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AEC RESEARCH AND DEVELOPMENT REPORT

NONDESTRUCTIVE TEST OF CARBON BEDS FOR REACTOR CONTAINMENT APPLICATIONS

PROGRESS REPORT
JANUARY - MARCH 1964

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Savannah River Laboratory
Aiken, South Carolina

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NONDESTRUCTIVE TEST OF CARBON BEDS FOR
REACTOR CONTAINMENT APPLICATIONS
PROGRESS REPORT: JANUARY-MARCH 1964

by

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ABSTRACT

Development of a standardized, nondestructive and more generally applicable test for installed carbon beds that are used in reactor containment facilities was started by the Savannah River Laboratory. Tests showed that the limitations imposed on air velocity and on H₂O content of the carbon by an earlier testing method were reduced significantly when "Freons" with higher boiling points were substituted for "Freon-12".

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INTRODUCTION

Radioactive particles and halogen vapors (principally ^{131}I) that might be released accidentally into the buildings of Savannah River Plant (SRP) reactors are removed by passing the exhaust ventilating air through particulate filters and carbon beds⁽¹⁻³⁾. Standardized tests are available for evaluating the particulate filters (dioctylphthalate-aerosol penetration tests⁽⁴⁻⁶⁾). The carbon beds are tested for absence of leak paths by a nondestructive technique in which "Freon-12"* is used as a stand-in for iodine. Development of this technique by the Savannah River Laboratory (SRL) is discussed in progress report DP-870⁽⁷⁾.

The "F-12" technique, however, is limited to testing carbon beds before field installation, with air at a maximum velocity of 20 ft/min, and with the carbon containing no more than 5% adsorbed H_2O . In actual plant installations, air velocities up to 70 ft/min and relative humidities up to 75% ($\sim 20\%$ H_2O content of carbon at equilibrium) are encountered. Leaks amounting to 0.1% of the total flow are difficult to detect, because over 0.1% of adsorbed "F-12" desorbs from the carbon in approximately 1 minute when exposed at these maximum plant conditions. About 5 minutes is required to evaluate the performance of installed carbon beds. Because of these limitations, work was undertaken to develop a standardized nondestructive test that is more generally applicable for installed carbon beds. This report and subsequent quarterly reports will discuss the progress and results of the new work.

* "Freon" and combinations of "Freon-" or "F-" with numerals are Du Pont's registered trademarks for its fluorinated hydrocarbons.

SUMMARY

Tests showed that "Freons" with boiling points higher than "F-12" reduces significantly the limitations of air velocity and H₂O content of the carbon imposed by the current "F-12" technique. "F-114" (b.p. 3.6°C) and "F-11" (b.p. 23.8°C) did not desorb from carbon beds nearly as rapidly as "F-12" (b.p. -29.8°C). (See Table I for summary of properties.)

The adsorption efficiency of small-scale carbon beds for "F-11", after 5 minutes of exposure to a mixture of "F-11" and with 25°C air flowing at a velocity of 70 ft/min was:

- 99.99+% when the carbon was dry (<0.5% adsorbed water)
- 99.93% when the carbon contained 12.5% adsorbed water (relative humidity in air of ~50% at equilibrium)

Although "F-11" desorbs more slowly than "F-12", a second leak test with "F-11" could be made after the "F-11" adsorbed on the carbon during an initial test was desorbed by exposing the carbon to normal air flow for 4 days. "F-114" penetrated carbon beds more rapidly than "F-11" but less rapidly than "F-12". Evaluation of other "Freons" is in progress.

The minimum sensitivity for detection of the "Freons" in air with an electron capture type instrument⁽⁷⁾ was 0.03 ppm for "F-12" and "F-114" and <0.001 ppm for "F-11". The sensitivity of the detector for halogens generally increases considerably with increasing atomic weight of the substituting halogen atoms. Similarly, the sensitivity increases with an increasing number of the halogen atoms in the compound. Because of the improved sensitivity for detection of "F-11", the tests were made with an inlet "F-11" concentration in air of 5 ppm, as compared to 500 ppm with "F-12" and "F-114".

A facility was designed for the development of a standardized nondestructive test for iodine adsorbers. The adsorption and desorption characteristics of various "Freons" can be evaluated on full-size carbon beds under the following test conditions:

- | | |
|---------------------|-----------------|
| • Air flow | 100 to 2000 cfm |
| • Air temperature | 15 to 120°C |
| • Relative humidity | 10 to 95% |

The facility was also designed for testing full-size beds with stable iodine vapor to develop correlations between "Freon" and iodine vapor penetration of carbon beds.

