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AEC RESEARCH AND DEVELOPMENT REPORT

STANDARDIZED NONDESTRUCTIVE TEST OF CARBON BEDS FOR REACTOR CONFINEMENT APPLICATIONS

FINAL PROGRESS REPORT
FEBRUARY TO JUNE 1966

D. R. MUHLBAIER

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FINAL PROGRESS REPORT: FEBRUARY TO JUNE 1966

by

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July 1967

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ABSTRACT

A standardized nondestructive in-place test for detecting leaks in installed carbon beds was developed and demonstrated by the Savannah River Laboratory. This work was previously reported in DP-1053(5), and the test was demonstrated recently at the Seventh and Eighth AEC In-Place Filter Testing Workshops*. This report presents detailed information and a correlating equation for general application of the test to carbon beds of different design.

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* Conducted at the Health and Safety Laboratory, New York Operations Office, Atomic Energy Commission.

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STANDARDIZED NONDESTRUCTIVE TEST OF CARBON BEDS FOR REACTOR CONFINEMENT APPLICATIONS

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INTRODUCTION

Halogen vapor (principally ^{131}I) that might be released accidentally into the building of Savannah River Plant (SRP) reactors would be removed by normal, routine passage of the exhaust ventilation air through carbon beds. New carbon beds are tested for absence of leaks by a nondestructive technique in which "Freon-12"* is used as a tracer. Development of this technique by the Savannah River Laboratory (SRL) is discussed in progress report DP-870(1). The "F-12" technique is limited to testing new carbon beds with air at a maximum velocity of 20 ft/min and with carbon containing no more than 5% sorbed H_2O. In addition, the test is unsatisfactory for carbon that has been in use, because adsorbed impurities cause the "F-12" to desorb too quickly (in less than one minute) for a definitive measurement of the leak path.

Because of these limitations, work was undertaken to develop a standardized nondestructive test generally applicable for in-place leak testing of installed carbon beds that have been in use. The limitations imposed on air velocity and sorbed H_2O were reduced significantly by the use of "F-112", a halogenated hydrocarbon less volatile than "F-12" (DP-910(2), 920(3), and 950(4)). The fundamentals and application of the test in the SRP confinement system are described in DP-1053(5). This final report discusses work (February-June 1966) for standardization of the in-place leak test and presents details for its application.

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

SUMMARY

An in-place leak test for installed carbon beds was developed by SRL and demonstrated in the SRP reactor confinement system(5). The "F-112" Leak Test is now used routinely by SRP personnel to measure any leakage flow in the SRP systems. The test was standardized for general application to carbon beds of different design, and a correlating equation was developed to predict satisfactory test conditions for new carbon beds packed with different carbons. The correlating equation predicts the effect of face velocity, carbon moisture content, particle size, bed thickness, and external surface area of particles. Information for both impregnated and unimpregnated carbons is presented. The correlating equation is also valuable in comparing the performance of different carbon beds.

The standardized test procedure is presented for general application of the test. Detailed drawings and troubleshooting aids are also presented for the construction and maintenance of the "F-112" Leak Test apparatus.

DISCUSSION

LEAK TEST STANDARDIZATION

The "F-112" Leak Test is designed to measure leakage flow through or around installed carbon beds. The fundamentals, application, and results of the test in the SRP confinement system are discussed in DP-1053.(5) The test has been standardized to permit measurement of leakage flow through carbon beds of different design in different systems by the use of a correlating equation (discussed below) and a standardized test procedure (see appendices).

Development of Correlating Equation

An equation was developed that predicts the required test conditions with "F-112" tracer for detecting leaks of $\geqslant 0.01\%$ in new carbon beds. The method employs a modification of the Dole and Klotz equation(6), which is based on external diffusion as the controlling mechanism for adsorption. Generally, three mechanisms are involved in physical adsorption: (1) interparticle diffusion (external diffusion) of the adsorbate from the gas stream to the immediate vicinity of the adsorbent particle, (2) transport of the adsorbate through pores in the adsorbent to the interior surfaces, and (3) deposition of the adsorbate molecules on the surface. Previous experiments showed that "F-112" adsorption by activated carbon was affected by the velocity of the air stream. Velocity affects only external diffusion; consequently, external diffusion must be a significant resistance in controlling the rate of "F-112" adsorption.

The value of the modified Reynolds number for the conditions studied (and conditions for most leak test applications) was generally less than 40 which represents laminar flow as predicted by Gamson, Thodos, and Hougen.(7) Thus, the correlating equation was developed for laminar flow as outlined by Dole and Klotz;(6) the mass transfer coefficient for laminar flow was used as given in Reference 7. The resulting equation is

$$\log \frac{C_b}{C_o} = - \frac{7.3ApX}{\left(\frac{D_pG}{\mu}\right)\left(\frac{\mu}{\rho D_v}\right)^{\frac{2}{3}}} \qquad (1)$$

where

$\frac{C_b}{C_o}$ = adsorbate concentration ("F-112" in this work) downstream of carbon bed divided by upstream concentration, in consistent units

A = effective outer surface area of carbon particles per unit weight of carbon, cm^2/g

p = average packing density, weight of carbon per unit volume of bed, g/cc

X = bed depth, cm

D_p = average particle diameter, cm

G = mass velocity of gas stream*, g/cm^2-sec

μ = absolute viscosity of gas stream*, g/cm-sec

ρ = density of gas stream*, g/cc

D_v = diffusion coefficient of vapor in gas stream*, cm^2/sec

A more rigorous treatment of the system equation for adsorption of a gas from a flowing gas stream is presented by Masamune and Smith.(8,9,10) However, the previous equation was used to develop the correlation, because it provided satisfactory results for the range of interest (initial adsorption) and is more useful for field application.

Equation 1 contains two dimensionless quantities; the Schmidt number $(\frac{\mu}{\rho D_v})$ and the modified Reynolds number $(\frac{D_p G}{\mu})$. The quantities in the Schmidt number were not varied significantly during the tests; less than 3% variation occurred in the Schmidt number (2.18), because the tests were conducted between 21 and 32°C and 0 to 85% relative humidity. The absolute viscosity (μ), which appears also in the modified Reynolds number, was not varied significantly. Other quantities (X, A, D_p, and G) in the equation were varied during the tests to evaluate their effect. The packing density (p) was varied only as required to maintain a pressure drop that was representative of firmly packed beds.

The effective outer surface area of carbon particles per unit weight of carbon (A) may be reduced if a significant quantity of material (such as H_2O) is adsorbed by the carbon. Consequently, A'y was substituted for A to allow for adsorption of gases on the surface of the carbon.

---

* "F-112" vapor in air for this work.

$$\log \frac{C_b}{C_o} = -c_1 \frac{A'ypX}{D_pG} \qquad (2)$$

where

$c_1 = \dfrac{7.3\mu}{\left(\dfrac{\mu}{\rho D_v}\right)^{\frac{2}{3}}}$

A' = maximum effective outer surface area of the carbon (i.e., no adsorbed gases on the carbon)

y = fractional free outer surface area of the carbon (i.e., the fraction of outer area that contains no adsorbed gases)

Equation 2 was developed on the basis that mass transfer is the controlling mechanism for irreversible adsorption. But desorption of "F-112" (which is physically adsorbed on the carbon) does occur at various rates depending on test conditions. However, the equation is still applicable, because desorption is of minor significance during the first few minutes of the "F-112" test (the time of interest for leak testing).

Analyses of the test results showed that the "F-112" penetration was relatively insensitive to changes in the average particle diameter (D_p) in the modified Reynolds number. Mass transfer across the boundary film was the major resistance to "F-112" adsorption. Apparently, D_p (in the range tested) did not affect significantly the properties of the boundary film. Therefore, Equation 2 was modified to include a mean value of D_p in the constant c_2:

$$\log \frac{C_b}{C_o} = -c_2 \frac{A'ypX}{G} \qquad (3)$$

where

$c_2 = \dfrac{c_1}{D_p}$

D_p = 0.18 cm (mean value investigated in this work)

Comparison of the test results for the small-scale carbon beds with previous results for full-size beds showed different "F-112" adsorption characteristics. The small-scale beds are flat with the faces perpendicular to the air flow; full-size SRP beds contain 12 flat sections located at an angle to the main air stream (Figure 4). The geometry of the beds can affect the local velocity profile inside the carbon bed. In full-size carbon beds, other differences such as variations in bed thickness and packing density may also occur. These differences can affect the performance of carbon beds. Thus Equation 3 was modified to include a shape factor (ϕ) to compensate for these differences. The final form of the correlating equation is

$$\log \frac{C_b}{C_o} = -c_2 \frac{A'ypX}{\phi G} = -c_2K \qquad (4)$$

where

ϕ = shape factor* (for differences in bed geometry and packing density)

$K = \frac{A'ypX}{\phi G}$ (correlating K-factor)

The average shape factor for a pilot order of new full-size SRP carbon beds with stainless steel frames was about 2.1. The shape factor for earlier model SRP carbon beds(11) built to nearly the same specifications but with carbon steel frames was about 1.35. The difference in shape factor was attributed to a lower packing density for the beds with stainless steel frames due to more flexure of the screens. As a result of these tests, the design of the beds with stainless steel frames was modified and the method of packing was improved(14). The shape factor for beds with stainless steel frames built to the new specifications was $\leqslant 1.4$. Thus, the correlating equation was a valuable tool in evaluating the adsorption characteristics of newly designed carbon beds.

Analysis of Equation 4 shows that when K equals zero, C_b/C_o equals 1 (100%). Hence, if the active area in the bed is zero, all the adsorbate will penetrate, which of course must be true. In addition, a plot of the equation on semilog graph paper (with C_b/C_o on the logarithmic scale) will result in a straight line.

The test data were correlated with Equation 4 for "F-112" penetration (C_b/C_o) of carbon beds 8 minutes after "Freon" entered the bed. Equation 4 is also applicable at shorter times, but a better fit of the data was obtained at 8 minutes. The longer time resulted in greater "F-112" penetration that could be measured more accurately. This was especially true for tests

---

* The shape factor for the small-scale flat beds used in this work is 0.90. As discussed in Reference 11, about 10% of the main air stream flows through the baffled region in the small-scale test beds.

conducted at low velocity in which no measurable "F-112" penetration occurred during the first few minutes. The data were correlated from "F-112" adsorption tests of small-scale carbon beds (shape factor $\phi = 0.90$) with the following range of variables:

$X = 1.3$ to 5.1 cm

$y = 0.08$ to 0.63

$A' = 41$ to 110 cm^2/g

$p = 0.53$ to 0.62 g/cc*

$D_p = 0.10$ to 0.30 cm

$G = 20$ to 41×10^{-3} g/cm^2-sec (35 to 70 ft/min)

The upstream "F-112" concentration (C_o) was 20 ppm (by volume) in all tests.

Activated carbons of coconut shell and bituminous base (impregnated and unimpregnated) were used in the tests. No significant differences were observed for the unimpregnated carbons. However, the unimpregnated carbons showed better adsorption characteristics than the impregnated carbons. The differences are discussed under Evaluation.

Evaluation

Equation 4 contains three variables (D_p, A', and y) that are difficult to evaluate precisely. The average diameter of the carbon particles (D_p) was estimated by a sieve analysis of the carbon(12). A weighted average was determined for D_p from the measured particle distribution. For the particle-size distribution of Type 416 carbon** (used at SRP), D_p equals ~0.15 cm. D_p for other carbons studied was related to Type 416 carbon by the following equation:

$$\frac{D_{p_2}}{D_{p_1}} = \left(\frac{V_2}{V_1}\right)^{\frac{1}{3}} \qquad (5)$$

where

V = average apparent particle volume, cc

---

* These packing densities represent firmly packed carbon beds.

** Product of Barnebey-Cheney Co.

The particle volume was calculated by dividing the average weight of a representative number of particles by the apparent density of the carbon particle. Table I lists the calculated D_p and other physical properties for the various carbons studied.

The maximum effective outer surface area of the carbon (A') is dependent on the size and shape of the particles. For Type 416 carbon, A' was estimated as 75 cm^2/g.(11) A' for all other carbons studied was estimated (using Type 416 as a base) by the following equation:

$$\frac{A'_1}{A'_2} = \left(\frac{\rho_{c_2}}{\rho_{c_1}}\right)\left(\frac{D_{p_2}}{D_{p_1}}\right) \tag{6}$$

where

ρ_c = apparent density of carbon particle (by Hg displacement), g/cc

Equations 5 and 6 are valid for carbons of the same sphericity. The estimated maximum effective outer surface area for the various carbons studied are listed in Table I.

TABLE I

Physical Properties of Activated Carbons

Carbon		Apparent Particle Density, g/cc(a)	Avg Particle Wt, 10^{-3} g	Total Surface Area, m^2/g	Estimated Particle Diameter, cm(b)	Estimated Outer Surface Area, cm^2/g(c)
Type	Mesh					
416(d)	10 x 14(e)	0.87	2.04	1000-1100	0.15	75
592(d)	10 x 14(e)	0.87(f)	2.04(f)	1000-1100	0.15	75
BPL(g)	4 x 10(h)	0.80	14.8	1050-1150	0.30	41
416	12(h, i)	0.87	2.04	1000-1100	0.15	75
416	18(h, j)	0.87	0.84	1000-1100	0.11	100
BD(d)	4 x 10(e)	0.87(f)	11.3	1000-1100(f)	0.27	42
ASC Whet(k)	12 x 30(h)	0.87(f)	0.64	1200	0.10	110
75g-4(d)	10 x 14(e)	0.87(f)	2.04(f)	1000-1100(f)	0.15	75

(a) By Hg displacement.
(b) Calculated as a function of particle volume by formula (5), see text.
(c) Calculated as a function of particle diameter and density by formula (6), see text.
(d) Product of Barnebey-Cheney, coconut shell base. Type 592 carbon has high ignition temperature.
(e) Tyler Sieve.
(f) Assumed.
(g) Product of Pittsburgh Activated Carbon Co., bituminous base.
(h) U. S. Sieve Series.
(i) Through No. 10 screen, retained on No. 12 screen.
(j) Through No. 16 screen, retained on No. 18 screen.
(k) Bituminous base carbon impregnated with salts of copper, chromium, and silver.

