ARGONNE NATIONAL LABORATORY
9700 South Cass Avenue
Argonne, Illinois 60439

MEASUREMENT OF THE ADSORPTION COEFFICIENTS
OF ARGON ON ACTIVATED CHARCOAL

by

A. F. Panek, C. C. McPheeters,
and H. C. Welborn*

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*Chemistry Department, Centenary College, Shreveport, Louisiana.

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ABSTRACT

The adsorption coefficients for argon on activated coconut charcoal have been measured to aid in the prediction of the behavior of argon in a system for monitoring fuel-cladding failures. In this study, two separate procedures were used to determine the dynamic adsorption of argon on activated coconut charcoal at temperatures from -78 to 140°C . The adsorption isobars for argon obtained by the two methods are in close agreement; moreover, values for xenon and krypton determined by the same methods agree well with values published in the literature.

I. INTRODUCTION

Operation of fast breeder reactors such as the Fast Flux Test Facility (FFTF), currently being built at Richland, Washington, will require that fuel-cladding failures be detected rapidly and reliably. One method of detecting fuel-cladding failures currently under development involves sampling the reactor cover gas and analyzing it for the radioactive fission gases, xenon and krypton, which are released when a fuel-cladding failure occurs. Unfortunately, radioassay of the cover gas is complicated by the presence of ^{23}Ne and ^{41}Ar (activation products of ^{23}Na , ^{41}K and ^{40}Ar). These isotopes emit high-energy gamma rays and a Compton continuum over the low-energy region of the gamma energy spectrum. Most of the xenon and krypton isotopes of interest emit gamma rays in the energy range covered by this Compton continuum, and their energy peaks are very small relative to the very large Compton "background". The small peaks therefore become lost in the statistical variation of the Compton "background".

The currently proposed reference system for monitoring the argon cover gas of the FFTF for gaseous fission products includes a gas-chromatographic technique for the separation of ^{23}Ne and ^{41}Ar from samples of cover gas, which are taken automatically and repeatedly at regular intervals.¹ A sample is transferred into a stream of argon carrier gas that sweeps it onto the chromatographic column, and the carrier gas flow is continued until the ^{23}Ne and ^{41}Ar are eluted from the sample. The flow is then stopped and the xenon and krypton isotopes remaining on the column are counted with a lithium-drifted germanium [Ge(Li)] detector. The entire process is automated so that the sampling, elution, isolation, and counting are done repeated on a 5-min cycle. The time required for transfer of the gas from the reactor to the sample point is 2 min. Therefore, the maximum time for detection of an increase in cover gas activity is 7 min. The design specification for this system has been completed,² and a prototype is currently being installed on EBR-II for testing.

Although the chromatographic system provides the capability of sampling and analyzing the cover gas with very little time delay, it is a relatively complex system because it combines automatic operation with maximum flexibility. Hence, the system may require considerable maintenance for proper operation.

In an effort to reduce the complexity of the cover gas monitor, an alternative method was proposed by Holt.³ This method provides for a simple delay line between the reactor and the sampling point to allow the decay of ^{23}Ne (38-sec half-life) to an acceptable background level; in FFTF, the required delay time is 10-15 min. Because the half-life of ^{41}Ar is relatively long (1.83 hr), this delay does not give adequate separation of ^{41}Ar from the xenon and krypton isotopes. Hence, a small charcoal bed is provided near the Ge(Li) detector to delay the passage of xenon and krypton while allowing the ^{41}Ar to pass through relatively unretarded. Since the xenon and krypton isotopes pass through the bed at a much lower rate than ^{41}Ar , their concentrations are increased relative to the ^{41}Ar and their gamma count rates become acceptably high relative to the ^{41}Ar Compton background.

Although the delay-bed system offered apparent advantages in simplification of equipment, additional information in several areas was required before a complete evaluation of the two systems could be performed. One area in which no information was available was the adsorption on charcoal of ^{41}Ar from an argon carrier stream under the moderate temperature conditions of the proposed system, namely $40 \pm 2^\circ\text{C}$.

This report describes the measurement of the adsorption coefficients of argon on activated charcoal by two methods: a chromatographic method; the coefficients were determined as a function of temperature and carrier gas, *i.e.*, for each temperature, helium, neon, and krypton were used as carrier gases and the adsorption coefficient of argon was determined. These data were then interpolated to estimate the adsorption coefficient of ^{41}Ar from argon carrier gas on charcoal in a flowing gas on charcoal in a flowing gas system. In the adsorption-desorption method the adsorption coefficients for argon on charcoal were determined as a function of temperature. The studies were performed over the temperature range -78 to 140°C .