DISCUSSION

TEST EQUIPMENT

Small-Scale Facility

Screening tests of various "Freons" are being conducted in a small-scale facility designed to determine the major effects of face velocity, temperature, and H_2O content of the carbon on the "Freon" adsorption characteristics. The most promising "Freons" will be selected from these tests for further evaluation in a facility that is designed for testing full-size carbon beds for actual plant installations. The small-scale facility consists of three basic systems:

- Carbon Bed Test Center
- Humidification and Air Flow Control System
- "Freon"-Air Dilution System

All systems are shown schematically in Figure 1 and their functions are explained below.

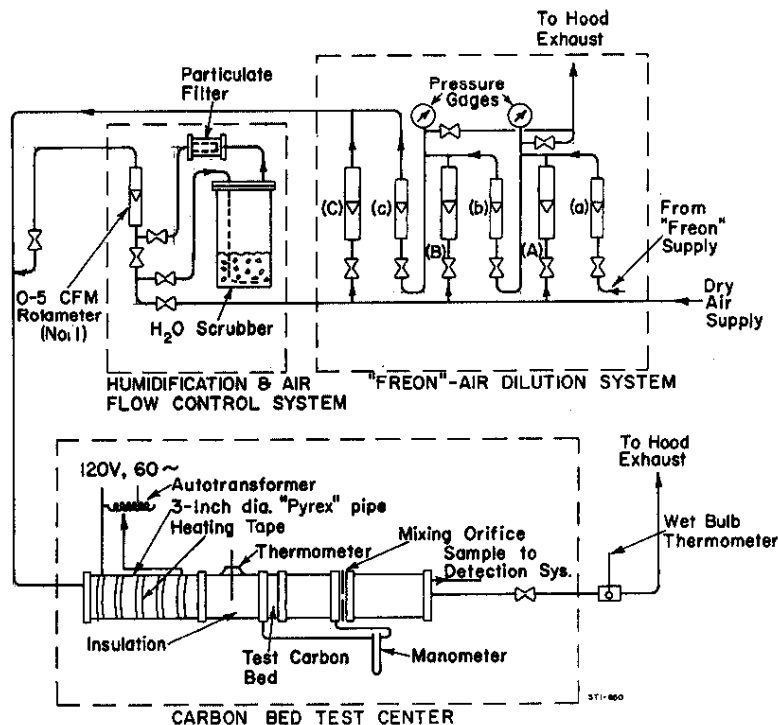


FIG. 1 DIAGRAM OF SMALL-SCALE FACILITY FOR EVALUATION OF "FREON" ADSORPTION CHARACTERISTICS

Carbon Bed Test Center

The test center consists of 3-inch-diameter "Pyrex"* pipe. Small-scale mockups of a section of the carbon bed from a full-size unit⁽³⁾ are installed between two flanges of the pipe. The test beds, shown in Figure 2, are 1 inch thick and are packed with activated carbon made from coconut shells; the physical and chemical properties of the carbon are discussed in DP-778⁽¹⁾. Supply air for the facility is preheated, if required, to the desired operating temperature (from 25 to 100°C) by an electrical resistance heater wrapped on the "Pyrex" pipe upstream of the test bed. A mixing orifice is located downstream of the bed to prevent possible channeling of "Freon" along the walls of the pipe. The downstream air, after sampling for "Freon", is exhausted to a hood and removed from the building.

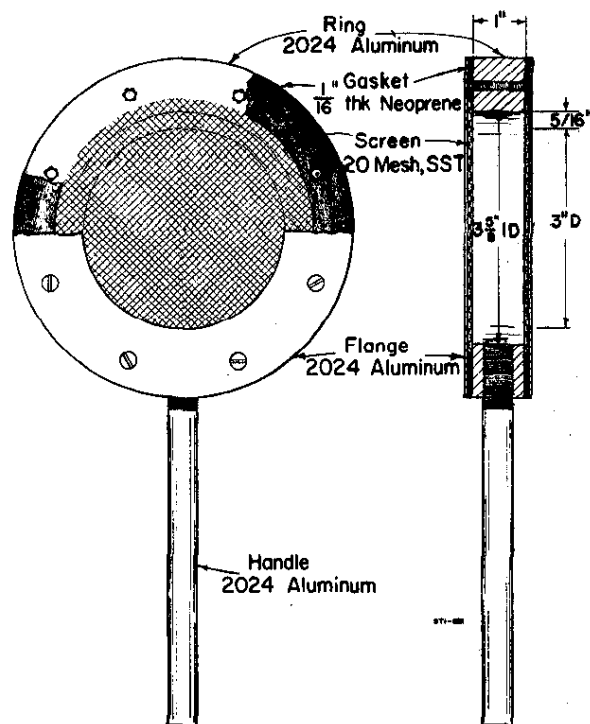


FIG. 2 SMALL-SCALE CARBON BED

Humidification and Air Flow Control System

Activated carbon adsorbs H_2O vapor from air in proportion to the relative humidity (R.H.). When exposed to air at 75% R.H., activated carbon adsorbs about 20%, by weight, H_2O at

