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IODINE TRANSPORT AND DEPOSITION IN A HIGH-TEMPERATURE, HELIUM LOOP FIRST TEST SERIES

F. H. Neill
D. M. Eissenberg
D. L. Gray

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F. H. Neill
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AUGUST 1965

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FORWARD

An integrated analytical and experimental program has been undertaken to investigate the transport and deposition of fission products. The program has as its objective the development of sufficient experimental information and a suitable mathematical model to permit the estimation of the contamination to be expected on primary coolant system components of a gas-cooled reactor. The problem is being approached initially by studying the deposition of fission products, both singly and in combination, in well defined geometries in out-of-pile loops. Subsequently, as the fundamental relationships of surface adsorption, desorption, decay, particle size and compound formation on regulating the transport and deposition of fission products are better understood, the program will be expanded to in-pile studies where the effects of radiation, release mechanisms, effect of short lived fission products can be evaluated.

Analytical studies, based on the heat-mass analogy, have been reported previously.[1,2,3,4,5] The first series of loop experiments, utilizing lightly irradiated fuel-graphite sources, was conducted in a loop at Battelle Memorial Institute (BMI-FPD loop) and have been reported.[1] The present report describes the first series of 21 tests in a fission-product deposition loop at the Oak Ridge National Laboratory (ORNL-FPD loop) which is designed to inject selective fission products into a flowing helium stream.

IODINE TRANSPORT AND DEPOSITION IN A HIGH-TEMPERATURE, HELIUM LOOP
FIRST TEST SERIES

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ABSTRACT

The results of an investigation of the transport and deposition of trace amounts of iodine in a high-temperature flowing helium stream are reported. All tests were conducted in the turbulent flow range with the deposition surface in the temperature range of 300 to 600°F. Four surface materials were studied — copper, silver, carbon steel, and stainless steel. Small-diameter silver wires were arranged in arrays perpendicular to the coolant stream to study the applicability of the heat-mass analogy to fission-product deposition studies.

The investigation indicated that the amount of iodine deposited per unit amount injected varied with different materials. A silver-plated surface had the highest deposition and the stainless steel surface had the lowest amount deposited. Desorption, under similar test conditions, was greater on the silver-plated surface than on the stainless steel surface. However, other tests indicated that desorption may not be uniform from a stainless steel surface maintained at isothermal conditions.

The silver wire tests indicated that the heat-mass analogy gave reasonable correlations with experimental results when calculating the ratio of deposition for various diameter wires.

INTRODUCTION

In this phase of the fission-product transport and deposition program, iodine in a liquid solvent was injected into a turbulent flowing helium stream and transported to a deposition section of the loop which contained a well-defined geometry maintained under isothermal conditions. The deposition of iodine in the test section was investigated by adding a radioactive iodine tracer to the injection solution.

The convective transport of molecular-sized fission products in the gas stream is analogous to that of heat. In the analysis of the experimental data from these tests the appropriate heat transport equations for

the various flow geometries were utilized. The interaction of the fission products with surfaces will differ, however, from that of heat principally because of four phenomena:

1. The probability of initial sticking to the surface may be less than unity.
2. The deposited activity may desorb with time.
3. The deposited activity will decay with time.
4. The surface reaction with the transported particle.

Each of these effects can vary with the nature of the fission product and of the surface. Such factors as the chemical and physical form of the fission product in the gas phase, the amount previously adsorbed on the surface, the amount of other contaminants on the surface, the surface temperature and roughness etc. can affect the deposition in a way not accounted for by the heat-mass transfer analogy.

The evaluation of the first of those phenomena in turbulent flow as well as the verification of the applicability of the heat-mass transfer analogy to turbulent flow is being carried out in the ORNL-FPD loop.

In the initial phase of the program, deposition of iodine under contr lled conditions, using simplified flow geometries, is being studied. Equations derived from a heat-mass analogy, were used to correlate the experimental data.

It is the purpose of this report to discuss the results from the first series of experiments in the fission product deposition (ORNL-FPD) loop facility which are characterized by the use of a source containing iodine with trace amounts of ^{131}I dissolved in a liquid carrier which was injected into an isothermal helium stream through a pitot tube injector.

DESCRIPTION OF FPD LOOP FACILITY

The FPD loop facility shown in Fig. 1 consists of: the high-pressure, high-temperature loop; a gas-addition system; a vent system; and an auxiliary cooling water circuit. The main loop includes a regenerative (HECT-II) compressor,[6] a pneumatically operated flow control valve, clamshell heaters, an orifice flowmeter, a fission product source injector,

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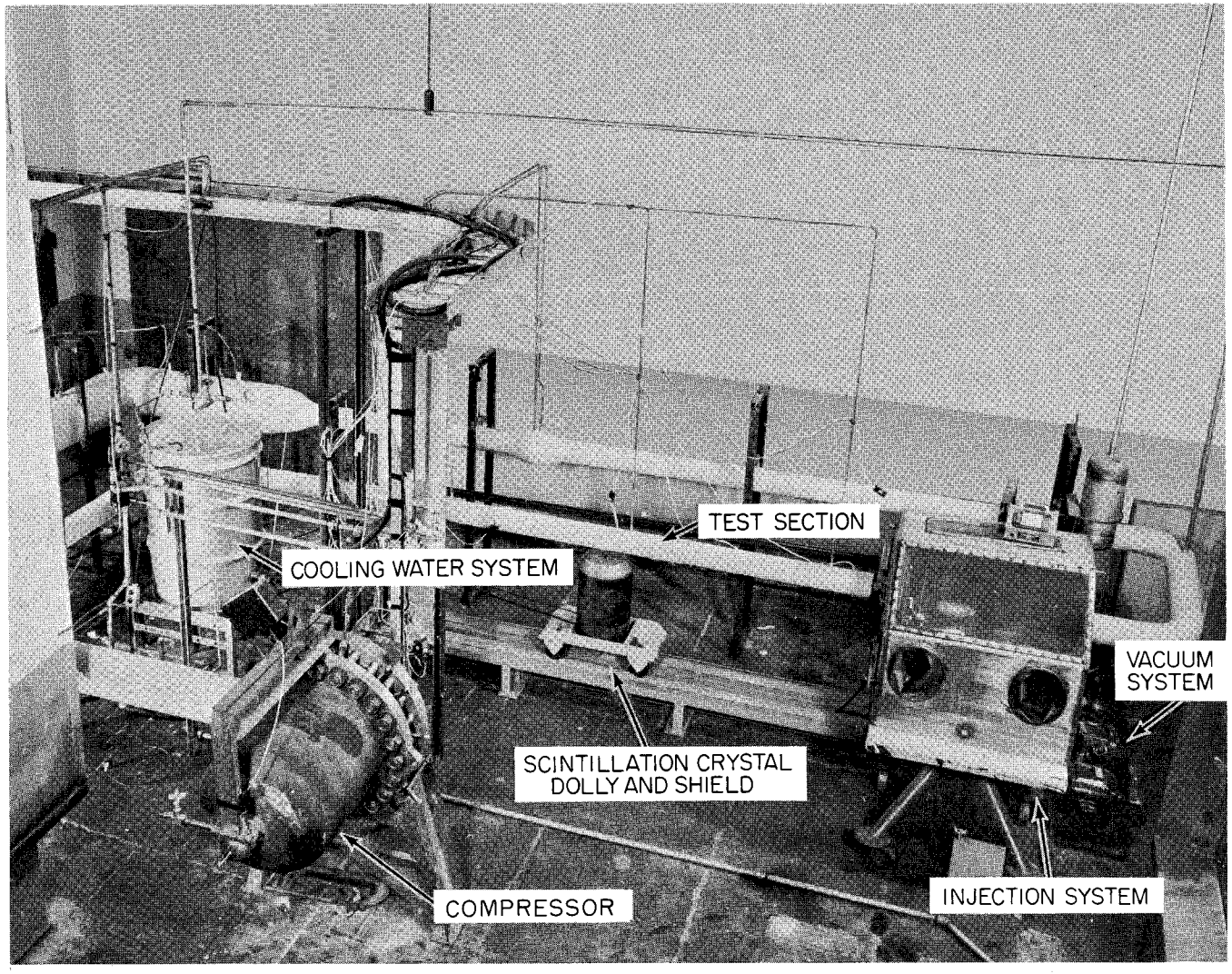

Fig. 1. Overall View of ORNL-FPD Facility.

and an 8-ft long deposition section. In the initial series of experiments, deposition took place directly on the pipe wall of the test section. In later experiments a liner, inserted into the deposition section and extending into the permanent loop piping, permitted the deposition surface to be extended from eight feet to approximately 12-ft-long.

The facility is completely self-contained with respect to utility services in order to eliminate the possibility of back flow of radioactive materials and contamination of normal building services. Other pertinent loop specifications are given in Table 1.

Table 1. ORNL-FPD Loop Specifications

Coolant	Helium
Design Pressure	400 psig
Design Temperature	600°F
Pipe Size	1 1/2-in. Sch. 40
Pipe Materials	Type 304 Stainless Steel
Heater Capacity	15 kw
Total Length	Approx. 70 ft
Total Surface Area	Approx. 26 ft^2 (Not Including Compressor)
Fission Product Detection System	Single Channel Analyzer with NaI crystal

The radiation monitoring equipment, suitably shielded from the source and the remainder of the loop piping, is mounted on tracks beneath the deposition section such that it can traverse the length of the test section.

A small, positive-displacement, piston-type pump is used to inject the liquid, containing trace amounts of radioactive iodine, into the helium gas stream at a controlled rate.

