

664526 88  
DP - 920

AEC RESEARCH AND DEVELOPMENT REPORT

# NONDESTRUCTIVE TEST OF CARBON BEDS FOR REACTOR CONTAINMENT APPLICATIONS

PROGRESS REPORT  
APRIL - JUNE 1964

SRL  
RECORD COPY



*Savannah River Laboratory*  
*Aiken, South Carolina*

## LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

Printed in USA. Price \$1.00

Available from the Clearinghouse for Federal Scientific  
and Technical Information, National Bureau of Standards,  
U. S. Department of Commerce, Springfield, Virginia

664526  
DP-920

Engineering and Equipment  
(TID-4500, 34th Ed.)

NONDESTRUCTIVE TEST OF CARBON BEDS  
FOR REACTOR CONTAINMENT APPLICATIONS

PROGRESS REPORT: APRIL - JUNE 1964

by

David R. Muhlbaier

Approved by

E. C. Nelson, Research Manager  
Pile Engineering Division

Issue Date: November 1964

E. I. DU PONT DE NEMOURS & COMPANY  
SAVANNAH RIVER LABORATORY  
AIKEN, SOUTH CAROLINA

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

### ABSTRACT

A standardized nondestructive and more generally applicable test for installed carbon beds that are used in reactor containment facilities is being developed by the Savannah River Laboratory. Continuing tests show that the permissible ranges of air velocity and  $H_2O$  content of the carbon are increased significantly by substituting "F-11" or "F-114B2" for "F-12" - a more volatile "Freon" that was used in an earlier testing method.

## NONDESTRUCTIVE TEST OF CARBON BEDS FOR REACTOR CONTAINMENT APPLICATIONS

PROGRESS REPORT: APRIL - JUNE 1964

### INTRODUCTION

Radioactive particles and halogen vapors (principally  $^{131}\text{I}$ ) 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<sup>(1-3)</sup>. Standardized tests are available for evaluating the particulate filters (dioctyl-phthalate-aerosol penetration tests<sup>(4-6)</sup>). 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<sup>(7)</sup>.

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  $\text{H}_2\text{O}$ . In actual plant installations, air velocities up to 70 ft/min and relative humidities up to 75% (~25%  $\text{H}_2\text{O}$  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 about 1 minute when exposed at these maximum plant conditions. About 5 minutes is required to evaluate the performance of installed carbon beds.

Because of the above limitations, work was undertaken to develop a standardized nondestructive test that is more generally applicable for installed carbon beds. As discussed in progress report DP-910, the limitations imposed on air velocity and adsorbed  $\text{H}_2\text{O}$  were reduced significantly by the use of "F-11" ( $\text{CCl}_3\text{F}$ ) which is less volatile than "F-12" ( $\text{CCl}_2\text{F}_2$ ). 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 trademark for its fluorinated hydrocarbons.

## SUMMARY

The adsorption characteristics of "F-114B2" ( $\text{CBrF}_2\text{-CBrF}_2$ , b.p.  $47.3^\circ\text{C}$ ) on activated carbon were shown to be significantly better than those of "F-11" (b.p.  $23.8^\circ\text{C}$ ). When carbon beds containing ~20% adsorbed  $\text{H}_2\text{O}$  were exposed to air at  $\sim 30^\circ\text{C}$  flowing at 70 ft/min, the adsorption efficiency for "F-114B2" was 99.93+% after 5 minutes of exposure. The adsorption efficiency for "F-11" was 99.89+% when evaluated at the same conditions with carbon containing only ~13%  $\text{H}_2\text{O}$ .

The efficiency for adsorption of "F-114B2" on carbon decreases rapidly at  $\text{H}_2\text{O}$  content above 20%. Moderate decreases in air velocity increase the time required to reduce the adsorption efficiency of "F-114B2" to 99.90%.

The minimum sensitivity for detection of "F-114B2" in air with an electron-capture-type instrument was 0.0005 ppm; it was 0.0003 ppm for "F-11". All tests were made with an inlet "Freon" concentration of 5 ppm.

The adsorption efficiency of "F-11" on carbon decreased rapidly at air temperatures above  $30^\circ\text{C}$  and when evaluated at an air velocity of 70 ft/min with carbon containing ~13% adsorbed  $\text{H}_2\text{O}$ .

