

SORPTION OF CESIUM BY GRAPHITES  
AS A FUNCTION OF STRONTIUM AND BARIUM  
CONCENTRATION AT HIGH TEMPERATURES

Progress Report

For Period

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## ABSTRACT

Tentative cesium desorption isotherms on H-451 graphite have been obtained for sample temperatures of 800°C, 900°C, 1000°C and 1100°C. Definite hysteresis has been noted at cesium vapor pressures below  $5 \times 10^{-6}$  atmospheres on the 1000°C isotherm. A cesium background experiment was conducted which indicated that the sorption tube background contribution is not a major factor in assessing the cesium behavior within the higher vapor pressure range of our investigations. One strontium desorption experiment was conducted, using H-451 graphite impregnated to 2.28  $\mu\text{moles Sr/gm C}$  to verify the retention of strontium in the absence of cesium vapors. Two sorption experiments on strontium impregnated H-451 graphite ( $\approx 5 \mu\text{moles/gm}$  and  $\approx 1 \mu\text{mole/gm}$ ) are about to begin.

A preliminary analysis has been made of the kinetic behavior observed in the sorption apparatus - sample arrangement and the characteristic diffusion times for cesium under various conditions. It has been concluded that the rather lengthy times required for the graphite sample to equilibrate (3 to 12 days) is determined by the diffusion of cesium atoms from graphite pore surfaces into the graphite grains. One sorption sample was radially profiled upon completion of a 1000°C sorption experiment. This profile was analyzed, using the coupled fast-slow diffusion model to further investigate the processes governing the transport of cesium in the H-451 graphite samples.

In support of the strontium impregnation technique and to assess the diffusion behavior of strontium in graphites, concentration dependent strontium diffusion studies on H-451 graphite have begun at a diffusion temperature of 1600°K. An initial uniform strontium concentration of 10  $\mu\text{gm Sr/gm C}$  was used in the first experiment. Axial profiling of the sample is under way.

Electron micro-probe qualitative analyses were conducted on H-451 graphite samples to obtain a preliminary evaluation of sample preparation and detectable impurity content. Metallic element micro-distribution investigations are continuing as time permits.

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## I. INTRODUCTION

The purpose of this research is to quantitatively determine the equilibrium vapor pressures of cesium as a function of its concentration and the graphite sample temperature for various mixtures of cesium, strontium and barium sorbed by two typical nuclear grade graphites, the "near isotropic" (Great Lakes H-451) and the needle coke (Great Lakes H-327), and a well-characterized high purity spectroscopic grade of graphite powder (Union Carbide SP-1C). Cesium sorption isotherms will be obtained for concentrations of strontium and barium ranging from that corresponding to approximately mono-layer coverage to zero concentration for the particular graphite sample over the sample temperature range of 700°C to 1100°C with cesium vapor pressures ranging from approximately  $10^{-9}$  atm to  $10^{-4}$  atm. The experimental information obtained will be analyzed to obtain a model by which the vapor pressures of cesium for mixtures of sorbed fission products may be predicted from single component sorption data.

## II. SUMMARY OF PROGRESS AND RESULTS

### A. Cesium Sorption Isotherms

Three cesium sorption experiments on H-451 graphite have been completed during this report period. The experimental conditions and resulting cesium concentrations are tabulated in Tables 1, 2 and 3. Cesium desorption isotherms have been obtained for sample temperatures of 800°C, 900°C, 1000°C and 1100°C (refer to Figures 1, 2, 3A, 3B and 4). Tentative parameters obtained by a non-weighted, linear least squares fit of the best desorption isotherm data for each sample temperature are tabulated in Table 4. Reproducibility of the 900°C and 1000°C isotherms is quite good (see Figures 2, 3A and 3B). The 800°C

and 1100°C isotherms have not yet been checked for reproducibility. The 1000°C cesium isotherm of Figures 3A and 3B is a composite obtained from the three experiments indicating very good agreement between experiments. Definite hysteresis exists at this sample temperature at cesium sample vapor pressures below  $5 \times 10^{-6}$  atmospheres. The presence or absence of hysteresis has yet to be verified experimentally at the other sample temperatures.

The cesium background experiments were conducted in order to evaluate the importance and the kinetic behavior of cesium accumulation on the sorption tube (Inconel-600) and protective sleeve (without a graphite sample); one contained a tantalum sleeve similar to what is presently in use, and one contained a molybdenum sleeve, which is being considered for future use to further minimize the tube background contribution. Unfortunately, the experiment containing the tantalum sleeve was contaminated, resulting in severe oxidation and attack of the tantalum, as noted after terminating the experiment. During the course of this experiment, a continuous increase in cesium sorption was observed at the sample end of the tube. This increase was only very weakly dependent upon the sample-end temperatures and cesium vapor pressures in the range under investigation. The experiment containing the molybdenum sleeve performed quite well (see Figures 5 and 7 and Table 5), indicating that the sorption tube background contribution may not be a major factor in assessing the kinetic behavior and cesium equilibrium concentrations of the sorption isotherms within the higher vapor pressure range of our investigations. However, another background experiment using a tantalum sleeve will be conducted to confirm this.

Two cesium sorption experiments using strontium-impregnated H-451 graphite samples are in the final stages of startup. The sample rods in these experiments have been grooved to a core diameter of 3.5 mm with 1.0 mm groove widths, leaving "fins" of 2.0 mm in thickness by 7.94 mm in diameter along the length of the rod, thereby increasing the surfact-to-volume ratio of the bulk sample. The finned rods were initially surface loaded with Sr-85 tagged dilute  $\text{Sr}(\text{NO}_3)_2$  solution and acetone to an approximate concentration of 5  $\mu\text{moles Sr/gm C}$  and 1  $\mu\text{mole Sr/gm C}$ . The actual concentration obtained after outgassing in vacuo and after the impregnation diffusion anneal will be determined in situ upon completing the pre-startup calibration of the sorption monitoring system, which is presently under way. The sorption experiment on strontium impregnated H-451 graphite ( $\approx 2.28 \mu\text{moles Sr/gm C}$ ) mentioned in the last report as being prepared at that time was not conducted. It was used instead to conduct a strontium desorption experiment in order to obtain information on the strontium desorption behavior in the absence of cesium vapor.

#### B. Cesium Sorption Kinetic Behavior

Kinetic information has been accumulated during the course of the three cesium sorption experiments completed to date. The characteristic behavior is displayed graphically in Figure 6. This information is routinely collected during an experiment to verify the attainment of an equilibrium condition prior to obtaining the cesium concentration for the sorption isotherm. The time to reach equilibrium normally varied from 60 to 280 hours; the longer times being at the lower source vapor pressures, lower sample temperatures, or after large changes in source pressure. In contrast to these rather lengthy

equilibrium times, the times to reach equilibrium for the background experiment were on the order of 200 to 600 minutes, as displayed in Figure 7.

In an effort to obtain a better understanding of the processes governing the transport of cesium in the graphite samples, selected sorption samples will be radially profiled upon completion of the sorption experiments. The profile for Experiment 5 is displayed in Figure 8. This profile has been analyzed, using the coupled fast-slow diffusion model<sup>(1)</sup>. The solid line in Figure 8 represents the fit obtained using this model. It is felt desirable to obtain and analyze a few more such profiles (such as from Experiments 7 and 9), if possible, to see if the coupled diffusion model may be properly applied to the kinetics of the pseudoisopiestic experiments.

#### C. Strontium Desorption, Impregnation and Diffusion

One strontium desorption experiment was conducted, using an H-451 graphite sample rod impregnated with 2.28  $\mu$ moles Sr/gm C. This sample was placed in an evacuated sorption apparatus without a cesium source and held at 900°C for 7 days, 1000°C for 19 days and 1100°C for 10 days. During the course of this experiment, the desorption of strontium was monitored in situ and was insignificant. The uniform radial profile obtained after completion confirmed this.

Three additional impregnation-diffusion anneals were conducted, using the King furnace to further assess and refine our experimental techniques for homogeneously impregnating the graphite sample rods with strontium. Based upon the radial profiles obtained (see Figures 9, 10

(1) L. R. Zumwalt and J. S. Phelps III, Trans. Am. Nucl. Soc. 22, 212 (1975)

and 11), the optimum annealing conditions are 3 to 5 hours at 1400°C, which occur with little or no sample surface roughening, if properly outgassed beforehand.

