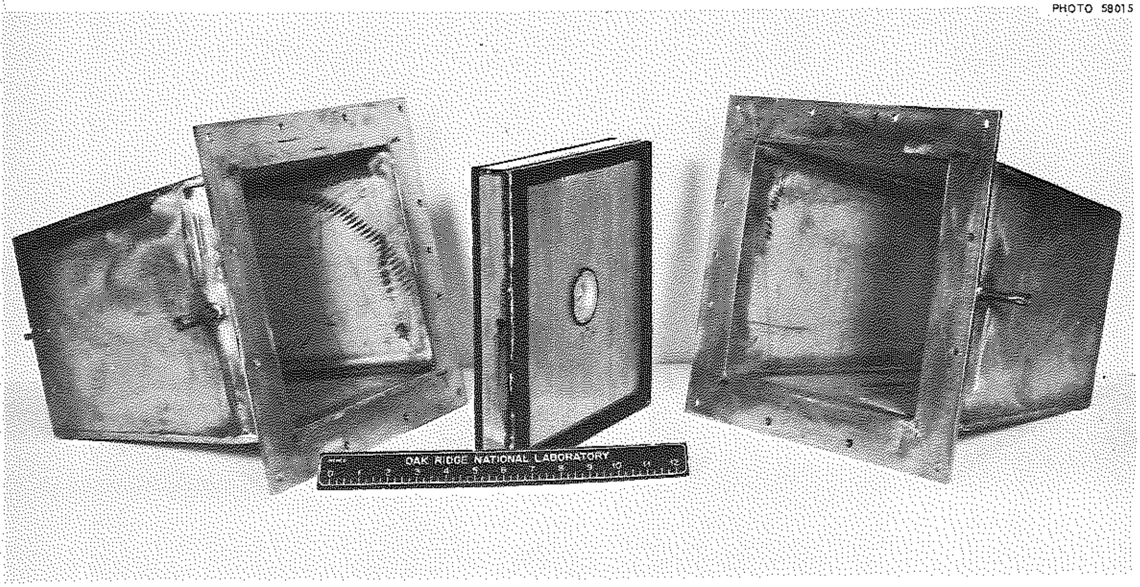

Fig. 3. Reduced-Scale Adsorber (11 by 11 in.).

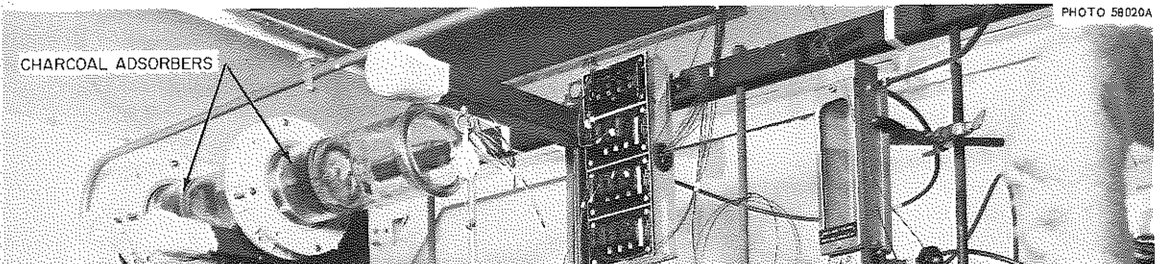

Fig. 4. Laboratory Apparatus for Iodine Adsorption Study of Reduced-Scale Adsorbers. Upstream side.

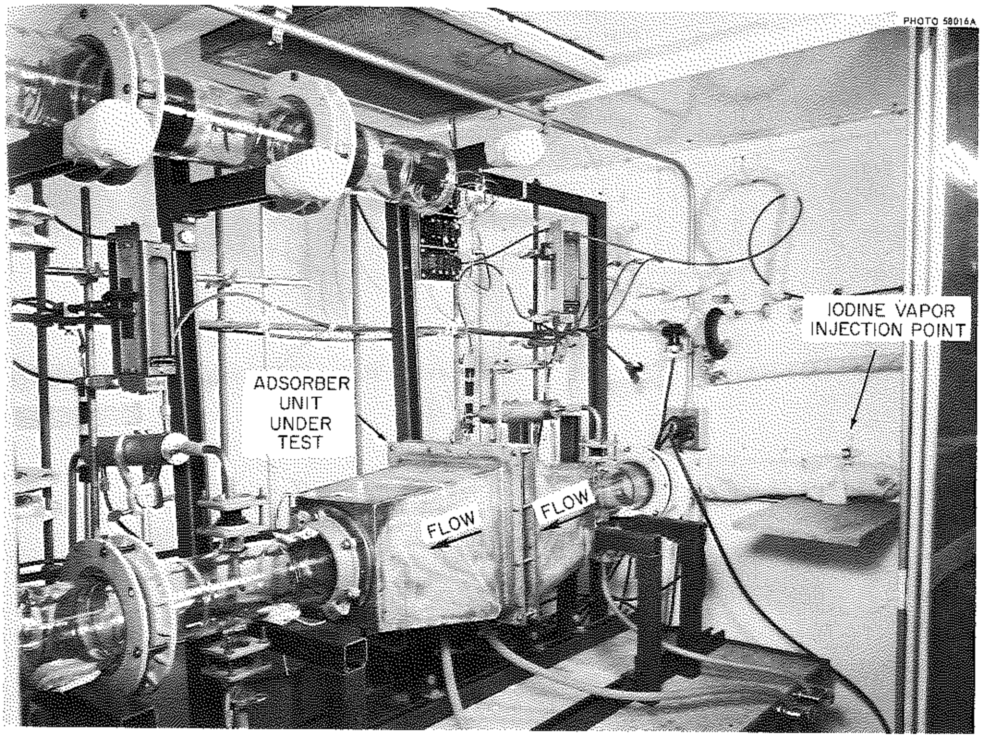

Fig. 5. Laboratory Apparatus for Iodine Adsorption Study of Reduced-Scale Adsorbers. Downstream side.

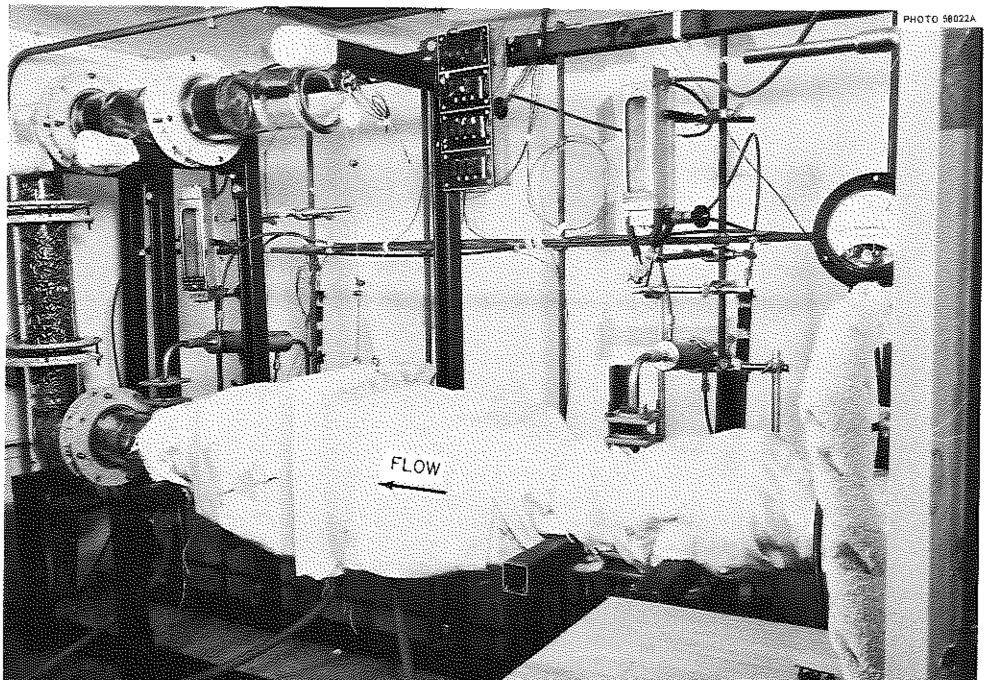

Fig. 6. Laboratory Apparatus After Heating Tapes and Insulation Were Installed.

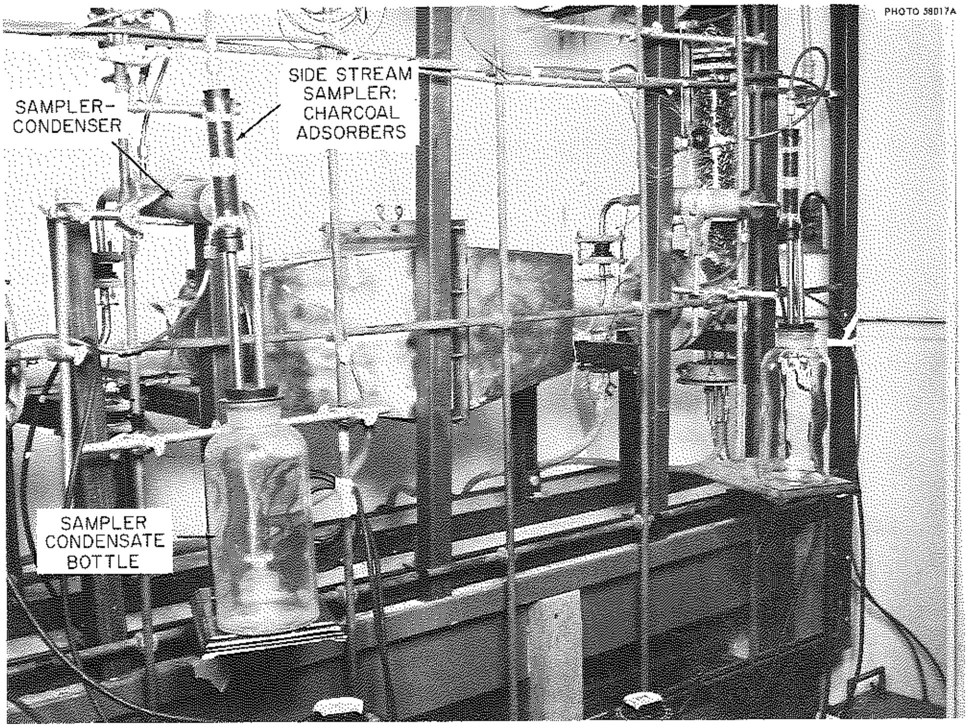

Fig. 7. Laboratory Apparatus for Iodine Adsorption Study of Reduced-Scale Adsorbers, as Installed.

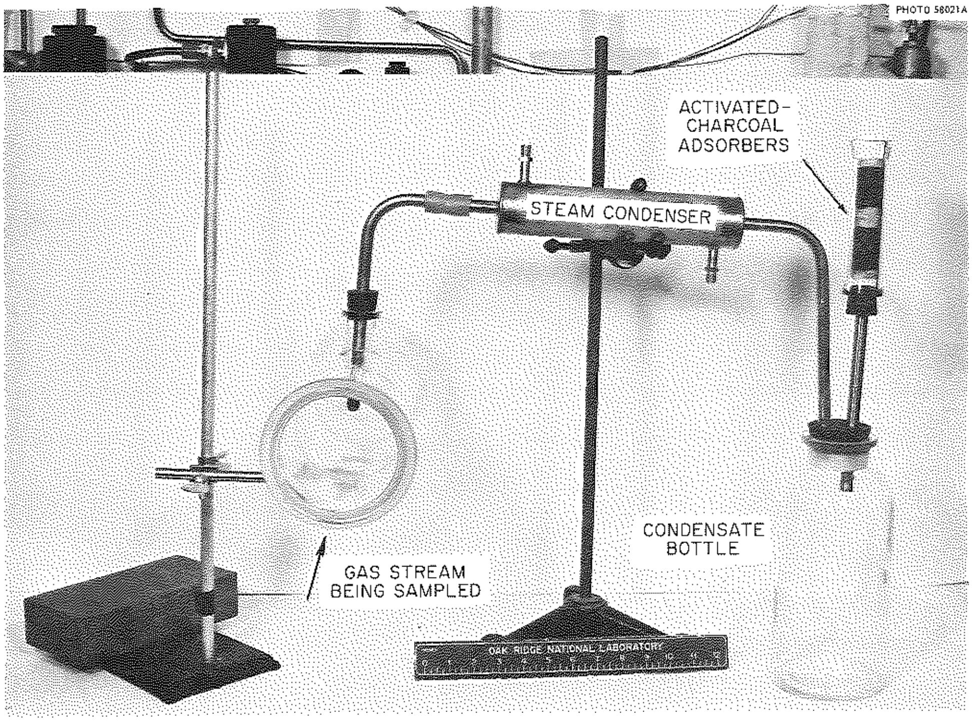

Fig. 8. Detail of Side Stream Sampler Arrangement for Reduced-Scale Adsorber Study.

unit. A woven fabric material is located at the inner face between the aluminum and the charcoal mass to prevent small particles of charcoal from being blown out by air flow. A slightly flexible cement material is used to seal the space between the two halves of the perforated aluminum box. Figure 4 shows the test system from the upstream side before the flexible heating tapes and asbestos insulation have been added. It should be noted that gas flow in the diagram in Fig. 2 is from left to right, while in Fig. 4 it is from right to left. Figure 5 is a view from the downstream side. Iodine vapor is injected into the system at the glass ball joint located above the wooden shelf in the right of the photo. The test system after installation of the flexible heating tapes and insulation is shown in Fig. 6. The small sampling systems, as installed, can be seen in Fig. 7, while a mockup shows details in Fig. 8. The steam condenser in the main stream was composed of a water-cooled pipe surrounded by a column packing of small bits of metal tubing. In Fig. 9 the condenser shell and water-cooled pipe are shown before the bits of metal tubing were added (see Fig. 4). The steam-air mixture enters from the bottom through the wire-screen basket. Cooled and dried air passes from this zone through the right-angle pipe at the top and enters a series of two room-temperature charcoal adsorbers, a closeup of which is shown in Fig. 10.