EXPERIMENTAL PROCEDURE

All tests in this series of experiments used iodine with ^{131}I tracer as the depositing species. The source consisted of a solution of approximately 5 mg of iodine, containing approximately 1 mc/mg of ^{131}I, dissolved in from 5 to 10 ml of either benzene (experiments 1 through 8) or cyclohexane (experiments 9 through 21).* The solution was injected, generally over a 1 1/2 to 2 hr period, through a pitot tube into the main loop near

*See Appendix B.

the center line of the gas stream. The loop coolant was maintained at nearly isothermal conditions during the injection and scan period of each run. The loop pressures, temperatures, and other pertinent operating conditions for each of the experiments are tabulated in Table 2. When the injected solution came in contact with the high-temperature helium gas, the iodine and the solvent vaporized and/or atomized, releasing the iodine which was carried by convection into the deposition section. Following run 15, a full-flow silver filter was installed upstream of the point of injection to prevent recirculation of iodine remaining in the gas stream after a complete loop cycle. Measurements made to determine its effectiveness are discussed in Appendix A.

During and after injection, the deposition section of the loop was scanned with a gamma-ray scintillation type detector suitably enclosed in a lead shield with a slit so located that the detector could "see" a one-inch long length of pipe. The detector signal was read-out on a single-channel analyzer. Normally activity measurements were made at 6-in. intervals along the test section. The scan measurements were calibrated by sectioning the deposition section after several runs and obtaining a radiochemical analysis. On some runs desorption from the test surface was studied qualitatively by continuing to scan after the source injection was complete. The effect of surface temperature on desorption was noted in this manner by increasing the loop temperature in known increments above the adsorption temperature.

ANALYSIS OF EXPERIMENTAL DATA

The amounts of the ^{131}I tracer which deposited at the various axial positions along the test section were obtained from scan counts by subtracting the background reading* (taken just before the start of a run) and by adjusting the resulting counts for the radioactive decay since the start of the run. The adjusted counts were related to deposited activity by comparisons with the radiochemical analyses of sections of the same or a similar test piece. The resulting calculated deposited activities were plotted as a function of axial position for each scan taken during and following the run. These deposition curves formed the basis of the qualitative

*Normally negligible - in the range of 2 to 4 c/s.

Table 2.
ORNL-FPD Loop
Run Summary

	Run Number	2	3	5	6	7	8	9	10	11	12	13	14	16	17	18	19	20	21
	Date	7/15	7/28	8/14	8/25	9/15	9/22	9/29	10/6	1964 10/8	10/9	10/21	11/3	11/17	12/1	12/7	12/11	12/29	1/12/65
Source Concentration	mc/ml	.043	.155	.187	.420	.292	.310	.285	.330	.283	.238	.288	.172	.336	.360	.398	.534	.645	.550
Total Injection	mc	.861	3.57	3.74	7.14	5.96	6.26	17.62	6.66	5.77	4.64	5.93	3.34	3.97	3.60	4.86	5.55	6.77	0.55
Coolant Temperature	°F	600	600	600	600	300	300	450/600	400	400	400	400	400	400	400	400	400	400	400
Coolant Velocity	cm/sec	2359	-	1996	5246	1774	1753	5498	1310	1310	1310	1286	1286	1328	1328	1730	835	1730	925
System Pressure	psig	300	300	300	300	300	300	300	300	300	300	300	300	300	300	300	300	300	300
Deposition Tube		Cu-P	S.S.	S.S.	S.S.	S.S.	C.S.	S.S.	S.S.	S.S.	S.S.	Ag-P	Ag-P	S.S.	S.S.	C.S.	Ag-W	Cu-P	Ag-W
Coefficient of Diffusion	cm^2/sec	.0569	-	.0569	.0569	.0325	.0325	.0569	.0402	.0402	.0402	.0402	.0402	.0402	.0402	.0402	.0402	.0402	.0479
Mass Transfer Coefficient	cm/sec	4.81	-	4.19	9.37	3.39	3.35	9.63	.055	.055	.055	2.71	2.71	2.77	2.77	3.54	1.76	3.54	1.97
Reynolds Number	Re	32500	-	27500	70500	43900	43400	79500	26400	26400	26400	26000	26000	27000	27000	30300	25500	30300	24200
Schmidt Number	Sc	3.23	-	3.23	3.24	3.32	3.31	3.23	3.28	3.28	3.28	3.27	3.27	3.27	3.27	3.27	3.27	3.27	3.26
Stanton Number	St (x 10^{-3})	2.04	-	2.10	1.79	1.91	1.91	1.75	.042	.042	.042	2.10	2.10	2.09	2.09	2.05	2.11	2.05	2.13
P-Factor		> 1.0	-	.681	.726	.679	> 1.0	.489	> 1	> 1	> 1	.580	.580	.750	1.01	.590	.996	.420	> 1
Active Fraction					.005		.01				.02	.11	.10	.01	.001	.02	.01	.05	.02

Nomenclature

Ag	Silver
C.S.	Carbon Steel
Cu	Copper
P	Plated
S.S.	Stainless Steel
W	Wires

comparison of deposition and desorption behavior for different temperatures, deposition surfaces, pretreatments, etc. The curves also were analyzed in some cases by means of a material balance equation which was derived assuming that there was irreversible deposition on the test section, and that the deposition rate was controlled by the diffusion rate of the iodine through the laminar sublayer due to a concentration gradient. The deposition rate was calculated directly from the appropriate convective heat transfer equation for turbulent pipe flow assuming the analogy between heat and mass transfer.[5]

The material balance equation is given by:

$$M(x)_{predicted} = \int_0^T N_o(t)\, h_m\, e^{-\alpha' x}\, dt \qquad (1)$$

where $\alpha' = \frac{4h_m}{Ud_e}$

Assuming:

1. The deposition surface is below saturation for the depositing species.
2. No recirculation of the depositing species
3. $t \gg \frac{L}{U}$
4. $\lambda \ll \frac{4h_m}{Ud_e}$.

Equation 1 permits direct calculation of the slope of each deposition curve after any time, T, without needing the value of the gas phase concentration of iodine $N_o(t)$. In order to account for any experimental deviations from the predicted slope, an empirical correction factor P was inserted as a multiplier of the calculated mass transfer coefficient. The resulting expression:

$$M(x)_{exp} = \int_0^t N_o(t)\, Ph_m\, e^{-\alpha x}\, dt \qquad (2)$$

where $\alpha = \frac{4Ph_m}{Ud_e}$

is the equation used for correlating the experimental slope of the deposition curve assuming that it will follow an exponential dropoff with

distance. It yields values of the empirical parameter P which then can be compared for different flow conditions.

The value of $N_o(t)$ cannot be obtained from applying the experimental data to Eq. 2 since the activity was not necessarily added at a constant rate. However, an expression for the total activity added to the test section can be obtained from Eq. 2 by integration over an arbitrary length of the test section L, (provided the length started at the entrance to test section) and assuming $\lambda \ll \frac{4Ph_m}{d_e}$

$$\int_0^T N_o(t)\, U\pi \frac{d^2}{4}\, dt = \frac{\int_0^L M(x)\pi\, d_e dx}{1 - e^{-\alpha L}} \quad . \tag{3}$$

The total activity added to the test section, which is the left hand side of the equation, can thus be calculated directly from the experimental deposition data (right hand side of equation) without any further assumptions. This activity when ratioed to the total activity added to the loop, Q, yields a second empirical correction term, AF, the active fraction:

$$AF = \frac{\int_0^L M(x)\ \pi d\ dx}{(1 - e^{-\alpha L})(Q)} \quad . \tag{4}$$

Thus the analysis of deposition behavior based on the heat-mass analogy has yielded two empirical correction terms, P and AF. These terms account for deviations of the deposition of I_2 from the assumptions of irreversible deposition and diffusion through a concentration gradient to the wall.

The values of the mass transfer coefficient for use in Eqs. 1 and 2 were obtained from a modified Dittus Boelter

$$\frac{h_m d_e}{D} = 0.023 \left(\frac{Ud_e}{\nu}\right)^{0.83} \left(\frac{\nu}{D}\right)^{0.44} \quad . \tag{5}$$

The diffusion coefficients needed in that equation are obtained from an expression based on the Chapman Enskog kinetic theory.[7]

$$D = 0.185 \times 10^{-2}\, \frac{\theta^{3/2}}{p}\, \frac{1}{\sigma^2 \Omega} \left(\frac{1}{M_1} + \frac{1}{M_2}\right)^{1/2} \quad . \tag{6}$$

Values of P and AF for the runs described in this report are tabulated in Table 1. Since there is uncertainty as to the form of the diffusing iodine and therefore in the calculation of the diffusion coefficients, the numerical values of P are open to some question. However qualitative comparisons of trends are still probably valid and the value of AF is not affected by the value of the diffusion coefficient.

EXPERIMENTAL RESULTS AND DISCUSSION

Rate of Deposition

During one of the tests, a second radiation detector was mounted in a fixed location beneath the stainless steel test section, approximately two feet from the point of injection. Measurements were made comparing the rate of deposition with the rate of injection. Results of this test are shown in Fig. 2. Although the low count rate resulted in considerable scatter in the deposition data, the close correlation between injection and deposition is apparent — particularly the corresponding results during changes in the rate-of-injection. Direct comparisons in time between injection and deposition are not possible as an unknown but significant fraction of time is required for the injection solution to travel from the injector pump to the loop helium stream.