Water was the adsorbate that occupied most of the carbon's surface area during the tests, because nearly all the tests were conducted with humid air. A typical isotherm for H_2O on carbon (surface area = ~1100 m^2/g) is given in Figure 1. About 35 wt % H_2O was sorbed on Type 416 carbon when in equilibrium with air at 90% relative humidity (RH); most of this moisture was probably in a monomolecular layer. About 33 wt % H_2O was calculated to form a monomolecular layer on Type 416 carbon. Above 90% RH, formation of multimolecular layers and/or condensation of H_2O in the pores of the carbon probably begins as indicated by the rapid increase in capacity for H_2O. The free area of all carbons that were tested was related to the quantity of adsorbed H_2O by the following equation:

$$y = \frac{W-Z}{W} \tag{7}$$

where

W = minimum wt % H_2O on carbon to occupy all adsorption sites

Z = wt % H_2O on carbon at time of test

Equation 7 is based on the assumption that y is a linear function of Z. This assumption is probably valid for the range of Z in which no multilayer adsorption occurs. For the unimpregnated

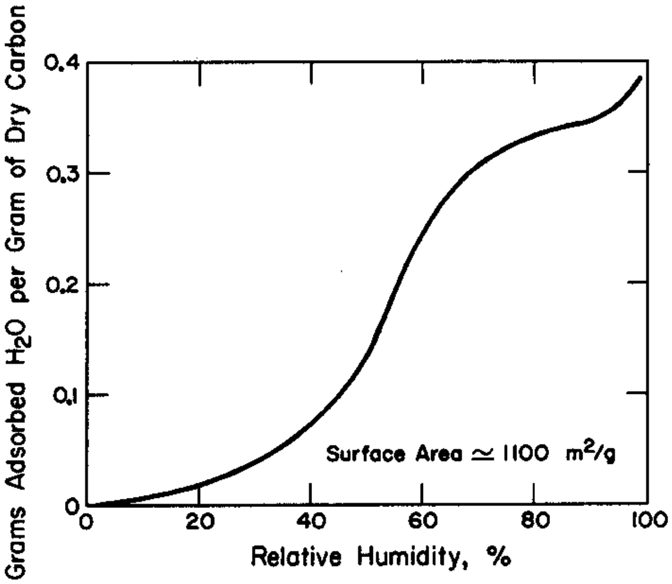

FIG. 1 TYPICAL ADSORPTION ISOTHERM FOR WATER VAPOR ON ACTIVATED CARBON

carbons, values of W of 33, 36, and 40 wt % were evaluated in the correlating equation; 40 wt % H_2O produced the least scatter in the data. For the impregnated carbons, the H_2O isotherm was different from that for the unimpregnated carbons. A relative humidity of about 65% was sufficient to maintain about 30 wt % H_2O on Type 416 carbon, but about 85% RH was needed to maintain the same amount of H_2O on ASC Whetlerite* carbon. For the Whetlerite carbon, values of W of 34 and 37 wt % were evaluated in the correlating equation; 34 wt % H_2O produced the least scatter in the data. The lower W value for the impregnated carbons was expected, because the impregnant occupies sites on the carbon.

The packing density of the carbon bed affected the "F-112" penetration, but this effect was not determined quantitatively. Small changes in the packing density affected the pressure drop across the beds and the "Freon" adsorption efficiency. However, reproducible results were obtained when the beds were packed firmly to produce consistent pressure drop across the various beds. The packing density was used only in the calculations of external surface area per unit volume and was not intended to account for any significant variations in the bed packing. The average pressure drop across the test beds (representative of firmly packed beds) are shown in Table II for an air velocity of 70 ft/min.

TABLE II

Average Pressure Drop of Small-Scale Test Carbon Beds

Bed Thickness, cm	Carbon Mesh	Calculated Particle Diameter (avg), cm	Avg ΔP, inches H_2O
1.3	10 x 14 (Tyler)	0.15	0.49
	18 (US)	0.11	0.68
2.5	4 x 10 (US)	0.30	0.34
	4 x 10 (Tyler)	0.27	0.38
	12 (US)	0.15	0.72
	10 x 14 (Tyler)	0.15	0.72
	12 x 30 (US)	0.10	1.50
5.1	4 x 10 (US)	0.30	0.61
	4 x 10 (Tyler)	0.27	0.57
	10 x 14 (Tyler)	0.15	1.42

---

* Impregnated bituminous base carbon; military specification MIL-C-13724A, May 4, 1960.

The data for 1.3-cm-thick beds tested with the larger carbon particles (0.30 cm) showed penetrations higher than predicted by Equation 4. In these tests, the bed thickness to particle diameter ratio $(\frac{X}{D_p})$ was probably too low to prevent "F-112" from channeling through the bed. Thus, $\frac{X}{D_p} \geqslant 8.5$ is a limit.

As previously stated, Equation 4 was developed for laminar flow (Reynolds number less than 40). However, data with Reynolds numbers up to 70 are adequately correlated by Equation 4.

The correlating test data are summarized in Table III and plotted with the use of the correlating equation in Figures 2 and 3. Figure 2 presents the data for unimpregnated carbons, and Figure 3 shows the results for impregnated carbons.

Unimpregnated coconut shell carbon was the basis for the correlating equation. Other carbons investigated were compared with the coconut shell carbon. The correlation for unimpregnated carbons (Figure 2) has a standard deviation of about 13% in the K-factor for the range of "F-112" penetration most accurately measured (0.01 to 0.3%). Scatter occurred in the data because of variations in the properties of the different types of carbon (differences in sphericity, packing density, H_2O isotherm, and base materials). The correlation for impregnated carbon (ASC Whetlerite, Figure 3) has a standard deviation of about 12% in the K-factor.

The curves of Figures 2 and 3 are not identical, because the W term of Equation 7 was changed. No adjustment in external surface area (A') was made for the impregnated carbon. This implies the impregnant does not significantly affect the external surface area. However, strongly adsorbing or chemisorbing materials may concentrate on the external surface causing some reduction in the number of active sites and some loss in effective area.

The curve of Figure 3 is based on the data points of ASC Whetlerite which contains about 9 wt % impregnants. Also plotted in Figure 3 are two data points for Type 75 g-4 carbon* (coconut shell base with 1 wt % I_2 impregnant). The points are plotted on the same basis as ASC Whetlerite ($W = 34$ wt %) and show less penetration than would be expected for Whetlerite with the same test conditions. This indicates that the amount and/or type of impregnant affects the effective external surface area as well as the H_2O isotherm.

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* Product of Barnebey-Cheney Co.

TABLE III

"F-112" Penetration Tests of Small-Scale Carbon Beds

	Carbon					Test Conditions					
Bed No.	Type[(a)]	Mesh[(b)]	Bed Thickness, inches	Packing Density, g/cc	ΔP at 70 ft/min, inches H_2O	Velocity, ft/min	Sorbed H_2O, %	Particle Diameter, cm	External Surface Area, cm^2/g	"F-112" Penetration at 8 min, %	K-factor, 10^3 $(cm^2)(sec)/g$
470	416	10 x 14	1	0.56	0.64	70	25.7	0.15	75	0.017	1.04
478	BPL	4 x 10	1	0.55	0.34	70	14.8	0.30	41	0.073	0.99
487	BPL	10 x 14	1	0.59	0.72	70	26.1	0.15	81	0.028	1.14
494	592	10 x 14	1	0.56	0.70	70	26.4	0.15	75	0.101	0.98
495	592	10 x 14	1	0.58	0.74	70	26.5	0.15	75	0.135	1.10
499	Whet	12 x 30	1	0.61	1.60	70	24.9	0.10	110	0.027	1.24
500	Whet	12 x 30	1	0.62	1.63	70	26.3	0.10	110	0.096	1.07
504	416	10 x 14	1	0.56	0.75	70	26.2	0.15	75	0.049	0.99
505	416	10 x 14	1	0.56	0.74	35	31.2	0.15	75	0.014	1.27
509	416	10 x 14	1/2	0.59	0.45	35	27.1	0.15	75	0.087	0.99
540	BPL	4 x 10	2	0.54	0.61	35	22.6	0.30	41	0.035	1.32
545	416	12	1	0.57	0.72	70	26.2	0.15	75	0.07	1.01
557	416	10 x 14	1	0.55	0.75	35	32.1	0.15	75	0.024	1.14
565	416	10 x 14	1/2	0.58	0.53	35	27.6	0.15	75	0.232	0.95
567	416	10 x 14	1	0.55	0.72	35	33.3	0.15	75	0.02	0.97
570	BD	4 x 10	2	0.54	0.61	70	25.0	0.27	42	0.014	1.18
571	BD	4 x 10	1	0.54	0.38	35	30.6	0.27	42	0.27	0.72
574	BD	4 x 10	2	0.53	0.54	70	27.1	0.27	42	0.041	0.99
577	416	10 x 14	2	0.54	1.42	35	36.9	0.15	75	0.145	0.87
581	416	10 x 14	1	0.56	0.74	35	33.0	0.15	75	0.026	1.01
583	416	10 x 14	1	0.58	0.73	35	34.6	0.15	75	0.14	0.81
591	Whet	12 x 30	1	0.61	1.50	70	26.0	0.10	110	0.161	1.09
603	Whet	12 x 30	1	0.56	1.33	70	25.3	0.10	110	0.160	1.09
605	416	18	1/2	0.56	0.68	35	30.9	0.11	100	0.01	0.89
606	Whet	12 x 30	1	0.56	1.52	35	27.5	0.10	110	0.028	1.63
620	Whet	12 x 30	1	0.58	1.44	35	29.4	0.10	110	0.096	1.18
624	75g-4	10 x 14	1	0.57	0.74	35	28.6	0.15	75	0.164	0.84
625	75g-4	10 x 14	1	0.54	0.71	35	28.3	0.15	75	0.024	0.84

(a) Type carbon identified in Table I.
(b) Mesh size and type given in Table I.

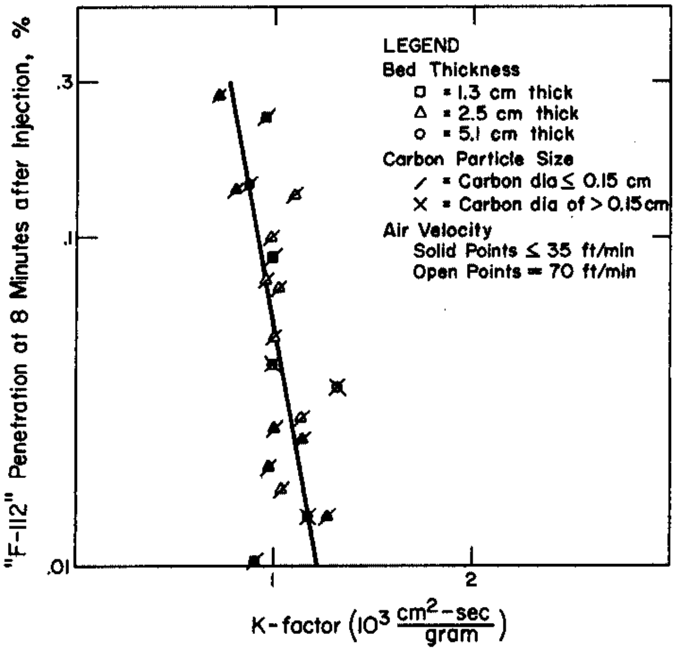

FIG. 2 CORRELATION OF "F-112" PENETRATION OF UNIMPREGNATED ACTIVATED CARBON

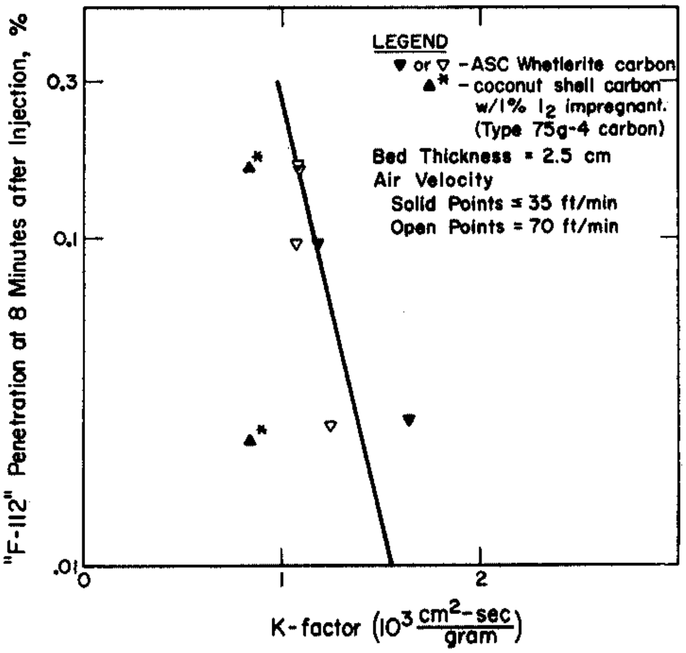

FIG. 3 CORRELATION OF "F-112" PENETRATION OF IMPREGNATED ACTIVATED CARBON

Construction of the curves of Figures 2 and 3 was based on the theoretical shape of the curve imposed by the correlating equation (a straight line passing through 100% penetration at K equals zero). The mean of the data points was used to draw the curve. Based on Figure 2, the value of the constant c_2 is 3.2×10^{-3} g/cm^2-sec. The theoretical value of the constant is 4.1×10^{-3} g/cm^2-sec for the actual test conditions. The agreement between the theoretical and measured values of c_2 verified the approach used in developing the correlating equation.

The data correlated by Equation 4 were obtained from tests of new carbon. It is also probably applicable for used carbon, because external diffusion should still be the controlling mechanism. Used carbon contains sorbed impurities that reduce the free external surface area, but sufficient data are not yet available from the full-size SRP beds (Figure 4) to determine quantitatively the effect of service on the free external surface area.

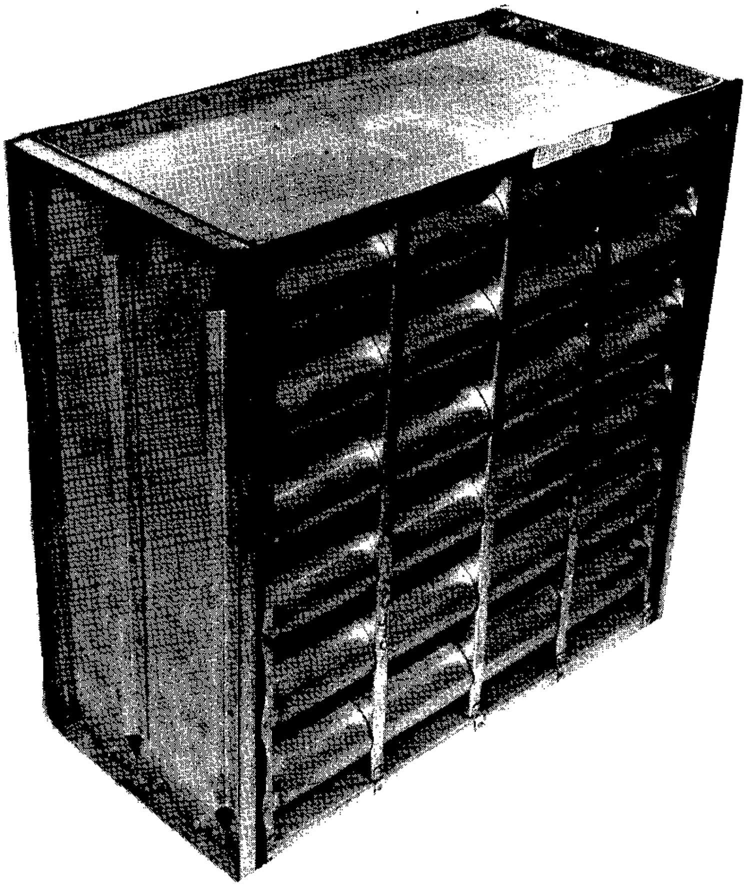

FIG. 4 ACTIVATED CARBON BED FRAME

Application of Correlating Equation

The correlating equation was used to evaluate a K-factor (defined by Equation 4) for known test conditions of new carbon beds. It is recommended that a K-factor $\geqslant 1.1 \times 10^3$ be used with "F-112" tests of unimpregnated carbons. Under such test conditions, excessive "F-112" penetration due to desorption will not occur, and the resulting efficiency-time curve can be extrapolated to time zero with confidence to determine leakage flow greater than 0.01%. For impregnated carbons with adsorption characteristics similar to ASC Whetlerite, a K-factor $\geqslant 1.4 \times 10^3$ should be used for measurement of leakage flow $\geqslant$0.01%. Figure 5 shows typical efficiency curves for satisfactory and unsatisfactory "F-112" tests that would result with acceptable and unacceptable K-factors. Curves A and B represent satisfactory tests, but the K-factor for curve B was somewhat lower than that for curve A. This accounts for the greater desorption with increased time. Curve C is unsatisfactory (K-factor too low), because the curve cannot be accurately extrapolated to time zero for measurement of leaks of 0.01%.