* Trademark of Corning Glass Works.

equilibrium conditions. Previous tests have shown that the H_2O content of the carbon has a significant effect on the adsorption characteristics of "Freon-12"⁽⁷⁾.

The Humidification and Air Flow Control System was designed to supply either dry or humid air to the test facility for evaluation of the effects of H_2O content of the carbon on the adsorption characteristics of candidate "Freon" compounds. When dry air is required, the supply air is passed directly to the test center (through rotameter No. 1, as shown in Figure 1). The R.H. of the supply air is 10% and is considered dry for test purposes. When humid air is required, part of the air is passed through a H_2O scrubber to saturate the air with H_2O . The R.H. of the main air stream is controlled by the fraction of air that is passed through the scrubber. The effluent from the scrubber is passed through a particulate filter to remove any entrained H_2O particles. At an air velocity of 70 ft/min upstream of the carbon bed, air with a R.H. up to 55% can be supplied to the test facility. The test carbon bed is exposed to test conditions until the H_2O content of the carbon is in equilibrium with the R.H. of the air.

"Freon"-Air Dilution System

This system is used to calibrate the electron capture type instrument⁽⁷⁾ for the candidate "Freon" compounds. The system has three stages of dilution (Figure 1) with two rotameters in each stage. Each stage has a maximum dilution factor of about 1000. Rotameters labeled with lower-case letters in Figure 1 are calibrated for a minimum flow of 15 cc/min; those labeled with capital letters are calibrated for a maximum flow of 15,000 cc/min. Concentrations of "Freon" in air from ~0.001 to 0.5 ppm (by volume) can be prepared with the system; the maximum error in the "Freon" concentration is $\pm 15\%$.

"Freons" with boiling points (b.p.) $< 10^\circ C$ are superheated gases at normal test conditions (air at temperatures of $25^\circ C$ and at pressures of 20 psia) and are supplied to the test facility in a gaseous state through a calibrated rotameter. Samples of "Freons" that are normally in a liquid state are prepared by vaporizing a measured quantity of the "Freon" into a cylinder containing a known volume of air; the unsaturated "Freon"-air mixture is then supplied to the test facility as described above. The physical properties of the "Freons" considered in the test program are summarized in Table I.

Physical Properties^(a) of Selected "Freons" Considered for Improved Adsorption Characteristics

	"F-12"	"F-C318"	"F-114"	"F-21"	"F-11"	"F-114B2"	"F-113"	"F-112"
Chemical formula	CCl ₂ F ₂	C ₄ F ₈ (cyclic)	CClF ₂ - CClF ₂	CHCl ₂ F	CCl ₃ F	CBrF ₂ - CBrF ₂	CCl ₂ F- CClF ₂	CCl ₂ F- CCl ₂ F
Molecular weight	120.9	200.0	170.9	102.9	137.4	259.9	187.4	203.9
Boiling point at 1 atm, °C	-29.8	-5.8	3.6	8.9	23.8	47.3	47.6	92.8
Freezing point, °C	-158	-41.4	-94	-135	-111	-110.5	-35	23.8
Critical temperature, °C	112	115.3	145.7	178.5	198	214.5	214.1	278
Critical pressure, atm	40.6	27.5	32.1	51	43.2	34.0	33.7	34
Critical volume, cc/mol	217	323	293	197	247	329	325	370
Critical density, g/cc	0.558	0.620	0.582	0.522	0.554	0.790	0.576	0.55
Density, liquid at 30°C, g/cc	1.292	1.480	1.440	1.354	1.464	2.148	1.553	1.634
Density, sat. vapor at b.p., g/l	6.33	9.62	7.82	4.57	5.85	-	7.38	7.02
Solubility of "Freon" in water at 1 atm and 25°C, wt %	0.028	-	0.013	0.95	0.11	-	0.017 ^(a)	0.012 ^(a)
Toxicity ^(b)	Group 6	Probably Group 6	Group 6	<Group 4 >Group 5	Group 5A	<Group 4 >Group 5	<Group 4 >Group 5	<Group 4 >Group 5

(a) Saturation pressure.