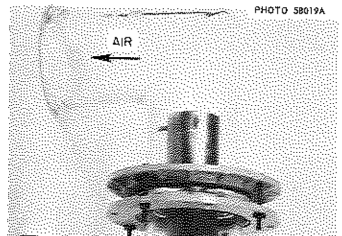

Fig. 9. Detail of Steam Condenser Before Addition of Metallic Column Packing.

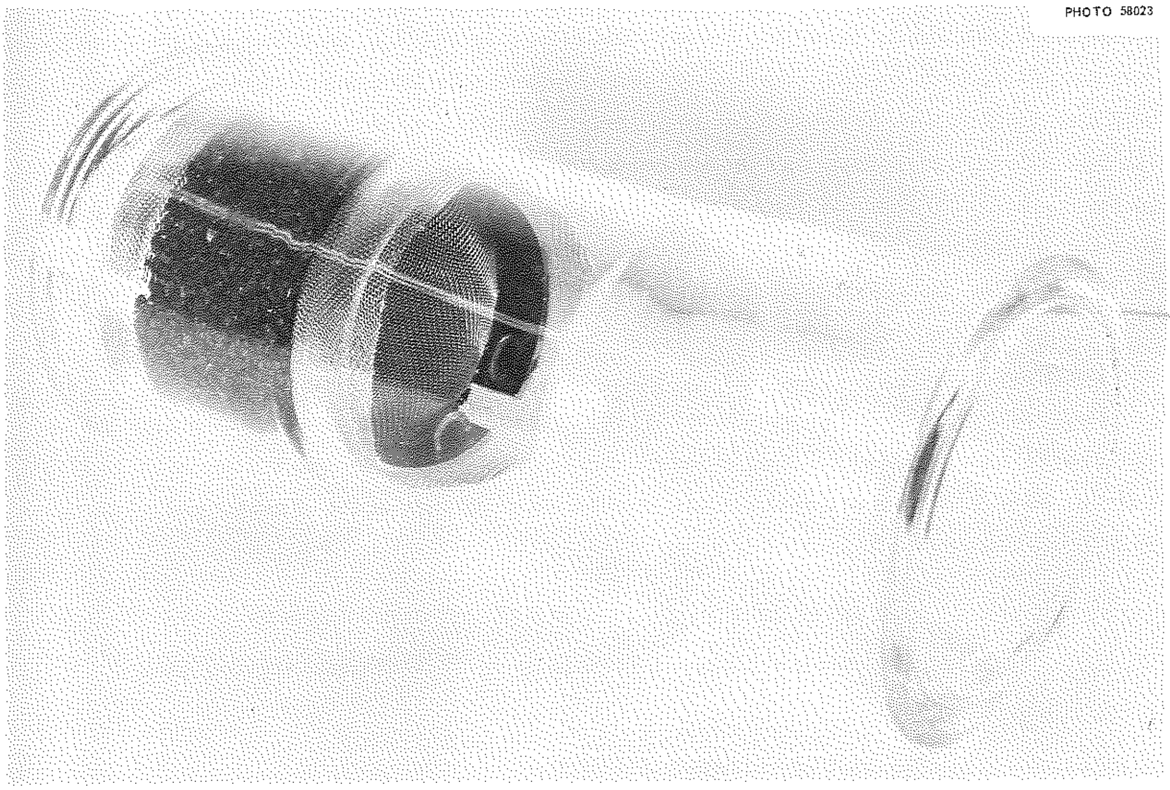

Fig. 10. Large Charcoal Adsorber, Assembled.

A typical test involved the following operations. Iodine vapor, labeled with sufficient ^{131}I for radioassay and consisting of enough ^{127}I carrier to yield a concentration corresponding to that expected in a reactor accident, was introduced continuously into the air-steam mixture being metered into the system. Any iodine vapor escaping from the charcoal adsorption unit under test was collected (1) in the condensate which was drained periodically from the system, (2) on the small pieces of metal tubing comprising the steam condenser column packing, or (3) in the two room-temperature charcoal adsorbers. During the test, a small portion of the steam–air–iodine vapor mixture was passed through sampling systems located upstream and downstream of the charcoal unit under test. After completion of the test, the system was cooled, completely drained, and disassembled.

The iodine adsorption efficiency of the charcoal unit under test was determined by radiochemical assay. The parts of the test system between points *A* and *C*, as illustrated in Fig. 2, were completely disassembled whenever possible and totally converted into samples suitable for radiocounting. Larger pieces were decontaminated chemically, and the resultant liquor was counted. The charcoal unit was completely destroyed for assay. When the ^{131}I content of all the pieces had been determined, an iodine adsorption efficiency was computed by comparing the amount of ^{131}I contained in the charcoal unit with the total inventory found in the charcoal unit plus all downstream components, including steam condensates. In addition, an iodine adsorption efficiency was calculated from the data obtained by the two side stream sampling systems. In calculating this efficiency, allowances were made for the observed deposition of iodine on the walls upstream from the test unit but downstream from the first sampler. Detailed conditions and results of the 14 tests on the 11- by 11-in. charcoal units are listed in Table 1. The inventory method (which measures adsorber performance most directly) gave the most reproducible results, indicating an efficiency of 99.86 ± 0.07% at the 95% confidence level. Efficiencies determined by the small samplers gave less reproducible results and indicated slightly lower values, with an average of 98.66 ± 0.82% at the 95% confidence level.

These tests, conducted under simulated reactor accident conditions with continuous iodine injection of approximately 10^{-2} mg of iodine vapor per cubic meter of gas mixture at 96 to 100°C and with 80 to 90% saturated steam in air, showed the efficiency of the charcoal unit alone to be 99.86 ± 0.07% at the 95% confidence level.

Table 1. Iodine-Removal Efficiency Tests on 11- by 11-in. Charcoal Adsorber Units

Charcoal depth: 1.125 in.
Iodine vapor concentration: 10^{-2} mg/m^3
Charcoal type: Pittsburgh BPL, −12 +30 mesh

Run No.	Temperature (°C)	Face Velocity (fpm)	Steam Saturation (%)	Iodine-Removal Efficiency (%)	
				By Inventory	By Sampling
1	96	4.6	91.3	99.84	99.93
2	100	4.3	80.7	99.63	98.95
3	26	4.8		99.98	99.86
4	99	4.5	83.8	99.95	98.57
5	100	4.4	82.1	99.73	
6	98	4.8	88.9	99.67	99.50
7	99	4.5	85.2	99.95	99.12
8	99	4.8	86.3	99.95	97.38
9	98	4.8	89.0	99.86	98.04
10	99	4.7	86.1	99.90	97.84
11[a]	100	4.7	83.4	99.90	99.43
12	98	5.0	89.6	99.83	99.38
13[a]	98	4.9	88.6	99.95	99.56
14[a]	98	4.9	89.3	99.72	95.00
			Mean	99.86 ± 0.07%	98.66 ± 0.82%

[a] 24-hr test; all other tests 12 hr.

SHIPBOARD IN-PLACE TESTING PROGRAM

Concurrently with the laboratory work, a program was conducted to develop and apply test procedures and equipment for in-place tests on the shipboard reactor compartment ventilation systems. The purpose of these tests was to determine whether the installation of the iodine sorption units is such that the full iodine-removal efficiency of the charcoal can be realized. Since it would be highly impractical to conduct in-place iodine tests under accident conditions of high humidity and elevated temperature, the adequacy of installation was determined under the ambient conditions in the reactor compartment. If such tests show that the iodine sorption units are installed and functioning properly under these conditions, then one may assume that the system will produce at least the iodine sorption efficiency observed in the laboratory studies of the 11- by 11-in. units under the temperature and humidity conditions expected in a reactor accident.

Nine series of tests were conducted during the period from May 1962 to January 1964, and two methods of testing were involved. The two methods will be described briefly, and then the nine series of iodine tests will be discussed in chronological order. A log of these tests appears as Table 2. Personnel associated with the NS "Savannah" participated in these tests for the purpose of securing experience in test operations.

Table 2. Log of NS "Savannah" Iodine Tests

Series	Date	Location of Ship	Type of Test	Systems Tested
First	May 27–29, 1962	Yorktown, Va.	^{131}I	Emergency and main
Second	Aug. 15–16, 1962	Yorktown, Va.	^{131}I	Emergency and main
Third	Sept. 11–13, 1962	Norfolk, Va.	^{131}I, ^{127}I	Emergency only
Fourth	Nov. 8–11, 1962	Seattle, Wash.	^{131}I, ^{127}I	Emergency and main
Fifth	Apr. 15–18, 1963	Galveston, Tex.	^{131}I, ^{127}I	New system installed (F-1 and F-2)
Sixth	Aug. 12–13, 1963	Galveston, Tex.	^{127}I	F-1 and F-2
Seventh	Sept. 4–5, 1963	Galveston, Tex.	^{127}I	F-1 and F-2
Eighth	Oct. 7–10, 1963	Galveston, Tex.	^{131}I, ^{127}I	F-1 and F-2
Ninth	Jan. 18–19, 1964	Galveston, Tex.	^{131}I	F-1 and F-2

Test Method Using Radioactive ^{131}I

Basically, this test was conducted by injecting iodine vapor (^{127}I tagged with ^{131}I) into the intake of the reactor compartment ventilation system while withdrawing air samples both upstream and downstream of the iodine sorption unit as shown in Fig. 11. These air samples were processed through small sampling units containing activated charcoal to collect any iodine vapor present.

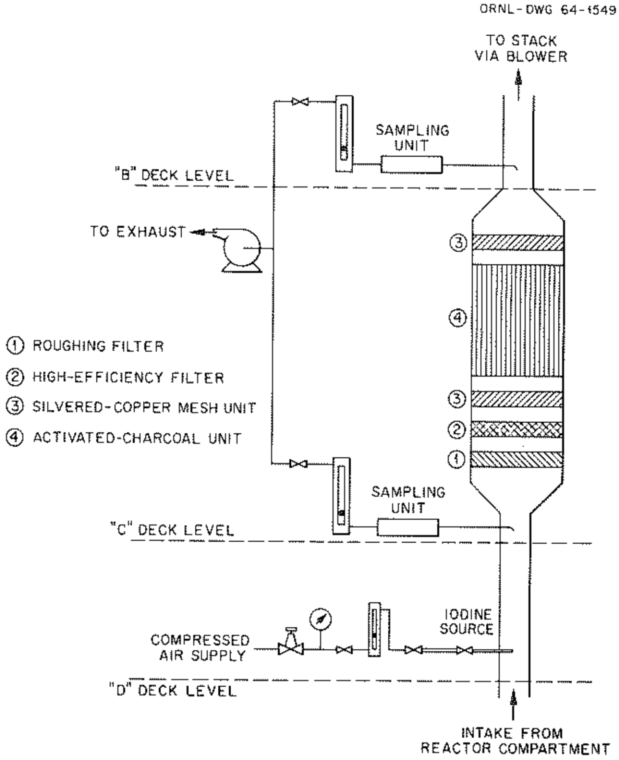

Fig. 11. Schematic of System for Shipboard Testing of Full-Scale Iodine Adsorbers.

The necessary information for computing the efficiency of the iodine sorption unit was obtained by radioassay of the sampling units for ^{131}I.

The success of testing in this manner depends upon a sufficient quantity of iodine penetrating the iodine sorption unit so that downstream sampling is feasible. To test a highly efficient iodine sorption unit, it is therefore necessary to inject a relatively large quantity of radioactive iodine vapor so that there is an amount of radioactivity downstream sufficient to measure. On the other hand, a supposedly highly efficient iodine sorption unit could be very inefficient due to internal damage or faulty installation and thus allow a major portion of the injected iodine vapor to pass through and be released into the atmosphere. To guard against this possibility while using biologically hazardous ^{131}I, the test procedure was conducted in two steps. The initial test was conducted using a small quantity of radioiodine which could not produce the desired test sensitivity but which would demonstrate that a larger quantity of radioiodine could be used safely in the succeeding test. In all cases steps were taken to ensure that the amount of ^{131}I issuing from the ventilation system would not exceed the established limits for the NS "Savannah" operation.

Test Method Using Nonradioactive ^{127}I

A method for determining the efficiency of an iodine-removal system, in place and by use of normal iodine, is of considerable interest for application to the ventilation systems on the NS

"Savannah." Normal iodine is preferred over ^{131}I tracer for in-place tests because of the elimination, by its use, of the need for precautions to prevent contamination of the ship. Tests using ^{131}I had to be performed while the ship was in a relatively unpopulated harbor or at sea, and these tests have sometimes interfered with the operating schedule of the ship. Tests using normal iodine can be performed while the ship is in any harbor or while carrying passengers.

Nonradioactive iodine tests were conducted in much the same manner as the radioactive tests, the main differences being that from 1 to 3 g of elemental iodine vapor (^{127}I) was injected into the duct and that the iodine content of the sampler was determined by neutron activation techniques. Whereas the concentration of iodine used in the test was greater than would occur in a reactor accident, the adsorbers were loaded so far below their capacity that the test was still realistic. The nonradioactive iodine tests were slightly less sensitive than the radioactive tests. Activated charcoal normally contains a small amount of iodine as an impurity, and in most cases the downstream samplers from iodine tests were found to contain iodine in amounts comparable to the impurity level. To ensure that the charcoal units were not overrated in their iodine-removal efficiency, it was assumed that all the iodine found in the downstream samplers actually came from iodine penetration of the unit under test and was not originally present in the sampler as an impurity. Thus, the efficiencies quoted from the nonradioactive tests represent lower limits, and the real efficiencies are in excess of the figures listed.