Strontium diffusion experiments are presently under way to determine the concentration dependent diffusion coefficients of strontium in H-451 graphite. The model used is that of a source disk of finite thickness (0.1 cm thick by 0.8 cm diameter) held in contact with a semi-infinitely long cylinder (0.8 cm diameter by 2.2 cm length). At the present time, one diffusion anneal has been completed on H-451 graphite having an initial uniform concentration of 10  $\mu\text{gm/gm}$  of stable strontium on the disk and cylinder, and 440  $\mu\text{gm/gm}$  of tagged strontium on the source disk. Axial profiling of the cylinder on the sample sectioning lathe arrangement has not been completed. It is anticipated that future diffusion experiments will be conducted in the concentration range of 10 to 1000  $\mu\text{gm Sr/gm C}$ .

#### D. Sample Characterization and Micro-Distribution Studies

The preliminary investigations needed to develop and to assess the various proposed methods for determining the metallic element micro-distribution are continuing as time permits. Efforts to develop a satisfactory auto-radiographic technique are continuing. Kodak SR-54 X-ray film was exposed to a 2.2  $\mu\text{Ci}$  source of Cs-137 for periods as long as 2 weeks with no significant results. Further attempts will be made, using faster, fine-grained films such as Kodak NS-54T and SB-54.

Electron micro-probe qualitative analyses were conducted on three H-451 graphite samples not previously exposed to cesium or strontium in an effort to evaluate the required sample surface preparation.

Trace amounts of calcium, phosphorous, potassium, chlorine and iron were found on both moderately polished samples. One polished sample also contained trace amounts of sulfur and silicon. Boron, vanadium and titanium trace impurities expected to be present at concentrations of less than 50 ppm were not detected. The sample with machined surfaces was too rough to provide any trustworthy results.

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### III. DISCUSSION

As evidenced by the results obtained during this contract period, we are continuing to direct our primary research efforts toward obtaining reliable cesium sorption information on H-451 graphite, initially, and to refine our experimental techniques before beginning an in-depth analysis of these results. An additional sorption experiment will be conducted on unimpregnated H-451 graphite to verify the present 800°C and 1100°C isotherms, and to investigate the possibility of hysteresis in the cesium vapor pressure regions below  $10^{-6}$  atmospheres. Finned samples, as previously mentioned, will be used in an effort to decrease the time required to attain equilibrium. The resulting kinetic behavior will be analyzed to provide further insight into the cesium transport mechanisms affecting this behavior.

Neglecting the end-of-run tube backgrounds, the isotherms obtained thus far are in good agreement. The concentration behavior of the three sorption experiments is inconsistent only during the cool-down stage upon termination of the experiments (compare Tables 1, 2 and 3). Consequently, the applicability of background corrections, based upon end-of-run conditions, is questionable at this time.

An analysis has been made of the kinetic behavior and characteristic diffusion times for cesium, (1) undergoing transport from the source to the sample by Knudsen diffusion through the tube of the sorption apparatus, (2) undergoing in-pore diffusion via the vapor phase and pore surfaces, and (3) undergoing bulk diffusion within the sample. It was found that Knudsen diffusion was the least rate-limiting step, with only 100 to 300 seconds required to complete a typical sorption or desorption step of the experiments. At source vapor pressure conditions of  $P_{(Cs)} = 10^{-4}$  to  $10^{-8}$  atmospheres, it

was found that in-pore vapor phase diffusion predominates over on-pore surface diffusion. In-pore diffusion times, taking into account the delaying effects of sorption, are estimated to be less than one hour, and may be no more than a few minutes. Thus it has been concluded that the delay process that determines the kinetics (corresponding to a few days being required to attain the saturation of the graphite sample) is the diffusion of cesium atoms from pore surfaces into the graphite grains. The characteristic diffusion time for this process is on the order of at least one day to at most, a few days.

Comparisons of the in situ cesium concentrations obtained by both the single channel analyzer (SCA) system data and by a least squares fit of the corresponding multi-channel analyzer data routinely agree to within 5% and always to within 10%. This agreement is to be expected, since the SCA system calibration is routinely checked at each sorption equilibrium point when the MCA is available.

The continuing research effort will be directed toward obtaining cesium sorption isotherms on strontium impregnated graphites in the priority order, H-451, H-327, and SP-1C at concentrations of approximately 300  $\mu\text{gm/gm}$  and below in the temperature range of 700°C to 1100°C. These will be followed by similar experiments, using barium impregnated graphites and barium-strontium co-impregnated samples to an extent determined by time and equipment availability. If further investigations prove fruitful, graphite sample characterization and the study of metallic element micro-distribution will be continued on a limited basis, using micro-radiography, electron microscopy and electron micro-probe analysis, as discussed previously. Also, the cesium profile and the concentration dependent strontium diffusion studies in H-451 graphite will be continued. The latter studies will initially be conducted at 1600°K in the strontium concentration range of 10 to 1000  $\mu\text{gm Sr/gm C}$ .



#### IV. CONTACTS WITH OTHER LABORATORIES

Contacts have been made and are continuing with laboratories interested in this research work. These include Brookhaven National Laboratory (Dr. Don Schweitzer), Oak Ridge National Laboratory (Dr. A. P. Malinauskas), Los Alamos National Laboratory (Mr. W. L. Kirk and Dr. B. L. Holian) and General Atomic Company (Dr. W. E. Bell). Although experimental sorption studies similar to ours are not presently being done at these laboratories, all of these parties have an interest in the results which we expect to have forthcoming in the next several months. These interests are, in general, in connection with the analysis of reactor safeguards.

#### V. PERSONNEL

Students who have worked (1975-1976) and are working on this project do so in connection with graduate research thesis requirements. They are:

Mr. Timothy D. Pyecha - for a Ph.D. thesis based on this research (all aspects of the project).

Mr. Paul T. Williams - second year graduate student - M.S. thesis (cesium profiles and strontium diffusion studies).

Mr. Naseer Kazi - first year graduate student - M.S. thesis (sample preparation and sorption experiments).

Of the Department faculty, besides the Principal Investigator, Professor Thomas S. Elleman has given technical advice and administrative support, which is greatly appreciated.