Effect of Surface Material on Deposition

The effects of the surface material on deposition were investigated in a series of 7 tests using 4 different materials. Deposited activity as a function of distance along the test section is shown in Fig. 3. The type 304L stainless steel test section was fabricated from 1-in.-diam, schedule 40 pipe as were the copper- and silver-plated test sections. The plating in the latter two test pieces was approximately 0.002-in. thick. The carbon steel test piece was fabricated from grade 1010 tubing, 1-in.-diam by 0.035-in. thick wall. The carbon steel test section was degreased at 200°F in a vapor of perchloroethylene before installing in the loop. The stainless steel pipe and the plated test pieces were tested in the "as-received" condition. The results from both of the silver plated

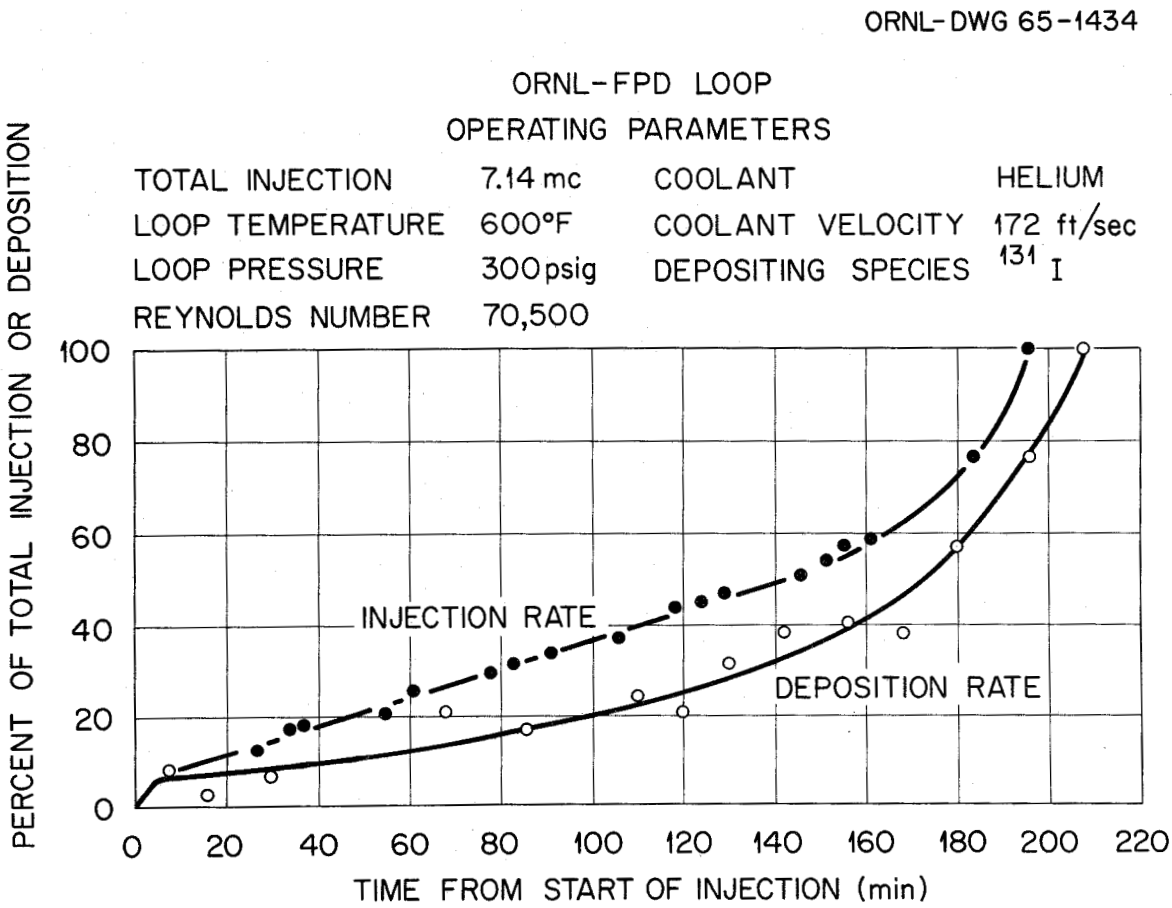

Fig. 2. Comparison of Injection Rate and Rate of Deposition.

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ORNL-FPD LOOP

OPERATING CONDITIONS

	SILVER SURFACE	COPPER SURFACE	CARBON STEEL SURFACE	STAINLESS STEEL SURFACE
Re NUMBER	26,000	30,000	30,000	79,500
SURFACE TEMPERATURE (°F)	400	400	400	450
GAS PRESSURE (psig)	300	300	300	300
GAS VELOCITY (ft/sec)	42	57	57	180

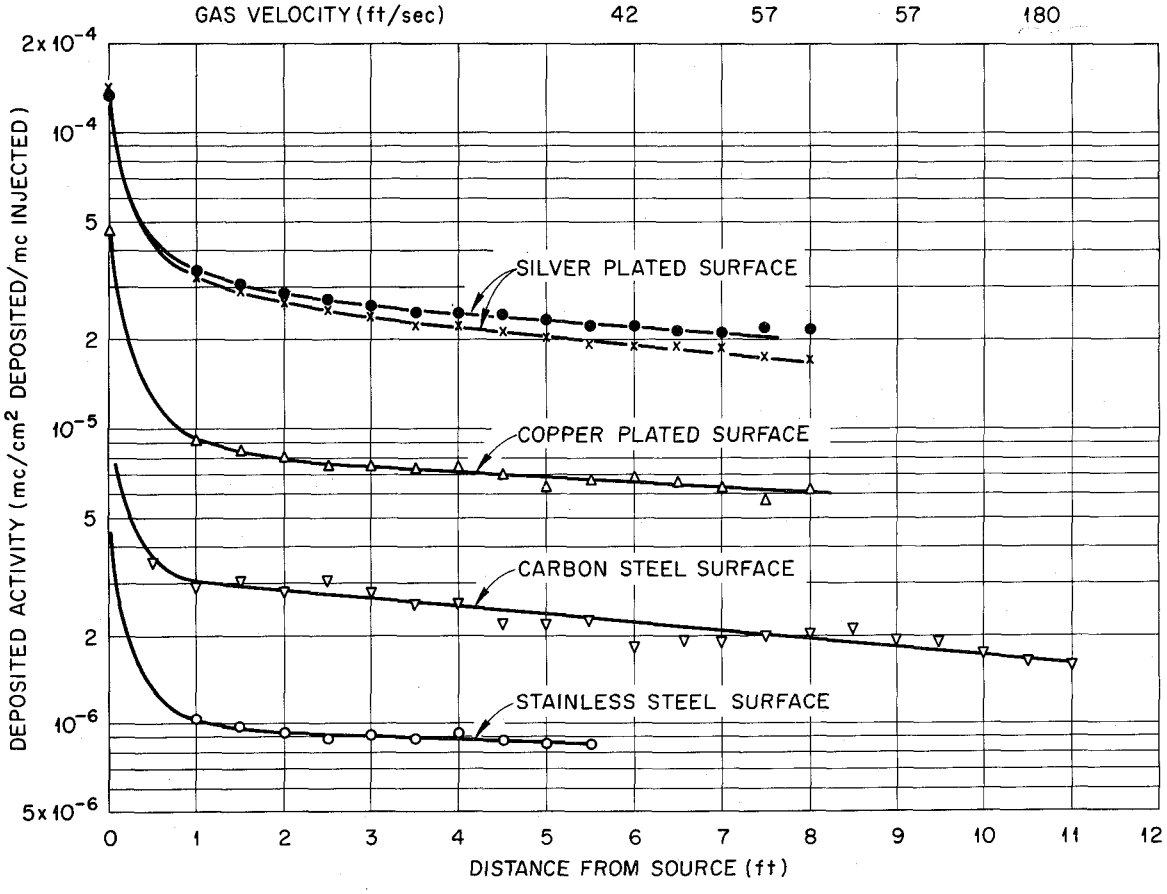

Fig. 3. Effect of Surface Material on Iodine Deposition.

fine?

reproducible?

surfaces are shown to indicate the replication of the silver surface data. Results from a earlier copper-plated deposition surface, not included in Fig. 3, was also in good agreement with the present data. The results of a second carbon steel deposition surface tested under slightly different flow and temperature conditions verified the present results; however they were not as consistent as the silver surface data.

These results agree qualitatively with experiments by the ORNL Nuclear Safety group on deposition of iodine from air by natural convection to the walls of a simulated nuclear reactor pressure vessel. They found, for example, that the deposition on carbon steel was a factor of 10 greater than that on stainless steel coupons[8] for similar exposure times. Similar investigations at the Atomic Energy Research Establishment, Harwell, England[9] also indicated deposition from air was influenced by surface material. In the above two investigations, the iodine was in ambient air and therefore can only be compared qualitatively with the FPD loop results. They are mentioned here only to indicate that other investigators also found deposition to be influenced by surface material.

Effect of Flow Conditions

Two tests were made in which all operating parameters were maintained constant with the exception of the flow rate. In the first run, the test section coolant velocity was maintained at 65.5 ft/sec; in the second run, the flow velocity was increased to 172 ft/sec. This gave a Reynolds number of 27,500 for the first run and 70,500 for the other run. Figure 4, a graph of the deposited activity as a function of distance along the test section, shows the comparison of results.

Two methods are available to compare the experimental data with Eq. 2. First, the ratio of the slopes of the two curves can be predicted from Eq. 2. Second, the ratio of the total deposition per unit area can be predicted from Eq. 2. The former method was not used because of inaccuracies in the profile of the deposition curve. Considering the latter method, it can be seen from Eq. 2 that the deposition per unit area is directly proportional to the mass transfer coefficient and hence should be expected to increase as the 0.83 power of the Reynolds number. However, this must be reduced by the ratio of the P-factor which is equal to[5]