Results of In-place Tests

The tests are described in some detail in the following subsections. Test results from all the in-place tests appear in Tables 3 to 6.

First Series – Yorktown, Virginia

Equipment and ^{131}I sources were shipped from ORNL to the NS "Savannah" for the initial series of tests at an isolated U.S. Coast Guard dock, Yorktown, Virginia.

The charcoal unit had been installed in the emergency ventilation system on December 14, 1961, and had been operated only intermittently for preliminary testing and system adjustment up to May 27, 1962, the date of this first test series. Prior to installation the units had been subjected to two Freon penetration tests at the Edgewood Army Chemical Center, Maryland, and to a stable-iodine (^{127}I) penetration test at the Armour Research Foundation, Chicago, Illinois. In the latter test, 200 g of iodine was adsorbed by the unit.

The iodine vapor was injected at a concentration corresponding to a reactor accident over a 15- to 30-min period, and the test was continued for 2 hr at a constant air flow rate. The temperature of the reactor compartment was between 95 and 100°F, and the relative humidity was very high. Details on iodine source preparation and iodine injection and sampling hardware are given in the Appendix. At the end of the test period, the small charcoal sampling units were recovered and disassembled. Each of the component parts was analyzed for its ^{131}I content

Table 3. In-Place ^{131}I Test – Original Ventilation Systems[a]

Test	Ventilation System	Flow Rate (cfm)	Iodine Source		Iodine Efficiency (%)
			^{131}I (mc)	^{127}I (mg)	
		First Series – May 1962			
1	Emergency	200[b]	5	1–2	99.93+
2	Emergency	200[b]	15	1–2	99.99+
3	Main	4000	5	10	95.0
		Second Series – August 1962			
4	Emergency	200[b]	15	2	99.7
5	Main	4000	5	10	90–95
		Third Series – September 1962			
6	Emergency	200[b]	15	2	99.99+
7	Emergency	200[b]	15	2	99.99+
		Fourth Series – November 1962			
8	Emergency	200[b]	7	3	99.97+
9	Emergency	200[b]	7	3	99.97+
10	Main	4000	2.5	10	98.6

[a] Test duration, 2 hr.

[b] System operated under emergency mode with maximum off-gas dilution.

using gamma spectrometry. Two tests were conducted using 5 and 15 mc of ^{131}I, and iodine-removal efficiencies of 99.95+ and 99.99+%, respectively, were obtained (Table 3). These tests indicated that the charcoal-containing iodine sorption units, as installed in the emergency system, were capable of realizing the iodine-removal efficiency observed in the laboratory tests under accident conditions.

A short time prior to this first series, it was decided to extend the test program to include the main reactor compartment ventilation system. The gas-cleaning provisions in this system do not include activated charcoal. Silvered-copper mesh was intended to remove whatever iodine had entered the system before the exhaust gases were diverted to the emergency system. The silvered-copper mesh unit had also been installed on December 14, 1961, and had been operated almost continuously up to the time of these tests. In testing this system, two downstream sampling units were used, one immediately following the mesh unit and the other on the open deck of the ship at the base of the off-gas stack. One test was performed using 5 mc of ^{131}I. Approximately equal amounts of ^{131}I were found in the two downstream sampling units. Comparison of this amount with that found in the upstream sampling unit yielded an iodine efficiency of 95%.

This result is consistent with laboratory tests on silvered-copper mesh in air at room temperature.[3,4]

Second Series – Yorktown, Virginia

The second series of in-place tests was conducted at the U.S. Coast Guard dock, as before, on August 15–16, 1962. Test procedures and sampling arrangements were the same as those used in the first test series. The iodine-removal efficiency of the sorption unit of the emergency ventilation system was found to be 99.7% (Table 3). The distribution of ^{131}I radioactivity among the components of the downstream sampler suggested strongly that the ^{131}I which had penetrated was predominantly associated with particulate matter. The distribution of radioactivity in the upstream sampler was indicative of iodine in the vapor state. This behavior was contrary to that observed during the first series of in-place tests in May, when an efficiency in excess of 99.99% had been determined. Strict comparison of the efficiency values may be subject to question, since the emergency system had been modified during the interval between tests. Modification included the installation of a metal transition piece between the original system duct and the particulate filter housing upstream of the charcoal unit. The purpose of this modification was to provide multiple intakes for this ventilation system. The opening for this transition piece was cut into the top of the metal filter housing while the filter was in place, and the filter sealing flange and filter frame were damaged. To reach the damaged area, it was necessary to remove the charcoal unit from the housing. Upon repair and replacement of the particulate filter, the charcoal unit was reinstalled. It was suspected that dust generated during these operations and deposited downstream from the filter but upstream from the charcoal had combined with 0.3% of the iodine and carried it through the adsorber.

In a manner similar to that used before, an efficiency test was conducted on the main reactor compartment ventilation system. Two downstream samplers were used, and the iodine efficiency was found to be between 90 and 95%, based on the amounts of ^{131}I radioactivity found in the two samplers. This was consistent with the previous test and also with laboratory results.

Third Series – Norfolk, Virginia

The third series of in-place tests was carried out on the emergency system during the night of September 12–13, 1962, while the ship was at sea off Norfolk, Virginia. No tests were conducted on the normal ventilation system at this time. The period of time between tests was shortened because of the apparently reduced efficiency noted in August during the second test

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[3] R. E. Adams and W. E. Browning, Jr., *Removal of Radioiodine from Air Streams by Activated Charcoal*, ORNL-2872 (Mar. 17, 1960).

[4] L. Silverman *et al.*, "Iodine Collection Studies," pp. 322–42 in *Sixth AEC Air Cleaning Conference, July 7–9, 1959*, TID-7593 (October 1960).

series. It was necessary to determine whether the iodine sorption unit was deteriorating before the ship's departure for Seattle, Washington.

Test procedures were identical with those of the previous tests. The iodine efficiency of the emergency ventilation system was determined to be at least 99.99% for the conditions prevailing at the time of the tests (Table 3). The distribution of radioactivity on the components of the upstream samplers was indicative of iodine in the vapor state. It was not possible to characterize the state of the iodine in the downstream samplers because the amount of radioactivity was not sufficient to produce a counting rate different from background. Overall behavior of these tests was identical to that of the first series of tests on May 27, 1962, and it was concluded that the apparent reduction of efficiency had been a temporary effect associated with the modifications and that the iodine sorption unit was not deteriorating.

Two in-place efficiency tests using normal iodine vapor and one "background" test in which no iodine was injected were conducted on September 11, 1962, to determine the utility of this method for measuring the iodine-removal efficiencies of the ventilation systems of the reactor compartment. Details of the iodine source preparation, construction of the small charcoal samplers, and the technique of activation analysis may be found in the Appendix. Nonradioactive elemental iodine vapor (approximately 1 g) was injected into the intake duct leading from the compartment into the iodine sorption unit of the emergency system. Samples of the air stream were taken before and after the iodine unit during the 2-hr test. Similar samples had been taken for a 2-hr period prior to injection of the iodine vapor. The samplers from these tests were returned to Oak Ridge and analyzed for iodine content by neutron activation techniques. Each of the samplers from the background test contained approximately 0.5 μg of iodine. This amount agrees with that found in samples of the charcoal which had not been exposed to the air in the ship. The iodine-removal efficiencies, in both tests, were determined to be greater than 99.9% (Table 4). These efficiencies compared favorably with the results obtained in the ^{131}I tests on the night of September 12–13, when efficiencies of 99.99 and 99.99+% were determined. These results indicated that a satisfactory testing procedure could be developed, making use of normal iodine and the analytical techniques of activation analysis.

Fourth Series – Seattle, Washington

This series of tests was conducted during the night of November 10–11, 1962, while the ship was under way in the Strait of Juan de Fuca, State of Washington.

The tests on the emergency ventilation system were conducted in the same manner as before. One portion of the activated charcoal from the downstream sampler from the first test was found to contain a very small quantity of ^{131}I, barely sufficient to produce a recognizable photopeak. The remaining pieces of the sample produced, in some cases, a count rate slightly above background, but no recognizable photopeak of ^{131}I was obtained. Assuming that this was due to ^{131}I, an iodine efficiency of 99.97+% was obtained (Table 3). The distribution of radioactivity on the components of the upstream sampler was indicative of iodine in the vapor state. On the whole,

Table 4. In-Place ^{127}I Tests – Original Ventilation Systems[a]

Test No.	System	Amount of I_2 Injected (g)	Amount of I_2 Collected (μg)		Iodine Efficiency (%)
			Upstream	Downstream	
		Third Series – September 1962			
1	Emergency		0.6	0.5	
2	Emergency	1.6	15,500	0.5	99.9+
3	Emergency	1.2	10,700	0.9	99.9+
		Fourth Series – November 1962			
4	Emergency		1.22	3.71	
5	Emergency	1.4	6,330	0.3	99.9+
6	Emergency	1.6	7,700	3.73	99.9+
7	Main		4.10	0.90	
8	Main	1.5	683	10.1	98.5
9	Main	1.3	636	18.3	97.1

[a] System flow: emergency – 200 cfm, main – 4000 cfm; test duration, 2 hr.

the behavior of this test was considered normal. The second, or duplicate, test was conducted in the same manner as the first. Again the components and pieces of the downstream sampler produced, in some cases, a count above background, but no photopeak representative of ^{131}I was observed. Assuming that this was due to ^{131}I, as before, an iodine efficiency of 99.97+% was obtained on this second test of the emergency ventilation system. These in-place tests indicated that the emergency-ventilation iodine-sorption unit, as installed, was capable of achieving the iodine-removal efficiency observed in the laboratory tests of 11- by 11-in. charcoal units.

Tests on the main ventilation system did not function as smoothly as those on the emergency system. In the previous series, a sampler was installed at the base of the off-gas stack; however, due to rain, wind, and heavy seas, the use of this sampling point was abandoned for this series. Only the single downstream sampler was installed, in the blower housing on the interior deck above the filters.

The first test was aborted when it was discovered that very little, if any, ^{131}I had been released from the glass ampul contained in the iodine injection hardware. A second attempt, using a new iodine source and new sampling units, was successful. Some ^{131}I was found in the downstream sampler, as expected, and the iodine efficiency was determined to be 98.6% (Table 3).

In-place tests using nonradioactive iodine (^{127}I) were conducted on both the emergency and the main ventilation systems of the NS "Savannah" at the time of the fourth series of ^{131}I tests. These tests were conducted at the Todd Shipyards, Seattle, Washington, on November 8–9, 1962, prior to the ^{131}I tests at sea.

Two in-place tests using normal iodine and one background test in which no iodine was injected were conducted on the emergency ventilation system. Approximately 1.5 g of elemental iodine, in the vapor state, was injected into the intake duct leading from the reactor compartment into the iodine sorption unit. Samples of the air stream were taken before and after the unit during the 2-hr test period. To establish the iodine background, similar samples were taken for a 2-hr period prior to the injection of iodine vapor into the system.

In a like manner, two normal iodine tests and a background test were conducted on the main reactor compartment ventilation system. An additional sampler, however, was installed at the base of the off-gas stack. The samplers from all these normal iodine tests were returned to Oak Ridge and analyzed for the ^{127}I content by neutron activation.

Samplers from the background tests were found to contain ^{127}I in amounts ranging from 0.9 to 4.79 μg. These amounts of iodine were larger than those determined in background samplers from previous in-place tests. At Norfolk, Virginia, the background samplers from the emergency system tests contained 0.5 and 0.6 μg of ^{127}I. Samplers exposed to the marine environment at Yorktown, Virginia, on August 15, 1962, contained 0.17 and 0.33 μg of ^{127}I. Comparable amounts of ^{127}I were found in unexposed samples of the activated charcoal. All the samplers used thus far in the program contained activated charcoal from a single batch. The amount of iodine present in the background samples, while larger than desired, has not interfered greatly with the sensitivity of the test procedure.

Assuming that the iodine found in the downstream samplers from the ^{127}I tests resulted from actual iodine penetration, then an iodine-removal efficiency of 99.9+% was measured for the emergency system (Table 4). This efficiency compared favorably with an efficiency of 99.97+% determined by the ^{131}I tests. By comparing the upstream and downstream samplers on the main ventilation system, efficiencies of 98.5 and 97.1% were obtained. Efficiencies of 99.1 and 99.1% were calculated by comparing the upstream sampler with the sampler at the base of the off-gas stack. These results compared favorably with the results of upstream and downstream sampling during the later ^{131}I tests, which indicated an iodine efficiency of 98.6%. Thus the normal iodine tests produced results consistent with those from the ^{131}I tests.

Fifth Series – Galveston, Texas

The fifth series of in-place iodine penetration tests was conducted on the charcoal halogen sorption units in the newly installed reactor compartment ventilation system of the NS "Savannah" during the night of April 17–18, 1963, while the ship was in the Todd Shipyards, Galveston, Texas.

This system contains two identical charcoal iodine sorption units mounted in parallel legs of the system. Temperature and humidity conditions were lower during tests conducted after modifications. Operating procedure indicates that one unit will be in constant use while the other unit is maintained in standby. For identification in this series of tests, the halogen sorption units were labeled F-1 (unit on starboard side of ship) and F-2 (unit on port side of ship). Two tests, using 5 and 15 mc of ^{131}I, were conducted on each of the two parallel units. Elemental iodine vapor labeled with ^{131}I was injected into the system duct at the "D" deck level, and samples of the air stream in the duct were taken before the iodine sorption unit at the "C" deck level and after the iodine sorption unit at the "B" deck level. After the 2-hr test period, the samplers were recovered and analyzed for their ^{131}I content by gamma scintillation spectrometry using the RCL-128 spectrometer aboard the ship. The charcoal removed from the downstream sampler of the first test (on system F-2) was found to contain only sufficient radioactivity to produce a count slightly above background. While the amount of activity was insufficient to produce a recognizable ^{131}I photopeak, the location of the radioactivity in the sampler assembly was where ^{131}I vapor would be expected. Assuming that this radioactivity was due to ^{131}I, an efficiency of 99.96+% was obtained (Table 5). A subsequent test on the same system (F-2) using a larger amount of ^{131}I indicated an iodine efficiency of 99.99+%. The two tests on system F-1 indicated efficiencies greater than 99.99% in both cases.