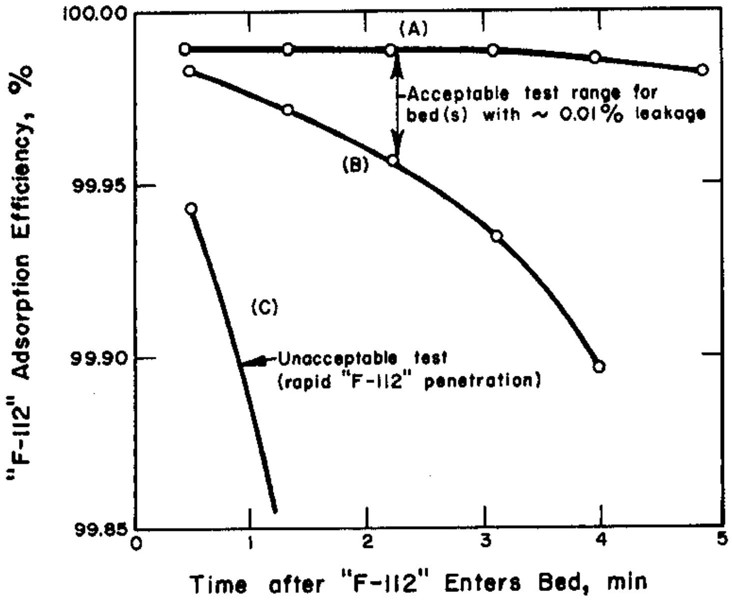

FIG. 5 ACCEPTABLE AND UNACCEPTABLE "F-112" TESTS

Appendix A presents sample calculations for determining K-factors. As shown in Appendix A, satisfactory test conditions for a 1-inch-thick bed (2.5 cm) with a shape factor of 1.8 and firmly packed with <u>new</u> unimpregnated carbon of 0.18-cm average particle diameter are 35 ft/min face velocity and 22% sorbed H_2O. If the carbon were impregnated to give the same adsorption characteristics as ASC Whetlerite and packed in the same bed, satisfactory test conditions would be 35 ft/min face velocity and 14.5% sorbed H_2O.

The quantities of the correlating K-factor that are associated with the carbon bed are constants and will not change with the exception of y (fraction free outer surface area; y is affected by adsorption of impurities that occurs with normal service of the carbon). Thus the correlation may be used also to compare the performance of different carbon beds and estimate the effect of service on the free external surface area. As previously discussed, the correlation is valuable in comparing shape factors (performance) of modified or newly designed carbon beds.

APPLICATION OF "F-112" LEAK TEST

Detailed application of the "F-112" Leak Test to the SRP Confinement System was discussed in DP-1053(5). Basically, there is no difference in the SRP test and the standardized test. Figure 6 shows a diagram for the general application of the test to a typical carbon bed installation. Figure 7 shows the test equipment and a diagram for the application of the test to the SRP systems. The sample points differ in the two diagrams, but this is not significant because adequate mixing occurs as discussed in DP-1053. Application of the standardized test requires use of the standard test procedure presented in Appendix B and satisfactory test conditions (face velocity and H_2O content of carbon) as established by the correlating K-factor. The limiting test conditions may be estimated for new carbon with the correlating equation and from SRP experience with used carbon. Recommended test conditions for SRP type carbon beds with ~3 years service are 10 ft/min velocity and <4% sorbed H_2O.

The leak test procedure is written for use with the "F-112"-air method of relative detector calibration because of its simplicity and greater accuracy. Details of this method of detector calibration are discussed under *Leak Test Apparatus* and in Appendix D. However, the detectors may also be calibrated with a "F-112"-hexane solution on an absolute basis if the concentration of the solution and sample injection volume are known. This latter method is also described in detail in Appendix D.

The appendices contain several aids that should be understood before application of the "F-112" Leak Test. Appendix C shows the data sheet and calculation form. Appendix E presents the technique for reading chromatograms containing the "F-112" peak. Troubleshooting aids and general precautions are discussed in Appendices F, G, and H. Equipment specifications are given in Appendices I and J.

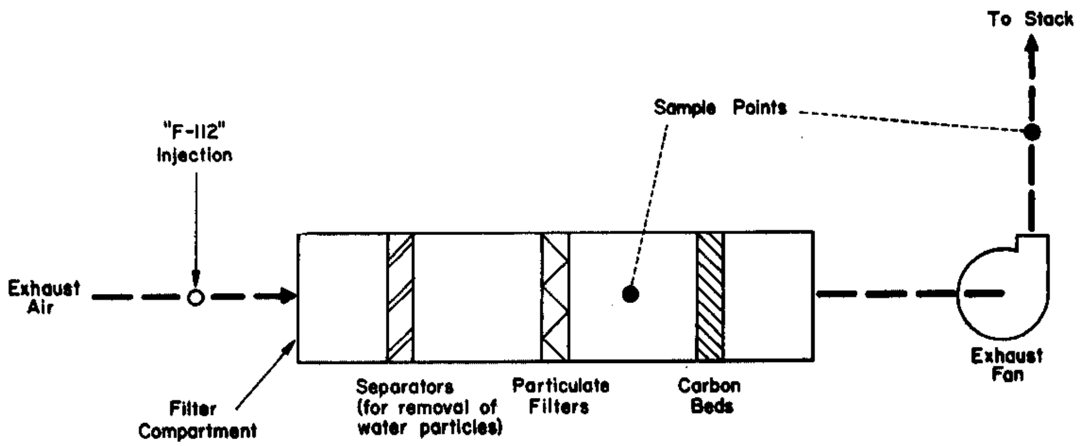

FIG. 6 DIAGRAM FOR "F-112" LEAK TESTING OF TYPICAL CARBON BED INSTALLATION

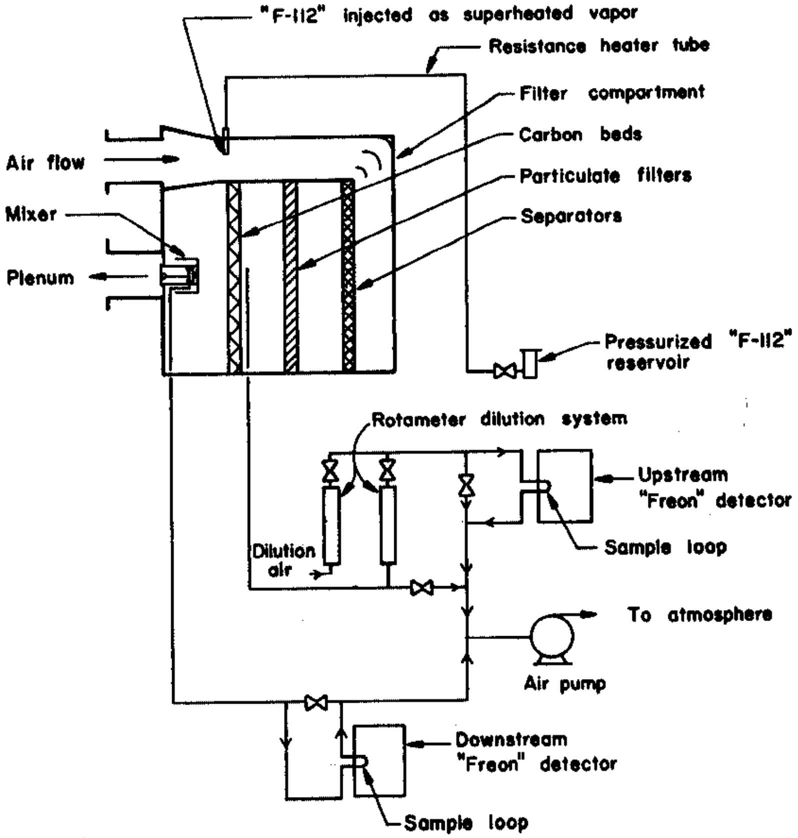

FIG. 7 DIAGRAM OF EQUIPMENT FOR TESTING INSTALLED SRP CARBON BEDS

LEAK TEST APPARATUS

Figure 7 shows a diagram of equipment for testing installed SRP carbon beds but is typical for any system. The basic requirement of the test apparatus is that it accurately measure the ratio of the "F-112" concentration upstream and downstream of the test carbon beds. This requirement can be broken into four major subdivisions:

1. A representative sample at the sample points in the duct.

2. Transport of sample from duct to test apparatus without change in concentration.

3. Accurate dilution of upstream sample before admission to upstream detector.

4. Relative calibration of "F-112" detectors.

These items were discussed in detail in DP-1053 and are briefly discussed below for review.

Item 1 is generally controlled by conditions in the duct, and requires thorough mixing of the "F-112" and air both upstream and downstream before sampling. Definitive measurement of leakage flow through a carbon bed cannot be accomplished without proper mixing and sampling. Methods of mixing and sampling in the SRP application are discussed in DP-1053.

Significant adsorption of the "F-112" tracer to the downstream duct wall between the carbon beds and sample point must be avoided. Otherwise, a representative sample will not reach the sample point. Therefore, the downstream sample point should be located as near the carbon bed as possible but must obtain a mixed sample. Adsorption of "F-112" tracer to the upstream duct wall is of no concern as long as the upstream sample point is located immediately upstream of the test carbon bed.

Transport of the sample from the duct to the test apparatus without change in concentration requires no significant adsorption of tracer to the sample tubing nor leaks in the tubing. Sample tubing of stainless steel or "Teflon"* was found satisfactory, because the "F-112" plateout was small. Sample transit times of less than 5 seconds should be used. In-leakage of air to the sample tubing must obviously be avoided, because it could change the sample concentration.

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* Du Pont's trademark for its fluorocarbon plastic.

The upstream sample must be diluted before admission to the upstream detector, because it is far too concentrated for quantitative analysis by the detector. The dilution is accomplished by calibrated rotameters to determine the exact dilution factor. Periodic recalibration of the rotameters is required for greatest accuracy.

Relative calibration of the "F-112" detectors requires that the response of both detectors to the same "F-112" concentration be known. The only restriction is that the detector response be in the linear range. Methods for relative calibration are discussed in Appendix D. The preferred method is a mixture of "F-112" in air admitted to both detectors simultaneously.

The "F-112" Leak Test apparatus that is used at Savannah River is shown in Figures 8 and 9. Detailed drawings are presented in Figures 10, 11, 12, 13, and 14.* The equipment case measures 31 by 22 by 13 inches and weighs about 90 lb. The "Freon" injection tube consists of stainless steel tubing (47 feet long by 3/32-inch OD with 0.010-inch wall) that is used as an electrical resistance heater (Figure 15).* A voltage of 115 volts AC is applied across the length of the tube, and the heat generated in the tube is used to heat the flowing "F-112" from a liquid to a superheated vapor. The tube is insulated electrically and thermally. The flow-ΔP characteristics of the injection tube are given in Figure 16. Injection tubes of shorter lengths may be used for lower capacity provided sufficient thermal energy is released in the tube. "F-112" may also be vaporized by bubbling air through liquid "F-112".

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* Copies of the drawings of the Carbon Bed Leak Tester (Figures 10 through 14) and the "Freon" Injection Tube (Figure 15) may be purchased from:

Clearinghouse for Federal Scientific and
Technical Information
U. S. Department of Commerce
5285 Port Royal Road
Springfield, Virginia 22151

These drawings should be requested by the ST drawing number.

FIG. 8 "F-112" LEAK TEST APPARATUS - FRONT
("F-112" Detector Included)

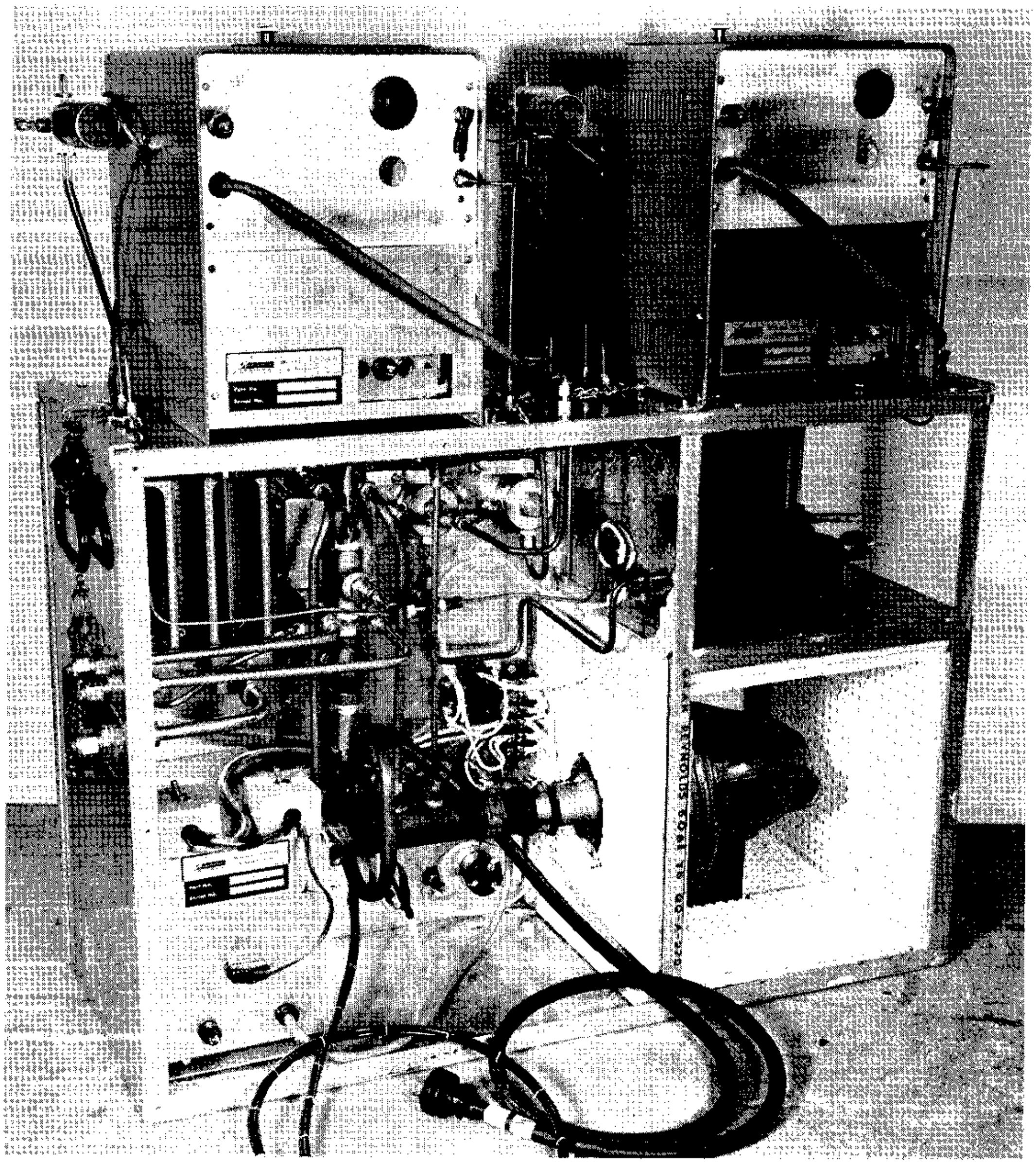
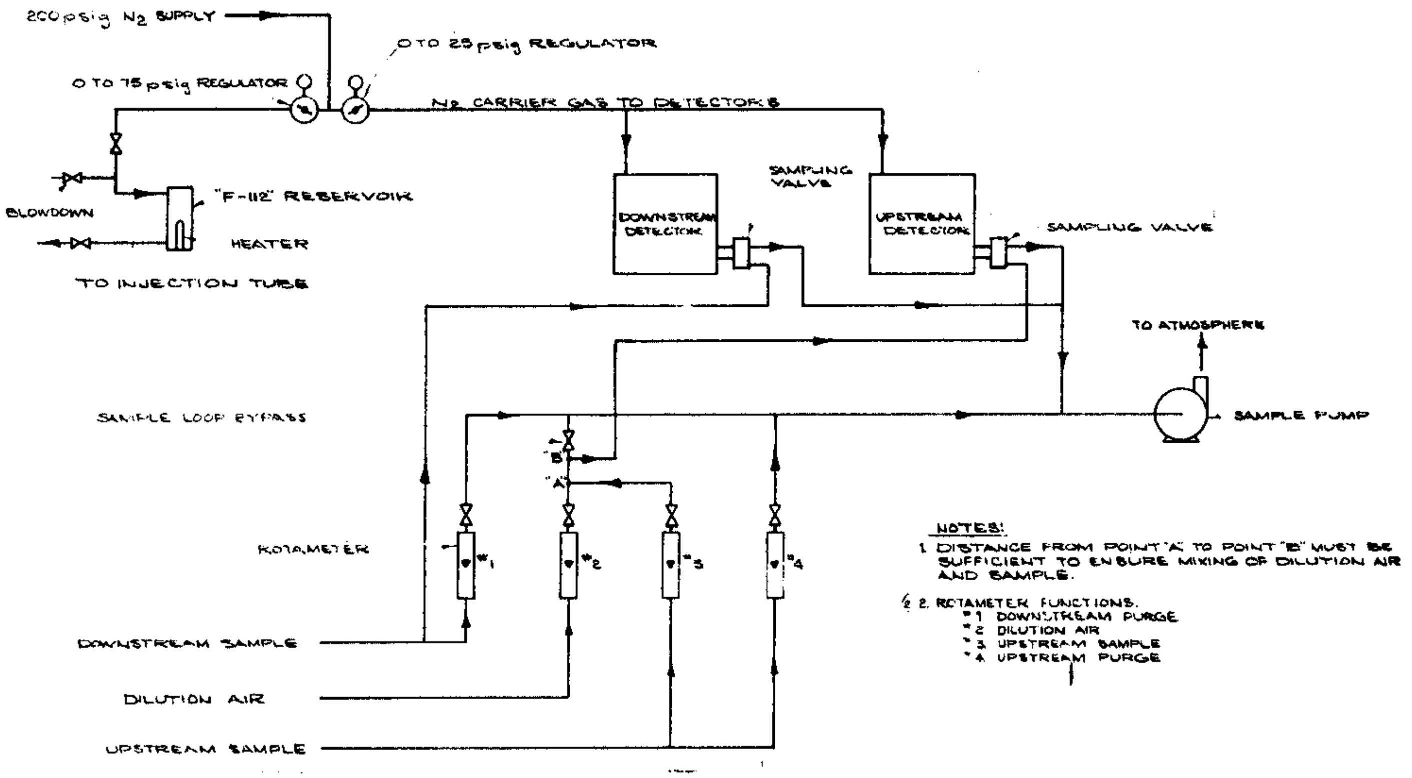