(b) Underwriters' Laboratories Classification of Comparative Life Hazard of Gases and Vapors (Group 1 is most toxic and Group 6 is least toxic). See Table III of Reference 8.

Test Development Facility for Full - Size Iodine Adsorbers

Design of a facility for the development of a standardized nondestructive test of full-size carbon beds was completed. A flow diagram of the facility is shown in Figure 3. The facility will be used for detailed evaluations of the adsorption and desorption characteristics of the most promising "Freons" on SRP production carbon beds (over-all dimensions: 24 x 24 x 12 inches deep), as well as beds of different designs, and for standardization of techniques under the following test conditions:

- Air flow 100 to 2000 cfm
- Air temperature 15 to 120°C
- Relative humidity 10 to 95%

The facility was also designed for testing full-size beds with stable iodine vapor (^{127}I) so that correlations could be developed between "Freon" and ^{127}I penetration of carbon beds. ^{127}I will be detected with the same instrument used for detecting "Freons" - the electron capture detector; the sensitivity of the detector for ^{127}I is estimated to be between 0.001 and 0.01 ppm.

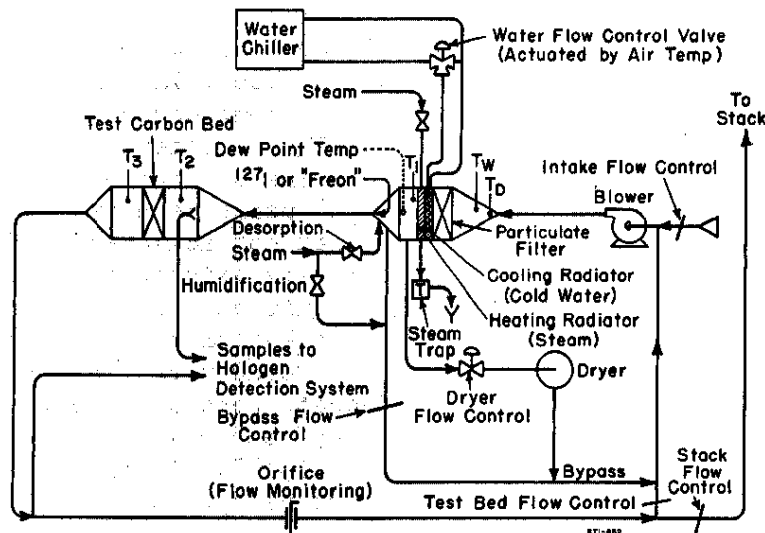


FIG. 3 FLOW DIAGRAM OF TEST DEVELOPMENT FACILITY FOR FULL-SIZE IODINE ADSORBERS

The facility was designed for total recirculation of conditioned air to reduce the refrigeration and drying requirements. Once-through operation was also provided to desorb "Freon" from test carbon beds and for purging of the system. The facility is composed principally of three systems: (1) the air flow system, (2) the air conditioning system (both temperature and humidity control), and (3) the "Freon" tracer system. A more detailed description of the facility is given in the Appendix.

RESULTS

"Freon" Detector Response

Determination of the response of the electron capture detector and chromatograph for the test "Freons" was started. The instrument and its operating details were discussed in DP-870. When sample concentrations of either "F-114" or "F-11" in air were injected into the instrument, the existing chromatograph column produced adequate resolution of O_2 and the test "Freon". The column was operated at $75^{\circ}C$ for "F-114" and at $120^{\circ}C$ for "F-11" as compared to $50^{\circ}C$ for "F-12"⁽⁷⁾. Higher column operating temperatures were required to elute more rapidly "Freons" with higher b.p. and to produce sharper peaks on the chromatogram. The decreased time for analysis permitted more rapid sampling, and the sharper peak increased the sensitivity of detection. Preliminary results indicate that the higher column temperature may shorten the life of

the column because of partial elution of the column stationary phase (SF-96*). However, the higher temperature reduced the drift in the base-line signal due to accumulation of small quantities of O_2 that was not eluted completely from the column when operated at $50^\circ C$ ⁽⁷⁾. The efficiency of the column was satisfactory for separating O_2 from either "F-114" or "F-11"; moderately sharp "Freon" peaks were recorded. Although the optimum sensitivity was not achieved with the present column, sufficient sensitivity existed for evaluation of the "Freons". Refinements in equipment and technique will not be started until after final selection of the best tracer compound. The existing column was found unsuited for "F-C318", because it failed to provide adequate separation between the O_2 and "F-C318". This lack of peak resolution prevented calibration of the detector for "F-C318". Other chromatograph columns were ordered to provide adequate separation of "F-C318".