These in-place tests indicate that the new charcoal iodine sorption units of the reactor compartment ventilation system, as installed, are capable of achieving the iodine-vapor-removal efficiency (99.86 ± 0.07%) observed in laboratory tests of 11- by 11-in. charcoal units.

Table 5. In-Place ^{131}I Tests – New Ventilation System[a]

Test	System	Iodine Source		Iodine Efficiency (%)
		^{131}I (mc)	^{127}I (mg)	
		Fifth Series – April 1963		
11	F-1	5	10	99.99+
12	F-2	5	10	99.96+
13	F-1	15	10	99.99+
14	F-2	15	10	99.99+
		Eighth Series – October 1963		
15	F-1	5	10	99.97+
16	F-2	5	10	99.96+
17	F-1	15	10	99.93+
18	F-2	15	10	99.96+
		Ninth Series – January 1964[b]		
19	F-1	5	10	99.95
20	F-2	5	10	99.98
21	F-1	15	10	99.91
22	F-2	15	10	99.97

[a] System flow rate: 1000–1100 cfm; test duration: 2 hr.

[b] See discussion of this test series in text.

Two in-place tests and one background test were conducted using normal iodine on each of the newly installed charcoal iodine sorption units. These tests were conducted on April 16, 1963, just prior to the fifth series of ^{131}I tests. Approximately 1.5 g of iodine, in the vapor state, was injected into the system duct at the "D" deck level, and sampling units were installed at the "B" and "C" deck levels. The charcoal iodine sorption units are located on "C" deck. To obtain information concerning ^{127}I background, samplers were operated for a 2-hr period just prior to injection of iodine vapor into the system. The sampling units were returned to ORNL and analyzed for ^{127}I by neutron activation.

Samplers from the background tests contained ^{127}I in amounts ranging from 1.42 to 8.66 μg, while the downstream samplers contained from 0.95 to 4.60 μg. The background values assumed more importance compared to earlier tests because the sensitivity of the test procedure had been reduced by the necessary mechanical and operational changes in the new ventilation system. By computing the standard deviation of the amounts of iodine found in the background samplers (1.61 μg), one can demonstrate that the quantity of iodine (4.60 μg) found in the downstream sampler of test 12 does not lie outside the 68% confidence interval of the background values (3.53 ± 1.61 μg). In computing the standard deviation of the iodine content of the background samplers, the 8.66-μg value was disregarded since it reflected iodine contamination in the common duct of the two iodine sorption systems introduced by the tests on system F-2. The iodine found in the downstream samplers was equivalent to 0.1% of the upstream amounts for three of the tests and 0.3% for test 12. Since these amounts are not significantly different from background, the efficiencies are reported as 99.9+ and 99.7+% respectively (Table 6). An attempt was made to increase the sensitivity in later ^{127}I tests, beginning with the seventh series, by increasing the amount of iodine injected rather than by increasing the sampling flow rate through the samplers.

Sixth and Seventh Series – Galveston, Texas

The sixth series of in-place iodine tests using normal ^{127}I was conducted August 12–13, 1963, on the F-1 (starboard) and F-2 (port) charcoal units of the reactor compartment ventilation system. The F-1 unit had been in continuous operation and the F-2 unit on standby since the last series of tests in April 1963. Normal testing procedures were followed, with one background and two iodine tests being performed on each charcoal unit. The results of this series of tests indicated that the iodine-removal efficiency of the F-1 charcoal unit may have been reduced appreciably, since efficiencies of only 89.2 and 91.5% were obtained (Table 6). The iodine-removal efficiency of the F-2 unit tested normal.

A review of the test operations and of the subsequent analytical determinations failed to produce sufficient information that would prove or disprove that the apparently reduced efficiency of the F-1 unit was caused by faulty tests or analyses. The good agreement of duplicate tests on each system favors charcoal unit damage as the logical explanation, while the unexplained appearance of approximately 50 μg of iodine in the downstream sampler from the background test

Table 6. In-Place ^{127}I Tests – New Ventilation System[a]

Test	System	Amount of I_2 Injected (g)	Amount of I_2 Collected (μg) Upstream	Downstream	Iodine Efficiency (%)
			Fifth Series – April 1963		
10	F-1		8.66	3.83	
11	F-1	1.5	1,840.3	0.95	99.9+
12	F-1	1.2	1,460.2	4.60	99.7+
13	F-2		1.42	5.33	
14	F-2	1.3	1,472.9	1.07	99.9+
15	F-2	1.8	1,820.5	1.33	99.9+
			Sixth Series – August 1963		
16	F-1		1.09	0.95	
17	F-1	1.3	1,550.5	168.1	89.2
18	F-1	1.6	2,381.8	202.9	91.5
19	F-2		9.18	49.7	
20	F-2	1.1	1,271.1	5.31	99.6+
21	F-2	1.3	1,574.0	6.54	99.6+
			Seventh Series – September 1963		
22	F-1		1.32	3.88	
23	F-1	1.2	1,250.8	0.86	99.9+
24	F-1	2.8	356.5	0.99	99.7+
25	F-2		1.53	0.80	
26	F-2	2.0	2,331.8	1.29	99.9+
27	F-2	1.8	2,378.9	2.94	99.9+
			Eighth Series – October 1963		
28	F-1		2.62	5.99	
29	F-1	2.7	3,000.2	2.44	99.9+
30	F-1	3.0	2,990.2	3.33	99.9+
31[b]	F-1	7.0	11,201	4.76	99.9+
32	F-2		2.58	1.72	
33	F-2	2.7	3,470.5	2.50	99.9+
34	F-2	2.5	2,650.1	3.44	99.9+

[a] Test duration: 2 hr; system flow rate: 1000–1100 cfm.

[b] Special 30-min test.

of the F-2 unit raises the question of iodine contamination either during the test or in the analytical laboratory.

Because of this situation, a retest of the charcoal units (seventh series) was conducted on September 4–5, 1963. The F-1 charcoal unit had been operated since the August tests, and no changes or adjustments had been made knowingly. The same test procedures were followed, with one background and two iodine tests being conducted on each charcoal unit. The samplers were filled with new activated charcoal expressly carried from ORNL for that purpose. The various manipulations required in the tests were performed carefully and were monitored to reduce the possibility of iodine contamination among the samplers. The samplers were returned to ORNL and analyzed by neutron activation following the same analytical procedure used previously. A sample of the charcoal used in the August tests was checked for possible iodine contamination, and none was found. The results of this series indicated efficiencies of 99.7+ and 99.9+% for the F-1 system and 99.9+% for the F-2 system (Table 6). However, the test results contain an inconsistency which tends to confound the situation. The reduced quantity of iodine found in the upstream sampler from test 24 on system F-1 appears abnormal, and the results of this specific test must be viewed with caution. This latest series of tests does not confirm the August test results on F-1, and both the F-1 and F-2 systems appear to have normal iodine-removal efficiencies. Unfortunately, these two series of ^{127}I tests did not produce the desired confidence in the F-1 iodine-removal system that is required.

During the seventh series of tests, the reactor watch log books for the period May through August 1963 were reviewed, and it was noted that the overall pressure drop for the F-1 system had, for no apparent reason, decreased the day after the sixth series tests and had remained low for several weeks before returning to the value noted before the August tests. This reduction in pressure drop could result from either damage to the charcoal unit or from improper valving in routing the air flow through the ventilation system.

An unknown material, foreign to the ventilation gases, was also encountered during the seventh test series. This material was found in significant quantities in the particulate filter paper contained in the upstream iodine samplers from all the tests except the background test on system F-1. To reach the filter paper, this material, which colored the filter paper brown, passed through the activated-charcoal section of the sampler. This behavior is suggestive of particulate, rather than gaseous, material. During the analysis for iodine, some evidence was obtained which suggested that this brown deposit probably contains manganese. The concentration of this unknown material in the off-gases must be quite high in order to explain the amounts found in the filter papers.

In summary, the results of these two series of ^{127}I tests do not completely agree, and some rather improbable circumstances must be assumed to make the results wholly compatible. The need for the eighth series of tests was apparent.

Eighth Series – Galveston, Texas

The eighth series of in-place iodine tests was conducted October 7–10, 1963, and both radioactive (^{131}I) and nonradioactive (^{127}I) tests were performed. The main purpose of this series was to attempt to clarify the results produced by the sixth and seventh test series.

The radioactive tests were conducted as previously, and the charcoal samplers were analyzed using the RCL-128 spectrometer aboard ship. The results for both the F-1 and F-2 units appeared normal, that is, greater than 99.9% (Table 5).

The nonradioactive tests were also conducted according to procedures established in previous test series. The samplers were returned to ORNL for analysis by neutron activation. Both F-1 and F-2 units produced iodine efficiencies greater than 99.9% (Table 6).

Attempts, shortly after the seventh series of tests, to obtain samples of the brown material previously observed were unsuccessful, and no evidence of this material was observed during this series.

It was concluded, based upon the results of the sixth, seventh, and eighth series, that the F-1 charcoal unit possessed an adequate iodine efficiency. The F-2 charcoal unit had consistently produced adequate efficiencies. It was suggested that the operation of the reactor compartment ventilation system be continued in the normal fashion. Replacement of the F-1 unit did not appear necessary.

Ninth Series – Galveston, Texas

The ninth series of in-place iodine tests, using only ^{131}I, was conducted January 18–19, 1964. The test procedure was the same as before, with one exception. Normally, the small samplers are recovered after the 2-hr test period and analyzed for ^{131}I using the RCL-128 gamma spectrometer aboard ship. However, since the spectrometer was not in a satisfactory operating condition during this test period, a modification in the test procedure was necessary. At the completion of the first test on the F-1 system, the two sampling units were disassembled and the component parts were taken to the University of Texas Medical School in Galveston, where a single-channel gamma spectrometer was used to determine the ^{131}I content of the downstream sampling unit. The spectrometer, used for low-level counting, was not readily adaptable for analyzing the larger quantities of ^{131}I contained in the upstream sampler. After it was determined that no appreciable quantity of ^{131}I had penetrated the charcoal units of the ventilation system, it was decided that the second iodine test on the F-1 system, using a larger quantity of ^{131}I, could be conducted safely. This same procedure was followed for the tests on the F-2 system the following day. Since a suitable facility for gamma counting the upstream samples was not available in the Galveston area, the eight sampling units were returned to ORNL for analysis. The results indicated adequate efficiencies for both systems (Table 5).

As indicated earlier, the ^{131}I content of the sampling units is usually determined using the RCL-128 gamma spectrometer aboard ship. In previous tests the amount of radioactivity in the downstream sampling unit was not sufficient to produce a photopeak recognizable as ^{131}I but did produce a count slightly above background. In computing efficiencies, it was assumed, however, that all counts represented ^{131}I; therefore, the efficiencies reported represented the lower limit and a "plus" symbol was added to indicate that the reported efficiency was not significantly different from 100%. In the case of the ninth test series, all the downstream samplers were found to contain ^{131}I in sufficient quantities to produce well-defined photopeaks. The component parts of the sampling units were counted at ORNL using a Technical Measurements Corporation (TMC) model 404 gamma spectrometer, which has a greater sensitivity than the older spectrometer aboard ship, and ^{131}I photopeaks were observed in the downstream samplers from both the F-1 and F-2 systems. Therefore, the values listed for these tests represent actual efficiencies and not lower limits as in some previous tests.

Conclusions from Shipboard Tests

Based on the results of the in-place tests conducted on board the NS "Savannah," it was concluded that during the period May 1962 to January 1964 the reactor compartment ventilation system, as installed, was capable of achieving at least the iodine-removal efficiency observed in a series of 14 laboratory tests of 11- by 11-in. charcoal units under accident conditions. These tests, described elsewhere in this report and conducted with continuous iodine injection at 96 to 100°C and with 80 to 90% saturated steam in air, had shown the efficiency of these charcoal units to be 99.86 ± 0.07% at the 95% confidence level. There was no evidence of deterioration of efficiency of the adsorbers after ten months in service or after ten months in standby under shipboard conditions.

Considerable experience was gained in conducting in-place iodine-removal efficiency tests under a variety of conditions. The conditions included two isotopes of iodine (^{131}I and ^{127}I), two types of sorber materials (charcoal and silvered copper), four different ventilation systems, times when the ship was moored in a quiet harbor and times when it was at sea in a storm, operation of the ship by two different contractors, location of the ship in two oceans and the Gulf of Mexico, and occupancy levels varying from being almost abandoned during a labor dispute to being toured by AEC officials and guests. This experience has shown that such in-place tests are feasible, and that they can perform a useful function of maintaining an awareness of the condition of a vital safeguard in addition to proving its effectiveness.

The testing method using radioactive ^{131}I proved to be highly sensitive and relatively immune from interference. It gave results within a few hours after the test but suffered the disadvantage of requiring precautions to avoid accidental release of the ^{131}I sources.

The testing method using nonradioactive ^{127}I with activation analysis proved to be safe and simple to conduct but was somewhat subject to interference by variations in the amounts of ^{127}I in the environment or in the materials used. This interference limited the sensitivity of the method slightly. The most serious shortcoming of the ^{127}I method was the time delay of one or two weeks required for activation analysis.