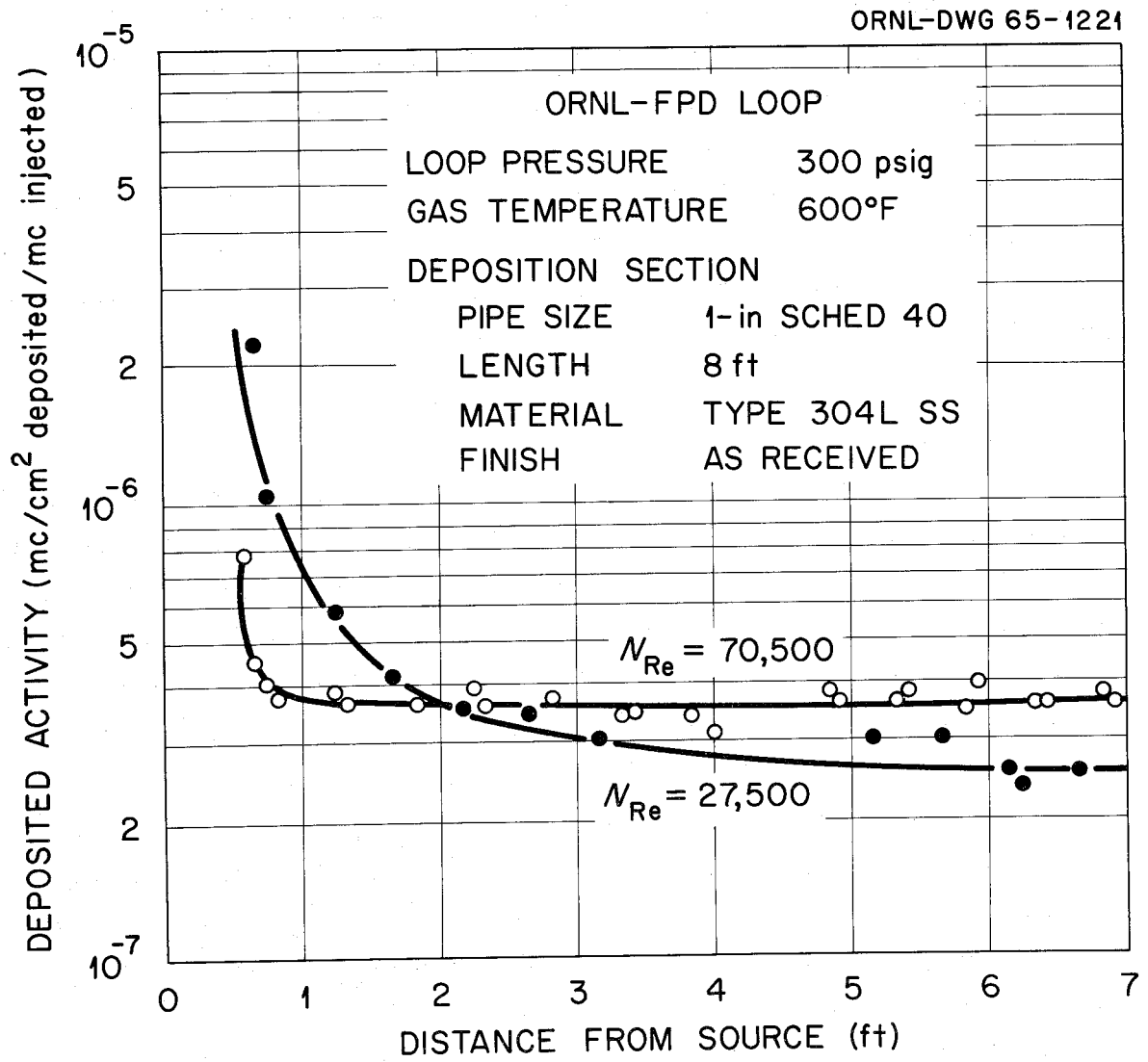

Fig. 4. Effect of Reynolds Number on Iodine Deposition.

$$P = \frac{1}{1 + h_m/h_w} \quad . \tag{7}$$

N_o is determined from the injection rate and the active fraction, AF, as explained in a previous section. The active fraction, however, is determined from the profile of the deposition curve and for these runs could not be accurately calculated. However a qualitative comparison can be made by assuming the AF to be the same in both tests. Using the mass transfer coefficients and P-factors shown for runs 5 and 6 in the tabulation in Table 2, a deposition ratio of 1.20 to 1 was calculated. The experimental ratio from Fig. 4 is approximately 1.45 at a point sufficiently far downstream to minimize entrance effects. This analysis assumed that the reflection term does not vary with flow rates.

Desorption Study

In two experiments, the effects of fission product desorption was observed. In both experiments stainless steel deposition surfaces were used. In the first, the loop was maintained at a gas temperature of 400°F, and approximately 5 mg iodine, dissolved in cyclohexane, was injected into the system over a 50 minute period. After the injection, loop operation was continued at 400°F for a period of 20 hours. The loop helium temperature was then increased to 500°F and operation was continued for an additional 124 hours. Six scans measuring the activity along the sufrace of the test section were made during the above period. The results of this experiment, shown in Fig. 5, indicate desorption from an initially uniformly covered surface does not necessarily occur uniformly over the section. The initial level of activity is shown as the solid line.

In a subsequent experiment in which the same test section was installed in reverse, non-uniform desorption was again noted at the same location on the test section (at the 6-ft location shown in Fig. 5), independent of the direction of flow. It seems likely that the non-uniform desorption was due to a surface effect rather than a flow condition.

The qualitative effect of two different surface materials on desorption is shown in Fig. 6. In this experiment, a 1-in.-diam, Sch 40 silver-plated, stainless steel pipe was maintained at a surface temperature of

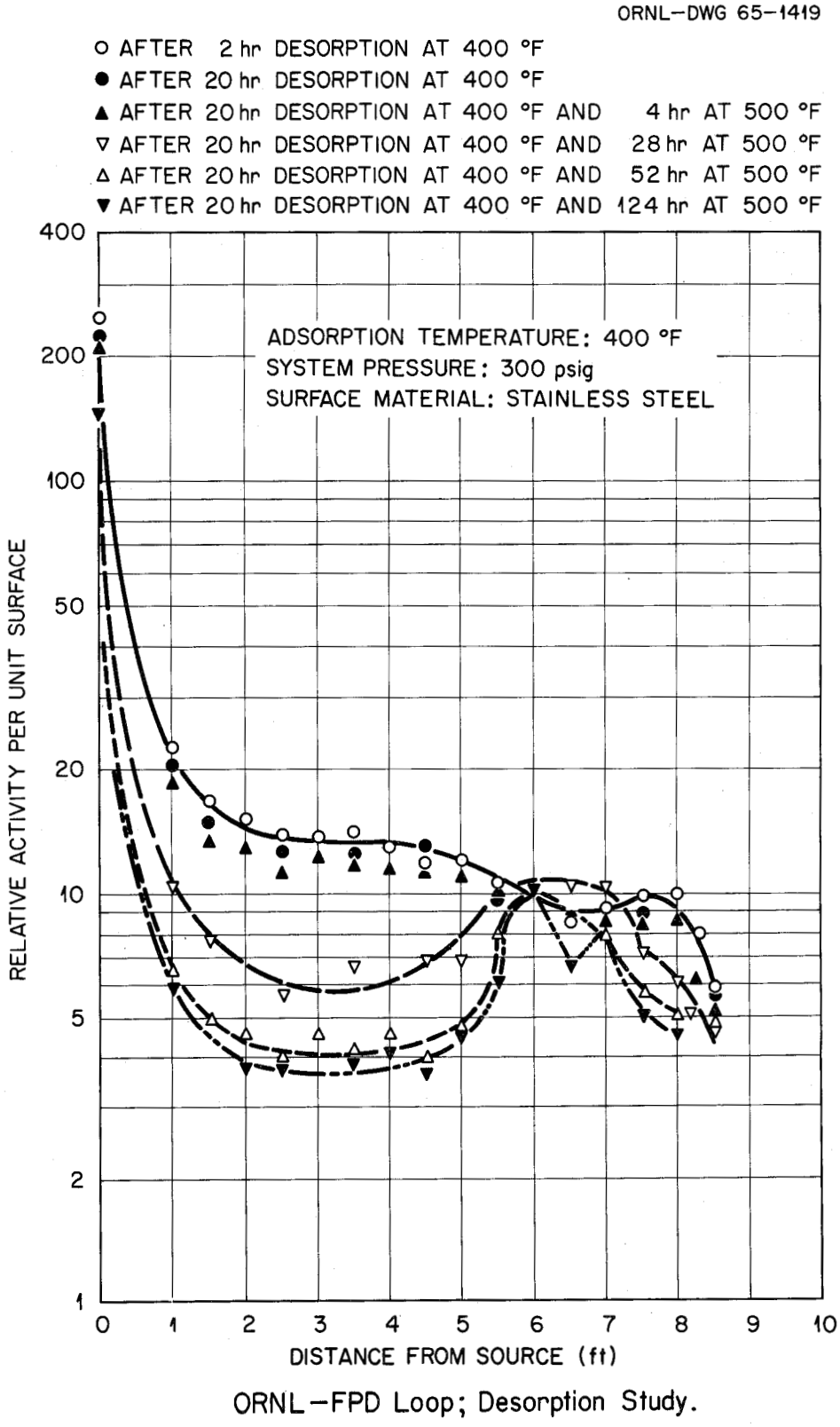

Fig. 5. Desorption from a Stainless Steel Surface.