Calibrations of the electron capture cell for "F-114" and "F-11" are shown in Figures 4 and 5. For comparison, the calibration curve for "F-12" is shown in Figure 6. A comparison of the calibration curves reveals that the minimum sensitivity for detection of the "Freons" in air with the

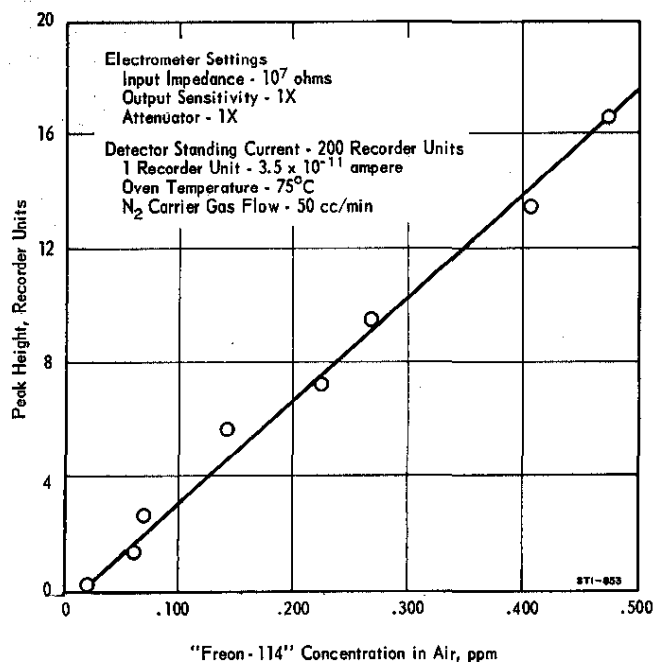


FIG. 4 "FREON-114" CALIBRATION OF ELECTRON CAPTURE DETECTOR

* 30% SF-96 on 45/60 mesh "Chromosorb" P made by Johns-Manville Company.