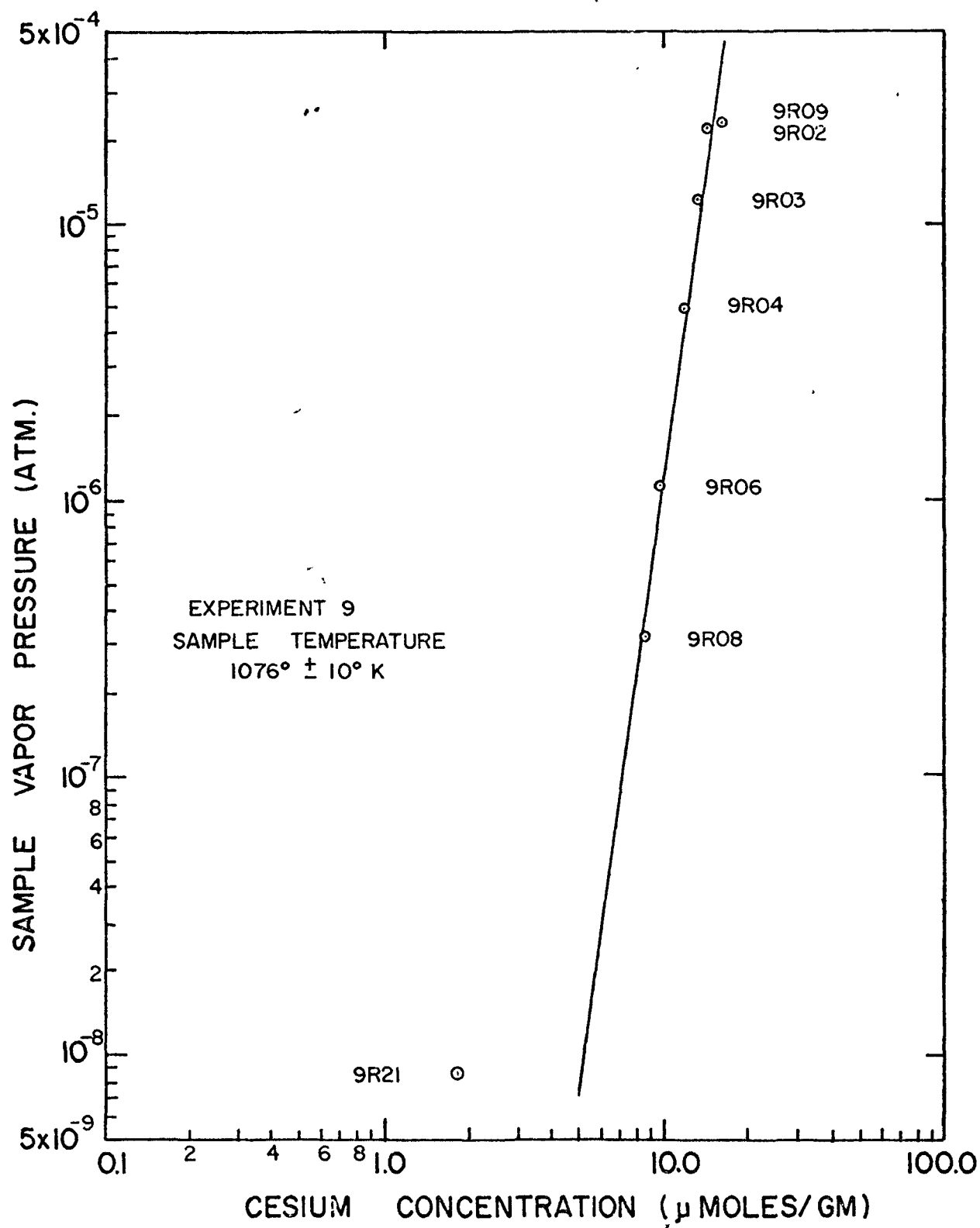


Figure 1: Cesium Sorption by H-451 Graphite at  $1076^{\circ} \text{ K}$

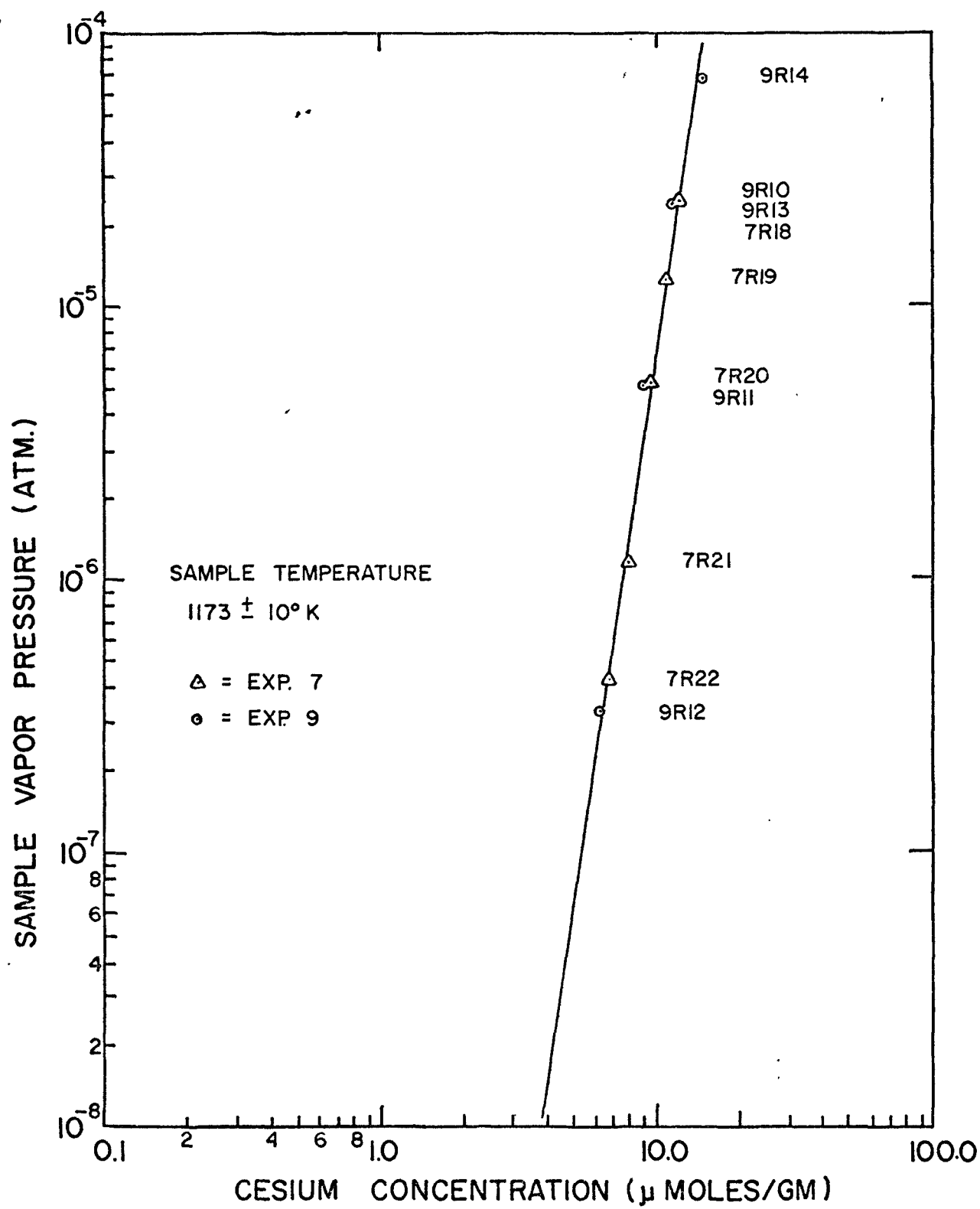


Figure 2: Cesium Sorption by H-451 Graphite at  $1173^\circ \text{K}$ .