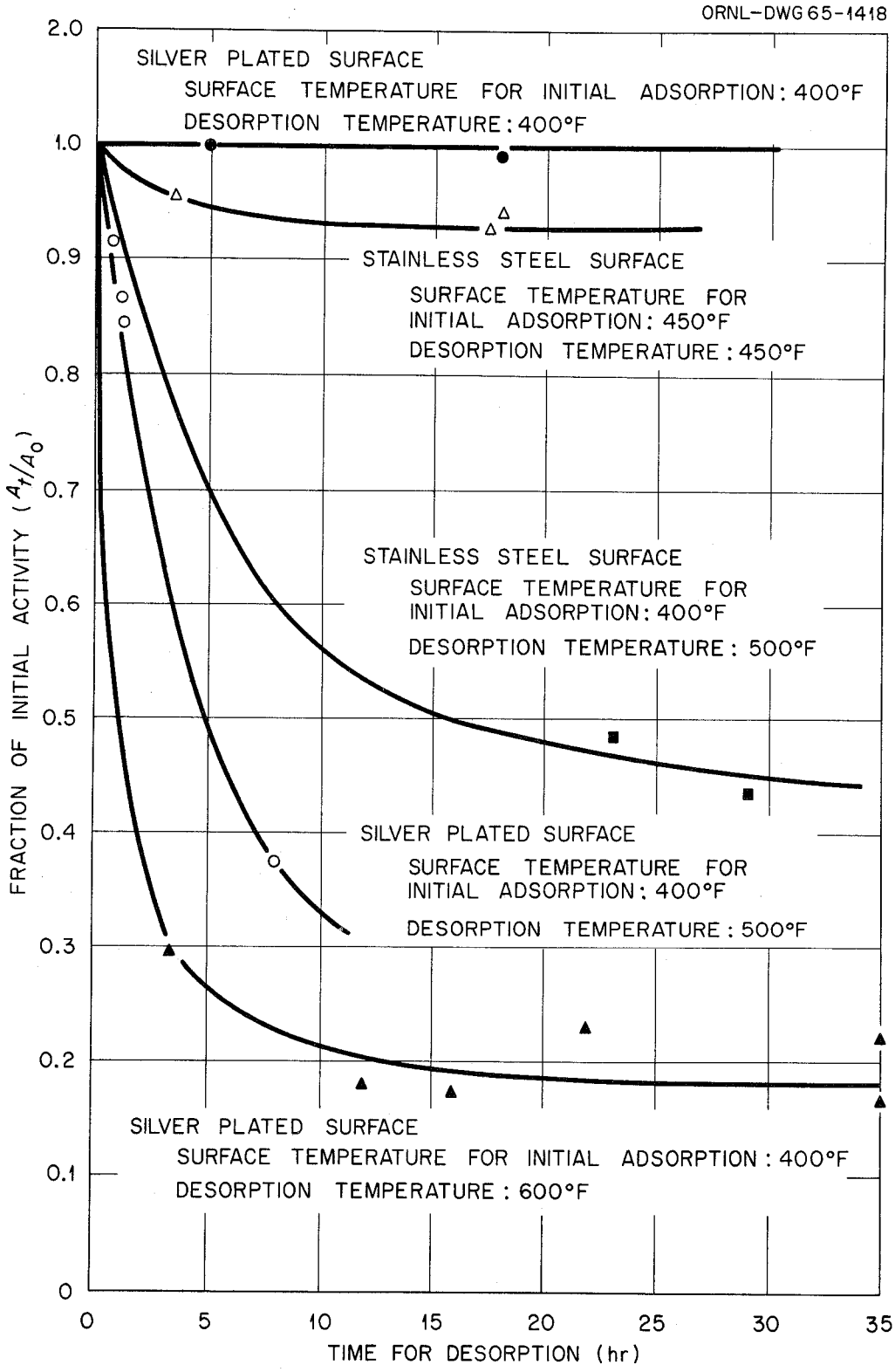

Fig. 6. Comparison of Desorption from Silver-Plated and Stainless Steel Surfaces.

400°F while injecting approximately 5 mg of iodine in a cyclohexane solution into the system. After the injection was completed, the surface temperature was raised to 500°F and subsequently to 600°F. Scans were made of the test section as a function of time after each temperature change. A similar procedure was then followed for a stainless steel test section. Results of a third experiment also with a stainless steel test section, are shown; however, in this test the temperature during adsorption and desorption was maintained constant at 450°F. The greater desorption at 500°F from the silver-plated surface than the stainless steel surface may have been due to decomposition of AgI which had formed during the deposition at lower temperatures since it has been reported[10] that "...AgI begins to decompose at a temperature in the range of 450°C (662°F) to 550°C (1022°F), and that the half-time for the rate is less than 1/2 hour." These experiments were conducted in an oxygen-water vapor atmosphere but may provide a clue that decomposition of the AgI may be taking place.

Effect of a Heat-Affected Zone

To observe deposition patterns in a heat-affected zone, such as might be caused by a pipe weld, three simulated welds were made on 1 1/2-in.-diam pipe test sections. In each test, two-bead welds were made on the outer surface of the test piece but did not penetrate to the inner surface of the pipe, thereby simulating a heat-affected zone without introducing a change of material or roughness of the deposition surface. The surface was blanketed with helium during welding to minimize oxidation of the test surface.

The first test was operated with a surface temperature of 400°F and a coolant flow rate of 27.4 ft/sec with one simulated circumferential weld at approximately the mid-point of the test section. The results are shown on the left side of Fig. 7. The second test, which operated at a surface temperature of 500°F and a flow rate 30.4 ft/sec, had two simulated weld sections included in the test section. These results are shown on the right side of Fig. 7.

From Fig. 7, it can be seen that the effect on deposition is substantial; in the first experiment the deposited activity was reduced to approximately 25% of the value in the non-affected heat zone, in the second

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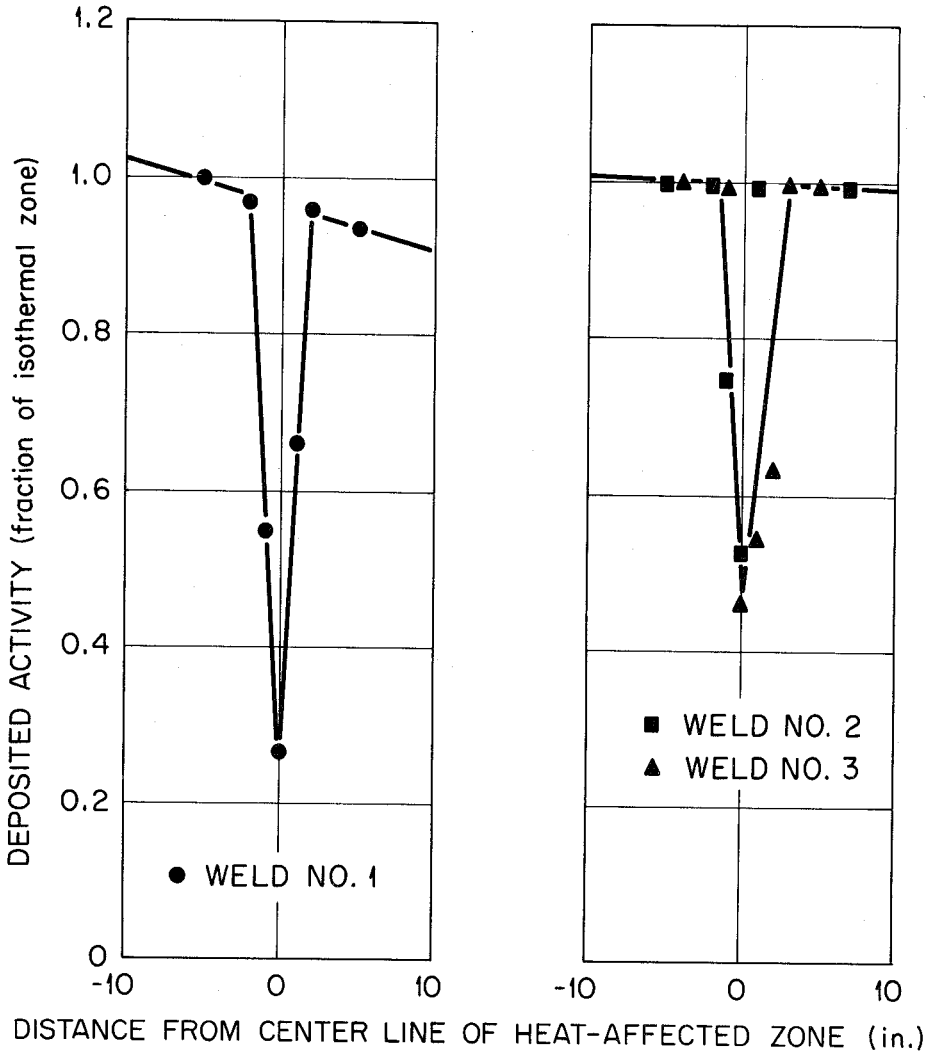

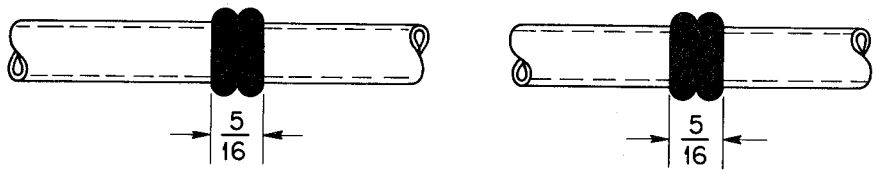

OPERATING CONDITIONS

400	SURFACE TEMPERATURE (°F)	500
5.5	INJECTION (mc)	0.55
25,500	REYNOLDS NUMBER	24,200
He	COOLANT	He

Fig. 7. Effect of a Heat-Affected Zone on Deposition.

experiment the deposition was approximately 45% of the activity in the non-affected heat zone.

The results of this experiment would be expected for iodine,[11] and similar non-metals, if the deposition surface was oxidized during the welding procedure. Additional care was taken during the second test to minimize any oxidation of the surface which might account for the reduced effect on the deposition ratio.

Replication of Data

A series of three experiments were made to determine the degree of replication of data possible in the loop under similar operating conditions. In the first, three pieces of 1-in.-diam schedule 40 welded pipe selected from the same heat were fabricated into test sections and installed in successive tests. Operating conditions between tests were maintained as nearly identical as possible. The results, on the basis of activity per unit area per millicurie of iodine injected, generally agreed to within a 30% deviation. However there was evidence of erratic deposition in all three test sections. The erratic deposition pattern may be due to the non-homogeneous surface in welded pipe or to the surface pre-treatment. The experiment was repeated using test sections with silver-plated surfaces. The operating conditions and results for these two runs are shown in Fig. 8. The data produced smooth curves within a 12% maximum deviation. The small difference in the absolute magnitude is not considered significant in this experiment as it is within the errors expected in measuring the total amount of radioactive iodine introduced into the system.

In a third series of experiments, a 1-in.-diam test section, fabricated from stainless steel, was used in three tests which were operated under identical temperature and flow conditions. The section was decontaminated between tests. From Fig. 9 it can be seen that the magnitude of deposited activity per unit of activity injected was reduced in each succeeding test.