Tests are in progress with "F-112" ( $\text{CCl}_2\text{F-CCl}_2\text{F}$ , b.p.  $92.8^\circ\text{C}$ ). Construction was started on a facility for "Freon" testing of large carbon beds. Tests with "F-21" ( $\text{CHCl}_2\text{F}$ , b.p.  $8.9^\circ\text{C}$ ) were terminated because of poor sensitivity of detection.

## DISCUSSION

### TEST RESULTS

Adsorption tests of "Freon" compounds that are less volatile than "F-12" (b.p.  $-29.8^{\circ}\text{C}$ ) were continued in the small-scale apparatus described in DP-910. New activated carbon beds (3 inches in diameter and 1 inch thick) containing up to  $\sim 22\%$  adsorbed  $\text{H}_2\text{O}$  were exposed to air velocities to 70 ft/min and temperatures to  $33^{\circ}\text{C}$ . All test beds were prepared in a similar manner to eliminate the bed characteristics as a variable. Results for "F-11" ( $\text{CCl}_3\text{F}$ , b.p.  $23.8^{\circ}\text{C}$ ), "F-21" ( $\text{CHCl}_2\text{F}$ , b.p.  $8.9^{\circ}\text{C}$ ), and "F-114B2" ( $\text{CBrF}_2\text{-CBrF}_2$ , b.p.  $47.3^{\circ}\text{C}$ ) are discussed below.

#### "Freon-11"

Previous tests (DP-910) showed that "F-11" could be used as a tracer at air velocities to 70 ft/min with carbon containing up to 12.5% adsorbed  $\text{H}_2\text{O}$ . New tests showed that air temperature has a significant effect on the efficiency for adsorption of "F-11" when tested at the limiting velocity and  $\text{H}_2\text{O}$  content.

Figure 1 shows the "F-11" adsorption efficiency of three carbon beds tested at 70 ft/min,  $\text{H}_2\text{O}$  content of  $\sim 12.5\%$ , and temperatures of 20.5 to  $33^{\circ}\text{C}$ . As the temperature increases, the adsorption efficiency at a given time decreases, but at temperatures above  $\sim 30^{\circ}\text{C}$ , the adsorption efficiency decreases rapidly. This effect is also shown

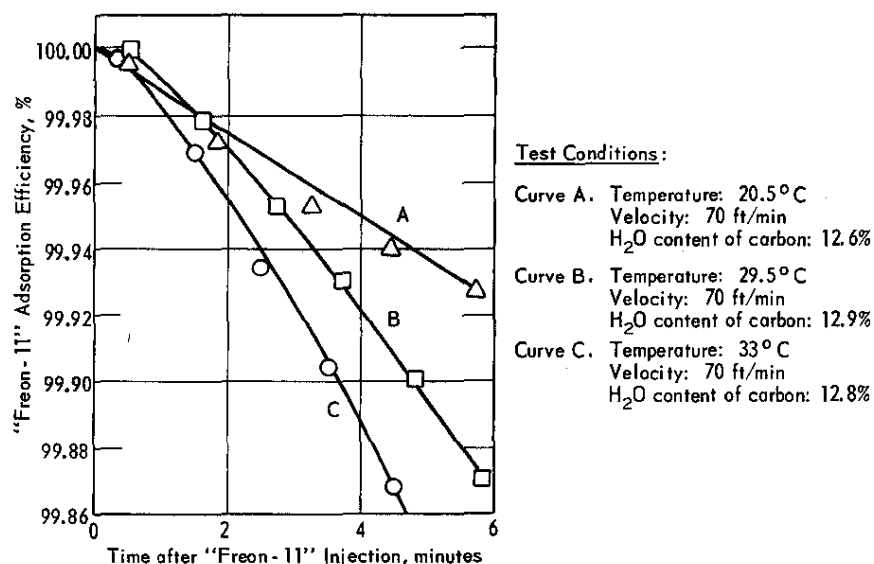


FIG. 1 "FREON-11" ADSORPTION EFFICIENCY

in Figure 2, in which temperature is plotted against the "F-11" adsorption efficiency 4 minutes after injection of "F-11" is started. No further work is planned with "F-11" because of the more promising results obtained with less volatile "Freons".