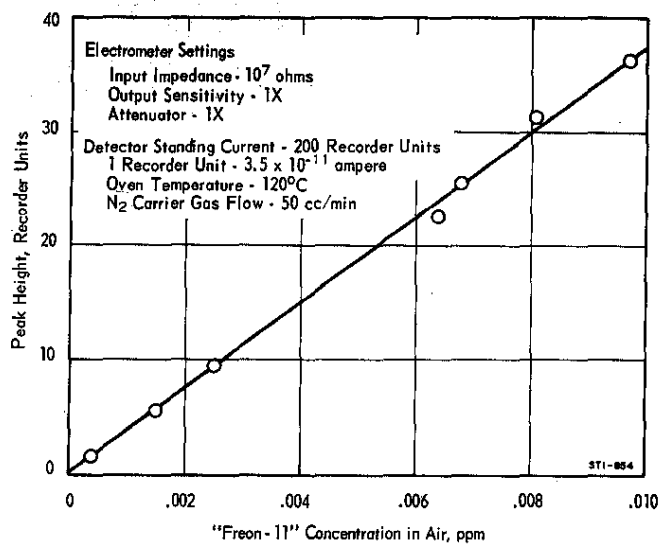


FIG. 5 "FREON-11" CALIBRATION OF ELECTRON CAPTURE DETECTOR

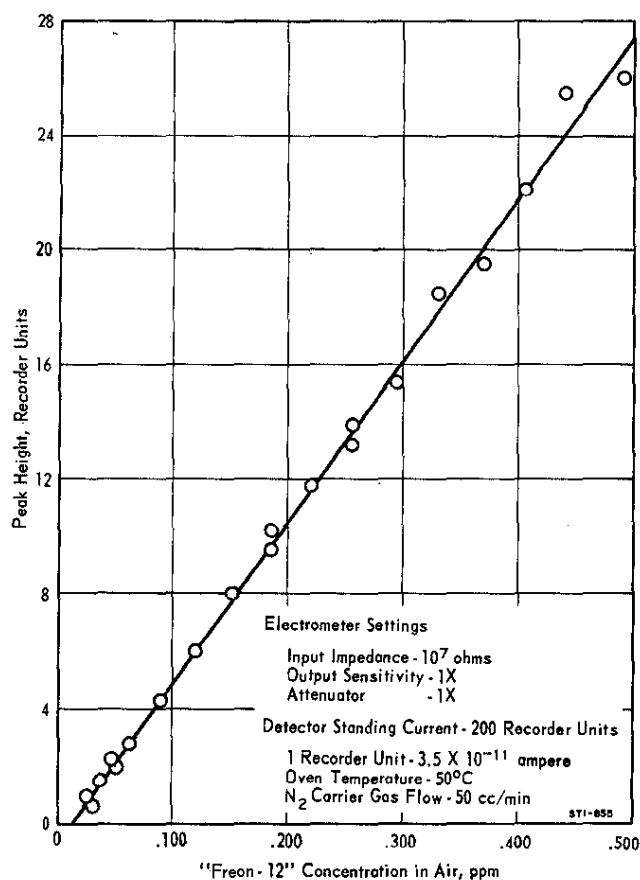


FIG. 6 "FREON-12" CALIBRATION OF ELECTRON CAPTURE DETECTOR

cell was ~ 0.030 ppm for "F-114" and "F-12" and < 0.001 ppm for "F-11". The minimum detectable "F-11" concentration was estimated to be ~ 0.0003 ppm. The increased sensitivity for "F-11" was expected, because the sensitivity of the detector for halogens generally increases with both atomic weight and increasing number of halogen atoms in the compound. The effect of atomic weight is large. This increased sensitivity for detection of "F-11" is significant. The upstream concentration of "F-11" in a test of a carbon bed is reduced by a factor of 100 below that used in the "F-12" test (5 ppm instead of 500 ppm), and the same capability to detect a leak path is maintained. The reduced inlet concentration of "F-11" would also reduce the total loading of "F-11" on the carbon and therefore reduce the time required to desorb the "Freon" with higher boiling points from the carbon in applications requiring frequent and repetitive tests.

Adsorption of "Freons" on Activated Carbon

Adsorption tests were started with "F-114" and "F-11" on small-scale carbon beds in the small-scale facility (Figure 1). The first series of tests was conducted with dry carbon and with air flowing at 25°C and at a velocity of 20 ft/min. Typical curves of the adsorption efficiency of "F-12", "F-114", and "F-11" under these conditions are shown in Figure 7. The adsorption efficiency (1) decreased to 99.90% for "F-12" in 10 minutes (Curve A), (2) decreased to 99.90% for "F-114" in 38 minutes (Curve B), and (3) was still in excess of 99.99% for "F-11" after 27 minutes when the test was stopped (Curve C). The tests were conducted with an inlet "Freon" concentration of 500 ppm for "F-12" and "F-114" and with 5 ppm for "F-11". The effect of inlet concentration on the adsorption of "Freon" on carbon has not been determined as yet. No significant effect is expected with dry carbon during the initial stages of adsorption, because mass transfer is probably the controlling mechanism.

The second series of tests was conducted with "F-11" at higher air velocities and with carbon containing a greater amount of H_2O . In Figure 8, the adsorption efficiency for "F-11" at three test conditions (Curves A, B, and C) is compared with "F-12" at similar conditions (Curve D). At an air velocity of 70 ft/min and after 5 minutes of exposure to "F-11" at an inlet concentration of 5 ppm, the adsorption efficiency was 99.99+% when the carbon was dry (Curve A) and 99.93% when the carbon contained 12.5% H_2O (Curve B). When the air velocity was reduced to 35 ft/min (Curve C), the "F-11" adsorption efficiency was 99.99+% after 5 minutes and with carbon containing 12.5% H_2O . The "F-12" adsorption