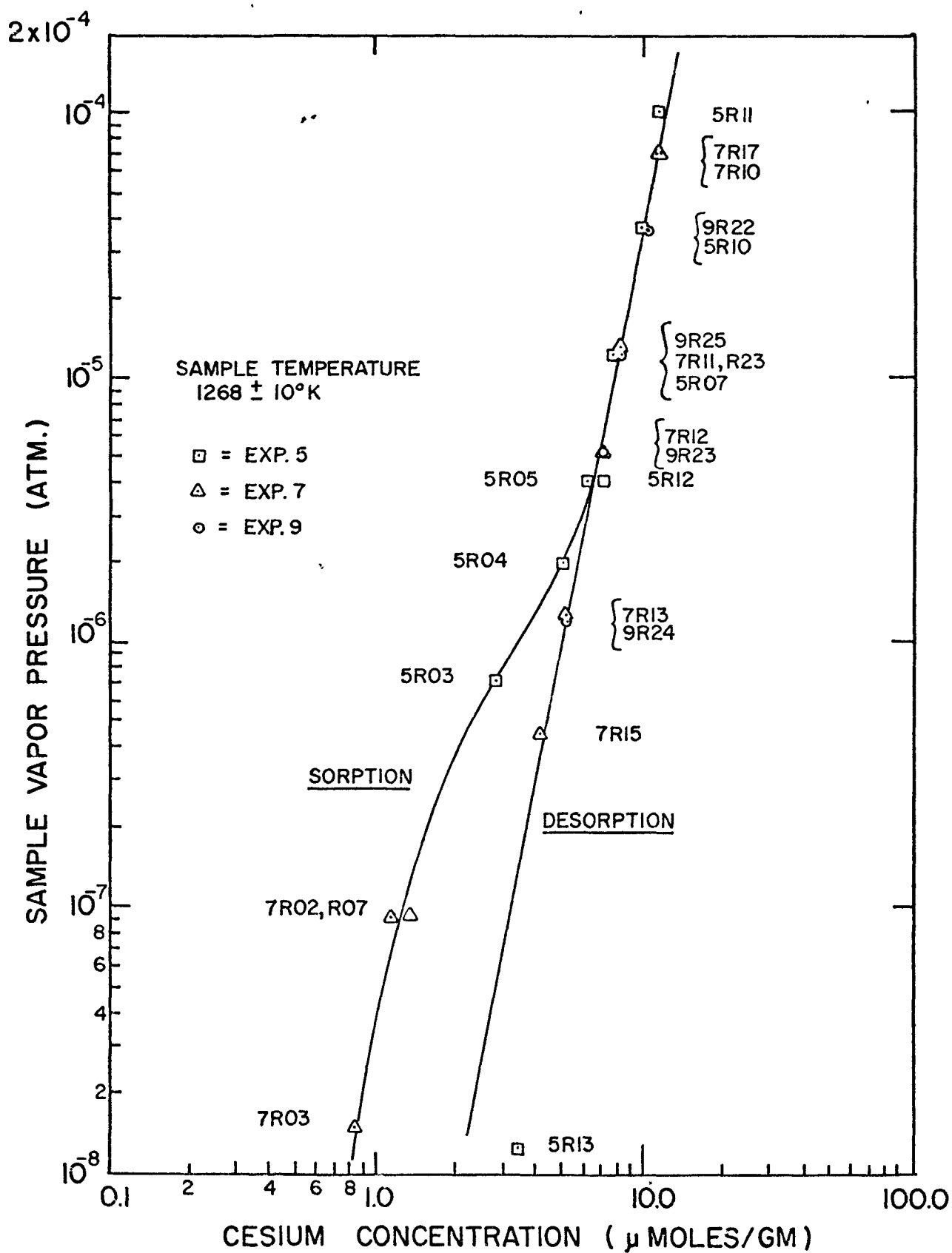


Figure 3A: Cesium Sorption by H-451 Graphite at  $1268^\circ\text{K}$

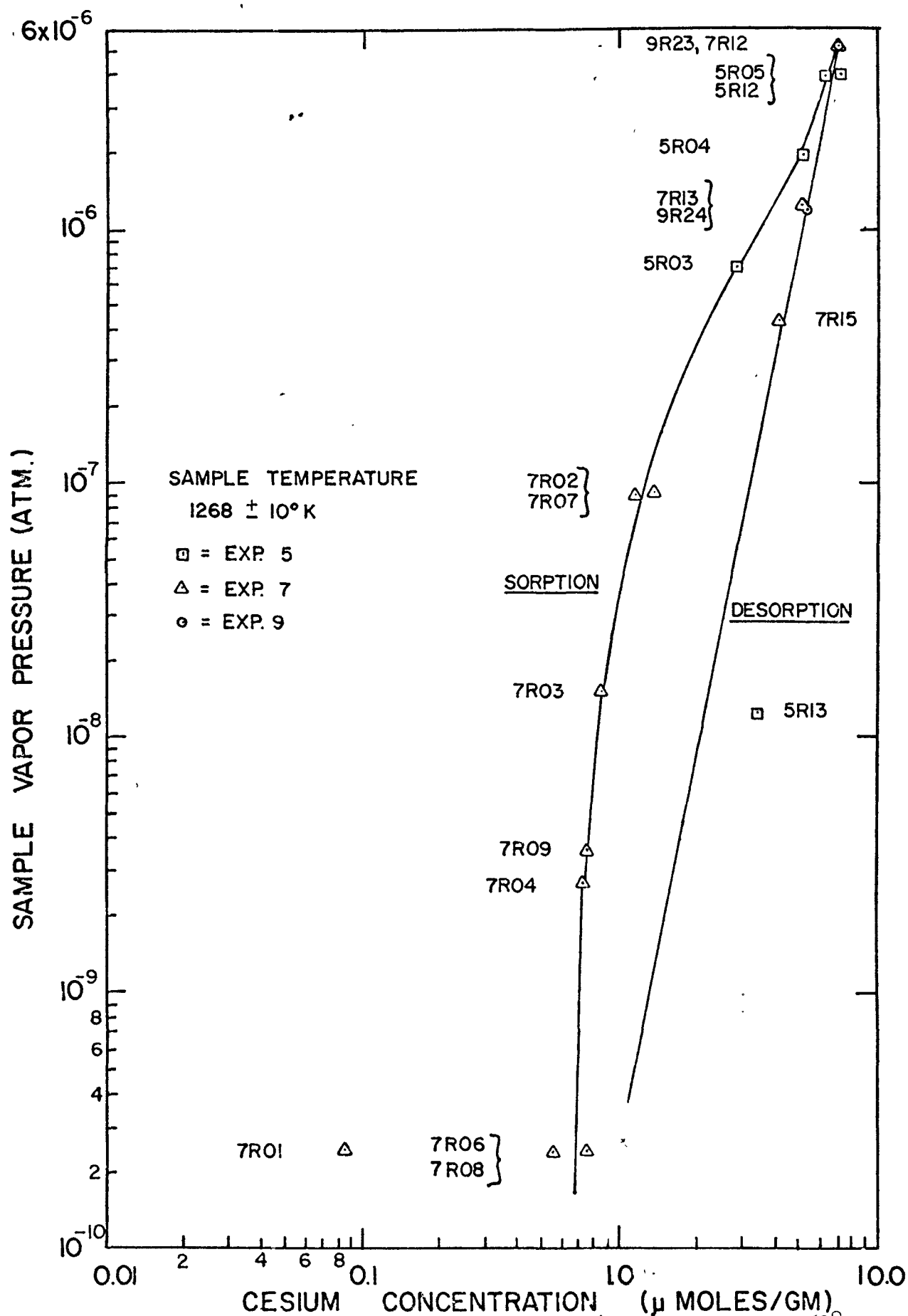


Figure 3B: Cesium Sorption by H-451 Graphite at  $1268^\circ \text{K}$

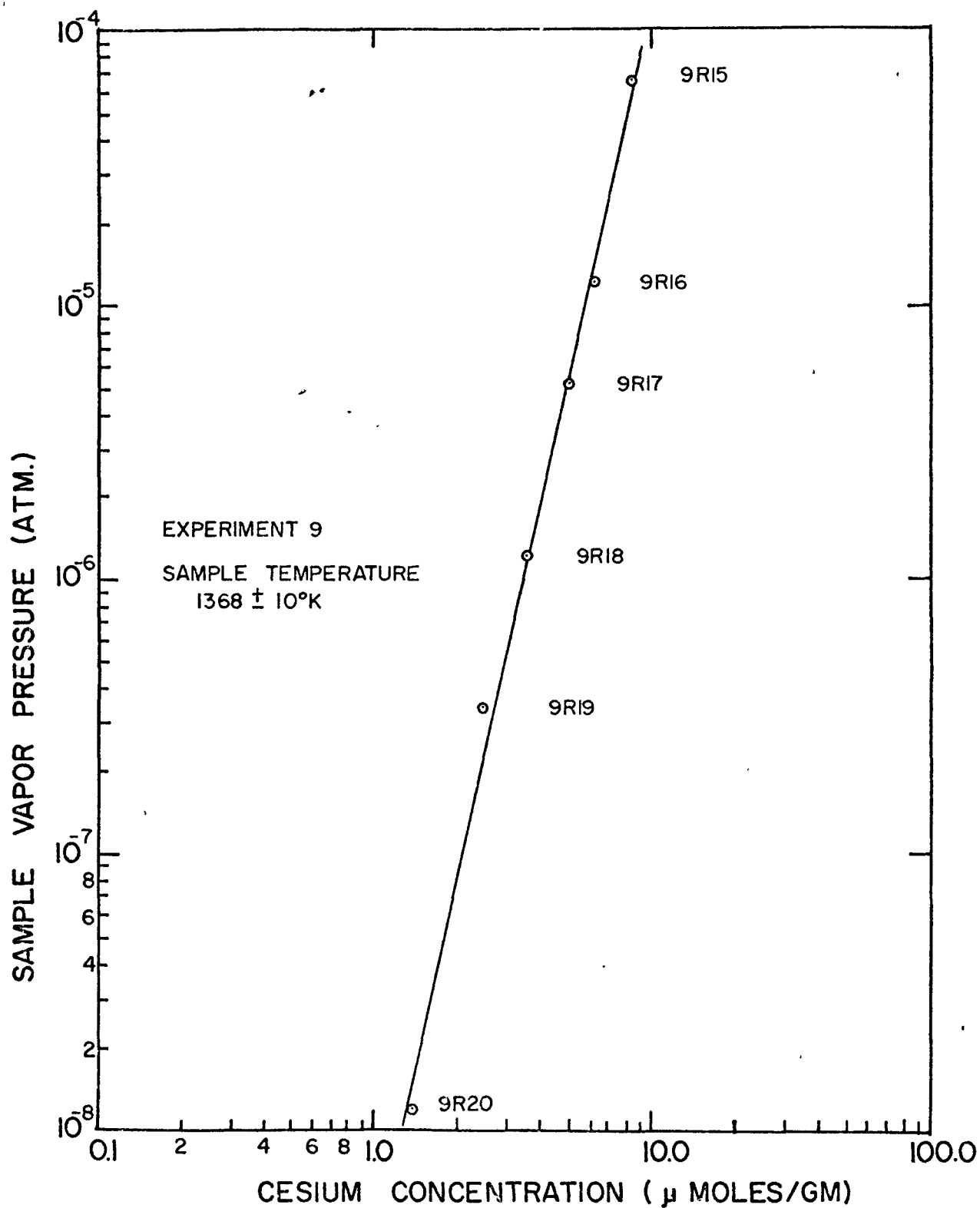