Thus the shipboard tests demonstrated the readiness and the capability of the reactor compartment ventilation system to retain iodine with high efficiency, and they showed that in-place tests of such engineered safeguards are feasible.

LABORATORY STUDIES OF IODINE ADSORPTION

Abnormal Iodine Penetration of Charcoal Adsorbers

Small-scale tests were conducted on samples of various charcoal materials to determine their iodine adsorption efficiencies under various operating conditions. This part of the study was originally intended to provide semiquantitative information on the behavior of various charcoals at design and off-design conditions; however, in some instances abnormal penetration occurred, and the study was modified to explore these phenomena.

The experimental system used initially is shown in Fig. 12. Incoming air was passed through the glass steam generator and then routed into the test system from the right in the photo. Iodine

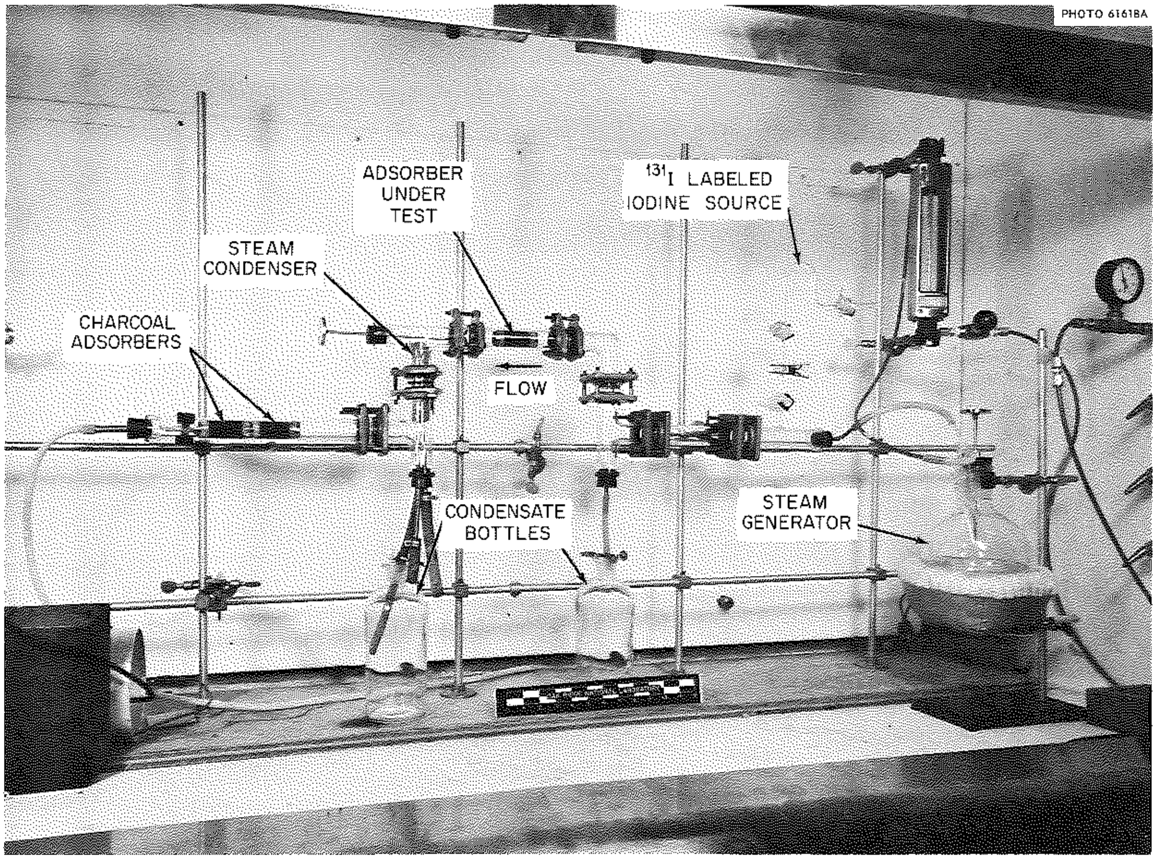

Fig. 12. Laboratory Apparatus for Investigation of Iodine Vapor Adsorption.

vapor ($^{131}I + ^{127}I$) was swept slowly from the glass U-tube (maintained at the temperature of solid CO_2) by a small flow of air, which was then mixed with the main stream. The gas mixture, air–iodine vapor–steam, was then passed through the charcoal adsorber under test (uppermost charcoal adsorber in the photo). Construction details of these 10-g-scale charcoal adsorbers are illustrated in Fig. 13. The test system was heated and insulated to a point just downstream of the charcoal adsorber under test to prevent excessive condensation of steam by the walls. After emerging from the test adsorber, the gas mixture was passed through silvered-copper mesh wrapped around a water-cooled steam condenser and finally through two room-temperature charcoal adsorbers. Drains were provided for steam condensate collection before and after the test adsorber. At the conclusion of the experiment, the test system was cooled and then disassembled up to the flange joint immediately upstream of the charcoal adsorber under test. The various parts, including condensates, were analyzed for their ^{131}I content. With these data an iodine adsorption efficiency for the charcoal adsorber could be calculated.

Forty-five tests were completed on four types of charcoal under various combinations of temperature and humidity. A summary of the results is presented in Table 7. Iodine vapor concentration was approximately 10^{-4} mg per cubic meter of sweep gas; the superficial linear velocity through the charcoal ranged from 21 to 33 fpm; and the bed depth was 1.2 in. For the most part, the charcoal exhibited what may be termed normal penetration by iodine considering the conditions specified for the test. However, abnormal penetration of iodine was observed in too many cases to be ignored. Variations in (1) the environmental conditions of the laboratory, (2) operating procedures for supposedly identical experiments, (3) materials used in construction of the experimental system, and (4) the iodine vapor source, either singly or in combination, must be considered as possible causes.

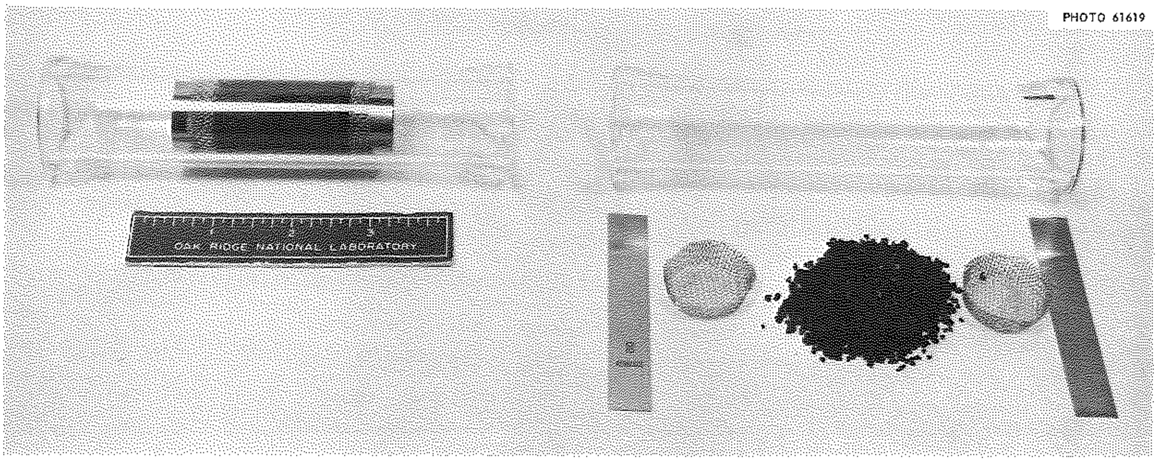

Fig. 13. Assembly of Charcoal Adsorbers for Laboratory Investigations.

Table 7. Results of Initial Small-Scale Iodine Adsorption Tests[a]

Charcoal	Number of Tests	Temperature (°C)	Steam	Efficiency Range (%)	Average (%)
BPL	16	97–100	Yes	19.9–99.7	80.0
PCB	8	97–100	Yes	50.7–99.9	86.8
Whetlerite[b]	3	98–99	Yes	82.9–97.6	92.2
GC	2	98–99	Yes	98.1 and 99.4	98.8
BPL	4	97–100	No	96.3–99.7	98.4
BPL	12	24–25	No	90.4–99.9+	98.5

[a] Iodine vapor concentration, $\sim 10^{-4}$ mg/m^3; superficial linear velocity, 21–33 fpm; charcoal bed depth, 1.2 in.; all charcoals −12 +30 mesh except GC, −8 +14 mesh; test duration, 5 hr.

[b] Whetlerite is BPL charcoal containing salts of silver, copper, and chromium.

A number of exploratory experiments were performed, yielding information which leads one to suspect that (1), (2), and (3) above were not the prime causes of this observed abnormal penetration. One cannot say, however, that these causes can be dismissed entirely. Very small particles, which appeared to be compounds of copper, were found in the compressed air supply. However, no difference in iodine penetration was noted when the air supply was passed through high-efficiency membrane filters to remove these particles and when it was not. This observation was further substantiated when bottled "breathing" air, which contained no observable particles, was used in place of the compressed air. No effect was noted by varying the materials of construction in the experimental apparatus. It was noted, in the 100°C steam experiments, that if a special effort was made to saturate the charcoal grains with water, then the iodine penetration would become large. This, however, was considered to be a special laboratory condition and not apt to have occurred in all the prior experiments where excessive penetration was observed. This saturation by water might be classed as a secondary cause but not the primary one. The result of these exploratory experiments was the isolation of the iodine source as the most probable primary cause of this abnormal iodine behavior.

Prior to this time, all the experiments had been accomplished with the charcoal bed under test having a nominal 1.2-in. depth of Pittsburgh BPL charcoal, −12 +30 mesh. In an effort to obtain additional information concerning the nature of the iodine source, the bed depth was increased to 8 in. These experiments, at room temperature, exhibited an increased iodine adsorption efficiency, as expected; however, the distribution of iodine within the charcoal mass was suggestive of a mixture of elemental iodine vapor and one or more forms of iodine which were more penetrating. The major fraction of the ^{131}I activity was stopped in the first 0.75 to 1.0 in. of charcoal, with a much smaller amount being found generally distributed throughout the remainder of the charcoal mass. A downstream high-efficiency filter and a secondary 8-in.-deep charcoal bed also contained small amounts of ^{131}I activity.

On the basis of these observations, an experimental system was designed which permitted a continuous measurement of the iodine-removal efficiency of the charcoal during the experiment. This system is diagrammed in Fig. 14 and is pictured in Fig. 15. Small upstream and downstream analytical samplers were monitored by 3- by 3-in. NaI crystals coupled to a multiple-input gamma spectrometer. The rate of increase of ^{131}I activity in the upstream sampler was related to the rate of release from the iodine source, which was thermostated to the temperature produced by a freezing mixture of solid CO_2 and trichloroethylene. At the end of each experiment, the main system and samplers were disassembled and analyzed for ^{131}I content. This refined apparatus was used to investigate the behavior of iodine as a function of time under several conditions which were suspected of causing or influencing the variations in efficiency. In the experiments where no moisture was added, the relative humidity was approximately 1 to 3%. In the experiments where moisture was added, the humidity was increased to approximately 60% by passing the incoming air sweep over water. The results, somewhat inconclusive, are shown in Table 8 and suggest that sometimes low iodine adsorption efficiencies can occur under moist conditions. It was noted that, when moisture was added, the distribution of ^{131}I in the components downstream of the charcoal adsorber became abnormal compared to the distribution when the air sweep was dry.

The effect of foreign particles in an iodine collection system was studied briefly using zinc oxide particles generated by passing a spark between zinc electrodes in air. Both nonradioactive and radioactive zinc electrodes were used in the experiments. The particle size was estimated

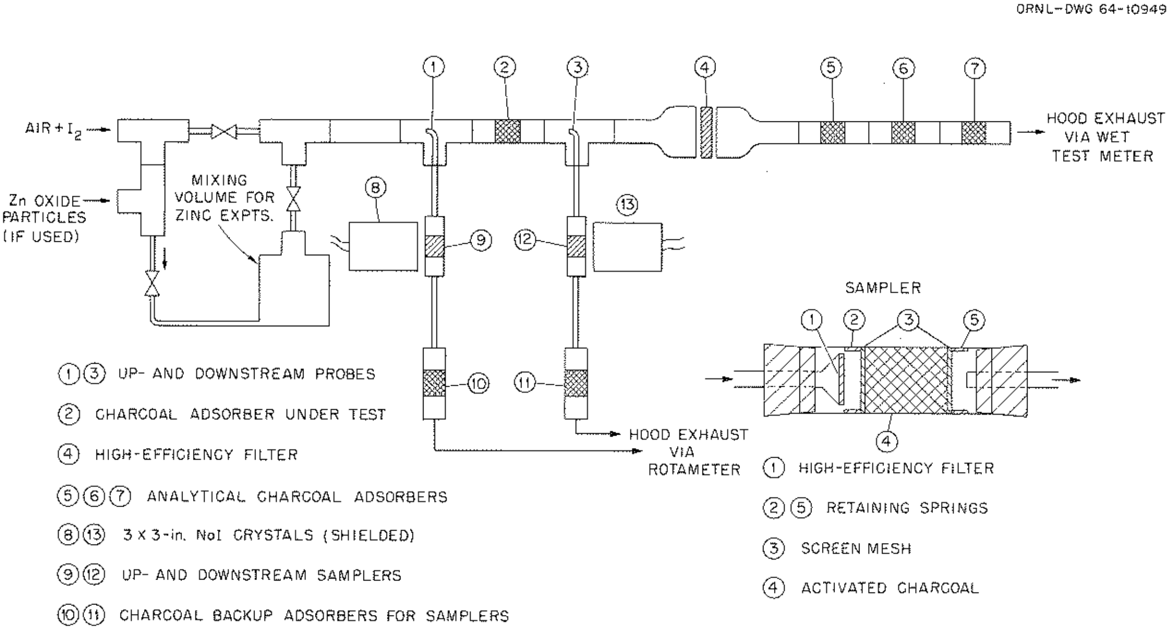

Fig. 14. Schematic of Laboratory Apparatus for Continuous Observation of Iodine Adsorption Efficiency.