II. THEORETICAL

The adsorption of gases on charcoal has been studied since the phenomenon was first observed by C. W. Scheele in 1773; however, only until after Polanyi⁴ postulated his potential theory in 1914 was there a basic thermodynamic understanding of the phenomenon. The Polanyi theory states that adsorption is caused by the attraction of the gas molecules by a potential which exists near the surface of the solid adsorbent. According to the theory, all adsorption data for a single adsorbent and adsorbate can be reduced to a single curve which is characteristic of the potential field of the solid and the magnitude of its interaction with the gas. The theory has been experimentally verified many times and is considered fundamentally sound, primarily because it attempts no physical description of adsorption.

The adsorption of argon on charcoal has been measured several times under various conditions. One of the earliest measurements was that of Peters and

Weil,⁵ in which the adsorption of argon on an unspecified type of charcoal was determined as a function of temperature and pressure. More recently, von Antropoff⁶ also determined the adsorption of argon on charcoal at various temperatures. The spread between the data of von Antropoff and that of Peters and Weil is greater than one order of magnitude, ranging from 22 cm³(STP) to 270 cm³(STP) at -78°C (von Antropoff's value is the lower). Collins studied the adsorption of noble gases on many types of charcoal. Although Collins⁷ and co-workers were primarily interested in measuring xenon and krypton adsorption from argon, they also determined the adsorption coefficient of argon. This argon adsorption isobar lies between the data of von Antropoff and that of Peters and Weil.

In 1959, Adams *et al*⁸ presented adsorption coefficients for krypton and xenon on charcoal in the range from 25 to 150°C and also presented evidence to indicate marked reduction in the adsorption coefficient occurs when the carrier gas is more strongly adsorbed than the minor constituent. Siegwarth *et al*⁹ corroborated the data of Adams *et al*, and indicated that, of those charcoals tested, Columbia G* exhibited superior adsorption properties.

We concluded that, of the many types of charcoal previously investigated, each type exhibited its own unique adsorption coefficients for the noble gases of interest. As a result of this variation, the values of adsorption coefficients vary over a range greater than one order of magnitude. On the basis of Siegwarth's work with xenon and krypton, Columbia G activated coconut charcoal was chosen for our study of the adsorption of argon.

The adsorption coefficients of the gases injected on a charcoal column can be calculated using the equation presented by Siegwarth:

$$t_r = \frac{m k}{F} (N - 1)/N \quad (1)$$

where

t_r = the true retention time on the column, sec

m = the mass of charcoal in the column, g

k = the adsorption coefficient, cm³(STP)/g

F = the carrier flow rate, cm³(STP)/sec

N = the number of theoretical plates in the column.

The relationship for calculating the number of theoretical plates, N , is $N = 16 (t_r/t_w)^2$, where t_w is the observed peak width. In the chromatograms, obtained in this study, the retention times were always observed to be more than ten times the peak width; therefore, because N was always large, the $(N - 1)/N$ term was assumed to be unity in all cases. According to Coates¹⁰ this assumption is valid for long columns.

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*Trademark of Union Carbide Corporation, Carbon Products Division.

III. EXPERIMENTAL

Several methods are available for measuring the adsorption coefficients of gases on charcoal.¹¹ The methods considered in this work are modifications of published methods and, for purposes of this paper, are called the "chromatographic" and "adsorption-desorption" methods. Because of the short half-life of ⁴¹Ar (1.83-hr), it was considered impractical to use gamma-counting techniques to measure the adsorption directly. The two methods mentioned above were used in this study to provide a cross-check of the data obtained by each method and also to allow a comparison with data for xenon and krypton published in the literature.

In the chromatographic procedure, the retention times of the noble gases on a column of activated charcoal at various temperatures were used to determine their dynamic adsorption coefficients. The method consisted of injecting small gas samples into helium, neon, argon, and krypton carrier gases flowing through a Columbia G activated charcoal column. The effects of temperature and carrier gas on the adsorption coefficients were observed. The coefficients were then calculated from Eq. 1 using the mass of charcoal, the flow rate of the carrier, and the retention time of the injected gas.

The adsorption-desorption method consisted of flowing argon, krypton, and xenon gases through a small bed of activated charcoal at various temperatures until the bed was saturated, desorbing the argon, and then calculating, by means of basic gas pressure-volume formulas, the volume of gas originally adsorbed on the charcoal sample. The adsorption coefficients were determined by dividing the volume of adsorbed gas at each temperature by the mass of the charcoal in the bed.

