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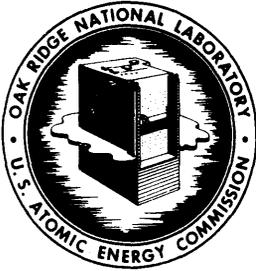
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SUBJECT: Removal of Fission Product Gases from Reactor Off-Gas Streams
by Adsorption (Presented at American Nuclear Society Meeting,
Detroit, Michigan, December 10, 1958).

TO: Listed Distribution

FROM: W. E. Browning, R. E. Adams, and R. D. Ackley

Abstract

In the operation of nuclear reactors, nuclear fuel reprocessing plants and in-pile experiments, special provision must be made for disposal of gaseous fission products to prevent contamination of the atmosphere to an unacceptable degree. A disposal process is described in which the noble gas fission products, krypton and xenon, are delayed relative to the sweep gas by physical adsorption as they pass through an adsorbent such as activated charcoal. A theoretical expression describing this process has been developed, using a theoretical plate analysis, and has been verified experimentally. The retention time for a gas present in trace concentration is proportional to the amount of charcoal in the adsorber bed and to the adsorption coefficient which is evaluated experimentally for a particular combination of materials and conditions. The retention time is inversely proportional to the volume flow rate of the sweep gas. The retention times of experimental adsorbers have been measured by a radioactive tracer technique using krypton-85 and xenon-133 to typify fission gases. Retention times have been measured for various adsorbents including different grades of activated charcoal, silica gel, activated alumina, and molecular sieve materials. Activated charcoal was the most effective adsorbent. Retention times were measured using helium, hydrogen, argon, nitrogen, oxygen, air, CO₂, Freon-12, and krypton as sweep gases, individually. Water vapor reduces the retention time for krypton on charcoal as does CO₂. Krypton interferes negligibly up to 1000 microns partial pressure. Provision must be made for the dissipation of heat from radioactive decay. Retention time decreases logarithmically as temperature increases. Ignition temperatures in oxygen were determined and methods for prevention and control of charcoal fires were investigated. Experimental results described have been used to design off-gas adsorber systems for in-pile experiments and to analyze the performance of an adsorber for a homogeneous circulating fuel reactor. These adsorber systems have performed satisfactorily for times in excess of one year.

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Introduction

In the operation of nuclear reactors, nuclear fuel reprocessing plants and in-pile experiments, special provision must be made for the disposal of gaseous radioactive fission products to prevent contamination of the atmosphere. Figure 1 shows the important fission gas isotopes. Their half-lives range from a few minutes up to ten years. Among the krypton isotopes the longest lived is krypton-85, approximately ten years, but the next longest is only four hours. The longest-lived xenon isotopes are Xe-131m and Xe-133, having half-lives of 12 days and 5.27 days, respectively; the shorter-lived one being more abundant. The longest-lived iodine isotope is I-131, having a half-life of eight days.

When a reactor accident occurs, all the radioactive isotopes shown in Figure 1 can appear in the gas system, even after removal of particulate matter. In reactors and in-pile experiments having circulating fuel, fission gases are released having the entire range of half-lives all the way down to a few seconds. By the time the off-gas reaches the disposal system, the age is such that only isotopes having half-lives greater than a few minutes are present to an appreciable extent. When solid fuels from reactors and in-pile experiments are dissolved after aging, only ten year Kr-85 and five and twelve day xenon and eight day iodine require consideration.

Various methods have been used to handle the noble gases, krypton and xenon. They have been vented to the atmosphere(8) or stored in large containers for decay. Solvent extraction has been applied successfully (9). This paper is concerned with the application of the process of dynamic adsorption of noble gas fission products.



Radioactive iodine has been removed by caustic scrubbers or by hot silver reactors. Using I-131 tracer, we have found that it is removed at least 99.9% by charcoal adsorber beds. A bed of this type for 1000 cfm of air would be 2.5 feet in diameter and 10 inches deep. This work is reported in CF-58-5-59 (1).

Dynamic Adsorption

In the process of dynamic adsorption, the noble fission gases, krypton and xenon, are physically adsorbed from a moving sweep gas, by a material such as activated charcoal in a manner similar to that used in gas chromatography. Adsorption equilibrium exists at every point within the adsorber. Only a small fraction of the krypton and xenon remain in the moving gas phase, and as a result, they are delayed relative to the sweep gas. The delay times are characteristics of the bed, which may be measured experimentally. In some applications the adsorber is regenerated continuously by radioactive decay of the adsorbed fission gases.