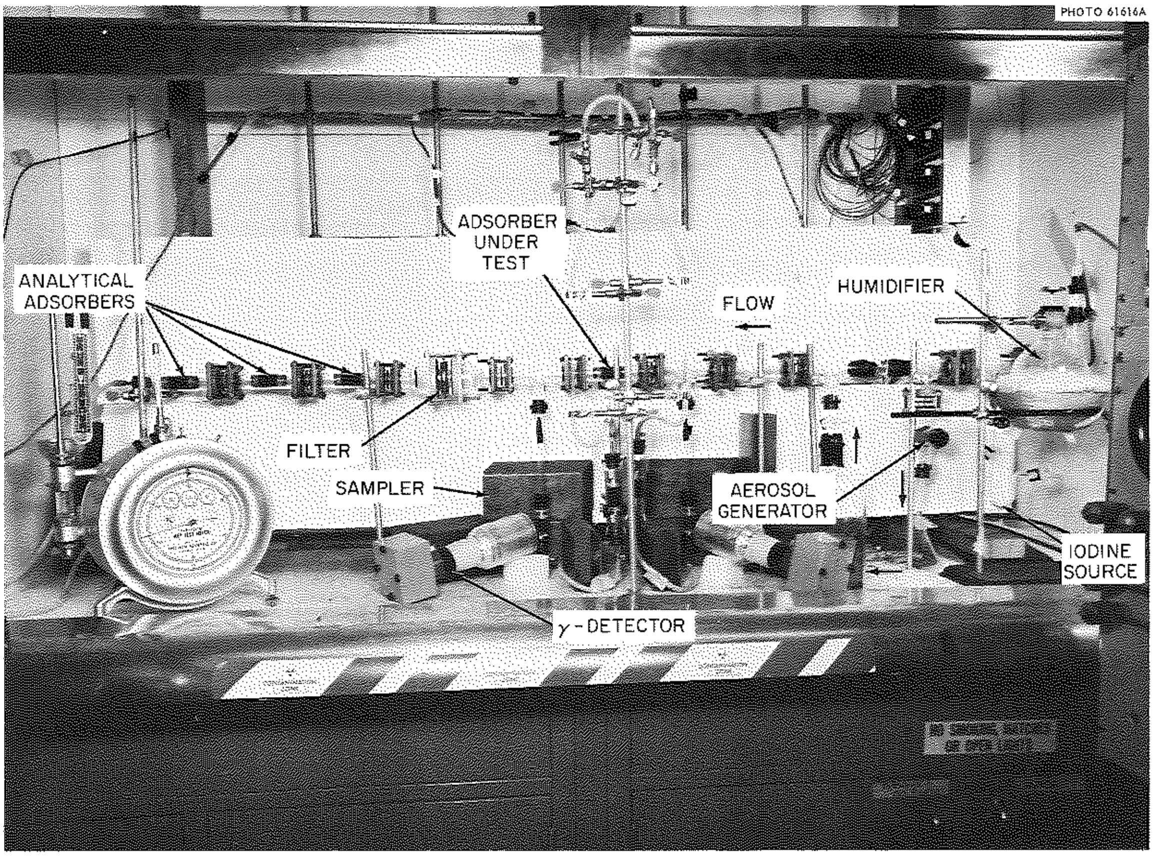

Fig. 15. Laboratory Apparatus for Continuous Observation of Iodine Adsorption Efficiency.

to range between 20 and 300 A, based on the experience of others using this type of particle generator.[5] These results are also shown in Table 8 and indicate that these particular particles do not interfere with the iodine efficiency of a charcoal adsorber.

It was observed that in some experiments there was a rapid initial transport of ^{131}I activity from the source, followed by a slower, reasonably constant rate of transport. This and other observations indicated that the iodine sources contained, but not necessarily at all times, two or more components. The initial rapid release of ^{131}I activity from a source at a temperature around –70°C was suggestive of an iodine compound more volatile than elemental iodine. The gradual release of ^{131}I activity after this initial release may be characteristic of either elemental iodine or a compound slightly more volatile than elemental iodine. Calculations, using an estimated iodine vapor pressure of 10^{-6} mm, indicated that sufficient elemental iodine vapor

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[5] W. E. Browning, Jr., and M. D. Silverman, "Measurement of Characteristics of Radioactive Aerosols Using Fibrous Filters," *Nucl. Safety Program Semiann. Progr. Rept. December 31, 1962*, ORNL-3401, pp. 50–55.

Table 8. Tests to Investigate Anomalies in Iodine-Removal Efficiency

All tests at 23–25°C

Pittsburgh BPL, -12 +30 mesh, charcoal in test unit (1.25 in. deep)

Superficial linear velocity, 22–25 fpm

Iodine vapor concentration, 10^{-2} to 10^{-3} mg/m^3

Test Number	Duration of Test (hr)	Air Supply	Added Particles	Added Moisture	Overall Efficiency (%)
1	18.0	Filtered	None	No	99.91
2	14.0	Filtered	None	No	99.81
5	17.5	Filtered	None	No	99.92
6	11.3	Filtered	Zn fume	No	99.93
7	13.6	Filtered	Zn fume	No	99.77
8	12.5	Filtered	^{65}Zn fume	No	99.91
9	12.0	Filtered	^{65}Zn fume	No	99.93
10	12.0	Not filtered	None	No	99.98
11	12.0	Not filtered	None	Yes	76.22
12	18.5	Not filtered	None	Yes	91.88

could be transported from the source into the system to account for the ^{131}I radioactivity. However, observations of the behavior of this radioactivity in the charcoal lead one to suspect that the less volatile fraction may be composed of elemental iodine mixed with some small amounts of an iodine compound. Such a compound could result from reaction of the iodine with trace impurities at some stage during the preparation or transfer of the source. The occurrence, in the source, of a compound of iodine volatile at dry-ice temperature can explain the anomalous behavior observed in the small-scale tests. The use of a dry-ice bath to regulate the transport of iodine from the source could have concentrated the volatile compound in the initial tests.

Behavior of Iodine Compounds

The difference in volatilities of the compounds of iodine sources was used as the basis for separating them by fractional vaporization at about –70°C. It thus became possible to investigate the individual behavior of these iodine substances.

Initial results of the study of this behavior are presented in Table 9, and the experimental apparatus is illustrated in Fig. 16. The behavior of iodine in tests 1 and 2 was representative of that in a charcoal system when no effort is made to separate the two fractions of the iodine source. Tests 3 to 8 illustrated the efficiencies that are obtained when the iodine source is divided into two fractions, the first being that part which is vaporized in less than 10 min at –70°C, and the

Table 9. Efficiency of Charcoal in Removing Iodine and Volatile Iodine Compounds from Air[a]

Test	Iodine Fraction[b]	Charcoal	Temperature (°C)	Relative Humidity[c] (%)	Efficiency (%)
1	A + B	BPL	23	~60	90–95
2	A + B	BPL	23	1–3	99.9+
3	A	BPL	23	1–3	98.0
	B	BPL	23	1–3	99.9+
4	A	BPL	23	~60	50.3
	B	BPL	23	~60	97.9
5	A	BPL	100	1–3	98.5
	B	BPL	100	1–3	99.9+
6	A	BPL	100	~60	99.1
	B	BPL	100	~60	99.9+
7	A	Whetlerite[d]	24	~60	90.5
	B	Whetlerite	24	~60	99.8
8	A	Whetlerite	100	~60	99.9+
	B	Whetlerite	100	~60	99.9+

[a] Bed depth, 1.5 in.; gas velocity, 25 fpm; duration of test, 4 hr; charcoal mesh size, -12 +30.

[b] A is the volatile fraction (low-molecular-weight iodine compounds) and B is the less volatile fraction (primarily elemental iodine) when the iodine source is at -70°C. A is injected during the initial 10 min of test at ~5×10^{-2} mg/m^3, B over 5 hr test duration at ~1×10^{-2} mg/m^3.

[c] Relative humidity of air sweep evaluated at room temperature.

[d] BPL charcoal impregnated with salts of silver, copper, and chromium.

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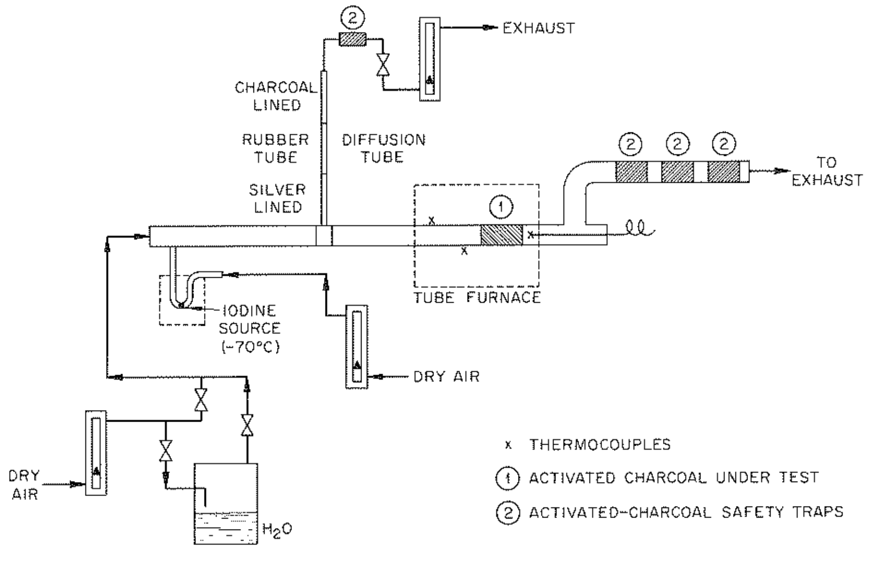

Fig. 16. Schematic of Laboratory Apparatus to Study Retention of Iodine Compounds by Charcoal.

second being that which is vaporized after 10 min. They indicated, to a small degree, the variation of efficiency for each fraction with the parameters of humidity, temperature, and type of charcoal. Evidently, the volatile fraction was adsorbed by charcoal less efficiently, and moisture affected the behavior of the volatile fraction to a much larger extent than it did the less volatile fraction. This was consistent with the observation [in earlier experiments where the two fractions of iodine were not distinguished (Table 8)] that with moisture added to the air sweep gas the iodine efficiency of the charcoal unit was sometimes decreased significantly, and the distribution of ^{131}I in the components downstream of the charcoal unit became abnormal compared to the distribution when the air sweep was dry. Elevated temperature and the presence of metallic impregnants in the Whetlerite charcoal together apparently enhanced the adsorption or retention of the volatile iodine fraction by charcoal.

In an attempt to identify the components of the iodine sources, the analytical technique of mass spectrometry was applied. The residue of an iodine source which had been used in the laboratory was found to contain two rather high-molecular-weight compounds, neither corresponding to the mass of I_2. A freshly prepared iodine source which was not allowed to reach a temperature greater than that of solid CO_2 was found to contain masses corresponding to those of HI, ICl, and I_2. Other iodine sources have contained CH_3I in addition to these compounds. Contemporary reports of investigations being conducted in England indicated the presence of CH_3I, and other iodine compounds, in many or possibly all of their iodine sources.[6]

Diffusion channel analysis[7] was also used to investigate these compounds of iodine. This analytical method involves the measurement of the distribution of radioactivity in a special channel after gas containing the radioactivity of interest has passed through it under conditions of laminar flow. Analysis of this distribution resulting from diffusional deposition yields diffusion coefficients which are related to molecular properties of the radioactive vapor. The diffusion channels are composed of a series of three tubes having different inner surfaces. The first surface is silver, the second is rubber, and the third is charcoal dust which had been applied with an adhesive. Measurements under "dry" conditions showed that the more volatile fraction deposited on a charcoal-lined diffusion tube with a diffusion coefficient of $\sim$0.1 cm^2/sec but not on silver or rubber (Fig. 17), while the less volatile fraction primarily deposited on silver with a diffusion coefficient of 0.08 cm^2/sec, characteristic of molecular iodine (Fig. 18). This fraction also contained other compounds of iodine. Under "moist" conditions the diffusion patterns were destroyed in the charcoal-lined diffusion tubes, and the deposition on the silver surfaces was affected. At this time it is not clear whether the moisture "poisoned" the surface of the tubes or changed the molecular or physical characteristics of the iodine compound.

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[6] D. H. F. Atkins and A. E. J. Eggleton, *Iodine Compounds Formed on Release of Carrier-Free Iodine-131*, AERE-M-1211 (May 1963).

[7] W. E. Browning, Jr., and R. D. Ackley, "Characterization of Radioactive Iodine Compounds by Diffusional Deposition," *Nucl. Safety Program Semiann. Progr. Rept. June 30, 1963*, ORNL-3483, pp. 26–28.