A. The Chromatographic Method

1. Apparatus

Figure 1 shows a simplified schematic diagram of the apparatus used in the chromatographic method. It consists of a Varian-Aerograph Model 90-P gas chromatograph equipped with a column of activated charcoal and a thermal conductivity detector. The activated charcoal was contained in a copper tube, (1/4 in. in diameter by 4 ft. long), which was coiled into a configuration to fit the chromatograph oven. The temperature of the column was controlled to within $\pm 2^\circ\text{C}$ and was measured by both a mercury thermometer and an iron-constantan thermocouple. Sample injections were made at the injection port, which was positioned to minimize the distance between the injector and the column. The detector bridge circuit was connected to a variable-speed strip chart recorder, and a carrier flow rate of 60 ml/min was maintained using a Matheson* series 8110 electronic flowmeter connected to the column outlet.

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* Matheson Gas Products, East Rutherford, New Jersey.

Fig. 1. Chromatographic Apparatus.
ANL Neg. No. 308-3672

2. Procedure

The copper tube, which had been previously weighed, was tightly packed by vacuum with 60/80 mesh Columbia G coconut-base activated charcoal and then reweighed to determine the mass of adsorbent present. The column was placed in the chromatograph and activated* at 150°C under helium purge for 15 hr. After activation of the column, a sample was injected into a flowing stream of carrier gas and passed through the column. The output of the thermal conductivity detector was recorded, and the retention time was determined by measuring the distance on the recorder chart from the injection point to the peak response and then dividing by the chart speed of the recorder. A typical chromatogram showing the non-adsorbed peak and the adsorbed peak are included in Fig. 1. Retention times for argon, krypton, and xenon were determined as a function of carrier gas and temperature of the column (0 to 140°C).

In the chromatographic method, an absolute error of +5% was considered to be possible in the measurement of flow rates, retention times, and temperature. Successive measurements were reproducible to a precision of +2%.

B. The Adsorption-Desorption Method

1. Apparatus

A diagram of the apparatus used in the adsorption-desorption method is shown in Fig. 2. The apparatus consists of a vacuum manifold equipped with a pressure gauge ($0-2 \pm 0.01$ Torr), several one-liter flasks with stopcocks (for adjusting the effective volume of the manifold), and ports for attaching the adsorption and standardization loops. The adsorption loop was mounted in an arrangement which allowed rapid switching between the adsorption and desorption modes by means of special four-way valves. The design of the adsorption loops and the operation of the four-way valves are also shown in Fig. 2.

2. Procedure

A 1-g sample of Columbia G 60/80 mesh activated charcoal was sealed in a gas loop as shown in Fig. 2. After degassing and reactivating the charcoal by evacuating and heating the adsorption tubes, the void volume of the loop was measured. This measurement was done by filling the adsorption loop with helium at a known temperature and pressure (room temperature and atmospheric pressure) and then measuring the pressure change upon expanding the loop into a known volume consisting of the manifold and appropriate one-liter flasks. A standard-volume loop (volume determined by mercury displacement), was similarly filled with helium under the same temperature and pressure conditions, expanded into the same manifold volume, and the pressure change noted. From these data, the volume of the adsorption loop was calculated using the ideal gas laws.

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*Although the charcoal is activated during manufacturing to provide maximum surface area, the term "activate" as applied here means to regenerate the charcoal to its original adsorptive capacity after storage or use.

Fig. 2. Adsorption-Desorption Apparatus.
ANL Neg. No. 308-3674

The evacuated adsorption tube was first equilibrated for several minutes at the selected temperature, while the argon, krypton, or xenon flowed through the by-pass to thoroughly purge the system. The noble gas being tested was then allowed to flow through the adsorption tube ($15 \text{ cm}^3/\text{min}$) for 5 min after a constant flow rate was established. The flow was stopped, and 2 min were allowed for equilibration at atmospheric pressure. After equilibration, the atmospheric pressure was recorded, the gas in the adsorption loop was expanded into the standard volume, and the final pressure of the system was noted. Heating of the charcoal section of the adsorption loop was necessary to completely desorb the gases; the final pressure measurements were made after removing the charcoal-containing portion from the system (by valving off), and cooling the associated tubing to ambient temperature.

After desorption, the pressure of noble gas in the manifold was measured. The volume of the gas was taken to be the known volume of the manifold minus the volume of the loop (determined earlier). The gas volume at standard temperature and pressure was then calculated from ideal gas relationships. Since the loop contained exactly one gram of charcoal, this volume under standard conditions was numerically equal to the adsorption coefficient in $\text{cm}^3(\text{STP})/\text{g}$.