FIG. 9 "F-112" LEAK TEST APPARATUS - BACK
("F-112" Detector Included)



NOTES:

1. DISTANCE FROM POINT "A" TO POINT "B" MUST BE SUFFICIENT TO ENSURE MIXING OF DILUTION AIR AND SAMPLE.
2. ROTAMETER FUNCTIONS.
   - #1 DOWNSTREAM PURGE
   - #2 DILUTION AIR
   - #3 UPSTREAM SAMPLE
   - #4 UPSTREAM PURGE

ST-CMX-2-645

FIG. 10 PIPING DIAGRAM OF "F-112" LEAK TEST APPARATUS

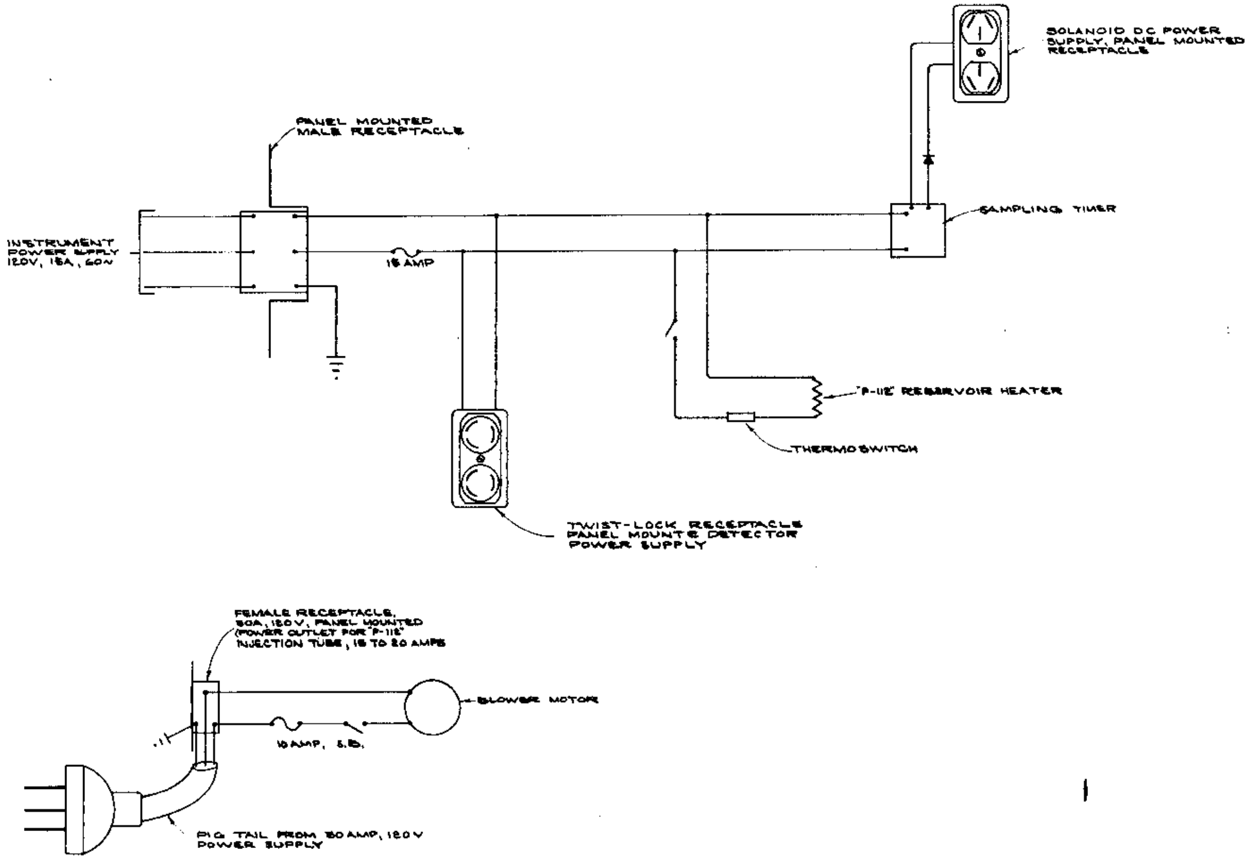
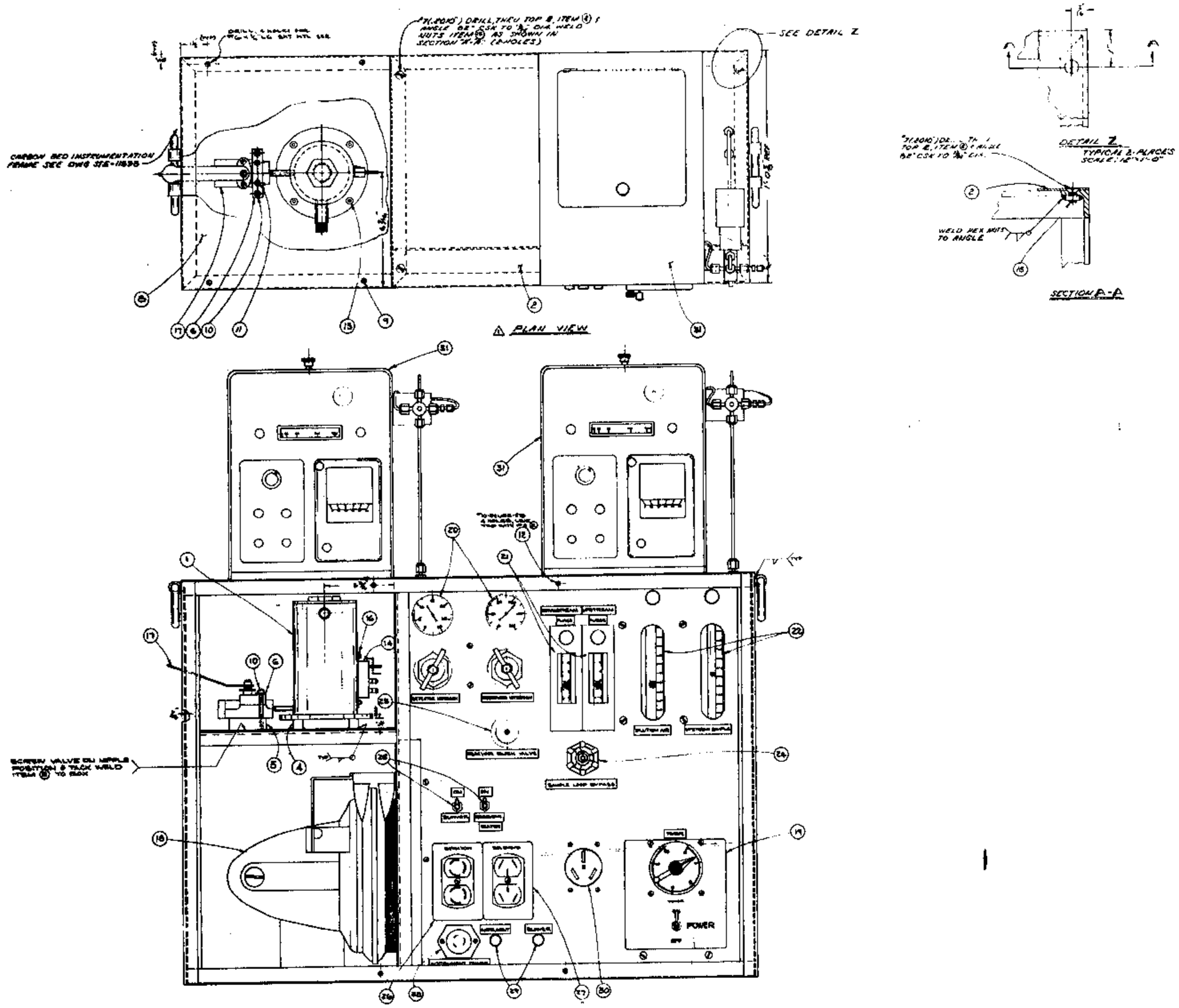