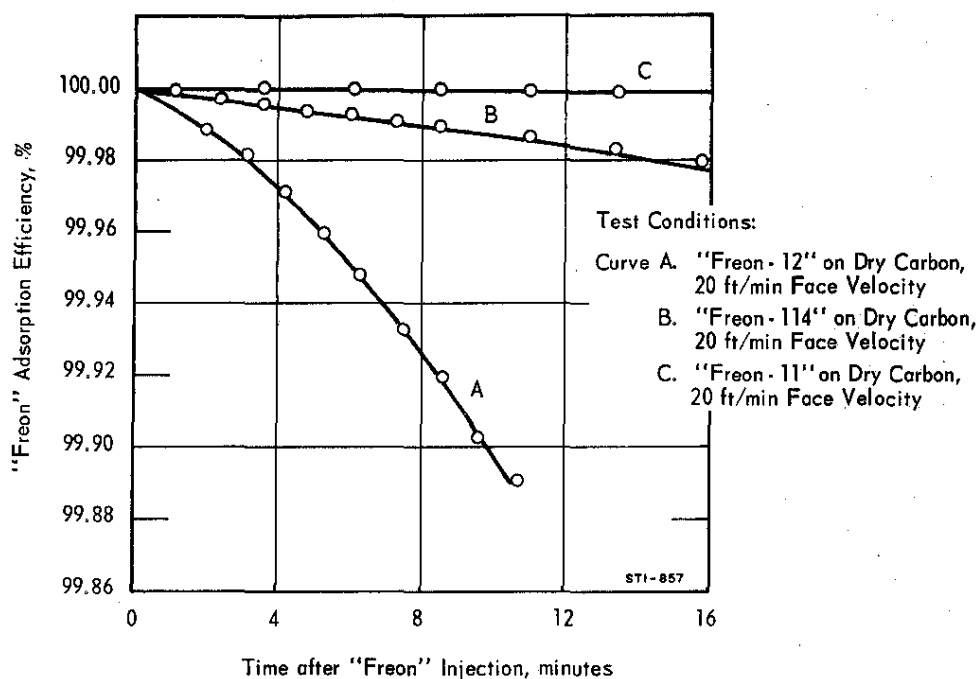


FIG. 7 ADSORPTION EFFICIENCY OF "FREONS" ON DRY CARBON

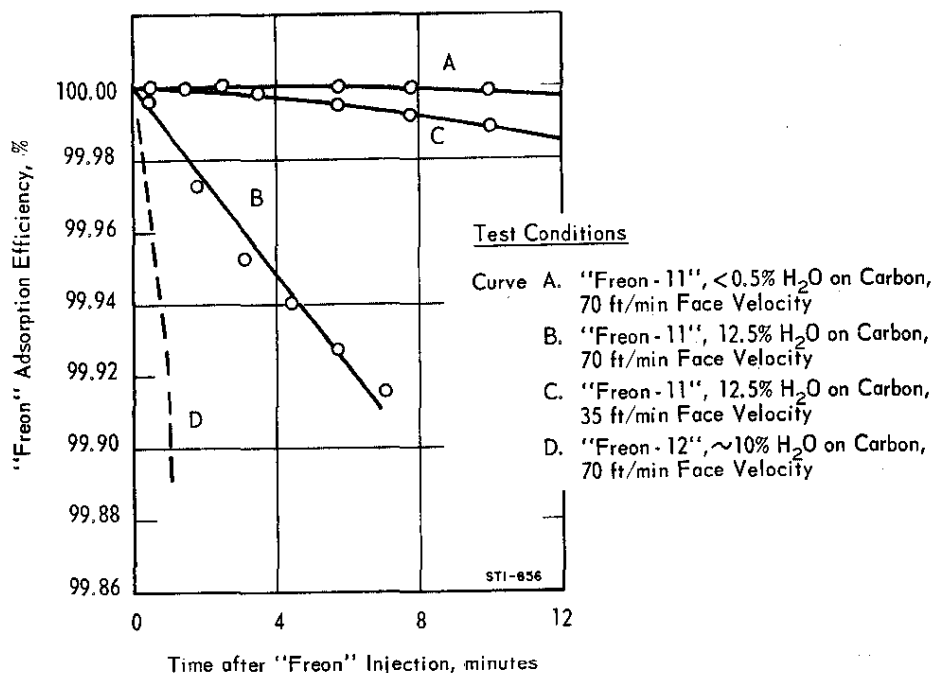


FIG. 8 EFFECT OF INCREASED FACE VELOCITY AND H₂O CONTENT OF CARBON ON "FREON-11" ADSORPTION EFFICIENCY

efficiency was 99.90% (Curve D) in ~1 minute when tested under similar conditions. All of the tests were conducted with air at a temperature of ~25°C. From these tests, it is apparent that "F-11" reduced significantly the limitations of velocity and H₂O content of the carbon on the existing "F-12" technique. Further tests of "F-11" with carbon containing greater quantities of adsorbed H₂O and tests of "F-114B2" (b.p. 47.5°C) are in progress.

A test with "F-11" also showed that a satisfactory repetitive test of a carbon bed can be made after the "F-11" adsorbed on the carbon during the initial test is partially desorbed. The second test was made on the same carbon bed and under the same test conditions (70 ft/min face velocity and 12.5% H₂O content of carbon). After the first "F-11" test, the carbon was exposed for 4 days to ambient air flowing at 70 ft/min and with a relative humidity of ~50%. Over 95% of the "F-11" was desorbed, and the residual concentration of "F-11" in the air downstream of the bed was about 1 ppb at the end of the desorption period. The adsorption efficiency of the carbon bed for "F-11" in the second test was almost identical to that measured in the first test when corrected for the residual "F-11" concentration.