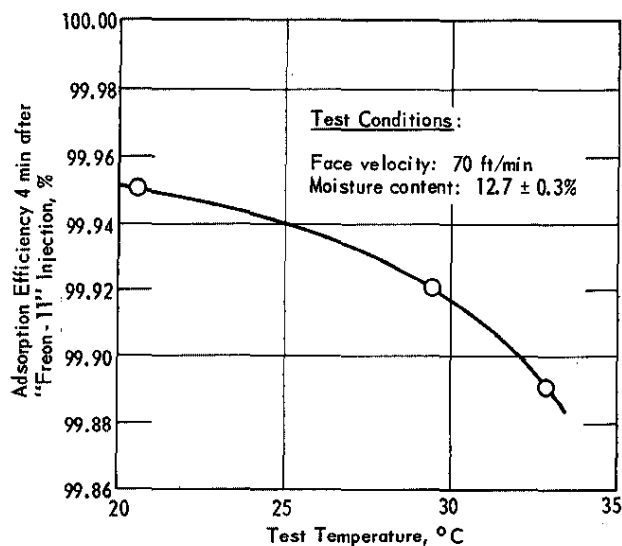


FIG. 2 EFFECT OF TEMPERATURE ON "FREON-11" ADSORPTION EFFICIENCY

#### "Freon-21"

Preliminary tests of "F-21" showed that the minimum detectable "F-21" concentration in air was ~1 ppm, which is relatively insensitive when compared to that of "F-11" (0.0003 ppm). No further tests are planned with "F-21" because of this decreased sensitivity and the large volumes of "F-21" that would be required for in-place testing of large carbon bed installations.

#### "Freon-114B2"

The adsorption characteristics of "F-114B2" on activated carbon were significantly better than those of "F-11". Satisfactory tests were obtained with "F-114B2" when carbon beds containing up to ~20% adsorbed water were exposed to air at ~30°C flowing at 70 ft/min. The improved adsorption characteristics are probably due to the higher boiling point of "F-114B2" (47.3°C as compared to 23.8°C for "F-11"). The previous maximum H<sub>2</sub>O content of carbon for an acceptable test was ~12.5% with "F-11".