Figure 4: Cesium Sorption by H-451 Graphite at  $1368^\circ\text{K}$

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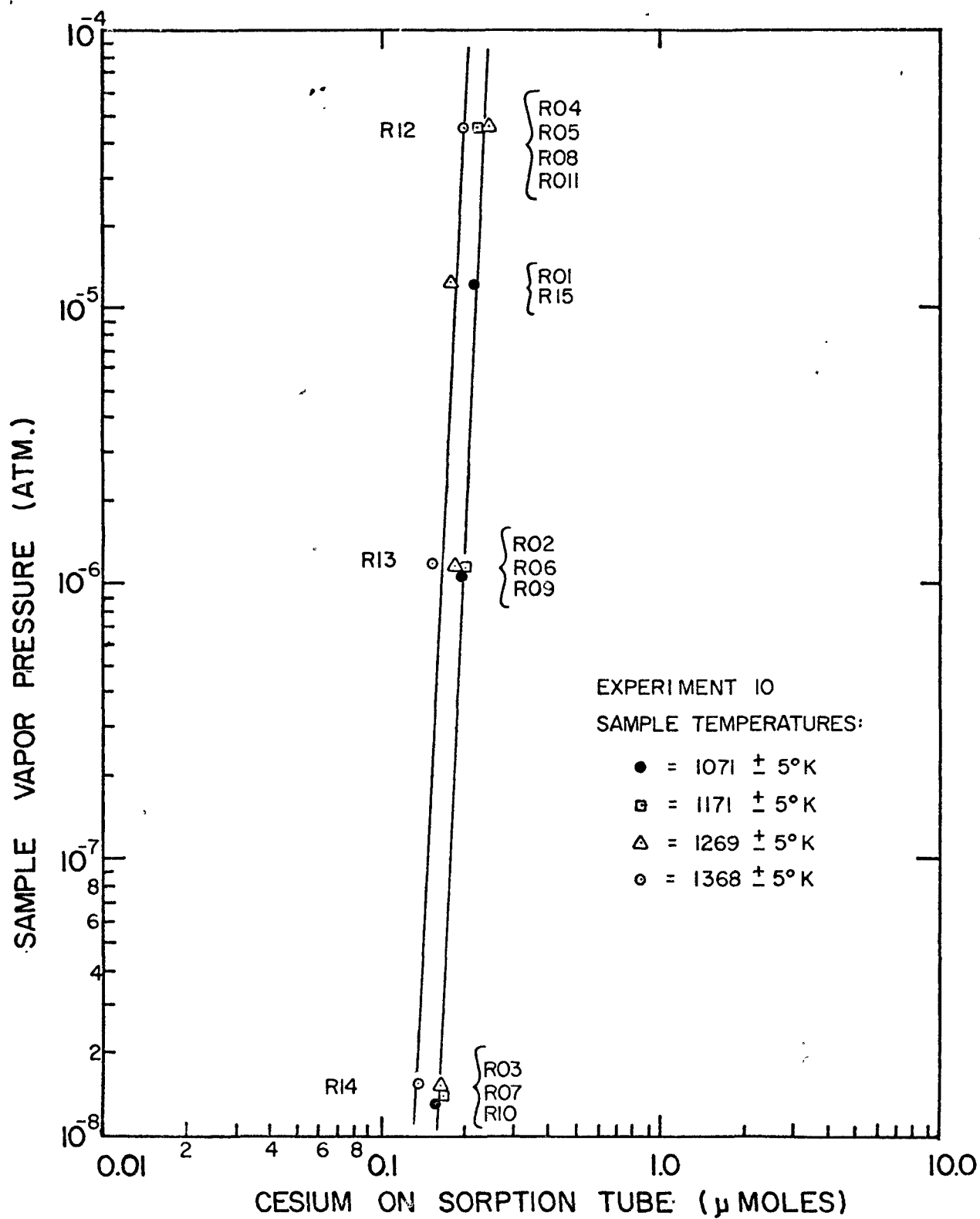


Figure 5: Cesium Sorption by Sorption Tube with Molybdenum Sleeve (No Sample)