One problem that was encountered in this procedure was the determination of the correct free volume of the loop to be subtracted from the total argon volume. The volume of the loop was originally measured at room temperature, but during the desorption, the part of the loop containing the charcoal was heated to 250°C . Thus, part of the loop was at 250°C and part was at room temperature. The loop volume was, therefore, corrected for temperature based on the approximate fraction of the loop volume that was at the higher temperature. This procedure probably introduced a small error in the adsorption coefficients. This error is significant only in the higher-temperature determination where the adsorption coefficient is low and the volume error becomes significant in relation to the volume of gas adsorbed.

Adsorption coefficients obtained by this method have a maximum error of $\pm 5\%$, due primarily to the combined error in pressure and temperature measurements. Measurements of adsorption coefficients using this method are routinely reproducible with 1-3% precision.

IV. RESULTS

The results of the studies with chromatographic method are shown in Table 1. The adsorption coefficient for each gas (argon, krypton, and xenon) is given as a function of the carrier gas from which the sample was adsorbed and as a function of the adsorption temperature. As can be seen from these data, the adsorption coefficients increase with decreasing temperature and decreasing molecular weight (or molecular size or boiling point) of the carrier gas.

The data shown in Table 1 are plotted in Fig. 3, along with the data of previous investigators. In general, the agreement between this work and that of Siegworth⁹ is very good for the krypton and xenon values. The values for argon obtained by different investigators vary widely, depending upon the type of charcoal used.

TABLE 1. Adsorption Coefficients: Chromatographic Method (Columbia G charcoal, 60/80 mesh)

Adsorbed Gas	Temp., °C	Adsorption Coefficient, cm ³ /g from Indicated Carrier Gas			
		Helium	Neon	Argon	Krypton
Argon	0	18.3	---		9.62
	30	9.34	9.70		6.70
	60	5.59	5.74		4.68
	100	3.16	3.29		3.06
	140	2.15	2.19		2.16
Krypton	0	141	---	99.9	
	30	53.3	55.0	45.8	
	60	25.2	25.6	22.9	
	100	11.5	11.9	10.8	
	140	6.35	6.69	6.12	
Xenon	0	---	---	---	---
	30	860	---	653	---
	60	280	278 ^a	245	173
	100	86.6	97.2	80.5	71.5
	140	37.1	38.7	34.7	34.7

^aMeasured at 61°C.

The adsorption coefficients for argon determined by the adsorption-desorption method are presented in Table II and shown in Figure 4. The same temperature trend is noted in these data as was seen in the data for the chromatographic method. Within the experimental uncertainty of the adsorption-desorption method ($\pm 11\%$ at 100°C and $\pm 1\%$ at 30°C) the data for argon determined by the two methods are in reasonably good agreement. However, the krypton and xenon adsorption coefficients obtained by adsorption-desorption are significantly lower than those obtained with the chromatographic method.

TABLE II. Adsorption Coefficients: Adsorption-Desorption Method (Columbia G charcoal, 60/80 mesh)

Temp., °C	Adsorption Coefficient, cm ³ /g		
	Argon	Krypton	Xenon
-78	173.0	184	279
0	12.6	44.7	127
30	7.33	25.8	83.3
60	4.58	15.2	55.8
98	2.72	8.33	32.0

Fig. 3. Adsorption Coefficients of Argon, Xenon, and Krypton: Chromatographic Method. ANL Neg. No. 308-3671

Fig. 4. Noble Gas Adsorption Isobars: Adsorption-Desorption Method
(1.00 g Columbia G charcoal, 60/80 mesh). ANL Neg. No. 308-3673

It is not necessarily expected that the data obtained by the two methods should agree because of the effect of gas partial pressure on the adsorption coefficient and because of the tendency of the charcoal to become saturated with sample gas when it is undiluted by a carrier gas. However, in the case of argon, the adsorption is low enough that the saturation effect does not seriously reduce the coefficient at high gas concentrations. The work of Collins *et al*⁷ has shown that adsorption coefficients decrease as the concentration of the gas increases. This effect is related to saturation of the adsorption sites available in the bed. Collins⁷ data, extrapolated to one atmosphere pressure, are in excellent agreement with our data.

V. DISCUSSION

In general, the data agree well with values published in the literature where comparison with similar charcoals can be made. The agreement between the values determined in this work and literature values for krypton and xenon supports the accuracy of the adsorption coefficients determined using Columbia G coconut charcoal.