FIG. 11 WIRING DIAGRAM OF "F-112" LEAK TEST APPARATUS



ST5-11897

FIG. 12 CARBON BED LEAK TESTER ASSEMBLY

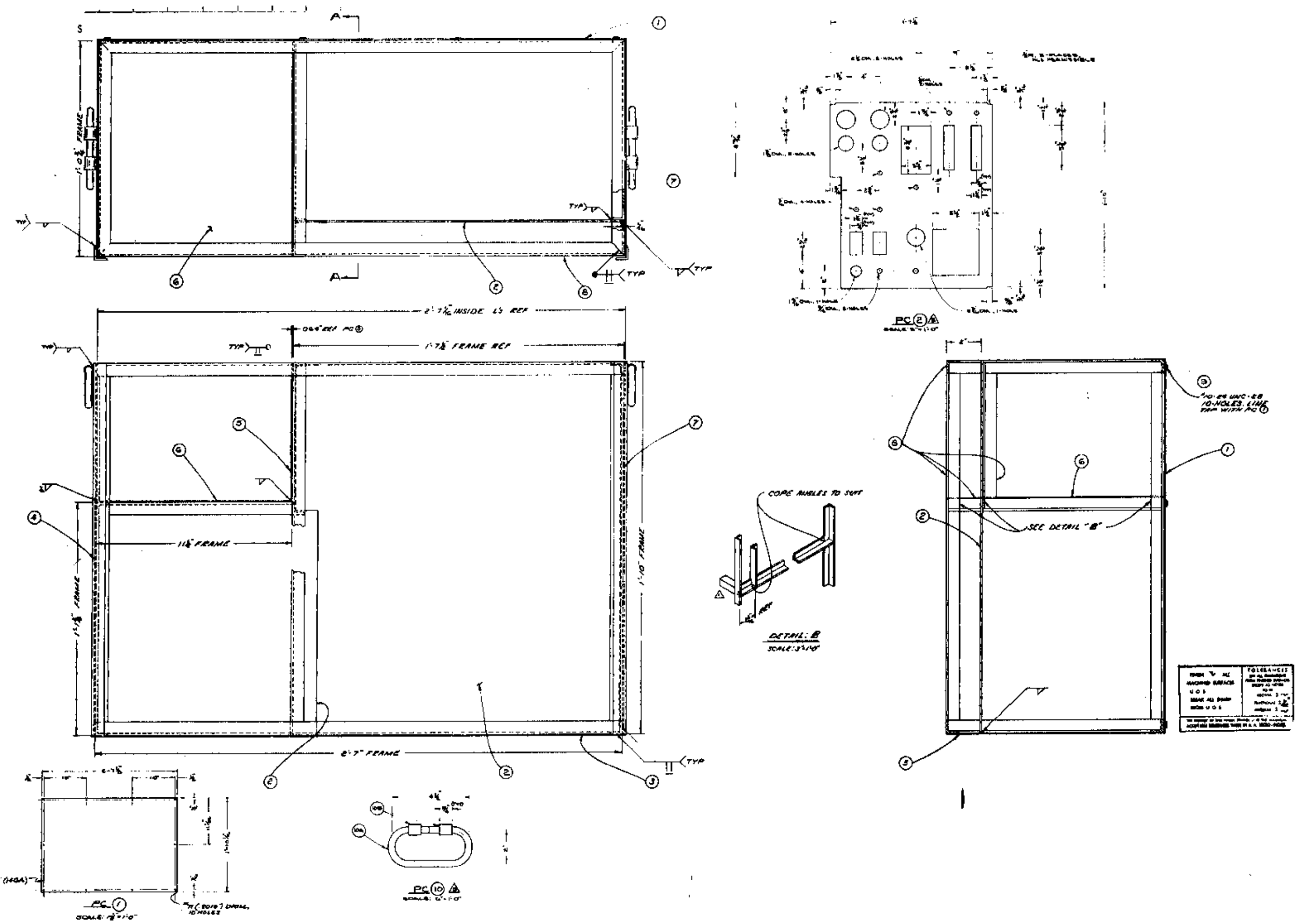


FIG. 13 CARBON BED LEAK TESTER FRAME

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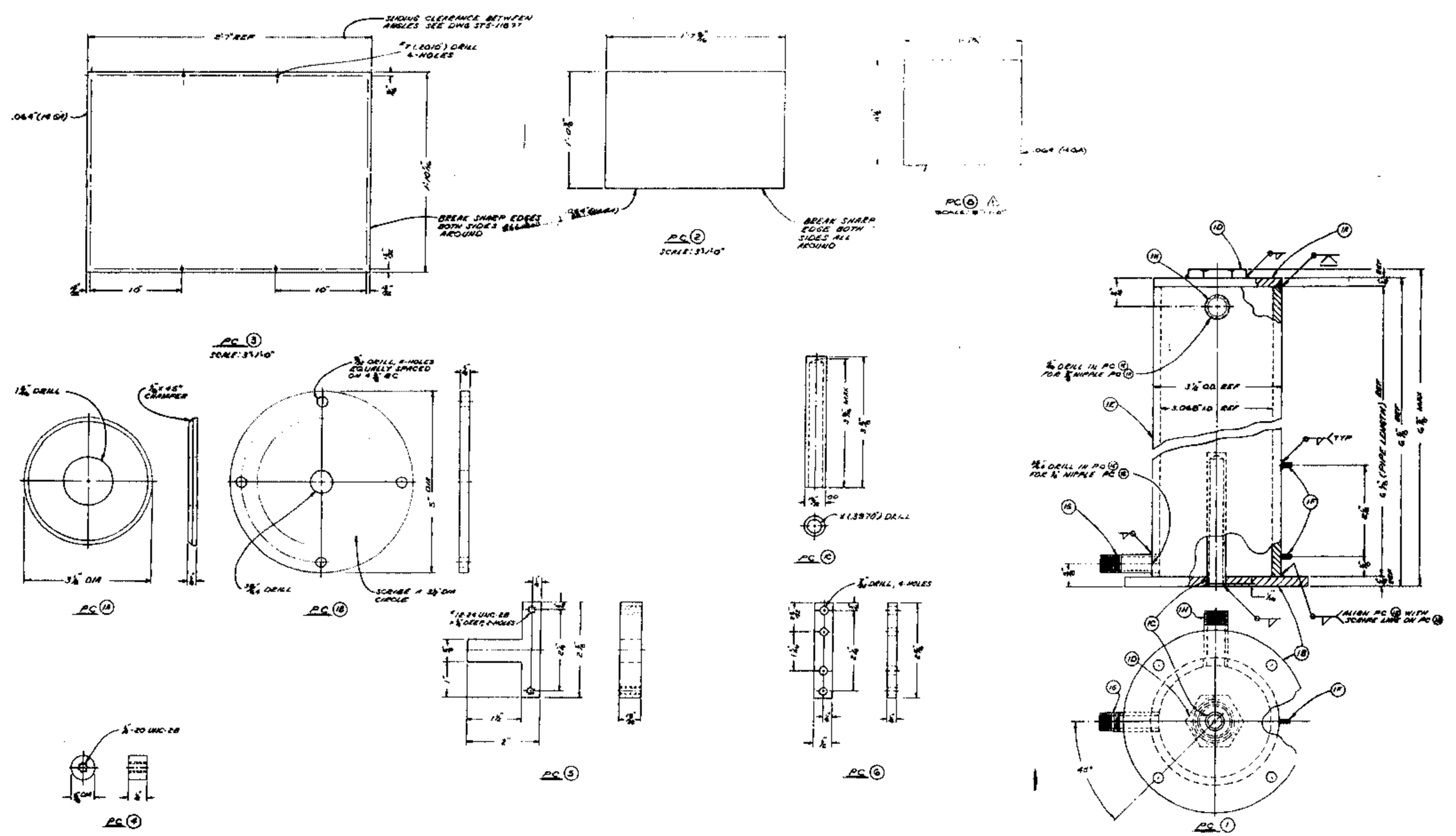


ST5-11899

FIG. 14 CARBON BED LEAK TESTER PARTS

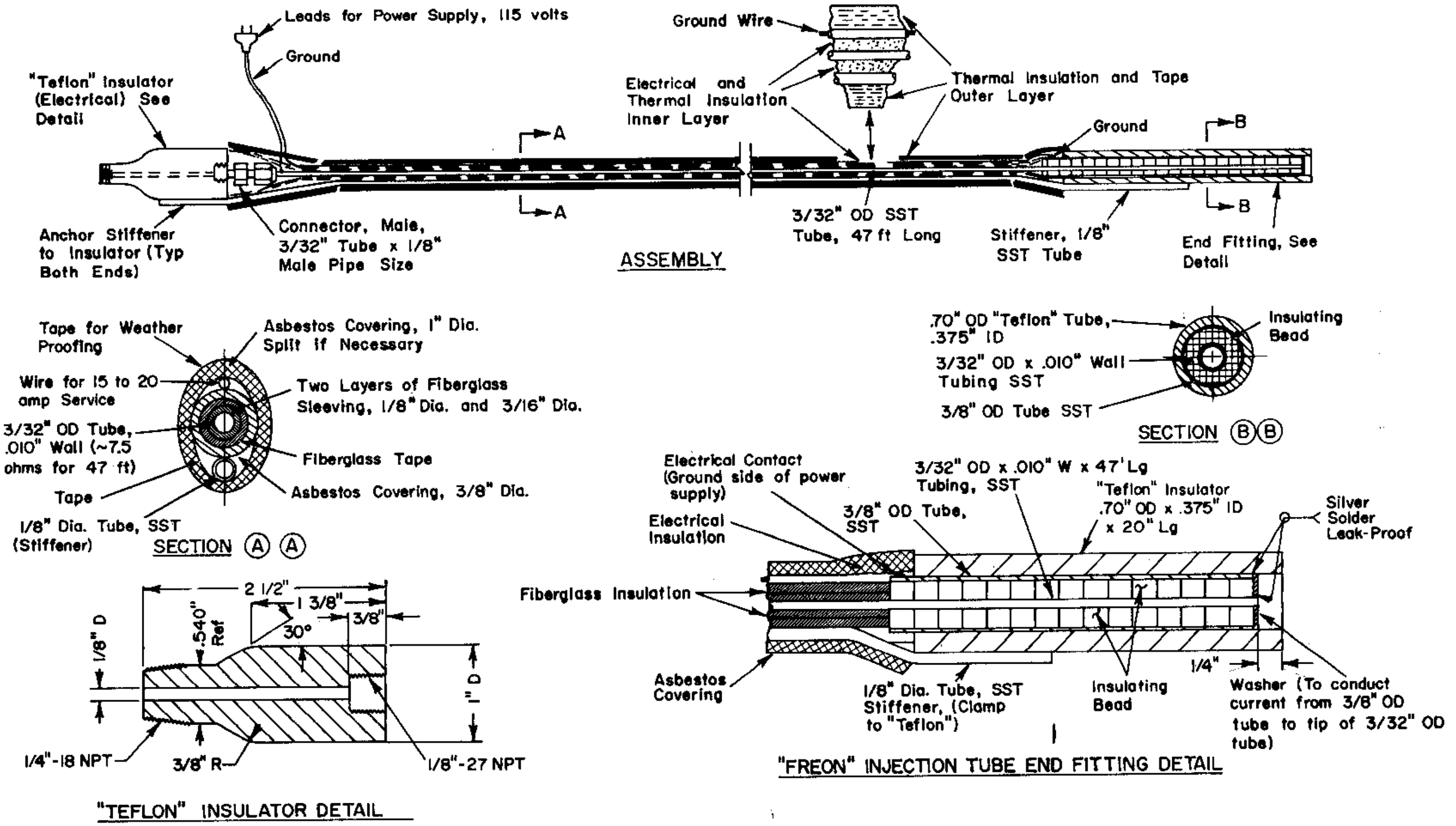
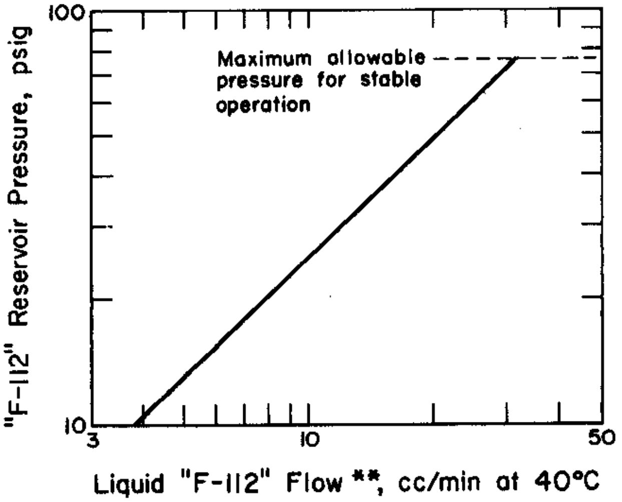


FIG. 15 "FREON" INJECTION TUBE-ASSEMBLY AND DETAILS

ST-CMX-2-646



* Injection tube about 47′ long by 3/32″ OD and 0.010″ thick stainless steel wall with 115 volts AC applied between ends of tube; current flow equals about 15 amperes (tube resistance equal to about 7.5 ohms).

** 20 cc/min of liquid "F-112" vaporized in 5000 ft^3/min of air at 80^0F equals a "F-112" concentration of about 27 ppm by volume.

FIG. 16 TYPICAL FLOW-PRESSURE DROP CHARACTERISTICS OF "F-112" INJECTION TUBE*

APPENDIX A

SAMPLE CALCULATIONS OF CORRELATING K-FACTOR

Case A

Carbon:	Unimpregnated coconut shell
Size:	No. 10 Tyler Sieve (through No. 9 mesh, retained on No. 10)
Particle density:	0.85 g/cc (by Hg displacement)
Bed thickness:	2.5 cm
Packing density:	0.55 g/cc

1. Calculate external surface area by first determining average particle diameter and substitute in Equation 6. Average particle diameter equals average of sieve openings.(12)

Tyler equivalent sieve opening sizes: No. 9 = 0.2000 cm
No. 10 = 0.1680 cm

$$D_{p_2} = \frac{0.2000 + 0.1680}{2} \simeq 0.18 \text{ cm}$$

Development of correlation was based on Type 416 carbon where

$$A_1 = 75 \text{ cm}^2/\text{g}$$
$$D_{p_1} = 0.15 \text{ cm}$$
$$\rho_{c_1} = 0.87 \text{ g/cc}$$

As previously determined,

$$D_{p_2} = 0.18 \text{ cm}$$
$$\rho_{c_2} = 0.85 \text{ g/cc} \quad \text{(given)}$$

Substitute in Equation 6

$$A'_2 = \left(A'_1\right)\left(\frac{\rho_{c_1}}{\rho_{c_2}}\right)\left(\frac{D_{p_1}}{D_{p_2}}\right) = \left(75 \text{ cm}^2/\text{g}\right)\left(\frac{0.87 \text{ g/cc}}{0.85 \text{ g/cc}}\right)\left(\frac{0.15 \text{ cm}}{0.18 \text{ cm}}\right)$$

$$A'_2 = 64 \text{ cm}^2/\text{g}$$

2. The correlating K-factor is $K = \frac{A'ypX}{\phi G}$

Unknowns: y (fractional free outer surface area)

ϕ (shape factor)

G (mass velocity of air stream, based on face velocity)

K (correlating K-factor)

Any one of the unknowns can be calculated if the other three are known or assumed.

Therefore,

Assume $\phi = 1.8$ (determine from several tests of carbon beds)

Let face velocity = 35 ft/min (maintain during test)

$$G \simeq 20 \times 10^{-3} \text{ g/cm}^2\text{-sec}$$

Determine maximum acceptable test conditions for measurement of leakage flow of $\geqslant$0.01%

$$K \geqslant 1.1 \times 10^3 \frac{\text{cm}^2\text{-sec}}{\text{g}} \quad \text{(unimpregnated carbon)}$$

What is maximum moisture content of carbon during test?

$$y = \frac{K\phi G}{A'pX} = \frac{1.1 \times 10^3 \frac{\text{cm}^2\text{-sec}}{\text{g}} \times 1.8 \times 20 \times 10^{-3} \text{ g/cm}^2\text{-sec}}{64 \text{ cm}^2\text{/g} \times 0.55 \text{ g/cc} \times 2.5 \text{ cm}}$$

$y = 0.45$

45% of the external surface area of the carbon must be free of any adsorbate (H_2O). Because new carbon may adsorb ~40 wt % H_2O with utilization of all adsorption sites (for mono-layer adsorption), the maximum moisture content of the carbon is 0.55 x 40 $\simeq$ 22 wt % H_2O.

Or by Equation 7,

$$y = \frac{W-Z}{W} \quad \text{or} \quad Z = W-yW$$

where W = 40 wt % (unimpregnated carbon)

Z = 40 - 0.45 x 40 $\simeq$ 22 wt % (maximum H_2O on carbon at time of test)

Case B

Calculate satisfactory test conditions for a carbon bed packed with impregnated carbon (new) with the same physical properties as in Case A but with the adsorption characteristics of ASC Whetlerite carbon. Assume carbon bed is of same size and shape factor as Case A. Therefore,

$$X = 2.5 \text{ cm}$$

$$A' = 64 \text{ cm}^2/\text{g}$$

$$p = 0.55 \text{ g/cc}$$

$$\phi = 1.8$$

Let $G = 20 \times 10^{-3}$ g/cm^2-sec (~35 ft/min face velocity)

But for a satisfactory test of leaks $\geqslant$0.01%,

$$K \geqslant 1.4 \times 10^3 \text{ cm}^2\text{-sec/g}$$

Determine maximum moisture content of carbon

$$y = \frac{K\phi G}{A'pX} = \frac{1.4 \times 10^{-3} \times 1.8 \times 20 \times 10^{-3}}{64 \times 0.55 \times 2.5} = 0.57$$

$$Z = W - yW$$

where

$W = 34\%$ (for ASC Whetlerite carbon)

$Z = 34 - 0.57 \times 34 = 14.5$ wt % H_2O

APPENDIX B

STANDARDIZED IN-PLACE "F-112" LEAK TEST PROCEDURE

NOTE: Part I of this procedure should be completed the day before the leak test is to be performed. Part II is used for the actual leak test. A diagram of the apparatus is shown in Figure 7.

Part I. Equipment Setup and Operational Check (Do not change or modify the test apparatus in any way except where noted.)

1. Locate test station and test equipment near carbon bed(s) to be tested.

2. Check "F-112" level in "Freon" reservoir; fill if necessary.

3. Install detectors on test station.

    a. Connect tubing from detector N_2 regulator to N_2 inlet at back of each detector.

    b. Connect sample-line tubing from rotameter system to gas sampling valves of each detector.

    c. Electrically connect solenoids of gas sampling valves to sampling timer.

    NOTE: Install detectors in proper position (as upstream or downstream detector) if using predetermined calibration conversion factor (see Appendix D).

4. Connect prepurified grade N_2 supply to test station.

    a. Install 0 to 300 psig regulator on N_2 bottle.

    b. Connect N_2 line from bottle regulator to test station N_2 inlet.

    c. Open all valves in the N_2 supply lines to the detectors including the toggle valve at the back of the detectors.

    d. Adjust regulator on N_2 bottle for 200 psig.

    e. Adjust regulator for detector N_2 supply for about 6 psig.