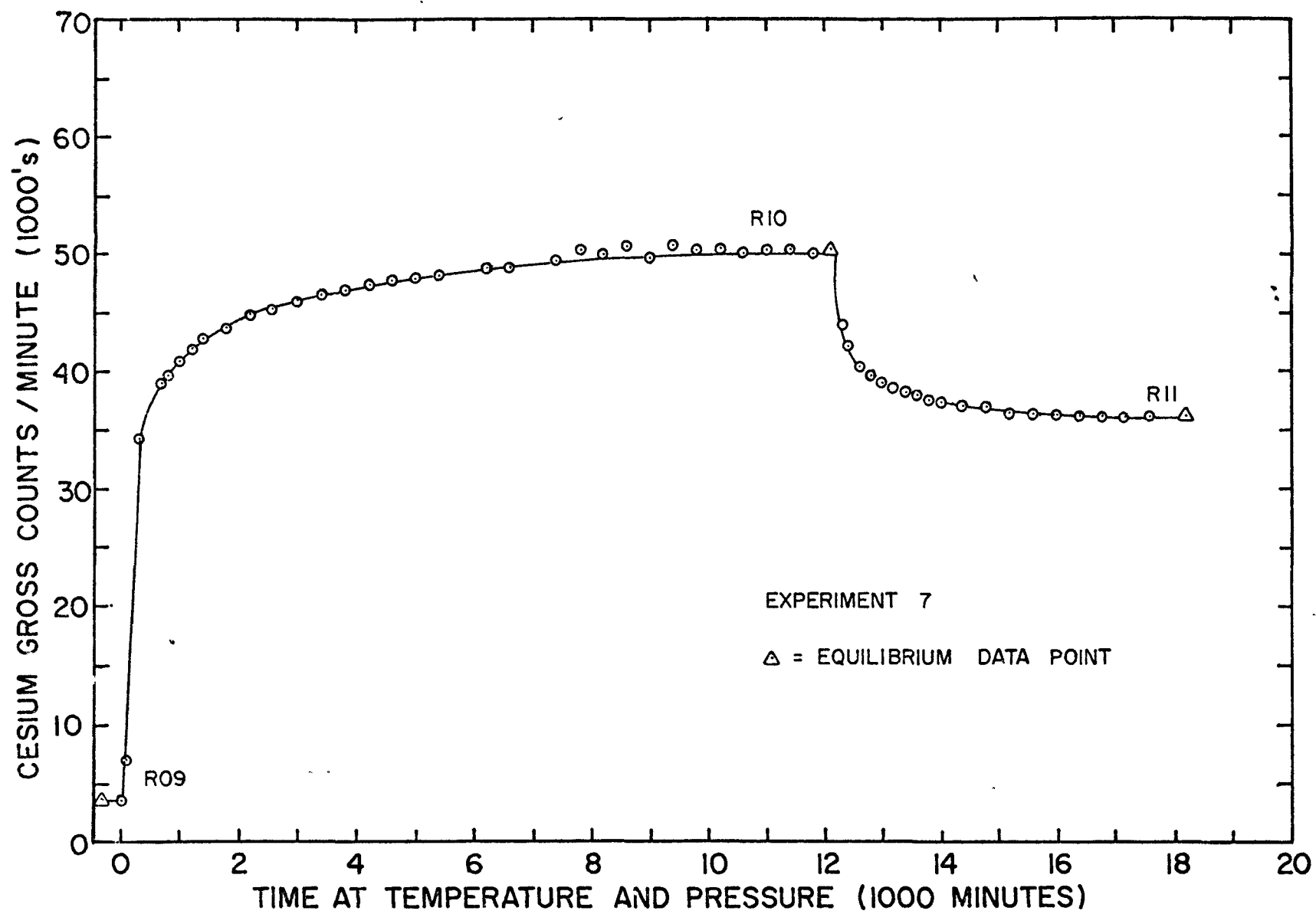


Figure 6: Typical Kinetic Behavior of Cesium on H-451 Graphite



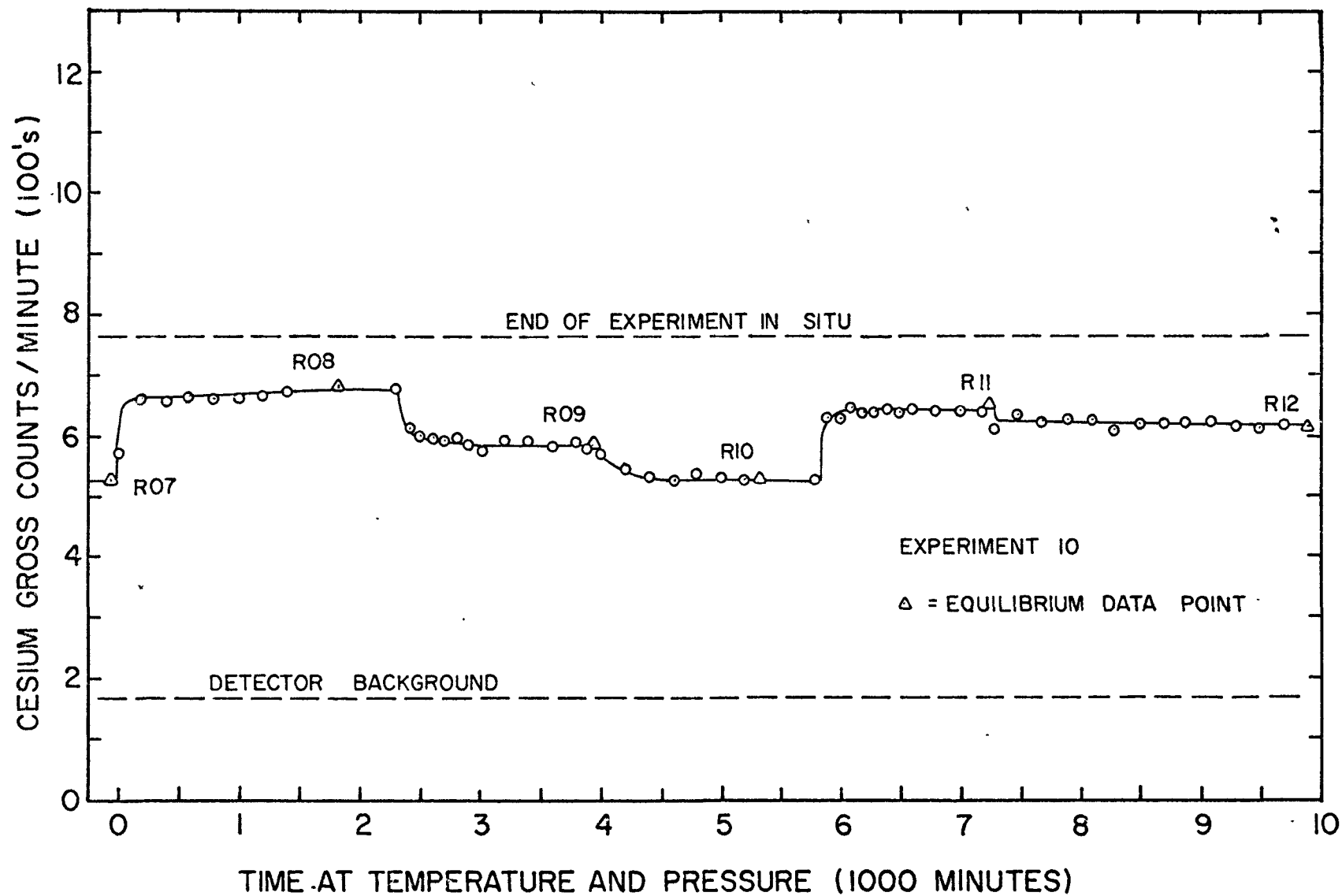


Figure 7: Kinetic Behavior of Cesium on Sorption Tube with Molybdenum Sleeve (No Sample)

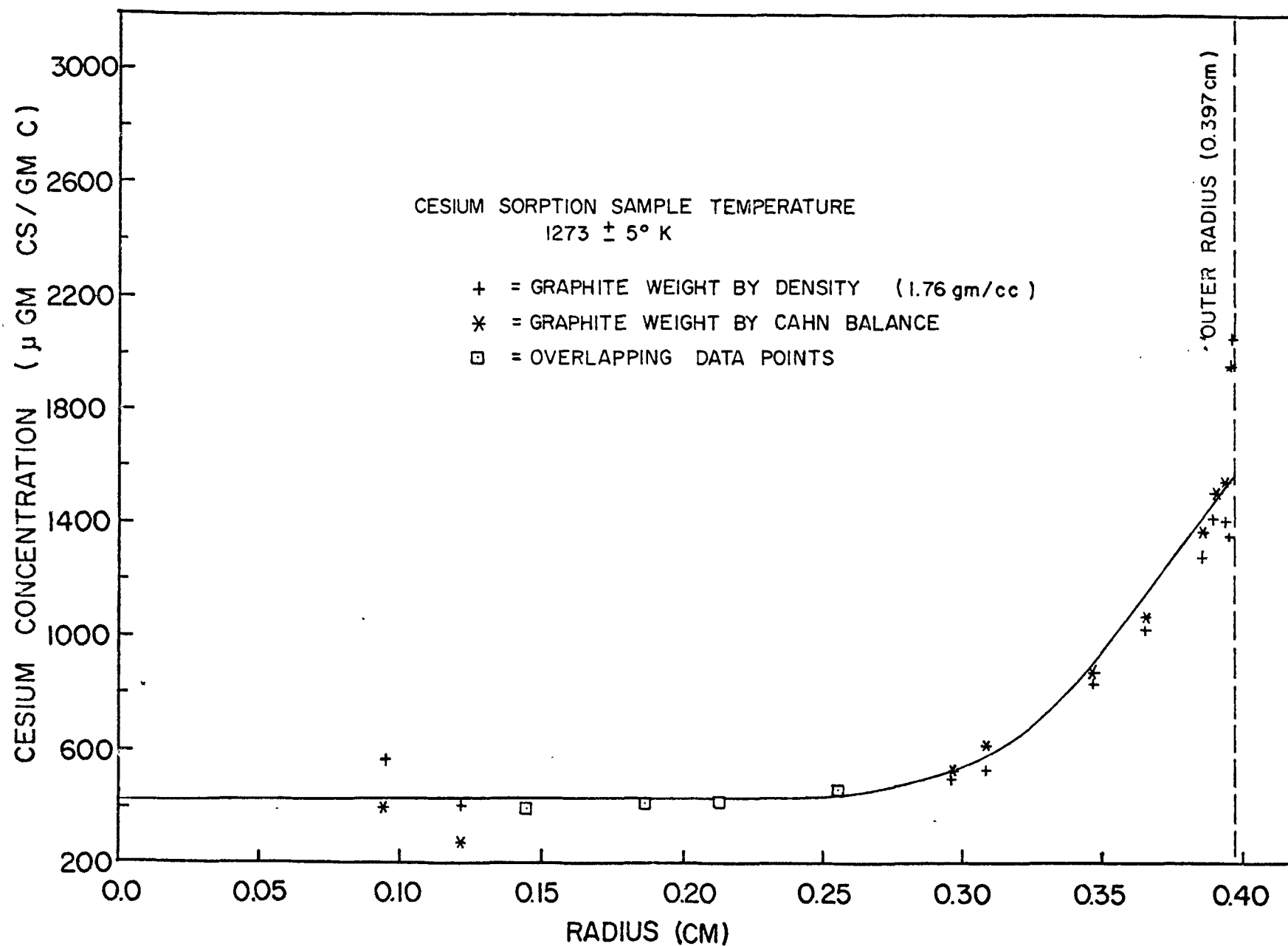


Figure 8: Radial Profile of Experiment 5: Cesium Sorption by H-451 Graphite

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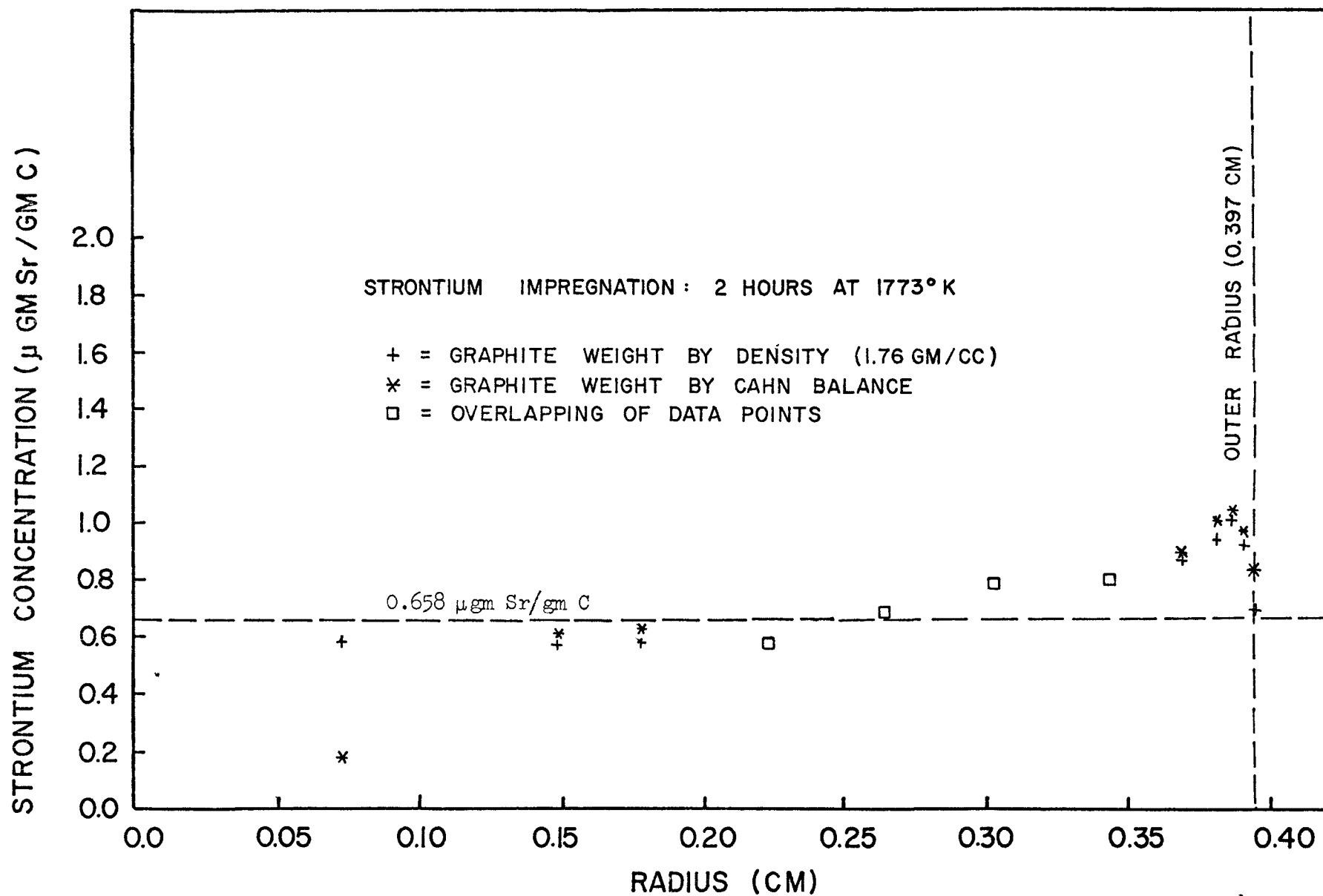