The decrease in deposition may be due to a passivation of the deposition surface due to the decontamination of the test section between tests. The decontamination procedure used was to contact the deposition surface

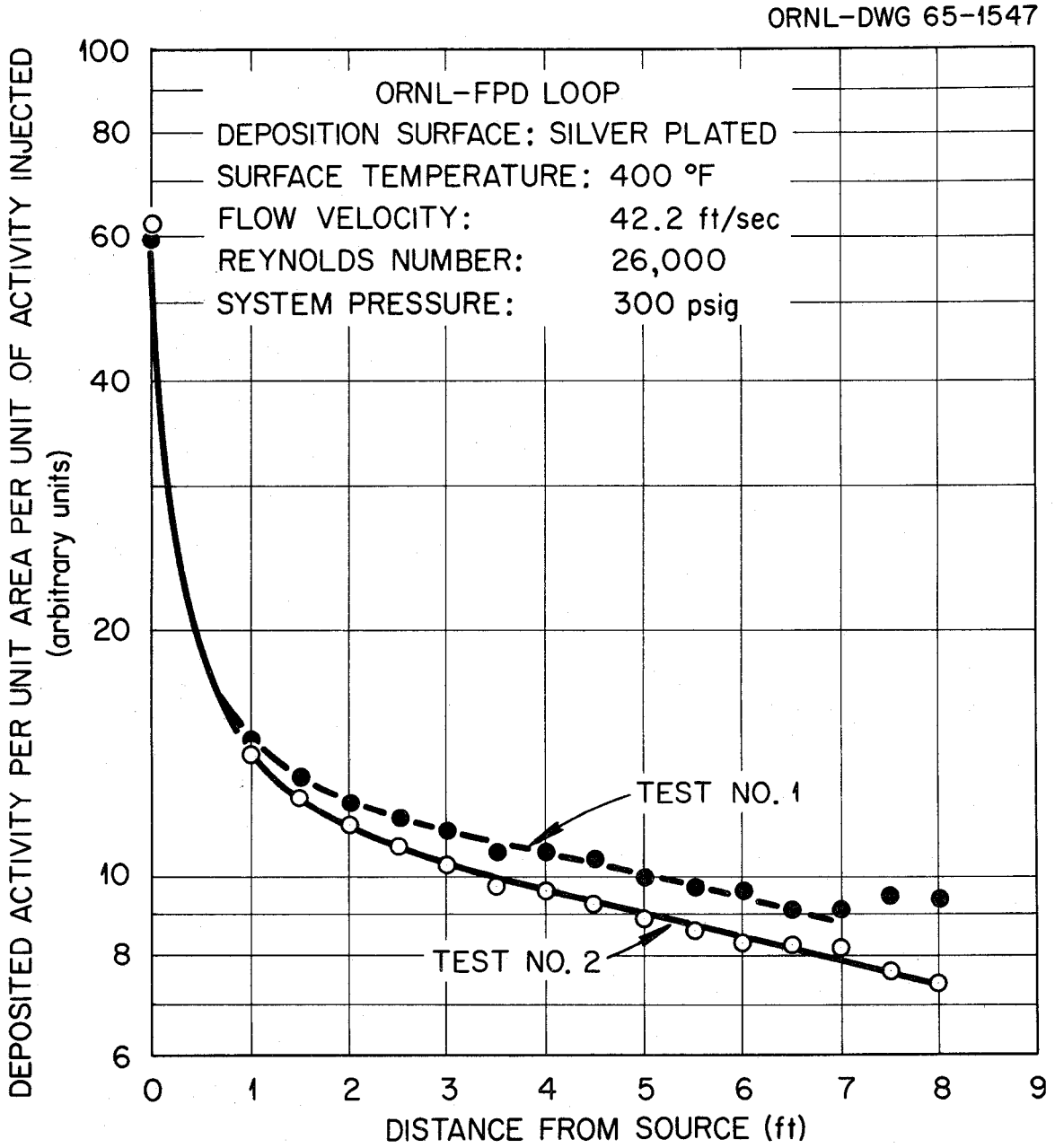

Fig. 8. Replication of Deposition Results on a Silver-Plated Surface.

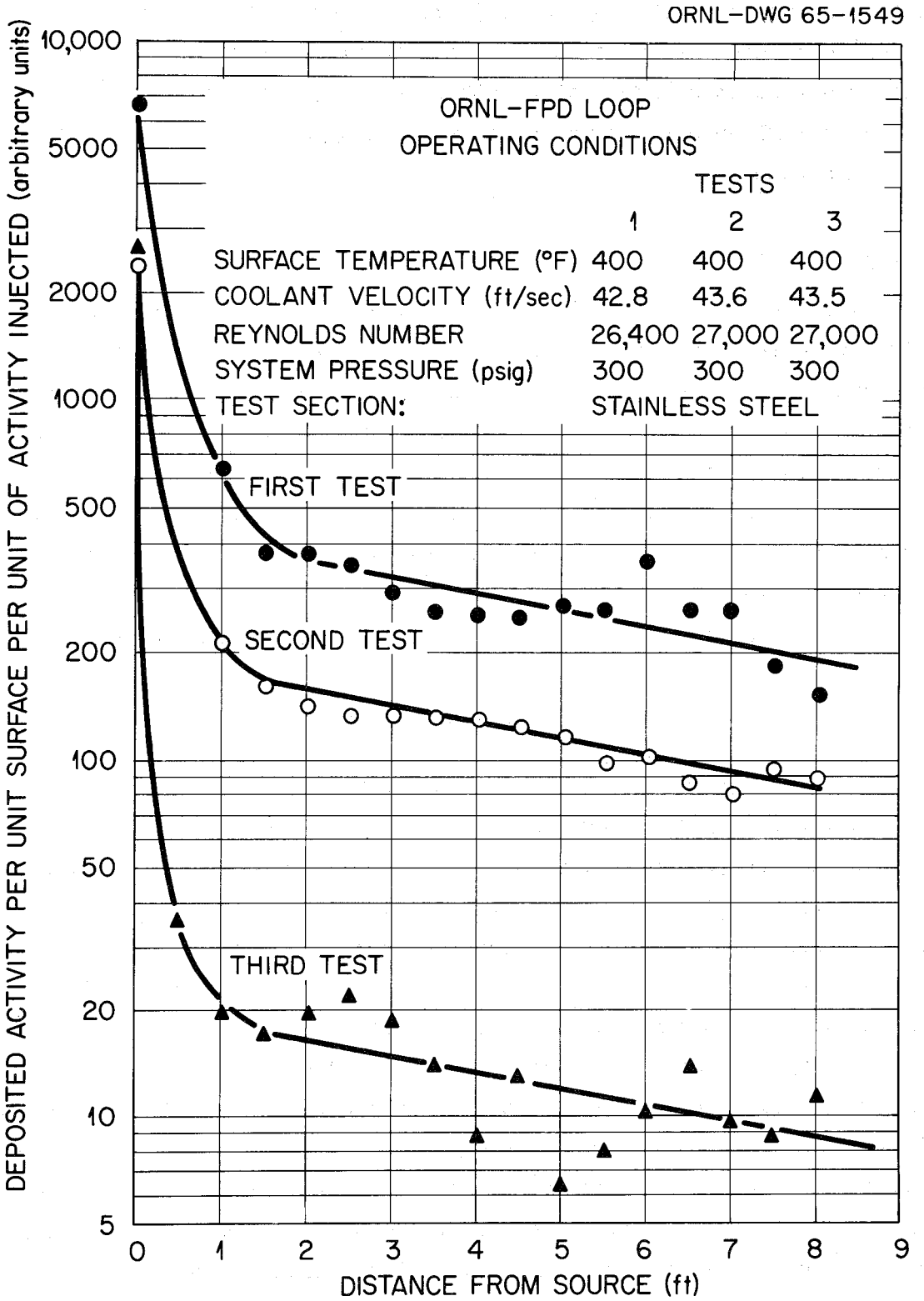

Fig. 9. Effect on Deposition of Surface Treatment.

with a solution of 50% (by volume) ammonium hydroxide and 50% hydrogen peroxide at ambient temperature. Additional testing will be required to better define this effect since other factors, such as rate of injection, may also influence the total deposition in a manner that is not presently well understood.

Silver Wire Experiments

A series of experiments were conducted in which small-diameter, silver wires were inserted into the flowing helium stream, positioned in arrays perpendicular to the direction of flow. One purpose for these experiments was to study the application of the heat-mass transfer analogy to fission-product transport under conditions substantially different from the idealized conditions existing in the smooth, straight test sections of previous experiments. The wire geometry was selected for test because heat-transfer data were readily available in the literature[12,13,14,15,16] for this geometry. The equation used for determining the Nusselt number (Nu) is of the form[12]

$$Nu = C(Re)^n \tag{6}$$

where C and n are constants of the equation for which values are given as a function of Reynolds number (Re) in the above reference.

Heat transfer coefficients for various diameter wires mounted in an array perpendicular to the gas stream were calculated from Eq. 6. These values were used to calculate predicted ratios of deposition for different size wires, and then compared with experimental data obtained from the loop. The absolute magnitudes of the activity of the deposited particles were also compared by estimating the total activity injected and subtracting the amount deposited on the wall between the source and the plane of the wires. It was assumed that the gas-stream activity depletion was proportional to the attenuation of the deposited activity on the wall. With this assumption, Eq. 6 was rewritten in terms of the Sherwood number. Equation 8 was used to determine the total calculated deposition on the silver wires.

$$M = h_m \bar{c} t \tag{8}$$

The first test consisted of two arrays of 3 silver wires each mounted perpendicular to the gas stream. The upstream array of three 0.010-in.-diam wires was located 5-ft, 9-in. downstream from the source injector. The downstream group was composed of 3 wires 0.004-, 0.010-, and 0.040-in.-diam placed in a plane 7-ft, 8-in. downstream from the point of injection. The upstream array of wires of constant diameter was installed primarily to test reproducibility of the experimental data.

In the second test, the geometry and all operating conditions were maintained the same as in the previous test, except that the location of the two sets of wires were interchanged. The results of both tests are shown in Table 3.