NOTE 1: The prepurified N_2 supply system must be kept clean for proper operation of the detectors. All junctions in the prepurified N_2 supply lines that are periodically disconnected should be supplied with fittings to plug the lines when disconnected. The plugs are necessary to keep out impurities that may enter the system from the atmosphere, hands, etc. The plugs must be kept clean when not in use and should never be interchanged with plugs for other systems (e.g., "Freon"-air sample lines). A low standing current or a rapid reduction in standing current for a newly installed clean cell may be due to impurities in the N_2 system that are transported to the cell.

NOTE 2: When the prepurified nitrogen bottle pressure reaches ~200 psig, change bottle supply. Use only prepurified N_2 and change only when detectors have operated long enough to attain normal standing current. Measure the standing current with 22 psig N_2 pressure on the detectors both before and after the N_2 bottle is changed. If the new N_2 supply causes a significant loss in standing current (greater than 10 to 15%), reinstall the old N_2 supply to clean the system. Discard the unsatisfactory N_2 supply and repeat the above procedure until a satisfactory prepurified N_2 supply is obtained.

5. Connect two electrical circuits to test station for power supply. Each circuit must be capable of delivering 30 amperes at 115 volts to the test station. If the circuit load reduces the voltage at the test station, the supply lines are not adequate. A low voltage at the test station for loaded circuits can invalidate the test by failure of the injection tube to vaporize "F-112".

   a. Use one circuit for sample pump and injection tube.

   b. Use the other circuit for both detectors, timer, and reservoir heater.

6. Turn on all electrical equipment described below and operate for 5 minutes to ensure that enough current is available. (However, this test does not ensure proper voltage at test station.) Then turn off all electrical equipment except detectors (electrometers and ovens) and "Freon" reservoir heaters (items (a) and (d)).

   a. Both detectors (electrometer and oven).

   b. Timer.

   c. Sample pump.

   d. "Freon" reservoir heater.

   e. "Freon" injection tube.

7. Connect upstream sample line from test station to sample point upstream of carbon bed(s).

8. Connect downstream sample line from test station to sample collector downstream of carbon bed(s).

9. Inspect flexible sample lines for crimping and damage; eliminate defects if present. Sample lines of thin wall "Teflon"* tubing, or equal, are recommended.

10. Connect "Freon" injection tube from "Freon" reservoir at test station to injection point upstream of test carbon bed(s).

    NOTE: Inspect injection tube for damage or crimping. Any defect must be repaired before using. For proper operation, the injection tube must completely vaporize the "F-112" flow required for a test.

    CAUTION: Handle injection tube with care; it conducts electrical power (e.g., about 15 amperes at 115 volts for SRL design).

11. Start sample pump and adjust flows through calibrated rotameters (Figure 10) to values required for test.

    Typical flows for SRP application are given below:

    Rotameter 1 = 0.7 cfm
    2 = 50,000 cc/min
    3 = 100 cc/min
    4 = 0.7 cfm

    NOTE: Flows through rotameters 1 and 4 are purge flows to permit rapid delivery of fresh sample to test station. Flow through rotameter 2 is clean air required to dilute

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* Du Pont's trademark for its fluorocarbon plastic.

the sample flow measured by rotameter 3. The ratio of flow through rotameter 2 to rotameter 3 is the dilution factor and must be known for calculation of the adsorption efficiency.

The dilution air flow through rotameter 2 must be free of any trace of "F-112". Consequently, the dilution air must not be obtained from the vicinity of the sample pump discharge because the air will contain "F-112" during a leak test. It is recommended that clean tubing be used to convey dilution air to rotameter 2 from a clean area upwind of the test station.

12. If all equipment performs satisfactorily, place on standby until ready to perform leak test. Do not turn off detectors (electrometer or oven) or detector N_2 supply.

13. Turn on heaters upstream of test carbon bed(s) and adjust air flow for an air temperature of 50 to 60°C downstream of carbon bed(s).

14. Operate heaters to maintain carbon bed(s) at 50 to 60°C for about one day before "F-112" leak test.

Part II. Test Procedure (Use after carbon bed(s) are dried for about one day. Do not modify test apparatus in any way except where noted. All instrument settings are based on Varian-Aerograph Model 680 detectors.)

1. Adjust detector N_2 pressure to between 22 and 23 psig.

2. Locate suction end of dilution air tubing on rotameter 2 in upwind air. The air must be free of any impurities that affect the detectors. Keep clear of water or debris. (Any foreign matter in this line will cause rotameter 2 to give false readings.)

3. If sample air contains contaminants, discharge pump effluent downwind of test station or return to upstream side of test carbon bed(s).

4. Adjust N_2 pressure on "Freon" reservoir to prescribed value for the desired test conditions. The approximate value of N_2 pressure may be calculated from known test conditions and injection tube capacity curve of Figure 16.

5. Check operation of detectors.

   a. Oven temperature should be adjusted to provide best separation of the "F-112" and oxygen peaks (about 75 to 100°C). (The oven thermostat may have to be adjusted between summer and winter conditions. See detector instruction manual.)

   b. Check that adequate chart paper is on recorder.

   c. The standing current for each detector must be greater than 25 recorder units (RU) at attenuator setting of 128 (see detector instruction manual for method of measuring). If the standing current does not exceed 25 RU, two possible causes are: 1) insufficient time for cell to warm up and purge clean with the hot N_2 flow, or 2) the cell is covered with nonvolatile substances that cannot be purged off. Therefore, cells that do not produce a standing current of greater than 25 RU (after 1 day of normal operation in a hot oven) should be returned to the manufacturer for cleaning or cleaned by following manufacturer's recommendation given in instruction manual.

6. Set up detector calibrator for "F-112"-air mixture.

   a. Connect "F-112"-air calibrator to upstream and downstream sample points on test station. Use 15-ft-long by 3/8-inch tubing and attach flow splitter at apparatus case.

   b. Locate calibrator upwind of sample pump.

   c. Start detector recorders and after stabilization, zero with attenuator at 128. Measure standing current and record on calibration data sheet. Then switch attenuator to 16. Adjust recorders to read between 10 and 20 with adjustment knobs. Label each chart as to detector, date, and test identification.

   d. Start sample pump.

   e. Adjust rotameter flow as below:

      Rotameter 1 = 0.5 to 0.7 cfm
      2 = 0 flow
      3 = 140+ cc/min
      4 = 0.5 to 0.7 cfm

   f. Close sample loop bypass valve (Figure 10).

7. Calibrate detectors.

   a. Start sampling timer and adjust time between each sample (if necessary) to exactly 50 seconds. Be sure both detector solenoids operate quickly and at the same time.

   b. Fully open 1/4-inch ball valve on calibrator.

   c. Record any background peaks.

   d. Open 1/8-inch needle valve 1/8 turn.

   e. Note recorders for "F-112" peak.

   f. Adjust valves of steps a and b to set "F-112" peak at about 25 to 50 recorder units. The peak height must not exceed 25% of the adjusted standing current (see Appendix H, item 5).

   g. When peak heights are consistent, record corresponding peak heights for each detector on "F-112"-air calibration data sheet.

8. After calibration, reconnect sample lines from upstream and downstream of test carbon bed(s) to test station.

9. Open sample loop bypass valve and set rotameter flows as per step 11 of Part I.

10. Note recorder traces for any unusual peaks that may interfere with the "F-112" peak in an air sample. The test cannot be conducted if other peaks interfere with the "F-112" peak. However, background peaks are permissible if they are consistent in size and less than the value of the standing current (see Appendix H).

11. Operate equipment for at least 5 minutes before proceeding with step 12. Be sure there is no irregular operation of equipment. Readjust recorders to read between 10 and 20 if necessary.

12. Adjust air flow through carbon bed(s) to desired face velocity for test.

    <u>NOTE</u>: If the air flow is changed, any background peak height on the downstream recorder may change and gradually level out at some consistent value.

13. Start current flow through "F-112" injection tube.

14. When equipment is operating stably and any background peak heights present are of consistent size, record data. If background peak heights vary significantly in size or if larger than the standing current, the leak test should not be performed. (Additional heating of carbon bed(s) will be required to desorb the impurity and reduce the background peak.)

15. Recheck settings for following equipment:

    a. Rotameter 2.

    b. Rotameter 3.

    c. N_2 pressure on "Freon" reservoir.

16. Review steps 17 through 23 before starting step 17. These steps should be completed within 5 minutes.

17. At the proper start time, inject "F-112" by quickly opening the valve between "F-112" reservoir and injection tube. The start time for "F-112" injection must be determined for each system. The injection start time should be selected to permit the "F-112" to flow from the reservoir through the system (assuming no delay by the carbon bed(s)) and reach the downstream detector about 0.6 minute* before the detector analyzes the first sample that could contain "F-112" (sample 1). Thus, results from sample 1 should be plotted on the "F-112" adsorption efficiency vs time curve at 0.6 minute after "F-112" reaches the carbon bed(s).

18. Label recorder charts for sample 1. Subsequent samples should be labeled in numerical order.

19. Observe and record any operation that could invalidate the test. If test must be halted, immediately stop "F-112" injection by closing valve at reservoir and proceed with step 23.

20. Check rotameters 2 and 3 and record any changes. <u>DO NOT ADJUST ROTAMETERS AFTER TEST STARTS</u>.

21. Note downstream detector peaks. If "F-112" peak goes off scale before completion of test, proceed directly to step 23.

22. Start step 23 exactly 4 minutes and 30 seconds after "Freon" injection was started.

23. Close valve between "Freon" reservoir and injection tube. Do not stop current to injection tube.

24. Let sampling system run for about 5 minutes after "Freon" injection is stopped. Then stop sample timer, sample pump, recorders, and "F-112" injection tube current.

25. Record data. Remove recorder charts and store for permanent record of test results.

---

* 0.6 minute is adequate time for an equilibrium sample to reach the detector in the SRP application.

26. Place test equipment on standby.

    a. Release nitrogen pressure in "Freon" reservoir.

    b. Set detector attenuators at 128. Do not turn detectors off.

    c. Set prepurified N_2 pressure at about 6 psig.

27. Calculate results of leak test.

28. If test results are unsatisfactory, the cause could be one or both of the reasons listed below.

    a. Test conditions too severe. The face velocity and/or moisture content of the carbon was too high during test. Dry the carbon for at least 3 or 4 days (to partially remove "F-112" adsorbed from previous test) and repeat the test at a lower face velocity. Excessive amounts of previously adsorbed "F-112" will create a high background concentration and may interfere with a repeat test. The carbon should be desorbed with hot air (about 50°C to 60°C).

    b. Leak path. Inspect carbon bed(s), mounting bolts, and gaskets for defective parts and faulty installation. If no defects can be found, repeat test of isolated sections of installation to locate leak(s).

       NOTE: When fewer carbon beds are tested, a smaller quantity of "F-112" must be injected into a lower air flow for consistent face velocity and upstream "F-112" concentration.

29. If the test was satisfactory, dismantle the test equipment and return the carbon bed(s) to normal service.

    NOTE: When the test equipment is disconnected, it is imperative that all detector prepurified nitrogen lines and instrument connections be plugged with clean tubing plugs and that all N_2 valves in the lines and on the detectors be closed. Plugs for these lines should be clearly identified and never used for any other lines. The prepurified N_2 regulator must also be kept clean. Other lines exposed to the weather should be plugged to keep out water and debris but these plugs must never be used for the prepurified N_2 lines.

APPENDIX C

"F-112" LEAK TEST DATA SHEET AND CALCULATION FORM

Date: ____________

Compartment: ________

I. "F-112"-Air Calibration

Downstream Detector	Upstream Detector
Identification No.: ______	Identification No.: ______
Standing current[1]: _____	Standing current[1]: _____
Oven temperature: _______	Oven temperature: _______

Calibration Sample[2]	Downstream Detector Response, RU @ 16X[3]		Upstream Detector Response, RU @ 16X[3]	
-3 (Background)[4]	________		________	
-2 (Background)	________		________	
-1 (Background)	________		________	
Average	________	(a)	________	(b)
1 ("F-112" & bkgd)[5]	________		________	
2	________		________	
3	________		________	
4	________		________	
5	________		________	
6	________		________	
Average	________	(c)	________	(d)

Relative calibration for "F-112" in air, $\frac{S_u}{S_d} = \frac{(d) - (b)}{(c) - (a)} =$ ______ (e)

---

[1] The standing current must be above 25 RU with attenuator at 128 (Varian-Aerograph Model 680 detectors) before calibration is conducted.

[2] Sample must be admitted simultaneously to both detectors for meaningful calibration of detectors. But calibration will occur when "F-112" peaks are consistent in size.

[3] The peak height should not exceed 25% of the adjusted standing current (see Appendix H, item 5).

[4] Any background peaks that occur on either detector must be recorded before calibration by the "F-112"-air method. However, the background peaks must be consistent in size and represent background during calibration. If background peaks are negative (cause recorder to move to left), a minus sign must be used and carried through all the calculations.

[5] The sample peak represents "F-112" injected for the calibration plus any background peak as measured in previous step.

Date: ______________

Compartment: __________

II. "F-112" Leak Test Data Sheet

Downstream Detector (Identification No. ______)

	Approx Setting	Exact Value
Standing current @ 128X(1)	25 plus	________
Oven temperature	75 to 100°C	________
Detector N_2 pressure	22 to 23 psig	________

Upstream Detector (Identification No. ________)

	Approx Setting	Exact Value
Standing current @ 128X(1)	25 plus	________
Oven temperature	75 to 100°C	________
Detector N_2 pressure	22 to 23 psig	________

Rotameter Readings (Air Flow)

Rotameter No.		Approx Setting(2)	Exact Value
1 (Downstream purge)		0.7 cfm	________
2 (Dilution air)	Upstream dilution system	50,000 cc/min	________ (3)
3 (Upstream sample)		100 cc/min	________ (3)
4 (Upstream purge)		0.7 cfm	________

---

(1) The standing current should be the same during the calibration and the leak test.

(2) Determine settings needed for particular application; flows listed are for SRP application.

(3) Consult rotameter calibration curve when applicable.

Date: ____________________

Compartment: ______________

II. "F-112" Leak Test Data Sheet (Continued)

Miscellaneous Data	Approx Setting	Exact Value
"F-112" reservoir pressure	(1)	
Ambient air temperature	-	
Compartment effluent air temperature at time of test	-	
Carbon bed air flow	(1)	
Sample interval time	50 sec.	
Attenuator setting:		
Downstream detector	16X(2)	
Upstream detector	16X(2)	

Peak Height During Test	Downstream Detector	Upstream Detector
Sample -2 (Background)	RU	RU
-1 (Background)		
+1(3)		
+2		(4)
+3		(4)
+4		(4)
+5		(4)
+6		(4)
+7		

---

(1) Determine desired value for particular test application.

(2) Different attenuator settings may be used for greater amplification of the tracer signal if desired. No adjustment in the efficiency equation is needed if the same attenuator settings are used during the calibration of the detectors. (See manufacturer's instruction manual for purpose and use of attenuator.)

(3) Sample +1 is the first sample to show "F-112" upstream of the test carbon bed. Samples before and after sample +1 are labeled in numerical order.

(4) Peak heights of about 35 to 50 RU on upstream detector are used for SRP applications.