Figure 9: Radial Profile of Strontium in H-451 Graphite: Impregnated for 2 Hours at 1773°K

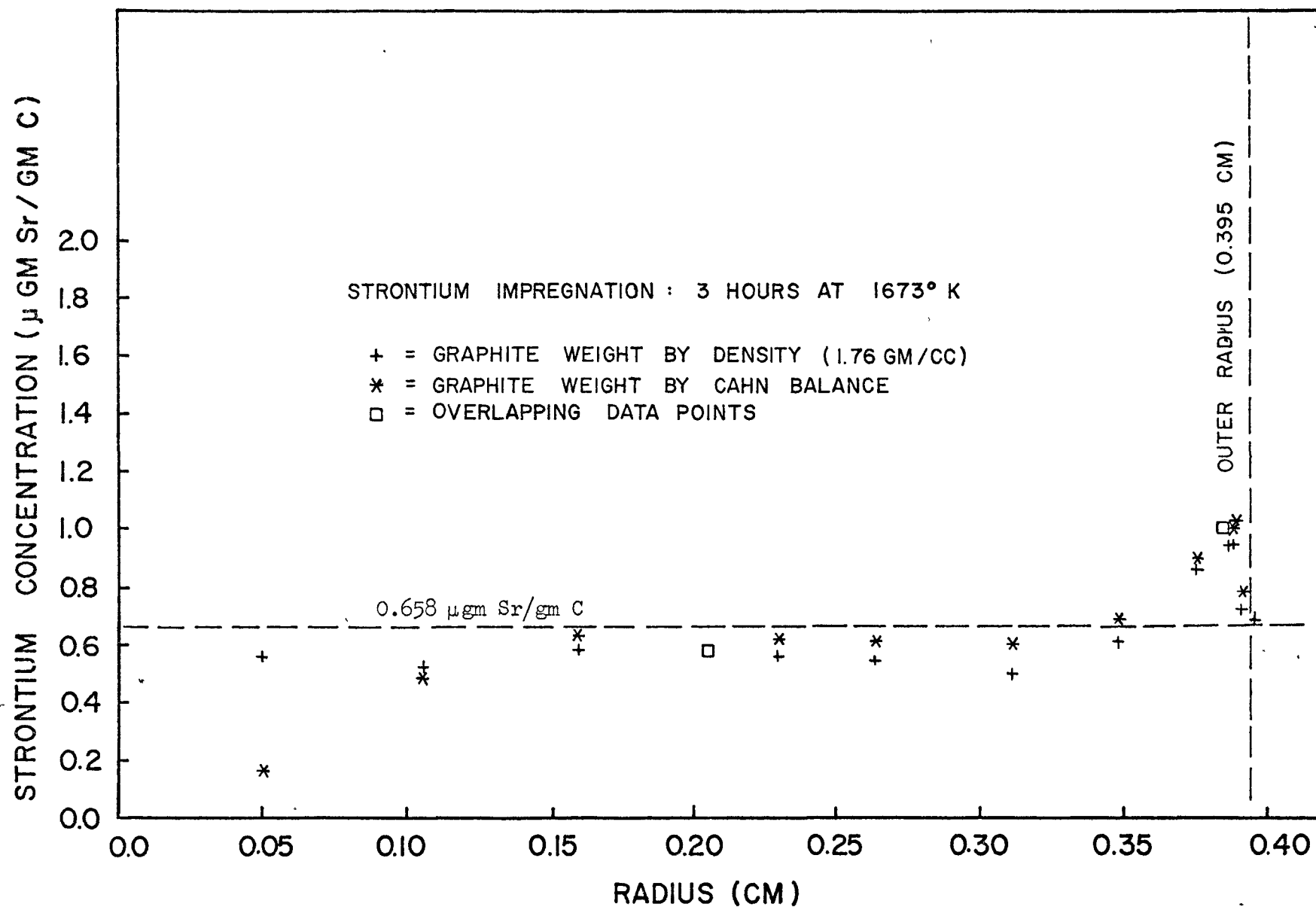


Figure 10: Radial Profile of Strontium in H-451 Graphite: Impregnated for 3 Hours at 1673°K

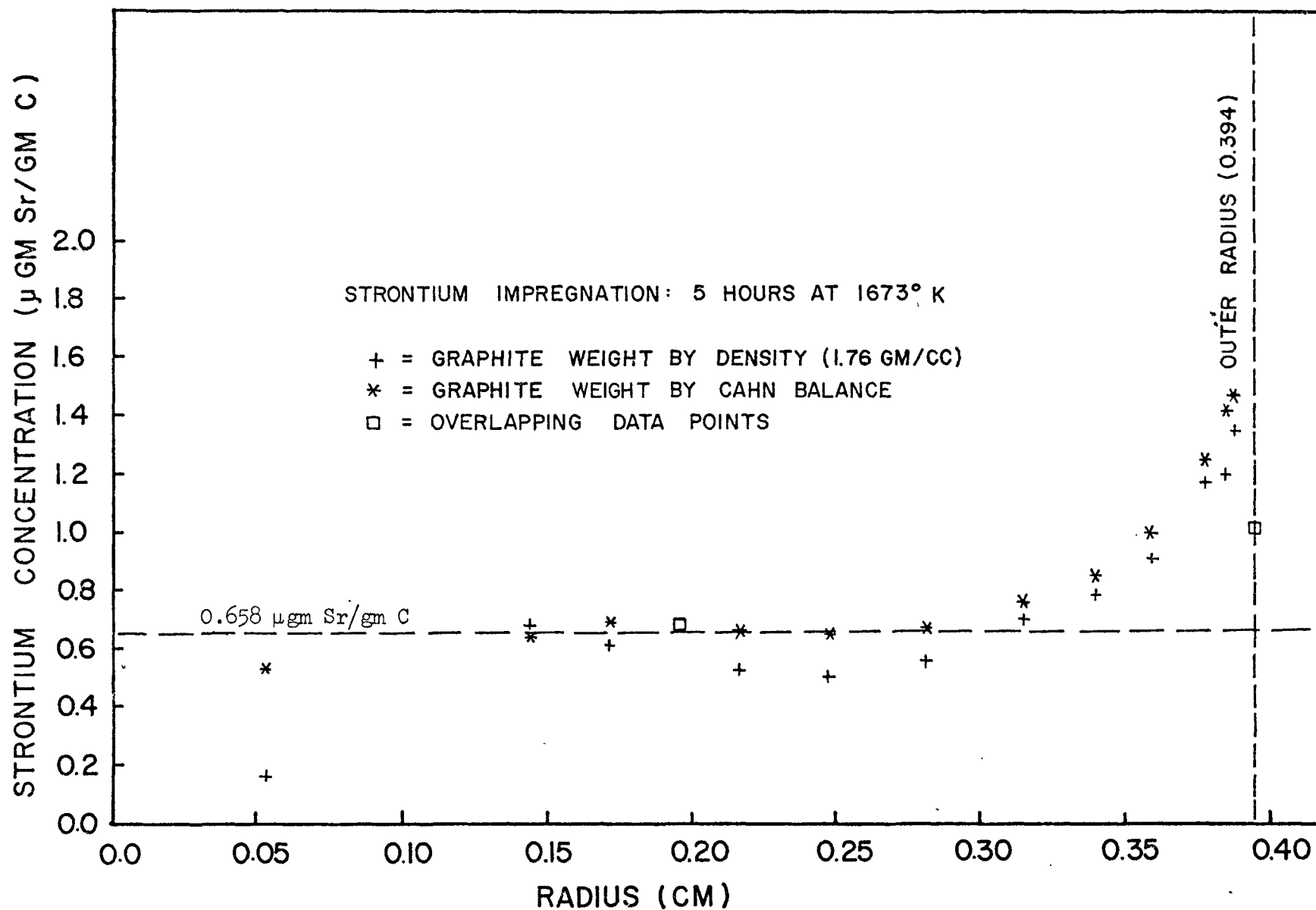


Figure 11: Radial Profile of Strontium in H-451 Graphite: Impregnated for 5 Hours at 1673°K