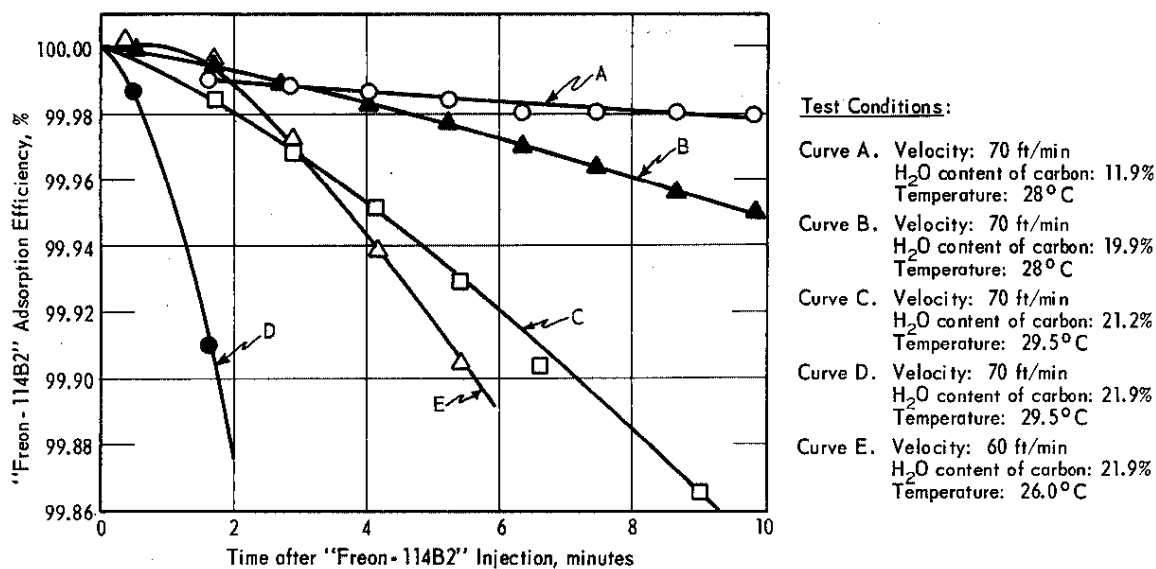


FIG. 3 "FREON-114B2" ADSORPTION EFFICIENCY

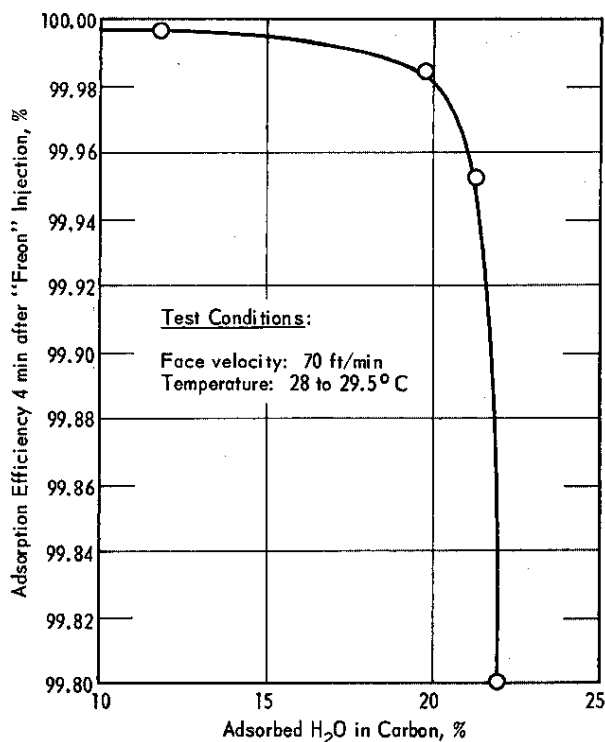


FIG. 4 EFFECT OF ADSORBED H<sub>2</sub>O ON "FREON-114B2" ADSORPTION EFFICIENCY

Figure 3 shows the results of four tests (Curves A, B, C, and D) on carbon beds tested with air at ~30°C flowing at 70 ft/min with carbon containing from 11.9 to 21.9% adsorbed H<sub>2</sub>O. Acceptable tests were obtained with carbon containing up to ~20% adsorbed H<sub>2</sub>O. Above ~20% adsorbed H<sub>2</sub>O, a slight increase in the H<sub>2</sub>O content reduced significantly the adsorption efficiency for "F-114B2". The effect of H<sub>2</sub>O content is shown more clearly in Figure 4 where H<sub>2</sub>O content is plotted against adsorption efficiency 4 minutes after the injection of "F-114B2" is started.

The limit on H<sub>2</sub>O content was extended slightly by decreasing the face velocity. When carbon containing ~22% adsorbed H<sub>2</sub>O was tested at a velocity of 60 ft/min, the "F-114B2" efficiency decreased to 99.90% in 5.5 min (Curve E, Figure 3) as compared to ~2 min when tested at 70 ft/min with the same H<sub>2</sub>O content (Curve D, Figure 3).

Calibration of the electron capture detector (DP-870) showed that the minimum detectable concentration of "F-114B2" in air is ~0.0005 ppm (Figure 5), which is comparable to that of "F-11". "F-114B2" was injected at the same upstream concentration (5 ppm) that was used in the "F-11" tests. Lower inlet concentrations of "F-11" and "F-114B2" reduce the carbon bed loading when compared with "F-12" (500 ppm injected upstream).