APPENDIX - DESCRIPTION OF TEST DEVELOPMENT FACILITY FOR FULL-SIZE IODINE ADSORBERS

Air Flow System

The air flow system is composed of a blower, duct, and butterfly valves for control of air flows from 100 to 2000 cfm. The blower is rated for a maximum air flow of 2000 cfm at a total developed head of 12 inches H_2O , gage. A flow diagram of the test facility is shown in Figure 3. Module No. 1 contains air conditioning units, described below, and Module No. 2 contains the test carbon bed. Module No. 2 was designed for testing SRP production carbon beds^(s) (over-all dimensions: 24 x 24 x 12 inches deep), but it can be readily modified for testing other beds of different design. The facility will be constructed of stainless steel sheet metal to prevent significant adsorption of high boiling point "Freons" and other tracer materials, such as ^{127}I vapor, on the duct work.

The system is normally operated on total recirculation by closing the stack and intake flow-control valves and by adjusting the bypass and test bed flow-control valves to produce the desired flow through the carbon bed. Flow through the bypass is required only when the test flow is less than that required to prevent surging of the blower (<500 cfm). To operate the system with once-through flow, the above position of the butterfly valves is reversed, and atmospheric air can be supplied continuously to the system for desorption of "Freon" from test carbon beds. When the system is operated with once-through flow, the air is exhausted to a stack. The air flow will be measured by a calibrated orifice located downstream of the test carbon bed.

Air Conditioning System

The air temperature and humidity are controlled in Module No. 1 (Figure 3), which contains a particulate filter, cooling coil, and heating coil.

The particulate filter is used to remove airborne particles from the supply air. Atmospheric dust in the air could adsorb "Freon" or ^{127}I and invalidate the test. The efficiency of the filter is 99.97+% for removal of particles 0.3 micron in diameter.

Cold H₂O is supplied to the cooling coil at constant temperature from a H₂O-chiller refrigeration system. The refrigeration system has a minimum capacity of 30,000 Btu/hr. Automatic control of the air temperature is provided only within the normal test temperature range of 15 to 50°C. Control of the air temperature within this range is accomplished by regulating the cold H₂O flow to the cooling coils. At equilibrium conditions, the cooling coil is required to remove only the heat input from the blower plus the heat gain to the system. Automatic temperature control is provided by a proportional controller with an adjustable set point to regulate the cold H₂O flow to the cooling coils.

Saturated steam at 150 psig is supplied to the heating coil to heat the air up to 120°C and at flows up to 2000 cfm when the facility is operated with once-through flow for desorption of "Freon". Because accurate control of the temperature is not required, the steam flow is regulated by manual adjustment of a throttling valve. Steam can also be supplied directly to the facility to produce steam-air mixtures for rapid desorption of "Freon".

Control of the R.H. of the air is accomplished by passing a side stream of air, discharged from Module No. 1, through a dryer. The dryer consists of a packed bed of molecular sieve material (Type 4A) that adsorbs H₂O vapor from the air. The dryer was designed to reduce the R.H. in the system air to less than 10% at 15°C, and it can be operated under the maximum drying requirements for 8 hours before regeneration of the molecular sieve is required. Regeneration is accomplished by heating the bed to ~150°C and purging with air to remove adsorbed H₂O from the molecular sieve. Control of the R.H. of the system air is accomplished by manual adjustment of the air flow through the dryer. The R.H. is determined by a dew point temperature indicator located in Module No. 1. When the required dew point temperature is reached, the valve is closed. If humidification of the air is required, a small quantity of steam is injected into the air stream in the suction side of the blower. The blower promotes mixing, and the particulate filter in Module No. 1 removes any entrained H₂O droplets so that only humidified air is supplied to the test carbon bed.

"Freon" Detection System

The "Freon" detection system is used to determine the adsorption efficiency of a test carbon bed. "Freon" is injected at a known rate upstream of a test carbon bed, and the "Freon" concentration downstream of the bed is measured with an electron-capture-type instrument. The procedure is identical to that described in DP-870. A "Freon"-Air Dilution System identical to that discussed previously in this report will be used for injecting "Freon" into the facility at concentrations from ~0.001 to 1 ppm for calibration of the "Freon" detector and from 1 to 500 ppm for adsorption tests of carbon beds.

The sampling system is constantly purged at a high flow rate to reduce the residence time of the "Freon" sample in the tubing lines. A dilution system is also provided in the sampling system to permit determination of the "Freon" concentration when it exceeds the calibrated range of the detector; this is accomplished by diluting the mixture with a known quantity of air.

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