Test Date: ____________

Compartment: ____________

III. Leakage Flow Calculation Sheet

A. Data Reduction

Downstream Detector

Sample	Downstream Recorder Units During Test	-	Background(1) (Avg of Sample -1 & -2)	=	(Column 1) Downstream RU Due to "F-112" Only
1		-		=	
2		-		=	
3		-		=	
4		-		=	
5		-		=	
6		-		=	

Upstream Detector

Sample	Upstream Recorder Units During Test	-	Background(1) (Avg of Sample -1 & -2)	=	(Column 2) Upstream RU Due to "F-112" Only
1		-		=	
2		-		=	
3		-		=	
4		-		=	
5		-		=	
6		-		=	

---

(1) See Appendix H, Note 5.

Test Date: ____________

Compartment: __________

B. "F-112" Adsorption Efficiency Equation

$$\text{"F-112" efficiency} = 100 \left\{ 1 - \left[\frac{\text{Downstream RU during test (Col 1)}^{(1)}}{\text{Upstream RU during test (Col 2)}^{(1)}} \times \frac{1}{\text{upstream dilution factor } (f_d)^{(2)}} \times \frac{S_u}{S_d} \text{ (Relative calibration for "F-112" in air)}^{(3)} \right] \right\}$$

---

(1) The "F-112" efficiency must be calculated for sample 1 through 6. Use the upstream and downstream recorder units during test (from column 1 and column 2) for corresponding sample numbers as shown in Section D.

(2) The upstream dilution factor (f_d) equals the flow through rotameter 2 divided by the flow through rotameter 3. This ratio must be determined for the actual test conditions and used in the equation. If the ratio changed during the test, the new ratio must be determined and used for the calculation of "F-112" efficiency of the appropriate sample in which the dilution ratio changed.

(3) The relative calibration for "F-112" in air $= \frac{S_u}{S_d} = \frac{C_u}{C_d} \times f_c$ as explained in Appendix D. The procedure and calculations are based on detector calibration with "F-112" in air (S_u/S_d). However, calibration by "F-112"-hexane method is possible by following details given in Appendix D.

C. Substitute Values into Equation of Section III B

$$\text{"F-112" efficiency} = 100 \left[1 - \frac{\text{Downstream RU during test}}{\text{Upstream RU during test}} \times \frac{1}{f_d} \times \frac{S_u}{S_d} \right]$$

$$\text{"F-112" efficiency} = 100 \left[1 - \frac{\text{D/S}^{(1)}\text{ RU}}{\text{U/S}^{(1)}\text{ RU}} \times \frac{1}{f_d} \times \frac{S_u}{S_d} \right]$$

$$\text{"F-112" efficiency} = 100 \left[1 - \frac{\text{D/S}^{(1)}\text{ RU (Col 1)}}{\text{U/S}^{(1)}\text{ RU (Col 2)}} \times \underline{\hspace{3cm}}\text{(f)} \right]$$

---

(1) D/S = downstream; U/S = upstream.

Test Date: ____________

Compartment: __________

D. Calculate "F-112" Efficiency for Each Sample of Section III A

$$\text{"F-112" efficiency} = 100\left[1 - \frac{\text{D/S RU (Column 1)}}{\text{U/S RU (Column 2)}} \times \underline{\hspace{3cm}}\,(f)\right]$$

Sample	$100\left[1 - \frac{\text{D/S RU (Col 1)}}{\text{U/S RU (Col 2)}} \times \underline{\hspace{2cm}}\,(f)\right]$		(Column 3) "F-112" Efficiency
1	100(1- ______________ x ____________)	=	________________
2	100(1- ______________ x ____________)	=	________________
3	100(1- ______________ x ____________)	=	________________
4	100(1- ______________ x ____________)	=	________________
5	100(1- ______________ x ____________)	=	________________
6	100(1- ______________ x ____________)	=	________________

Test Date: __________

Compartment: __________

E. Plot Curve of "F-112" Efficiency

Plot the percent "F-112" efficiency vs time on graph paper for each of the samples.[1]

Sample	Time After "F-112" Enters Carbon Bed, min.		(Column 3) "F-112" Efficiency
1[2]	0.6	vs	______________
2	1.4	vs	______________
3	2.3	vs	______________
4	3.1	vs	______________
5	3.9	vs	______________
6	4.8	vs	______________

---

[1] The leakage flow can be determined by drawing a smooth curve through the data points and extrapolating the curve to time zero. The value of the "F-112" penetration (100% - efficiency) at time zero equals the leakage flow. For the most accurate results, the "F-112" penetration (due to desorption at about 4 minutes after the "F-112" enters the carbon beds) should not be more than the value of the maximum acceptable leakage flow. For example, if maximum acceptable leakage equals 0.1%, then desorption should not exceed 0.1% during first 4 minutes of test.

[2] Sample 1 may not be an accurate measure of the "F-112" efficiency, but it is an excellent indication of leakage flow by the presence or absence of "F-112" downstream of the carbon bed(s).

APPENDIX D

RELATIVE CALIBRATION - APPLICATION AND TECHNIQUE

As presented and explained in DP-1053, the equation for determining the "F-112" efficiency of a given sample is:

$$\epsilon = 100 \left[1 - \left(\frac{R_d}{R_u} \frac{1}{f_d} \frac{C_u}{C_d} f_c \right) \right] \qquad \text{(D-1)}$$

where

ϵ = "F-112" adsorption efficiency, %

R_d = downstream detector response for "F-112" during leak test, recorder units (RU)

R_u = upstream detector response for "F-112" during leak test, recorder units (RU)

f_d = dilution factor of sample from upstream of carbon bed(s) to upstream detector, dimensionless constant

C_u = upstream detector response to standard calibration solution, recorder units per microliter (RU/μl)

C_d = downstream detector response to standard calibration solution, recorder units per microliter (RU/μl)

f_c = calibration conversion factor, dimensionless constant

$\frac{C_u}{C_d} f_c$ = relative calibration for "F-112" in air

Thus,

$$\frac{C_u}{C_d} f_c = \frac{S_u}{S_d} \qquad \text{(D-2)}$$

where

S_u/S_d = relative calibration for "F-112" in air; equals upstream detector response divided by the downstream detector response (with same concentration of "F-112" in air), recorder units per recorder units

The object of the relative calibration is to determine the response of each detector to the same concentration of "F-112" in air (S_u/S_d). Relative calibration of the detectors must be accomplished at the time of each leak test for the best accuracy in measurement of leakage flow. Two methods are available for determining the relative calibration at the test site: the "F-112" in air and the "F-112" in hexane. Details of the techniques are presented below in parts A and B. The "F-112" in air method is preferred because it yields S_u/S_d directly.

PART A

Technique for Calibration of Detectors by "F-112" in Air

A method of determining the relative calibration is to inject the same concentration of "F-112" in air to both detectors and measure the response of each detector. The relative calibration of "F-112" in air (S_u/S_d) then equals the upstream detector response divided by the downstream detector response. However, the "F-112"-air concentration must be low enough to permit the detectors to respond in the linear range (peak height less than 25% of the adjusted standing current). Thus the "F-112" concentration in air must be less than about 100 ppb by volume. The calibration device is diagramed in Figure 17 and is designed to mix a small quantity of "F-112" with air. The device operates by drawing air at high velocity (~60 ft/sec) through the 1/4-inch-diameter pipe which reduces the pressure in the pipe and draws a small quantity of "F-112" into the system. Capillary tubing is used to restrict the "F-112" flow, because very little "F-112" is needed to obtain the desired concentration.

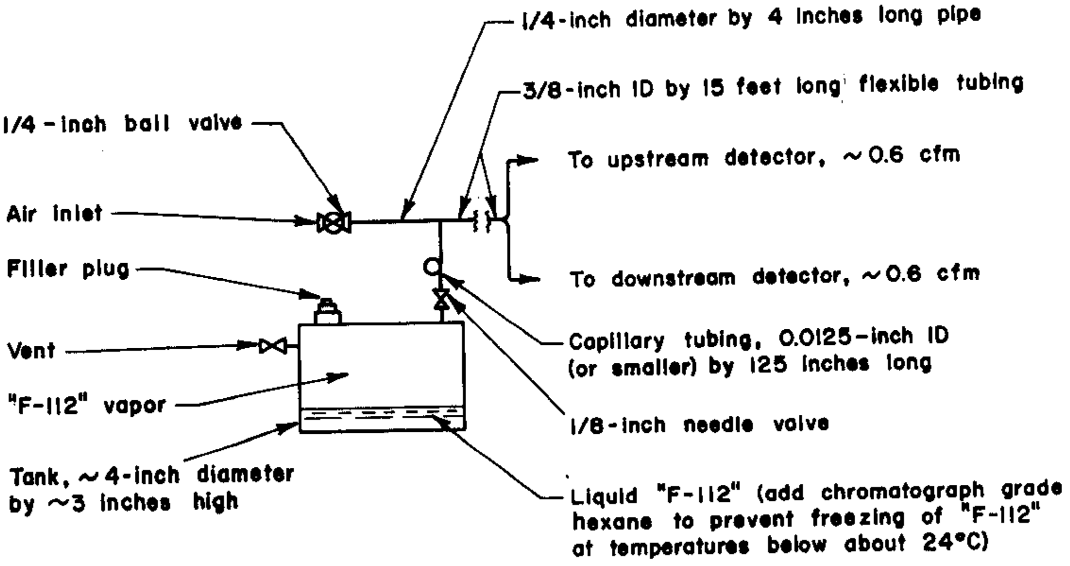


FIG. 17 DEVICE FOR CALIBRATION OF DETECTORS WITH "F-112"-AIR MIXTURE

After the "F-112" is mixed with the air, the flow is split at the Leak Test apparatus for simultaneous passage through each detector. It is not necessary to know the concentration of the "F-112"-air mixture, but it is necessary that the same concentration occurs simultaneously at each detector. Ideal conditions will exist when the "F-112" concentration does not vary with time. Long tubing between the calibrator and flow splitter is advantageous in damping minor changes in "F-112" concentration.

To perform the "F-112"-air calibration, locate the calibrator upwind of the sample pump so that the pump discharge will not be recycled. Attach an adapter to the sample entry connections of the Leak Test apparatus to split the flow from the calibrator for admission to both the upstream and downstream sample lines. Close the dilution air rotameter valve and sample loop bypass valve to prevent dilution of the sample for the upstream detector. Start the sample pump and adjust the flow through the system to about 1.2 to 1.4 cfm (total) as indicated by the purge flow rotameters. With the calibrator in normal operation, the ball valve should be wide open and the needle valve open about 1/8 turn. Further adjustments of "F-112" concentration may be necessary and can be accomplished by readjusting the ball and needle valves. The "F-112" reservoir should be vented before attempting to calibrate the detectors, because changes in temperature will affect the vapor pressure of the "F-112", which can affect the pressure in the reservoir. Smaller and longer capillary tubing may be used to reduce the "F-112" concentration.

If the calibrator is used at temperatures below the melting point of "F-112" (about 24°C), chromatograph-grade hexane may be added to the "F-112" to prevent it from freezing and possibly plugging the capillary tubing. The hexane will not interfere with the calibration, because the electron-capture type detector is not sensitive to hexane.

PART B

Technique for Calibration of Detectors by "F-112" - Hexane Solution

The "F-112" in hexane method of detector calibration yields the terms C_u/C_d of Equation D-2. This is accomplished by injecting a known volume of hexane solution (containing a small amount of "F-112", quantity known or unknown) into each detector and measuring the detector response per unit volume of injected solution (C_u and C_d). However, to determine the relative calibration of the two detectors for an air sample, the calibration conversion factor (f_c) must be known. f_c is a dimensionless constant for any two detectors used in the same test positions.

f_c may be determined by evaluating the quantities of Equation D-2 or by Equation D-3 given below.

$$f_c = \frac{\text{Volume of gas sample injected in upstream detector}}{\text{Volume of gas sample injected in downstream detector}} \qquad \text{(D-3)}$$

Equation D-3 can be easily evaluated if the detector manufacturer provides certification of the volume of sample injected to each detector. Notice that the factor (f_c) will change depending on which detector is used for upstream position and which is used for the downstream position.

The "F-112" in hexane method of calibration has the advantage of being able to calibrate the detectors on an absolute basis at the test site if the absolute concentration of "F-112" in hexane is known. Such standard calibration solutions may be purchased from commercial laboratories.

The detectors are calibrated by injecting a known volume of a standard solution of "F-112" in hexane*. The volume can be accurately measured with a microliter syringe. The use of the microliter syringe for this purpose is outlined in the Detector Instruction Manual and in the literature supplied with the syringe. (A care and maintenance guide is also supplied with the syringe.) Below is outlined a satisfactory calibration method. Observe all precautions. Each person calibrating the detector must develop his technique so that he can accurately reproduce sample injections.

Grasping only the syringe flange and plunger, pump out the air into the calibration solution and then overfill the syringe. Hold syringe vertically (needle up) and expel solution until approximately the desired quantity remains in the barrel. Lower the plunger until air appears above solution. Determine the exact volume of solution in syringe. Subtract any air bubbles appearing in the solution in the syringe. With the plunger in the same position (air above solution), guide the syringe needle through the septum and into the detector as far as possible. Quickly inject the solution and quickly withdraw the needle. Hold the syringe vertical (needle up) and withdraw the plunger far enough to be sure no more solution remains in the needle. Determine the exact volume of solution remaining in the syringe body and subtract it from the volume before injection to determine the exact volume of solution injected. Record the volume injected and the detector response to "F-112" only, in recorder units. Repeat the above procedure until enough data points are obtained to plot a reliable calibration curve.

---

* The calibration solution should contain about 0.2×10^{-10} gram of "F-112" per microliter of chromatograph-grade hexane.

Note the following precautions for good calibration technique.

1. Smoking is not permitted when calibrating detectors because the "F-112"-hexane solution is flammable and smoke may contaminate the solution.

2. Do not use lubrication in the syringe (grease may split barrel).

3. Do not touch the plunger. Perspiration and soil from one's fingers may cause the plunger to stick or freeze in the barrel.

4. Do not touch the main section of the barrel because the heat from the hand will affect the accuracy of the measurement.

5. Insert the needle into the detector as far as possible.

6. Always expel the solution <u>quickly</u>.

7. Inject at least one sample into detector to condition column before attempting to calibrate.

8. Change rubber septum on injection part of detector after every 30 injections. This must be done to prevent leakage through the needle holes in the septum.

9. When inserting the needle through the septum, hold the plunger from moving by applying pressure to the side of the plunger. Otherwise, the pressure in the injection chamber (about 22 psig) will force the plunger and solution out of the back end of the syringe.

APPENDIX E

TECHNIQUE FOR READING CHROMATOGRAMS

Typical chromatograms are illustrated in Figure 18. Traces A and B are ideal for "F-112" in air and hexane. Traces C and D are typical cases for "F-112" in hexane. Trace C differs from the ideal because there is inadequate separation between the hexane and "F-112" peaks. The problem is probably due to high oven temperature. A lower oven temperature should provide adequate separation of peaks. The detector should not be calibrated if moderate overlapping of peaks occurs. However, minor overlapping is permitted if the apparent peak height is corrected by one-half of the base drift (BD) as shown by trace C.

Trace D of Figure 18 differs from the ideal trace for "F-112" in hexane because the detector cell is dirty. The condition need not be corrected until the standing current is less than 25 recorder units (RU) at 128 attenuator setting. However, the apparent peak height should be corrected by one-half of the base drift as shown by trace D.