Data obtained from the chromatographic method (Fig. 3) indicate that, in general, the largest adsorption coefficients for the noble gases are obtained using the lighter (lower molecular weight) carrier gases and that the adsorption coefficient decreases as heavier carrier gases are used (in agreement with the studies of Rayney¹²). The notable exception to this trend is in the relative values obtained using helium and neon as the carrier gases. In every case, the adsorption coefficients were higher for neon carrier gas than for helium. If the molecular weight of the carrier gas were the only factor involved here, we should observe a continuous decrease in adsorption coefficients in going from helium carrier to neon to argon to krypton. Thus, it appears that properties of the carrier gas other than molecular weight affect the adsorption coefficient of the sample. Such properties as atomic radius, for example, could explain the discrepancy. The atomic radii of the noble gases increase from the smallest, neon, to helium to argon to krypton to xenon. The smaller neon atoms¹³ would be expected to interfere with adsorption of the sample gas slightly less than would helium.

As illustrated in Fig. 3, the adsorption isobar for xenon from an argon carrier falls between those from neon and krypton carriers. Also, the isobar for krypton from an argon carrier indicates less adsorption than for krypton from neon. Based on these data and the limits dictated by the degree of adsorption of argon from both neon and krypton, the adsorption of individual argon molecules from an argon carrier as a continuous process can be determined. This would be, in practice, equivalent to estimating the adsorption of ⁴¹Ar molecules in a flowing argon stream. The adsorption isobar for argon would fall between the neon and krypton carriers. This approximation is in very good agreement, over the range of temperatures studied, with the adsorption coefficients found for argon using the adsorption-desorption method as also shown in Fig. 3. The small differences found at high temperatures are probably the result of experimental limitations of the adsorption-desorption technique, in which the measurement of increasingly small amounts of desorbed gases were made with increasing possibility of error.

The nonlinear behavior of some of the adsorption isobars in Fig. 3 can be explained, in each case, by a consideration of the environment of the charcoal. Adsorption data obtained from high-molecular-weight carriers (less volatile and, thus, less strongly adsorbed) demonstrate the effect of the competition between molecules for favorable adsorption sites. In the case of xenon injected into helium or neon carriers, the large xenon molecule is energetically much more subject to adsorption than the helium or neon carrier. Since helium and neon molecules are negligibly adsorbed, their presence does not present a steric barrier to xenon adsorption. Thus, the isobar is linear in a semilogarithmic plot) within experimental error.

The effects observed when argon is injected into a stream of krypton carrier can be explained in the same manner; however, a different result is observed. The less strongly adsorbed argon molecules occupy the least favorable adsorption sites at low temperatures ($<0^{\circ}\text{C}$), resulting in a decreasing slope of the isobar. At high temperatures ($>30^{\circ}\text{C}$), however, more rapid adsorption-adsorbate exchanges and decreasing affinities of the injected gas and carrier for the charcoal allow the argon to occupy, on the average, the same number of strong adsorption sites as it does with the nonadsorbed carrier, helium. Thus, at low temperatures the isobars of gases from highly adsorbed carriers decrease in slope, and at high temperatures, the isobars closely resemble those from nonadsorbed carriers.

It can be noted that the adsorption coefficient values for argon obtained by the adsorption-desorption method agree with those obtained by the chromatographic method. Poorer agreement is seen, however, for krypton and the worst agreement for xenon.

Owing to differences in the gas concentrations and partial pressures between the two methods, it would be expected that adsorption coefficients determined by the chromatographic method would be greater than those determined by the adsorption-desorption method when conditions of low temperature and high adsorption prevail. In the chromatographic method, the noble gases are injected into carriers, which, depending on their relative volatilities, molecular weights, atomic radii, etc. may or may not serve as competitive adsorbers. This is, when the noble gas under investigation is adsorbed from a nonadsorbing carrier, for example, xenon from neon at low temperature, maximum adsorption coefficients are observed because the dilute xenon gas finds many adsorption sites available.

In the adsorption-desorption method, the molecules of the gas occupy sites which vary widely in adsorption potential. As the gas partial pressure increases, the most favorable sites are filled rapidly, leaving the less energetic sites available. This decrease in adsorption potential with increasing gas concentration (or partial pressure) is seen in the comparison of the data of the chromatographic method with that of the adsorption-desorption method.

VI. APPLICATION TO A FAST BREEDER REACTOR

As a result of the data obtained in our work, the concept of using a charcoal adsorption bed to increase the ^{41}Ar concentration for analysis has been found to be feasible. Plans are underway to test this system for possible utilization on FFTF.

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