Note in Table 3 the predicted and experimental ratio of deposition on the 0.004-in.-diam wire to that on the 0.010-in.-diam wire is in good agreement — a difference of approximately 2%. The 0.040-in.-diam wire had about twice the deposited activity expected, based on results from the other two wires. The reproducibility of data in the first test was good as shown by comparing the deposition on the set of three 0.010-in.-diam wires. In the second experiment, the predicted ratios were in reasonable agreement with the experimental ratios for all the wires.

The calculated values for the total activity deposited, based on Eq. 8, are not in good agreement with the experimentally obtained values. It is believed that some of the injected activity became tied-up by either a foreign compound in the gas stream — possibly the cyclohexane solvent — or by particulate matter in such a manner that it did not react with the deposition on the pipe wall discussed previously (see pp. 8-9). Additional work must be done in this area in order to better understand the significance of the active-fraction concept.

CONCLUSIONS AND RECOMMENDATIONS

This report presents data from a group of iodine deposition experiments in which the radioactive source was in the form of a liquid. The liquid source had two advantages; it provided a positive means of controlling the total amount injected into the system, and it permitted preselection of an injection rate. The major disadvantage to this type source

Table 3. Summary of Deposition on Silver Wires

Wire Size in.	Calculated Deposition mc/cm^2	Experimentally Determined Deposition mc/cm^2	Calculated / Experimental	Calculated Ratio	Experimental Ratio	Error %
			Test No. 1			
.004	4.45×10^{-3}	1.07×10^{-3}	4.15			
.010	2.71×10^{-3}	6.39×10^{-4}	4.75	1.70	1.67	1.8
.040	1.29×10^{-3}	5.97×10^{-4}	2.13	2.10	1.07	96.0
.010-1	3.16×10^{-3}	8.47×10^{-4}	3.74			
.010-2	3.16×10^{-3}	8.52×10^{-4}	3.71			
.010-3	3.16×10^{-3}	8.32×10^{-4}	3.80			
			Test No. 2			
.004	3.48×10^{-4}	1.13×10^{-4}	3.41			
.010	2.37×10^{-4}	7.61×10^{-5}	3.11	1.63	1.48	10.1
.040	1.12×10^{-4}	4.59×10^{-5}	2.44	2.11	1.66	27.1
.010-1	1.80×10^{-4}	5.69×10^{-5}	3.16			
.010-2	1.80×10^{-4}	6.70×10^{-5}	2.68			
.010-3	1.80×10^{-4}	5.47×10^{-5}	3.28			

is discussed in Appendix B – namely, the uncertainty on the results of the effect of introducing a foreign substance into the gas stream.

The research thus far has been principally concerned with studying the attenuation of deposited activity in straight, isothermal tubes. From the deposition curve, it has been possible to determine a P-factor and the active-fraction for a variety of surface materials, surface temperatures, and flow conditions.

The wire experiments have several advantages over tube wall deposition experiments for studying the application of the heat-mass analogy to fission product deposition. It permits several parameters to be tested in the same experiment, and minimizes the effect on deposition results of the non-uniform surfaces commonly found in pipes. Determination of sticking-fractions on reactor materials compared with silver are well adapted to wire experiments. The effects of Reynolds number and temperature can probably be best done with wire type experiments.

Other isotopes must be studied in detail. Analytical studies have been made to determine the species that are the most objectionable based on gamma energy, yield, release rate, to reactor maintenance (i.e., the level of activity when reactor is shut down). Such a list indicates that the fission product decay chain for the 140 mass number will be among the most troublesome because of the high energy gamma from lanthanum and the high yield and release of barium.

In the next series of experiments a PdI_2 source will be used in order to minimize the introduction of foreign compounds into the gas stream. The present work will be continued in more detail and will be expanded to study the effects of flow, temperature and surface material. The effects of surface saturation and its effects in limiting the total amount of deposition must be studied in more detail. A modified computer code that separates the effect of fission product reflection from a depositing surface from the desorption effect will be used to correlate the experimental data. Certain of the desorption effects will be studied in a packed-bed test section. Additional understanding is needed of surface reactions to separate and understand physical adsorption of fission products from chemisorption.

Some of the experimental program will be transferred to laminar-flow deposition tubes where flow conditions are better characterized.

NOMENCLATURE

$\bar{c}$ = Average concentration of particles in gas stream, particles/cm^3

d_e = Equivalent passage diameter, cm

h_m = Mass transfer coefficient for stream, calculated from a mass-heat analogy, cm/sec

h_w = Mass transfer coefficient at the wall surface, cm/sec

L = Length of test section, cm

M(x) = Concentration of particles deposited on the wall surface, particles/cm^2

$N_o(t)$ = Concentration in the gas stream at the origin due to an external source, particle/cm^3

p = Pressure, atmospheres

P = Ratio of experimentally determined deposition velocity to calculated deposition velocity (See Eq. 7)

T = Time of injection, sec

U = Mean velocity of stream, cm/sec

x = Distance along tube, cm

α = As defined in Eq. 1

θ = Temperature, °K

λ = Decay constant, sec^{-1}

σ = Collision diameter, Angstrom

ν = Kinematic viscosity, cm^2/sec

Ω = Collision integral

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Appendix A

APPENDIX A

Main Line Filter Experiment

Before the start of run 16, a main line filter was installed in the loop immediately upstream of the injection point. The purpose of the filter was to prevent particles that had deposited throughout the loop from desorpting during succeeding runs and being transported by the helium into the test section. The concern is two-fold: first, the desorbed particles add an unknown source term, and secondly, the ratio of stable iodine to radioactive iodine is not known for the desorbed particles.

The filter chosen was silver mesh composed of 0.010-in.-diam silver wires. The mesh was in excess of 10-in. deep in the direction of flow. X-ray pictures verified that the mesh was uniformly distributed throughout the filter volume.

To obtain a measure of the filter efficiency, 1.5 cc of radioactive iodine solution (approximately 0.75 mc of ^{131}I) was injected into the loop downstream of the test section. The loop temperature during injection was maintained at 400°F, after scanning the test section for a build-up in activity the loop temperature was raised to 500°F and subsequently to 600°F. The results were encouraging: for the 400°F test, the increase in activity in the test section was about 10% above background; and for the 500°F test, the activity level raised approximately 15% above background when equilibrium was reached. When the 600°F test was run the activity level in the test section approximately doubled background indicating that the higher gas temperature was increasing the filter bed temperature and thereby allowing the bed to release iodine to the test section. As these values of activity are low compared to the amount of activity deposited from a source it was concluded that the filter performed satisfactorily for the present group of experiments.

Appendix B

APPENDIX B

The Stability of Solutions of Iodine in Cyclohexane and in Benzene

A. D. Horton A. S. Meyer

This research was undertaken to determine whether radioiodine solutions in benzene or in cyclohexane could be used as a means of introducing free iodine into a stainless steel reactor loop that was heated to 300 to 600°F and was pressurized to 300 psia with helium. Solutions containing 5 mc ^{131}I and 5 mg of I_2 carrier per 10 ml of solvent were being injected with an infusion pump through a stainless steel capillary extending into the helium stream. Since the purpose of the experiment was to study the rate of deposition of elemental iodine in a section of the loop, the formation of iodine compounds with the solvent would make analysis more difficult. Two methods of investigation, partition gas chromatography and spectrophotometry, were selected as the best available techniques to determine whether these solvents interacted with the iodine under loop conditions.

Partition gas chromatography is a technique in which vaporized samples are swept through a partition column where the components of the samples are separated into fractions according to boiling point and structure. A detector produces a signal for each component as it emerges from the column with the signal strength proportional to the concentration of the component in the mixture. The technique is empirical and tentative identification and exact quantitative analysis can be carried out only by comparison with chromatograms of known compounds as standards.

The most sensitive detector on this instrument is the flame ionization detector. This detector responds to traces of organic consituents but is insensitive to iodine. Iodine can be measured with an alternate detector (a thermal conductivity cell) but the sensitivity is not adequate for the measurements of these low iodine concentrations. Thus, the gas chromatographic measurements do not offer a direct measure of the free iodine but only inferential information unless iodine containing reaction products can be identified and measured quantitatively.

An alternate technique utilizes a modified Cary Model 14 spectrophotometer in which the absorbance of samples at elevated temperatures can be measured over the wavelength of 200 mμ to 2.5 μ. By sealing samples of the solutions in quartz ampoules and scanning the absorbance of the vapors in the heated ampoules, iodine, and in some cases the solvent, can be observed at the temperature of the loop tests. This technique is inherently less sensitive than the gas chromatographic method but provides a direct measurement of the free iodine.

The absorbance of any species at a particular wavelength ideally follows the Beer-Lambert equation which states that the absorbance, $\log_{10} \frac{I_o}{I}$, is proportional to the quantity of the species in the optical path. While the absorbance characteristics of some components may be modified by environmental conditions such as pressure and temperature, such variations in specific absorbance can be compensated for by comparing the absorption curves with reference standards. Ampoules containing vaporized solutions of iodine in carbon tetrachloride were used as reference standards in these spectrophotometric measurements.

Experimental

The aforementioned methods of investigation were used to determine the behavior of iodine solutions in benzene and in cyclohexane, under conditions as near as possible to those used in the loop experiments.

Preparation of Solutions

Solutions used in the loop tests contained a total of 5 mg of iodine per 10 ml of solvent. However, preliminary experiments in gas chromatography indicated that the amounts of possible reaction products would be too small to detect. Therefore solutions containing 200 mg of normal iodine per 10 ml of benzene, cyclohexane, or carbon tetrachloride were prepared. The carbon tetrachloride and cyclohexane solutions showed the violet color of uncomplexed iodine, whereas the benzene showed the brown color of pi-bonded iodine.

Gas Chromatography

A Micro-Tek Model 2500 R gas chromatograph was used to determine the possible reaction products of the iodine-solvent solutions. Helium passes through a heated injector port and flash vaporizer, carries the vaporized sample through a partition column, and then to a hydrogen-flame ionization detector. The flow of helium to the injector port can be interrupted by means of a linear gas sample valve thus allowing an injected sample to remain in the flash vaporizer at a set temperature for as long as desired. The flash vaporizer thus serves as a reaction chamber to approximately simulate the conditions in the test loop, and to provide a means of rather crude rate studies. In all cases, chromatograms of reacted iodine solutions were compared with chromatograms of pure solvent samples which had been subjected to the same reaction conditions.