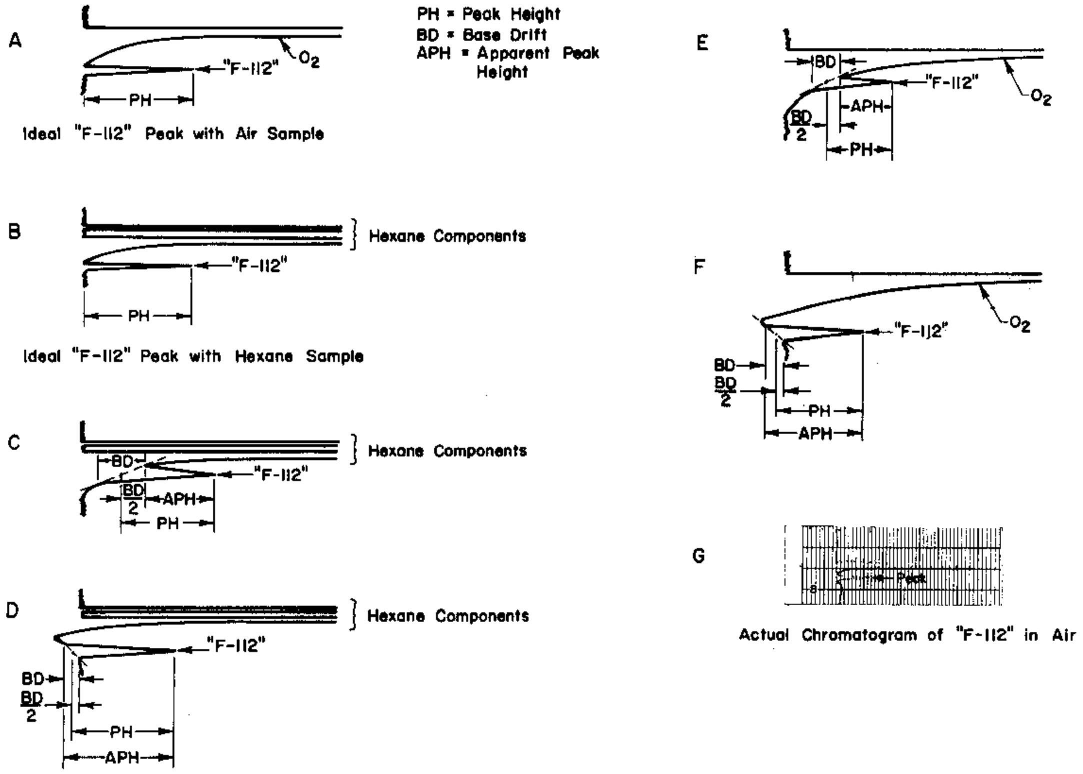


FIG. 18 TYPICAL CHROMATOGRAMS (WITH "F-112" PEAKS)

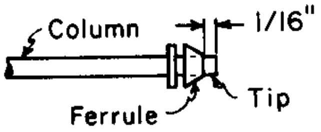
Traces E and F of Figure 18 illustrate (for "F-112" in air) the same conditions as traces C and D. Trace G is an actual chromatograph of an "F-112" air sample with the "F-112" peak height labeled.

APPENDIX F

TROUBLESHOOTING "F-112" DETECTOR
(GAS CHROMATOGRAPH WITH ELECTRON CAPTURE CELL)

Consult manufacturer's instruction manual for maintenance problems. Before the chromatograph is dismantled for maintenance, the electron capture detector cell should be removed and stored in a sealed container. The detector cell contains a fixed radioactive tritium source in a fragile glass tube. The chromatograph column should also be sealed with an end plug.

Troubleshooting aids are listed below to help locate and correct the cause of common problems. The test group should be familiar with the aids listed, because many of the problems are caused by improper installation or operation of equipment or failure to keep the system clean.

Possible Causes	Correction
I. Low or no standing current.	
1. Cell foil dirty.	1. Install newly cleaned cell and have old cell cleaned.
2. Chromatograph column grounded to base of cell. Confirm by loosening column nut at cell. Recorder needle should move as contact is made and broken. (For Model 680 detector only)	2. Install chromatograph column with tip length between edge of ferrule and end of column not longer than 1/16 inch. 
3. No prepurified nitrogen flow.	3. Start nitrogen flow. Be sure all valves in system and on back of instruments are open.
4. Nitrogen supply is of poor quality (contains too many impurities).	4. Use only prepurified grade nitrogen and when changing cylinders, be sure new supply is as good as the old supply (see procedure).
5. Dirty nitrogen supply lines.	5. Purge lines with prepurified nitrogen and/or install new clean nitrogen supply lines.
6. Detector cell electrodes not making contact.	6. Install cell so that it seats firmly in receptacle.

7. Detector cell assembled backward.	7. Assemble cell properly, see instruction manual. (Anode lead from base of cell goes to electrometer, cathode lead from main body of cell goes to -90 volt power supply.)
8. Cell foil worn out (insufficient radioactivity on foil).	8. Replace with new detector cell assembly.
9. Cell not connected to electrometer circuit.	9. Check leads to cell.

II. Inconsistent peak heights for a constant "Freon" concentration when using the gas sampling valve.

1. Faulty operation of gas sampling valve.	1. See instruction manual for proper operation.
a. Loose linkage between shaft and solenoid (nonuniform travel of shaft).	a. Repair linkage but be be sure it is precisely adjusted for travel and position.
b. Dirt in valve or insufficient lubricant causing sticking of shaft at critical point.	b. Clean and lubricate. Clean with alcohol and lubricate with high vacuum silicone grease. CAUTION: Use a very thin film of grease on the O-rings only. Any excessive grease will plug the miniature holes in the valve body and also adsorb and retain "F-112" and impurities that will gradually desorb and cause a background signal. Excessive grease can also contaminate the column and cell.
2. O-rings of gas sample valve leaking.	2. Install new O-rings ("Viton"* or equal) and/or lubricate O-rings with high vacuum silicone grease. See caution of item II-1b.

---

* Du Pont's trademark for fluoroelastomer.

III. Standing current is quickly reduced (within two weeks after installing clean cell).

1. Detector cell has not had enough time to warm up after installing new cell or after turning on oven and nitrogen.	1. Allow at least one day for system to warm up and purge clean before taking standing current of newly installed cell.
2. Nitrogen supply contains impurities.	2. Use clean prepurified nitrogen supply.
3. Impurities on nitrogen supply lines, gas sampling valve, and chromatograph column from nitrogen supply containing excessive impurities or from impurities reaching the system from dirty line plugs or exposure to air.	3. Purge with clean prepurified nitrogen and/or replace components where possible.
4. Column bleeding its components into cell.	4. Replace column or recondition column by purging with about 75 cc/min of prepurified nitrogen (regulator pressure set at 22 to 23 psig) and heating at 200°C for 24 hours. This can be done with the column in the oven if the thermostat is readjusted for 200°C. CAUTION: When the column is reconditioned or the oven heated to 200°C or above, the detector cell must be removed to prevent its contamination by impurities from the column or destruction from the heat.

IV. Signal noise (recorder will not trace steady line).

1. Shorting of leads to detector cell or to electrometer.	1. Eliminate shorting.
2. Shorting of chromatograph column to detector cell base.	2. Column tip from ferrule to end of column should be no longer than 1/16 inch. (See item I-2.)

3.	Electronic circuit faulty.	3.	Repair circuit.

V. Recorder will not trace on chart.

1. Same as items IV-1, 2, and 3.

VI. Low sensitivity for detection of "F-112".

1.	Detector cell assembled backward.	1.	See item I-7.
2.	Cell foil worn out (insufficient radioactivity on foil).	2.	Replace with new detector cell assembly.
3.	Sample loop on gas sample valve loose or too small.	3.	Repair or change.
4.	Gas sample valve operating improperly (full sample not reaching chromatograph column).	4.	Repair and adjust position of shaft with respect to holes in valve body.
5.	Incorrect or poorly functioning chromatograph column.	5.	Replace or recondition as necessary.
6.	Improper voltage on cell.	6.	See manufacturer's instruction manual. The sensitivity may be changed by changing the applied cell voltage.

VII. Other peaks interfere with "F-112" peak on chromatogram.

1.	Background concentration from previous tests.	1.	a. If peak is less than 12½% of standing current, treat as background concentration. b. If peak is greater than 25% of standing current, desorb carbon bed of "F-112" before testing (see note 5 of Appendix H).

2. Impurity on apparatus tubing (test by injecting air directly from atmosphere to sample loop).	2. Desorb and clean tubing with nonhalogenated solvent and purge with clean air.
3. Impurity in atmosphere.	3. Use different chromatograph column to separate "F-112" from interfering with impurity peak.

APPENDIX G

TROUBLESHOOTING "F-112" LEAK TEST APPARATUS

I. "Freon" injection tube will not vaporize "F-112".

Possible Causes	Correction
1. No "F-112" reaching tube; possibly empty "F-112" reservoir or lines plugged with trash or frozen "F-112".	1. Clear lines and fill reservoir.
2. Low or no current flow through heater tube.	2. a. No power supply. Broken wire or tube. Resistance across terminals of plug should be about 7.5 ohms for SRL design. b. Low voltage at injection tube. Voltage should be about 115 volts at injection tube plug when test station is in normal operation.
3. Wet insulation on injection tube which causes absorption of heat.	3. Dry out insulation by heating tube.
4. Pressure on "F-112" reservoir too high.	4. The injection tube is not designed to heat the "F-112" with more than ~75 psig on the reservoir.

II. Rotameter ball does not move freely. (<u>Do not conduct "F-112" Leak Test with this condition</u>!)

Possible Causes	Correction
1. Trash or dirt in rotameter tube.	1. Clean with alcohol. Be sure rotameter is resealed in system. A leak could invalidate the test.
2. Water in rotameters.	2. Start air pump to draw fresh air through the rotameters to dry them out.

APPENDIX H

GENERAL PRECAUTIONS FOR "F-112" LEAK TEST

The following items could produce error in determining leakage flow in carbon beds without obvious indication of malfunction. However, good technique and careful operation will prevent these errors.

1. The detectors should be calibrated at the same sample loop pressure that will occur during the actual leak test. If the sample loop pressures change, the detector calibration will change, because the detectors are sensitive to the total mass injected. The existing design has essentially eliminated this problem, because the discharge of each sample valve is connected to a common header which is connected to a low head sample pump.

2. The rotameter calibration must be valid during operation in the test apparatus. Oscillating air movement as created by a positive displacement pump could yield a different rotameter calibration than that for uniform flow. Use of a high speed centrifugal pump eliminated this problem.

3. System leakage must not occur because it could cause unknown dilution of the sample. All joints must be leakage free. Periodic testing is recommended by pressurizing the apparatus and using a soap solution to test for leaks.

4. The "F-112" calibration of the detectors must be accurate. The accuracy of the "F-112"-hexane calibration may be checked by plotting a curve of the sample size versus the detector response. All valid calibration points should fall near a smooth curve drawn about the points. For detector responses of less than 15 to 20% of the adjusted standing current (see note 5), the "F-112"-hexane calibration curve should be a straight line passing through zero. (The linearity limit for "F-112" in air occurs at about 25% of the adjusted standing current.)

5. Accuracy of the test is reduced if the peak height during the leak test exceeds about 25% of the adjusted standing current. (e.g., if the standing current is 30 RU at 128 attenuator setting, the adjusted standing current at 16 attenuator setting

is 128/16 x 30 = 240 RU. Then the peak height during the test should not exceed 25% of 240 or 60 RU.) The maximum recommended peak height can be composed of both background signal and "F-112". However, it is recommended that the background not exceed 12½% of the adjusted standing current at 16 attenuator setting. This value equals the value of the standing current when measured at 128. (e.g., 12½% x 128/16 x 30 RU = 30 RU.) Thus 12½% of the adjusted standing current is available to measure "F-112" concentration.

6. The equipment operation should be consistent from one test to the next. Any changes should be reflected on the data sheet and may indicate malfunctioning of equipment. The causes of inconsistency should be investigated.

APPENDIX I

CHROMATOGRAPH COLUMN FOR "F-112" DETECTOR - SPECIFICATIONS AND PROCEDURE FOR MODIFICATION

The chromatograph column is 5 ft long by 1/8-inch OD (3/32-inch ID) and originally packed with 30% SF-96 on 45/60 mesh "Chromosorb P"*. The column must be modified by baking in an oven at 350°C for 16 hours to remove most of the stationary phase (SF-96) from the "Chromosorb". Prepurified nitrogen at a flow of about 75 cc/min is purged through the column during the baking operation.

The chromatograph column may be purchased from the manufacturer** of the "F-112" detector although the purchaser may have to make the necessary modification. The purchaser may modify columns in the detector oven by following the procedure listed below.

1. Remove the electron capture detector cell from the oven of detector. If cell is not removed, high oven heat will damage the tritiated foil.

2. Install the 5-ft-long chromatograph column in the oven.

3. Set the prepurified nitrogen flow rate through the column at about 75 cc/min (about 23 psig N_2 pressure).

4. Adjust oven thermostat for 350°C oven temperature.

5. Heat and purge the column for 16 hours, then cool oven and column and reset thermostat to 95°C oven temperature. Satisfactory conditioning is evidenced by a blue powder eluded from the column.

6. To recondition† a previously conditioned chromatograph column, repeat step 1, 2, and 3. Then set oven temperature at 200°C and heat and purge for about 24 hours. Readjust system when reconditioning is complete.

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

** Varian-Aerograph, Walnut Creek, California.

† A chromatograph column may need reconditioning if it does not produce sharp "F-112" peaks or if it causes a gradual reduction in the standing current of the electron capture detector cell.

APPENDIX J

EQUIPMENT SPECIFICATIONS FOR "F-112" LEAK TEST

The following equipment specifications are for major components used in the SRL fabricated apparatus. Comparable equipment of different manufacturers should be satisfactory.

"F-112" Detection System (2 required)

The "F-112" detection system consists of a gas chromatograph with electron capture detector cell, solenoid operated gas sampling valve, and a suitable recorder. The SRL apparatus utilized an Aerograph Pestilyzer Model 680* gas chromatograph which has been discontinued by the manufacturer. Any comparable gas chromatograph such as the Aerograph Model A-600* may also be used. However, relative small chromatographs are desirable from the standpoint of portability.

Sampling Timer (1 required)

Automatic reset timer for 115 volts, 10-ampere service. When switched on, the timer must operate solenoids (on above described detectors) for periods of 5 seconds at adjustable intervals of 15 seconds to 5 minutes. After each operation of the solenoid, the timer must reset and continue to operate until switched off.

Upstream Sample Rotameter (1 required)

Brooks Flow Meter model 2-1110-6-MB-VH, needle valve on outlet, 148 cc/min maximum air flow, 316 stainless steel construction, "Teflon"** packing.

Dilution Rotameter (1 required)

Brooks Flow Meter model 2-1110-6-MB-VH, needle valve on outlet, 50,000 cc/min air flow, brass construction, "Teflon" packing.

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* Product of Varian-Aerograph, Walnut Creek, California.
** Du Pont's trademark for its fluorocarbon plastic.

Upstream and Downstream Purge Rotameters (2 required)

Brooks Flow Meter model 5-1350-VB, tube number 5-65A; 0.7 cfm air flow, needle valve on outlet, brass construction, "Teflon" packing.

Sample Pump (1 required)

Centrifugal air pump, hand type vacuum cleaner, Ideal Cleaner Catalog No. 22-110, heavy duty model with 1-1/3 hp motor, 52-inch H_2O static pressure, 115 volts AC.

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