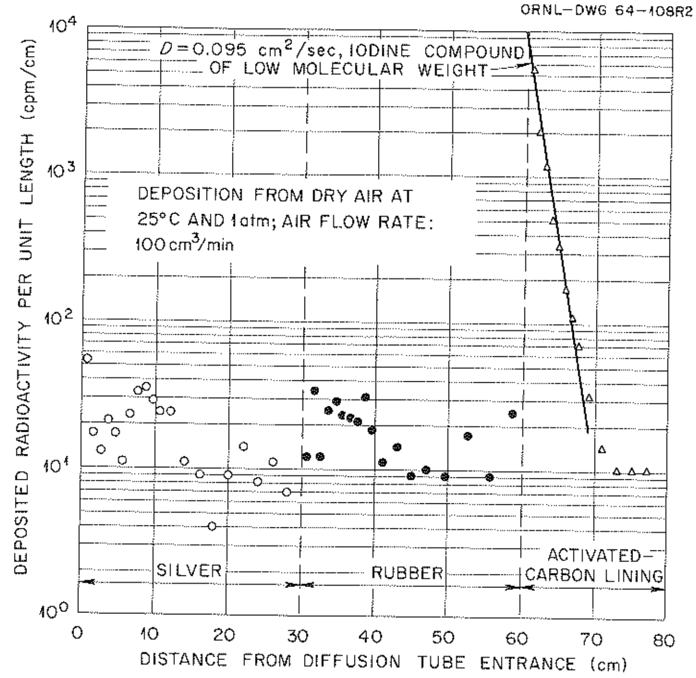

Fig. 17. Diffusional Deposition of a Volatile Iodine Compound of Low Molecular Weight.

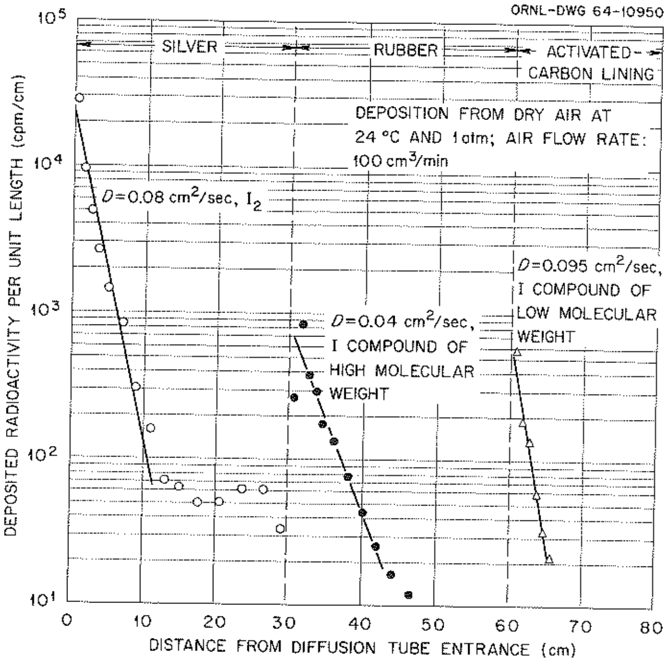

Fig. 18. Diffusional Deposition of Molecular Iodine and Other Iodine Compounds.

Efforts to identify fully the various forms of iodine and to characterize their behavior in activated-charcoal adsorbers are continuing. It is necessary to know the identities of these compounds and the circumstances of their occurrence so that an evaluation can be made of the possibility of their appearing in a reactor accident. Their behavior in adsorbers under accident conditions must be investigated so that appropriate adsorber efficiencies may be determined. This work is being continued as a part of the Nuclear Safety Program at ORNL and is reported semiannually.[2]

It is noted that the nonelemental compounds are by no means the major fraction of the iodine vapor sources. They apparently account for only a very small fraction of the iodine mass contained in the source. For example, in many sources only a fraction of 1% of the iodine is nonelemental. Thus, only moderate efficiencies for removal of these forms of iodine may be required to yield adequate overall iodine efficiencies. The tests on 11- by 11-in. reduced-scale adsorbers were conducted under realistic conditions and probably included the effects of iodine compounds. Thus the results of these tests are, at present, the best indication of the performance to be expected of adsorbers under accident conditions, even considering the possible occurrence of iodine compounds.

ASSOCIATED LABORATORY STUDIES

Investigation of Freon for Adsorber Testing

Each of the in-place test methods already described has one deficiency which prevents its convenient adaptability to the testing requirements aboard the NS "Savannah." The test procedure using ^{131}I, while being very sensitive and producing almost immediate test results, involves the risk of radioactive contamination of the ship or its environment. The ^{127}I procedure circumvents the contamination problem but with a resulting loss in sensitivity and with a large increase in the time period from conduct of the test until results are available.

Retention of Freon by activated charcoal was studied on a laboratory scale as a possible means for frequent nonradioactive testing. A reduction in the time normally required for a quantity of Freon to penetrate the charcoal unit would be indicative of either the deterioration of the charcoal adsorber or the development of leaks or channels in the charcoal mass. Qualitative information derived from these Freon tests would then be verified periodically by either ^{131}I or ^{127}I tests.

Laboratory tests were conducted under conditions simulating those present during normal operation of the full-scale ventilation system. Freon-12 (CCl_2F_2), -13 ($CClF_3$), -14 (CF_4), and -22 ($CHClF_2$) were used in these investigations. Freon-14 and -22 were dropped from the study because of their very rapid and somewhat erratic penetration of the charcoal test unit. Consequently, Freon-12 and -13 have received the most attention. Experimentally, the length of time (t_{max}) required for a rapidly injected amount of Freon gas (3–5 cm^3 STP) to reach a maximum concentration downstream of a 1.25-in. depth of either PCB or BPL, −12 +30 mesh, charcoal was measured.

Air at a superficial velocity of 6.5 fpm was passed through the charcoal mass, which was maintained at 23–24°C. The experimental apparatus used in this study is pictured in Fig. 19. The control panel at the right contained provisions for air flow control and Freon injection. The air was humidified by passage through the water vapor generator and then into the system. During injection of the Freon, a bypass arrangement prevented it from contacting the water. Immediately after the injection, the air stream was diverted through the water vapor generator. A General Electric halogen leak detector with a type H-3 probe, set to a sensitivity of 0.1 ppm Freon-12 in air, was used to detect the Freon gas downstream of the charcoal mass under test. The humidity of the system was computed from dew point readings taken with a Weighing and Components Controls, Inc., dew point indicator. With this instrument the dew point temperature is measured by manual observation of moisture condensation on a refrigerated mirror.

Temperature and humidity of the gas-charcoal system were selected as the principal variables for initial study since it would be difficult to control them aboard ship. Humidity of the gas stream (or, more specifically, the water adsorbed on the charcoal surfaces) was found to interfere with the adsorption of the various Freons to a degree larger than anticipated.

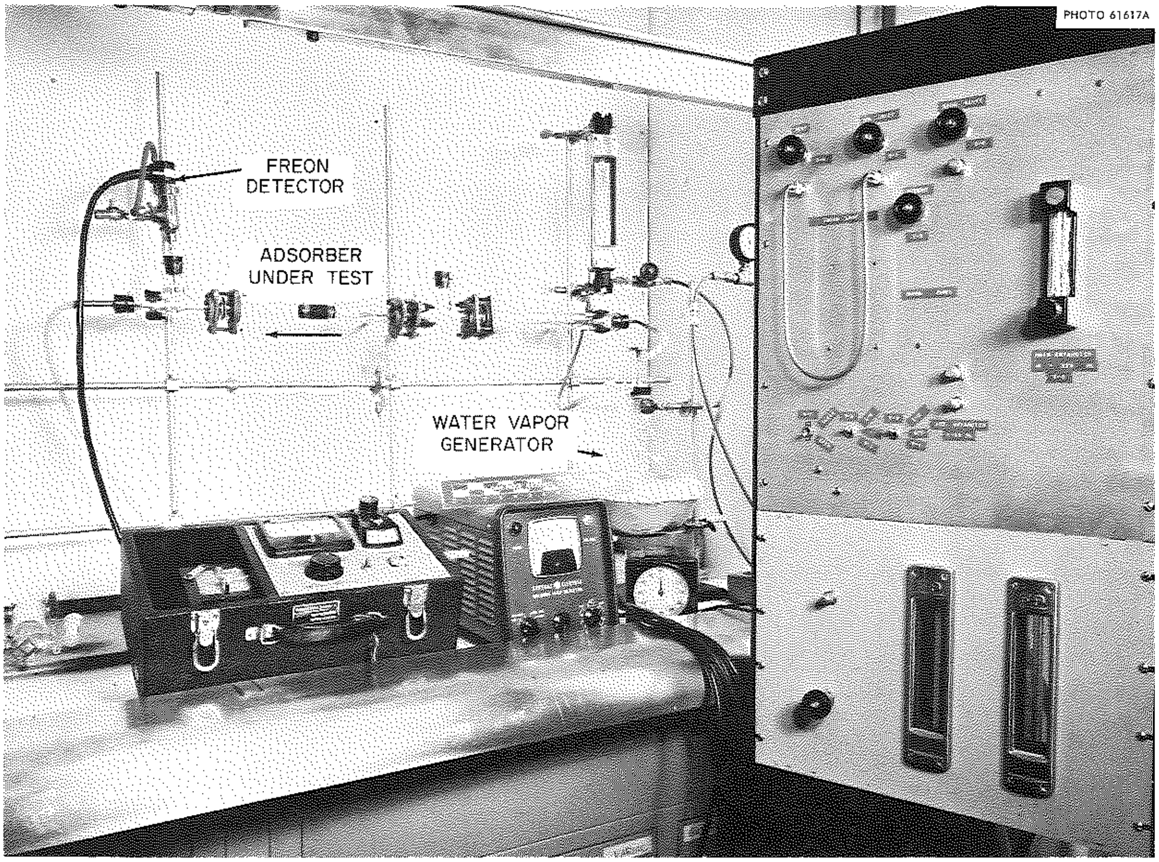

Fig. 19. Laboratory Apparatus for Investigation of Freon for Adsorber Testing.

Holdup time data obtained on Freon-12 and -13 are displayed in Fig. 20. To explain the curves obtained, one must refer to the characteristic adsorption of water vapor by activated charcoal. Unlike desiccant or molecular sieve materials, charcoal physically adsorbs water vapor to an equilibrium level depending upon the humidity of its environment. If the humidity changes, then the quantity of water adsorbed per unit weight of charcoal changes to reflect the new equilibirum relationship. Figure 21 depicts this equilibrium adsorption of water vapor by activated charcoals. Note the enhancement of adsorption of Freon in Fig. 20 over the humidity range of 40 to 60%.

This interference of Freon adsorption by adsorbed water vapor on the charcoal surface is of such magnitude that successful application of this method of testing the charcoal units was considered doubtful. The temperature and humidity of the reactor compartment are subject to wide variations, depending upon the geographical location of the ship and whether or not the reactor

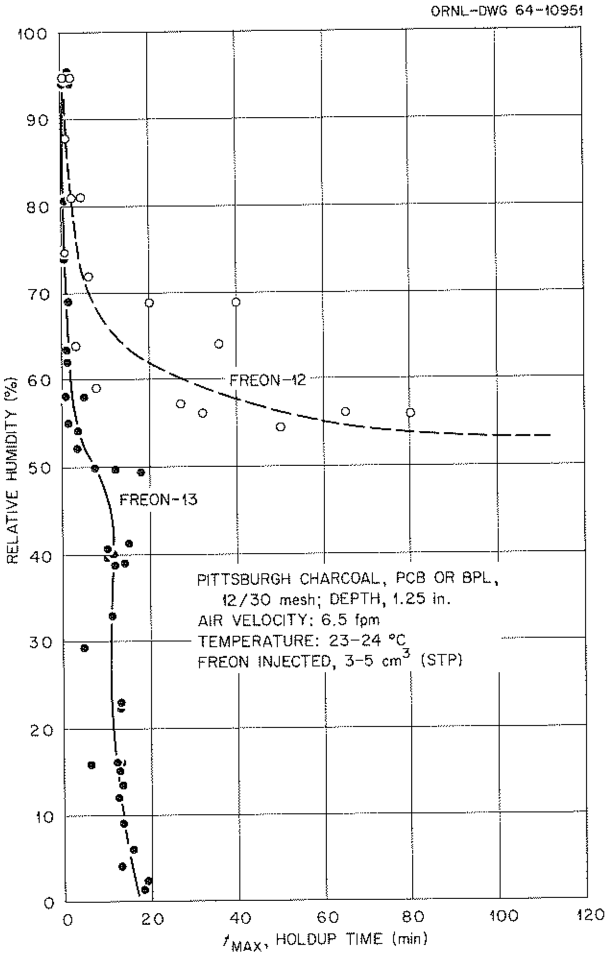

Fig. 20. Effect of Humidity on Holdup Time of Freon-12 and -13 by Charcoal.

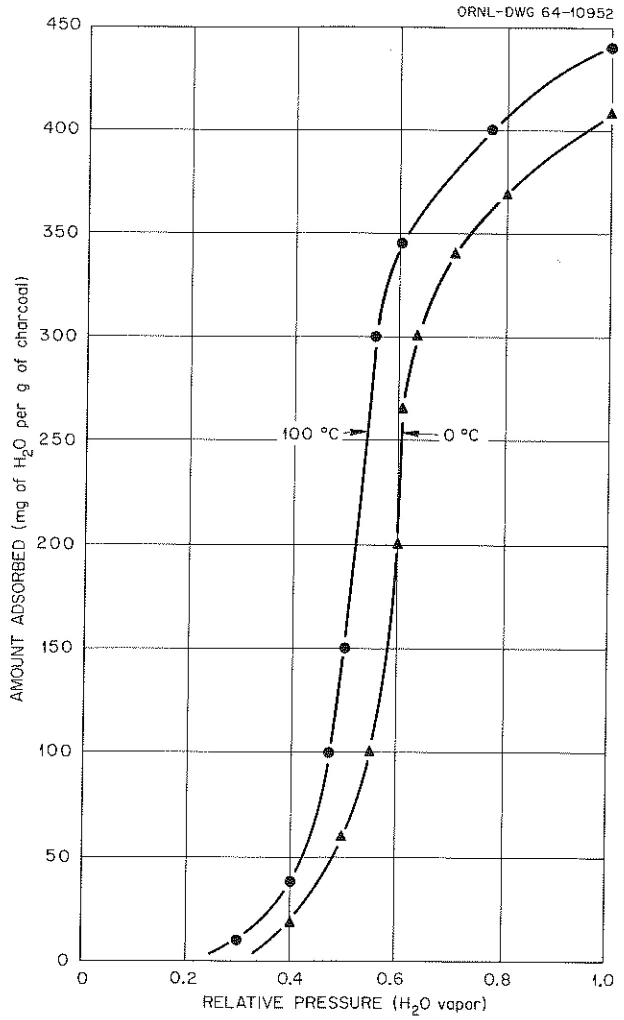

Fig. 21. Adsorption of Water Vapor by Charcoal. From S. Dushman, *Scientific Foundations of Vacuum Technique*, pp. 483–84, Wiley, New York.

is operating. Because of these changing conditions, one would never be certain of the water content of the charcoal in the iodine sorption units. High temperatures and humidities could cause such a reduction in Freon retention that one might suspect damage to the charcoal unit when, actually, none had occurred. Interpretation of the results of Freon tests aboard the NS "Savannah" would be difficult, at best. Application of Freon tests under carefully controlled conditions has been reported recently.[8]

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[8] A. H. Peters and D. R. Muhbaier, *Nondestructive Test of Carbon Beds for Reactor Containment Applications*, DP-870 (February 1964).