From the flash vaporizer the reacted sample was swept by a 50 cc per minute flow of helium into an 8-ft by 1/4-in. OD partition column packed with 20 wt % SE-30 on 30-60 mesh chromosorb W.

After injection of the sample, the column was operated at room temperature (~ 25°C) for 4 min , heated to 100°C during the next 2 min, then programmed at ~ 12°C per min for 12 min and held at 250°C for 16 min. Injection of 4 µl of sample and proper attenuation of the detector output gave a chromatogram suitable for the estimation of the concentration of all impurities except cyclohexane in the solvents and the new components formed by the reaction of iodine with the solvent. A 10-ft by 1/4-in. column of tricresyl phosphate on firebrick was used to separate cyclohexane from cyclohexene and the other impurities.

Spectrophotometry

Because of the design of the sample furnace it was necessary to measure the absorbance of the samples in cylindrical ampoules rather than in cells with optically flat windows. Quartz ampoules approximately 1-1/2-in. long x 1/2-in. OD with a path length of about 1 cm and a volume of approximately 2.5 cc were fabricated. To each of these ampoules approximately 10 µl quantities of either one of the pure solvents or one of the iodine solutions were added. Then the ampoules were immersed in liquid nitrogen evacuated

and sealed. This quantity of solvent was calculated to yield a total pressure below two atmospheres when the ampoules were heated to 600°F. Ampoules containing mg quantities of iodine alone were also prepared. Absorption spectra of all ampoules were taken in the visible and ultraviolet regions. Most significant absorptions were obtained in the region of 500 μc. Absorption spectra were observed for a period of hours at 300°F and during the period when the ampoules were heated to 600°F and maintained at the higher temperature.

Results

Gas Chromatography

The cyclohexane was determined to be 99.85% pure; the major impurity was cyclohexene.

A chromatogram of the cyclohexane-iodine solution showed a component which did not appear in the solvent, and which had a boiling point of approximately 125°C. The characteristics of the peak produced by this component were those of a highly polar compound that probably was not an organic iodide. The concentration of this compound was 0.7 ml % on the day the cyclohexane-iodine solution was prepared, and it increased to a fairly constant value of ~ 5% after 96 hours standing at room temperature. The concentration of this component decreased rapidly with residence time in the hot (600°F) injection port of the chromatograph. A residence time of 4 min decreased the concentration by ~ 80%. At the same time, another new component appeared which had an approximate boiling point of 170-180°C. This component was determined to be cyclohexyl iodide by comparison with a chromatogram of cyclohexyl iodide recorded under the same conditions used for the cyclohexane-iodine solutions. The identity was confirmed by infrared spectrophotometry. The concentration of this component reached a maximum of ~ 0.04 mol % when the solution was 48 hours old. After that, the concentration diminished with age to a constant value of 0.009 mol % at 96 hours. Table 1 shows the gas chromatographic data obtained at the various time intervals after the preparation of the solutions. Data are recorded for solution injected directly and for solution reacted for 4 min, the period which gave the maximum yield of cyclohexyl iodide.

Table 1. Concentration of Components Produced by the Reaction of Cyclohexane with Iodine
Gas Chromatographic Determination

Concentration of Iodine, 0.84 mol %						
History of Sample	Component BP 125°C, mol %		Cyclohexyl Iodide mol %		Compound BP 210°C, mol %	
	Direct Injection	4 min at 600°F	Direct Injection	4 min at 600°F	Direct Injection	4 min at 600°F
Fresh solution	0.72	0.14	0.006	0.018	Not found	Not found
Age - 24 hrs	1.72	0.93	0.001	0.023	"	"
Age - 48 hrs	3.52	0.63	0.005	0.036	"	"
Age - 72 hrs	4.48	0.74	0.003	0.009	"	"
Age - 96 hrs	4.60	0.86	0.001	0.009	"	"
Heated in sealed quartz cell to 600°F and 300 psia	Not found	-	0.10	-	0.70	-

The benzene was found to be 99.5% pure; the major impurity was toluene. Chromatograms of the benzene-iodine solution recorded under the same conditions as that of the cyclohexane-iodine solutions did not show any components that did not appear in the benzene alone.

Spectrophotometry

In the absence of solvents, iodine vapor yielded a very complex band spectra with a maximum absorbance at 501 mμ. When any one of the solvents, benzene, cyclohexane, and carbon tetrachloride, was present the maximum was shifted to 510 mμ and the fine structure of the spectra was modified. However, the absorption spectra were qualitatively identical in the presence of either of the three solvents. None of the solvents exhibited absorption spectra in the region of the iodine maxima and possible reaction products which should have absorbed in the ultraviolet were masked by the solvent vapor.

Initial additions of iodine solution to the ampoules were made with a Hamilton syringe. This syringe utilizes a stainless steel needle (type unknown) and a metal plunger (material unknown). The absorbance of iodine obtained after some of the injections of uncomplexed solutions (cyclohexane and carbon tetrachloride) was found to be substantially lower than that obtained after injection of the benzene solutions in which an iodine-benzene complex exists. Therefore, later samples were made by weighed additions from an all-glass capillary dropper. Ampoules filled from the glass dropper yielded substantially identical spectra at 300°F. Solutions of iodine in cyclohexane, measured spectrophotometrically in the liquid phase, were found to be unchanged after a one-hour contact with stainless steel wool (type unknown). It therefore appears that the iodine may have reacted with the metal plunger of the syringe rather than the needle.

Absorbance data for iodine vapor at 300°F are given in Table 2.

The absorbance of iodine in the presence of either benzene or cyclohexane was essentially constant for a period of hours when the vapors were held at 300°F. When the temperature of cyclohexane-iodine vapors was increased the iodine color faded rapidly when the temperature reached about 500°F to yield colorless vapors at 600°F. An ampoule containing sufficient cyclohexane-iodine solution to yield a pressure of 300 psia was heated to

Table 2. Absorbance of the Vapors of Iodine and Solvent at 510 mμ at a Temperature of 300°F

Solution	Cell Volume, ml	Wt. of I_2, mg	OD	OD/mg/ml
I_2, only	2.42	1.095	0.943	2.08
I_2 - CCl_4	2.34	0.198	0.221	2.44
I_2 - C_6H_6 (1)	2.10	0.200	0.219	2.21
I_2 - C_6H_6 (2)	2.03	0.200	0.201	2.12
I_2 - C_6H_{12} (1)	2.24	0.200	0.186	2.08
I_2 - C_6H_{12} (2)	2.30	0.200	0.168	2.49

(1) Hypodermic syringe with stainless steel needle used to measure sample.

(2) All-glass capillary dropper used to measure sample.

600°F and produced a colorless liquid with a pale yellow solid. The solution was examined gas chromatographically and results are included in Table 1.

When the benzene-iodine vapors were heated from 300 to 600°F the iodine absorbance decreased approximately 6% and continued to decrease at the rate of 6% per hour when the temperature was maintained at 600°F. One ampoule was inadvertently overheated to about 800°F. Approximately 75% of the iodine color was discharged.

Discussion and Conclusion

Since the gas chromatograhic technique does not offer a direct measurement of the iodine concentration, the spectrophotometeric results are considered to be the most relevant. Also in the gas chromatographic measurement some weakly bound iodine compounds could have been decomposed by reaction with the stainless steel walls of the flash vaporizer or with the column packing.

The spectrophotometric results are relatively imprecise for several reasons. Firstly, the absorbancies measured are relatively low and, with some uncertainties in the estimation of the baseline, errors of several

per cent are introduced. Additional errors are introduced as a result of uncertainties in the optical path when small cylindrical cells are used so that the overall photometric error may be as high as 10%. To this must be added the errors inherent in the experimental difficulties of sealing such small volumes of liquids in the ampoules. Accordingly, the absorbance data for all solvents (Table 2) is considered equivalent within experimental error. Conversely, changes in the concentration of iodine in a particular ampoule can be measured with reasonable precision. For example, the rate of decrease in iodine absorbance in benzene at 600°F was measured with an error of only about one per cent.

Since identically shaped absorption spectra were obtained with three diverse solvents it is reasonable to conclude that the observed spectra correspond to free iodine, and that the differences between these spectra and the spectrum of iodine alone result from the pressure of the solvent molecules acting as an inert foreign gas. Two consultants, L. B. Rogers and P. H. Emmett, concur with this interpretation of the data.

Both the spectrophotometric and gas chromatographic data indicate that a negligible proportion of the iodine (< 10%) is combined with the solvents at 300°F. At 600°F the benzene-iodine mixtures are still relatively stable while the cyclohexane-iodine mixtures react rapidly and completely. Fragmentary data also indicate that the pi-bonding of benzene may tend to stabilize solutions prior to vaporization.

It was necessary to carry out the analytical experiments under conditions somewhat different than those found in the test loop. In general, the analytical tests were performed at higher partial pressures than those within the loop. These higher pressures would, on the basis of the law of mass action, tend to yield an increase in compound formation. Possible catalytic effects from materials in the loop were outside the scope of this study and were not investigated.

Diffusion tube experiments on the loop gases (I_2 and benzene), carried out earlier by W. E. Browning, can be interpreted as indicative of the formation of a loosely bound iodine compound. L. B. Rogers has suggested that since benzene forms pi-bonded compounds with silver salts, the benzene may have been chemisorbed on the surface of the silver diffusion tube and thus inhibited the absorption of iodine. Both W. E. Browning and P. H. Emmett

agree that this could yield the observed diffusion tube results, and feel that, if so, adsorption of the solvent vapors could also modify the results of the loop experiment.

It is therefore concluded that benzene is the superior solvent for the introduction of uncombined iodine to the system, but that surface effects of the solvent could modify the loop results. An alternate technique of introducing the iodine as vapor in helium is to be recommended.

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