TABLE 1: Experiment 5: Cesium Sorption by H-451 Graphite

H-451 Sample Weight: 3.7134 gm  
 BET Surface Area: 0.42 m<sup>2</sup>/gm

Run Sequence Number	Time at Temperature (Hours)	Source Temperature ( $\pm 2^\circ\text{K}$ )	Sample Temperature ( $\pm 5^\circ\text{K}$ )	Cesium Sample Vapor Pressure (atm.)	Micromoles Cs Sorbed per Gm Graphite
R01	71.6	298	1273	3.602(-9) <sup>(a)</sup>	7.975(-2)
R02	98.2	308	1272	9.370(-9)	4.363(-1)
R03	236.6	365	1273	7.241(-7)	2.878
R04	236.4	383	1273	1.972(-6)	5.098
R05	163.4	398	1273	4.038(-6)	6.415
R07	192.5	423	1273	1.226(-5)	8.124
R10	189.5	448	1273	3.675(-5)	9.927
R11	92.7	473	1273	1.018(-4)	11.691
R12	117.9	398	1273	4.038(-6)	7.208
R13	240.8	311	1273	1.233(-8)	3.457
R14 <sup>(b)</sup>	236.7	295	295	1.285(-9)	7.378
Tube <sup>(c)</sup>					1.011
EOR <sup>(d)</sup>					6.287

(a) Read as  $3.602 \times 10^{-9}$

(b) End of Run In Situ (sample and tube)

(c) End of Run Tube Background (sample removed)

(d) End of Run Sample (change in counting system)

TABLE 2. Experiment 7: Cesium Sorption by H-451 Graphite.

H-451 Sample Weight: 3.7204 gm  
 BET Surface Area: 0.42 m<sup>2</sup>/gm

Run Sequence Number	Time at Temperature (Hours)	Source Temperature ( $\pm 2^\circ\text{K}$ )	Sample Temperature ( $\pm 5^\circ\text{K}$ )	Cesium Sample Vapor Pressure (atm.)	Micromoles Cs Sorbed per Gm Graphite
R01	97.3	273	1268	2.391(-10) <sup>(a)</sup>	8.862(-2)
R02	209.3	335	1273	9.176(-8)	1.175
R03	142.8	313	1273	1.477(-8)	8.645(-1)
R04	121.5	295	1273	2.668(-9)	7.233(-1)
R06	336.8	273	1273	2.396(-10)	5.643(-1)
R07	164.9	335	1268	9.301(-8)	1.361
R08	180.4	273	1268	2.391(-10)	7.746(-1)
R09	124.4	298 $\pm 5$	1268	3.595(-9)	7.477(-1)
R10	195.9	464	1268	7.140(-5)	11.423
R11	94.0	424	1268	1.294(-5)	8.253
R12	76.5	403	1266	5.145(-6)	7.026
R13	188.3	374	1265	1.233(-6)	5.213
R15	286.8	357	1264	4.384(-7)	4.173
R17	160.4	464	1265	7.285(-5)	11.598
R23	102.3	423	1275	1.266(-5)	8.041
R18	92.8	438	1179	2.390(-5)	12.319
R19	117.3	423.6	1181	1.263(-5)	11.188
R20	78.2	403.6	1178	5.222(-6)	10.013
R21	137.5	373.6	1173	1.168(-6)	8.118
R22	218.0	357	1173	4.244(-7)	6.838
R24 <sup>(b)</sup>	52.3	273	295	1.024(-10)	23.880
Tube <sup>(c)</sup>					4.554(-1)
EOR <sup>(d)</sup>					23.644

(a) Read as  $2.391 \times 10^{-10}$

(b) End of Run In Situ (Sample and Tube)

(c) End of Run Tube Background (sample removed)

(d) End of Run Sample (change in counting system)

TABLE 3. Experiment 9: Cesium Sorption by H-451 Graphite

H-451 Sample Weight: 3.7044 gm  
 BET Surface Area: 0.42 m<sup>2</sup>/gm

Run Sequence Number	Time at Temperature (Hours)	Source Temperature (+ 2°K)	Sample Temperature (+ 5°K)	Cesium Sample Vapor Pressure (atm)	Micromoles Cs Sorbed per Gm Graphite
R01	117.6	295	297	1.289(-9) <sup>(a)</sup>	2.847(-2)
R02	294.0	437	1073	2.285(-5)	14.555
R03	78.3	423	1077	1.231(-5)	13.557
R04	190.3	403	1076	5.054(-6)	11.883
R06	287.0	373.6	1078	1.134(-6)	9.695
R08	162.1	353.6	1078	3.236(-7)	8.692
R09	214.8	438	1077	2.387(-5)	16.331
R21	423.3	308	1076	8.621(-9)	1.835
R10	79.8	438	1173	2.390(-5)	12.008
R11	139.1	403	1173	5.104(-6)	9.315
R12	219.6	353	1175	3.252(-7)	6.371
R13	170.0	438	1173	2.390(-5)	12.097
R14	99.5	463	1173	6.857(-5)	15.407
R15	64.4	463	1367	6.860(-5)	8.584
R16	61.6	423	1367	1.242(-5)	6.228
R17	56.2	403.6	1366	5.300(-6)	5.141
R18	116.7	373.6	1366	1.229(-6)	3.554
R19	167.6	352.8	1368	3.422(-7)	2.496
R20	214.8	310.6	1368	1.222(-8)	1.379
R22	173.0	447.6	1271	3.605(-5)	10.065
R23	139.7	403.4	1271	5.205(-6)	7.169
R24	167.5	373.6	1271	1.200(-6)	5.310
R25	119.4	422.8	1271	1.225(-5)	8.308
R26 <sup>(b)</sup>	20.3	294	294	1.285(-9)	9.208
Tube <sup>(c)</sup>					5.212(-1)
EOR <sup>(d)</sup>					9.723

(a) Read as 1.289 x 10<sup>-9</sup>

(b) End of Run In Situ (sample and tube)

(c) End of Run Tube Background (sample removed)

(d) End of Run Sample (change in counting system)



TABLE 4. Cesium Desorption Isotherms on H-451 Graphite

Non-weighted linear least squares fit of the individual isotherms for:

$$\ln (P) = A + B \ln(C)$$

where

P = cesium sample vapor pressure (atm.)

C = cesium concentration (  $\mu$ moles Cs/gm C)

Sample Temperature ( $\pm 10^\circ\text{K}$ )	A	B
1076	-30.498	7.308
1173	-27.248	6.599
1268	-22.269	5.207
1368	-19.487	4.578

TABLE 5. Experiment 10: Cesium Sorbed by Sorption Tube with Molybdenum Sleeve.

Run Sequence Number	Time at Temperature (Hours)	Source Temperature ( $\pm 2^{\circ}\text{K}$ )	Sample Temperature ( $\pm 5^{\circ}\text{K}$ )	Cesium Sample Vapor Pressure (atm.)	Cesium Sorbed ( $\mu\text{moles}$ )
R01	49.2	423	1071	1.22(-5) <sup>(a)</sup>	0.2145
R02	100.9	374	1071	1.09(-6)	0.1920
R03	59.1	315	1071	1.36(-8)	0.1574
R04	53.4	452	1071	4.54(-5)	0.2412
R05	39.1	452	1171	4.54(-5)	0.2401
R06	66.5	373	1171	1.125(-6)	0.1948
R07	36.1	314	1170	1.42(-8)	0.1647
R08	31.8	452	1270	4.54(-5)	0.2354
R09	27.1	373	1268	1.156(-6)	0.1896
R10	24.8	315	1269	1.48(-8)	0.1637
R11	23.4	452	1269	4.54(-5)	0.2224
R15	85.3	423	1267	1.23(-5)	0.1777
R12	43.3	453	1363	4.54(-5)	0.2050
R13	43.0	373	1368	1.18(-6)	0.1530
R14	20.8	315	1368	1.54(-8)	0.1379
R16 <sup>(b)</sup>	28.7	295	295	1.285(-9)	0.2757

(a) Read as  $1.22 \times 10^{-5}$

(b) End of Run In Situ