Development of Adsorber for Environmental Iodine Monitoring

Environmental monitoring for the presence of radioiodine in the various compartments of the ship is accomplished by passing a measured volume of air through an activated-charcoal cartridge. Iodine radioactivity in the cartridge is then determined by one of several types of radiation detection instruments. High air flow rate and small sample size are necessary operational features; therefore, high linear gas velocities exist in the charcoal mass. Twelve experimental tests were performed under conditions of low mass concentration of iodine in the air and high linear gas velocity. Under linear gas velocities of 295 fpm and iodine concentrations of the order of 1 $\mu g/m^3$, efficiencies consistently greater than 90% were obtained for Pittsburgh PCB charcoal, −6 +16 mesh, in depths of 1.75 in. for a 45-min test period. The average efficiency was 97.4% under these conditions. Efficiencies in this range are considered adequate for environmental monitoring purposes.

ACKNOWLEDGMENTS

The assistance of W. M. Johnson, Reactor Chemistry Division, and F. V. Hensley, Plant and Equipment Division, in conducting the laboratory studies and shipboard tests; of H. T. Russell, S. T. Carroll, and E. Jacobs, Isotopes Division, in preparing the large number of iodine sources used; of E. I. Wyatt and R. R. Rickard, Analytical Chemistry Division, in providing neutron activation analysis; and of G. M. Watson, Reactor Chemistry Division, and H. C. McCurdy, Reactor Division, for support and encouragement is gratefully acknowledged. Numerous members of the NS "Savannah" ship personnel and shore staff contributed to the successful performance of the in-place tests. In particular, the contributions of R. O. Mehann, E. P. Resner, R. S. Deakin, J. Robinson, W. G. Thomson, and K. R. Holtzinger are appreciated.

APPENDIX

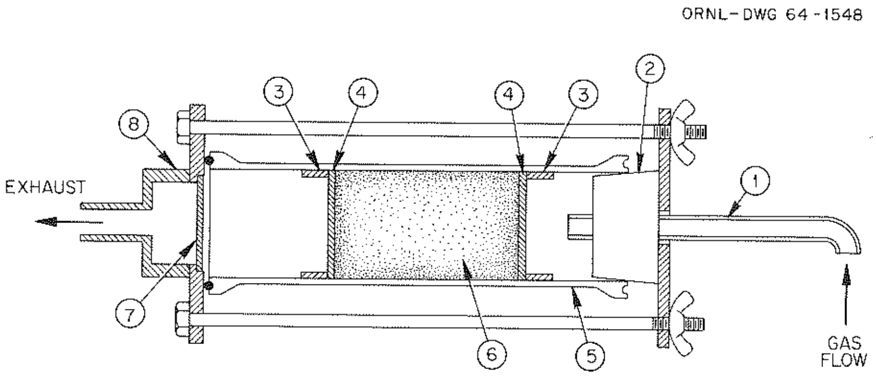
Analytical Methods

Iodine-131 Counting

The small sampling units, schematically illustrated in Fig. A.1, were disassembled at the end of the test, and the component parts or wipings were sealed in plastic dishes for counting. The flat 3-in.-diam plastic dish containing the sample was placed directly upon the top of the 3- by 3-in. NaI crystal coupled to an RCL-128 multichannel analyzer installed on board the ship. The relative amount of ^{131}I activity was determined by summing the counts found in the channels comprising the peak area representative of the 0.36-Mev gamma associated with the decay of ^{131}I into ^{131}Xe. Background counts were, of course, subtracted from the gross count on each sample. Comparison of the relative amounts of activities found in the two samplers yielded a measure of the iodine vapor efficiency of the activated-charcoal iodine unit under the ambient conditions existing during the test.

Iodine-127 Activation Analysis

A different style of sampling unit, diagrammed in Fig. A.2, was utilized for the ^{127}I tests. Since the amount of iodine contained in these samplers was to be determined by neutron activation techniques, an effort was made to construct the unit from materials that could be placed in a



KEY

1 STAINLESS STEEL PROBE, $\frac{1}{2}$ in. x 12 in.
2 RUBBER STOPPER
3 FLEXIBLE BAND RETAINER
4 SCREEN
5 GLASS PIPE, 2 in. ID
6 ACTIVATED CHARCOAL
7 HIGH-EFFICIENCY FILTER
8 END PIECE AND FILTER HOLDER

Fig. A.1. Detail of Sampling Unit for ^{131}I Shipboard Tests.

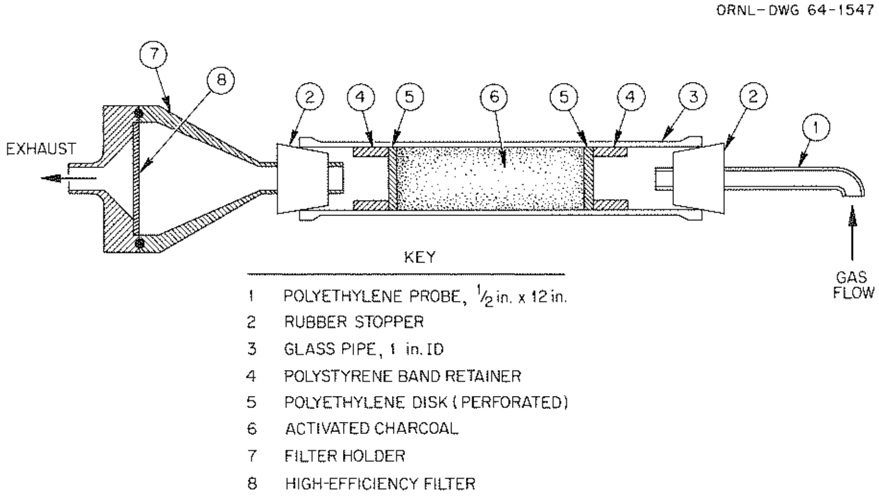


Fig. A.2. Detail of Sampling Unit for ^{127}I Shipboard Tests.

nuclear reactor. Direct neutron activation of the component parts prevented possible loss of iodine at this step. Various activated charcoals and plastics were neutron irradiated to find suitable materials. Polyethylene and polystyrene were acceptable, as well as an activated charcoal produced from coconut shells (Pittsburgh type PCB).

At the conclusion of the shipboard test series, the sampling units were returned to ORNL for determination of their ^{127}I content. This service was performed by a group within the Analytical Chemistry Division. A brief outline of their procedure follows:

1. The sampling units were broken into their component parts and placed in an irradiation container.
2. A known amount of ^{127}I was placed in a separate package within the irradiation container.
3. The container was irradiated in the ORNL Graphite Reactor* for 30 min in a thermal-neutron flux of 7×10^{11} neutrons cm^{-2} sec^{-1} with approximately a 4π irradiation geometry. The following reaction occurs: $^{127}\mathrm{I} + n \rightarrow {}^{128}\mathrm{I}$, $t_{1/2} = 25$ min.
4. The irradiation container was removed from the reactor and transported to the laboratory, where the ^{127}I "known" and the parts of the sampling unit were removed.
5. Iodine-127 carrier solution was added to the parts of the sampling unit. Each part was then leached with a solution of $NH_4OH + H_2O_2$ (1 to 1) three times. The three leach solutions were then combined. The total solution was heated to destroy the H_2O_2, concentrated, and acidified with HNO_3, and the oxidized iodine was stripped into CCl_4.
6. Normal iodine chemistry was then performed to rid the iodine of interfering impurities.
7. Finally, the iodine was precipitated and recovered as $\mathrm{Ag}^{128}\mathrm{I} + \mathrm{Ag}^{127}\mathrm{I}$ (carrier).

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*The ORNL Graphite Reactor was retired from service in November 1963, and provisions are being made in another reactor at ORNL for irradiations of this type.

8. The known from each irradiation container was carried through the same procedure.
9. By comparing the ^{128}I, determined by gamma spectrometry, of the known with that of the unknown, the original mass of ^{127}I in the unknown sample was determined.

For this particular application, this procedure was capable of detecting, quantitatively, as little as 0.3 μg of ^{127}I per sampling unit from the shipboard tests.

Preparation of Iodine Sources

Iodine-131 Sources for Laboratory Use

The elemental iodine sources used in this study were produced by a group within the Isotopes Division of ORNL and the ^{131}I sources for laboratory use were contained in glass U-tubes ready for injection into the test assembly. A U-tube may be observed in Figs. 12 and 15. A brief outline of the preparation procedure follows:

1. The ^{131}I was obtained as NaI contained in a basic sodium sulfite solution.
2. The desired amount of ^{127}I carrier was added as NaI.
3. The iodine content of the solution was precipitated as insoluble PdI_2 by the addition of acidified $PdCl_2$.
4. The PdI_2 precipitate was recovered and washed three times to remove chloride and sulfite ions.
5. The washed PdI_2 precipitate was dried at 120–130°C under vacuum.
6. The PdI_2 was then decomposed at 400–500°C under high vacuum.
7. The liberated $^{127+131}I_2$ was collected in the liquid-nitrogen-cooled U-tube.

Sources of this type were used in all the laboratory experiments.

Iodine-131 Sources for Shipboard Tests

The ^{131}I sources used in the shipboard tests were produced in the manner just described. The difference existed only in the iodine container. Since the sources had to be prepared at ORNL and shipped by air to the location of the ship, it was not considered advisable to contain the iodine solely in glass U-tubes. A metal device was constructed which doubled as a protective container and also as an injection device (Fig. A.3). The elemental iodine source was sealed in a glass ampul which was placed in a length of thin-wall stainless steel tubing closed at the ends by two valves. For use this assembly was fitted into the duct of the ventilation system and the walls of the stainless steel tubing were deformed slightly. As a result of this deformation, the glass ampul was crushed, and the iodine vapor was released to the air stream passing through the tubing and into the duct. Usually more than 90% of the ^{131}I activity was transferred into the duct within 15 min. In some cases the intake duct was extended to ensure adequate mixing before the upstream sampler.

Iodine-127 Sources for Shipboard Tests

The ^{127}I sources (normal iodine tests) were prepared by simply weighing the required quantity of elemental iodine crystals and placing this amount in the small glass injection device illustrated in Fig. A.4. To inject the iodine vapor, air was passed over the iodine mass, which was heated by the small heater. Approximately 1.5 hr was required for complete injection of 3 g of iodine vapor into the ventilation duct.

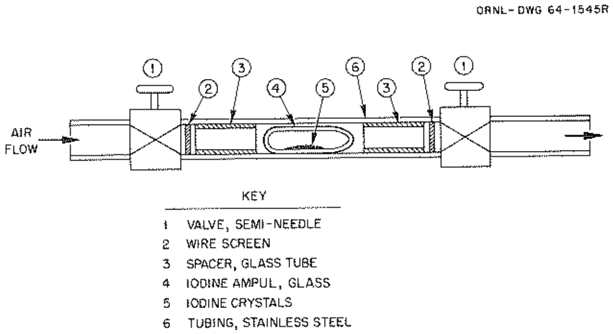

Fig. A.3. Device for Injecting ^{131}I During Shipboard Tests.

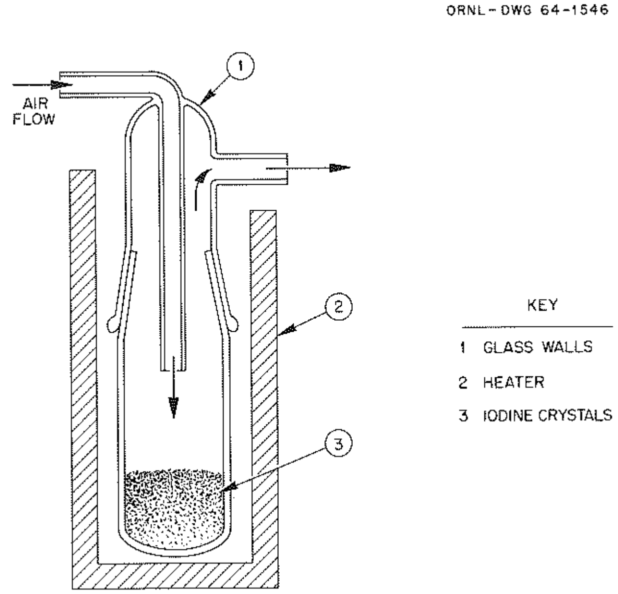

Fig. A.4. Device for Injecting ^{127}I During Shipboard Tests.

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