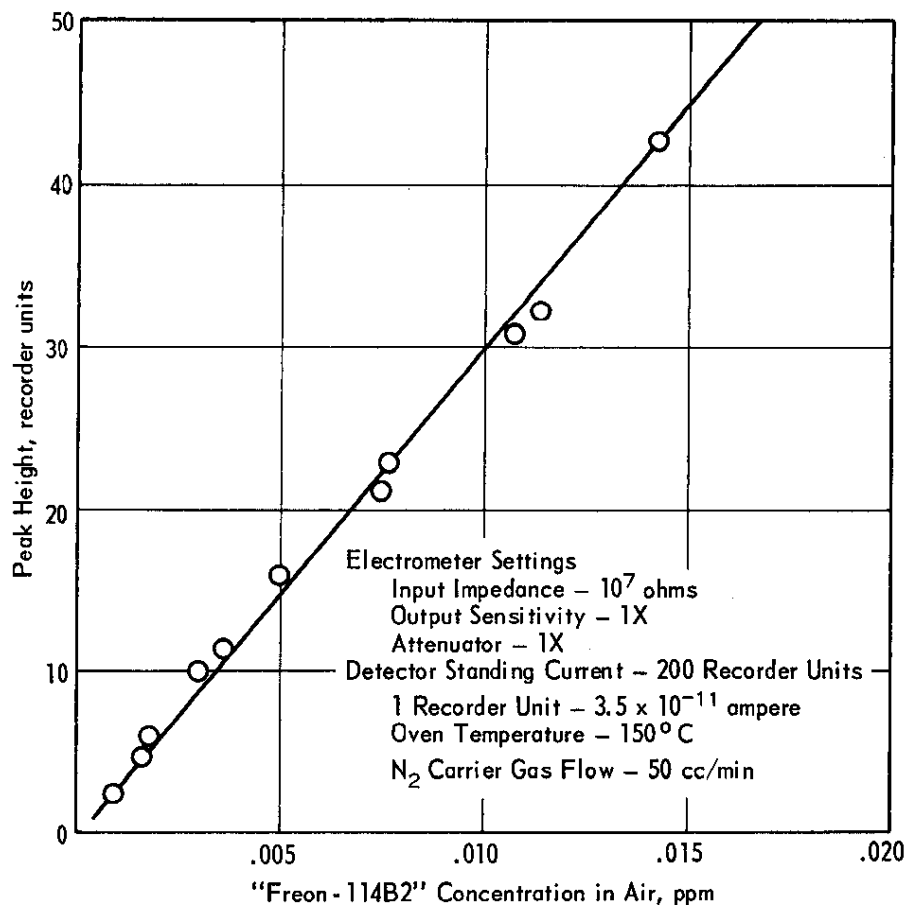


FIG. 5 "FREON-114B2" CALIBRATION OF ELECTRON CAPTURE DETECTOR

Chromatograph columns used to separate "F-114B2" from O<sub>2</sub> in air samples were the same as was used previously. They consist of a stainless steel tube (10 feet long and 3/32 inch in ID) packed with 30% SF-96 on 45/60 "Chromosorb"\* P. All previously examined "Freons" required 1 minute to complete the separation; "F-114B2" required ~1-1/4 minutes. No attempt was made to improve the separation time, but it is believed the time can be reduced by either increasing the N<sub>2</sub> flow rate, increasing the oven temperature, decreasing the column length, or decreasing the per cent of SF-96 on the column.

#### TEST DEVELOPMENT FACILITY FOR FULL-SIZE IODINE ADSORBERS

Construction was started on the facility described in DP-910 to develop a standardized nondestructive test for full-size iodine adsorbers. It was ~30% complete as of June 30, 1964.

---

\* "Chromosorb" is a registered trademark of Johns-Manville Company.

## REFERENCES

1. G. H. Prigge. Application of Activated Carbon in Reactor Containment. USAEC Report DP-778, E. I. du Pont de Nemours & Co., Savannah River Laboratory, Aiken, S. C. (1962).
2. A. H. Peters. Application of Moisture Separators and Particulate Filters in Reactor Containment. USAEC Report DP-812, E. I. du Pont de Nemours & Co., Savannah River Laboratory, Aiken, S. C. (1962).
3. J. W. Walker and A. H. Peters. "Filters for Reactor Containment." Mech. Eng. 85, 46-54 (1963).
4. MIL-STD-282, Filter Units, Protective Clothing, Gas-Mask Components and Related Products: Performance - Test Methods, United States of America War Office, Military Standard (28 May 1956).
5. E. L. Etheridge. Field Leak Testing of HAPC Reactor Confinement Filter Installations. USAEC Report HW-71083, General Electric Co., Hanford Atomic Products Operation, Richland, Wash. (1961).
6. H. W. Knudson and Locke White. Development of Smoke Penetration Meters. U. S. Naval Research Laboratory Report P-2642 (1945).
7. A. H. Peters and D. R. Muhlbaier. Nondestructive Test of Carbon Beds for Reactor Containment Applications - Progress Report: June 1962 - December 1963. USAEC Report DP-870, E. I. du Pont de Nemours & Co., Savannah River Laboratory, Aiken, S. C. (1964).
8. D. R. Muhlbaier. Nondestructive Test of Carbon Beds for Reactor Containment Applications - Progress Report: January-March 1964. USAEC Report DP-910, E. I. du Pont de Nemours & Co., Savannah River Laboratory, Aiken, S. C. (1964).