We have applied a radioactive tracer technique to measure retention times. A short pulse of radioactive krypton-85 is injected into the flowing gas stream at the entrance of the adsorber. The concentration of radioactive krypton is measured at the outlet of the bed as a function of time. Figure 2 shows a typical elution curve for a mixture of Kr and Xe. The ordinate is the relative concentration of radioactive gas and the abscissa is the time after injection of radio-krypton at the inlet. For each of the two elements, krypton and xenon, there is a characteristic breakthrough time, and a time to peak which for symmetrical elution curves is the average time for the passage of individual fission gas atoms through the adsorber. The breakthrough time is used for conservative design; however, the average retention time is easier to predict.

Theory

The transport of fission gases through an adsorber may be treated theoretically as shown in Figure 3 by considering a small element of length of the adsorber, dy . The rate of change of concentration of a fission gas, dP/dt , in the element dy is proportional to the concentration gradient, dP/dy , and to the volume flow rate, F , and is inversely proportional to the equilibrium adsorption coefficient, k , and to the unit loading of the adsorber m/L .

P = concentration or partial pressure of fission gas in the gas phase
(atm)

t = time at which P is observed (min.)

F = flow rate of sweep gas (cc/min.)

L = length of adsorber (cm)

m = amount of adsorbent in adsorber (gm)

k = adsorption coefficient for fission gas on adsorbent $\left(\frac{\text{cc} \cdot \text{atm}}{\text{gm} \cdot \text{atm}}\right)$
or $\frac{\text{cc(STP)}}{\text{gm} \cdot \text{atm}}$

y = distance from entrance of adsorber at which P is observed (cm)

f_0 = function which describes arbitrary variation of P at inlet of adsorber.

In the theoretical derivation k is the equilibrium adsorption coefficient relating the amount of fission gas adsorbed to the local partial pressure of fission gas. In the application of experimental results k is the dynamic adsorption coefficient which relates the average retention time to m/F . In the experimental test, f_0 describes an instantaneous pulse of radioactive fission gas. t is the time after injection of the radioactive pulse. The solution in Figure 3 states that the pulse will emerge unchanged in shape, but delayed by a time, km/F . This simple treatment yields a useful value for the time to peak. However, it indicates

erroneously that there will be no distortion of the pulse as it travels through the bed, because this mathematical treatment ignores pulse broadening processes that occur in the bed.

We have used a theoretical plate model to treat dynamic adsorption while providing for the pulse broadening processes (2). In this analysis the adsorber is imagined to be divided into a number of theoretical chambers, N , with adsorption equilibrium in each one. The differential equation in Figure 4 describes the concentration of a fission gas as a function of time in the first one of these theoretical chambers, after injection of an instantaneous pulse of fission gas. The symbols have the same significance as in Figure 3. The quantity N is the number of theoretical chambers in an adsorber having an amount of charcoal, m . The rate of change of fission gas concentration is proportional to the flow rate, F , and to the instantaneous concentration, P , and is inversely proportional to the adsorption coefficient, k , and to the weight of charcoal in a theoretical chamber, m/N . The solution for the fission gas concentration at the outlet of the last chamber is given by the second expression in Figure 4. The time to peak concentration at the outlet is shown in the third expression and is similar to the last equation in Figure 3. The time to peak or average retention time is proportional to km/F and is relatively insensitive to the number of theoretical chambers, N . The second equation in Figure 4 describes the shape of the elution curve and may be used to predict the breakthrough time.

Figure 5 compares the theoretical plate equation with experimental data. The theoretical curve has been fitted to the experimental curve by selecting values for k and N . The fit of the curves in the breakthrough region indicates the usefulness of the theoretical plate model. Using this analysis, experimental data may be reduced to two elementary parameters, k and N .

Experiments

The information on retention of fission gases under various conditions has been obtained experimentally using the apparatus shown in Figure 6. A constant flow of sweep gas, in this case oxygen, passes through an adsorber, a flow meter and past a Geiger-Müller tube. The radioactive tracer is injected at the krypton holder and the counting rate, which is proportional to concentration of tracer, is observed as a function of time by the G-M counter. Figure 7 shows the adsorbers on the right, the counters in the middle background, and instruments for registering counting rates on the left. Figure 8 shows an end-window Geiger-Müller counter tube equipped with a gas cell. Using apparatus of this type, elution curves shown in Figure 9 were obtained. Here the relative concentration of radioactive krypton in the effluent gas is plotted versus time after injection of the krypton pulse at the entrance of the adsorber. The adsorber contained 782 grams of charcoal and the sweep gas was oxygen at a flow of 250 cc/min. Elution curves are shown at four different temperatures. The lower the temperature, the longer the retention time. Refrigerated beds would be desirable except for possible failure of refrigeration. These data are analyzed to obtain the adsorption coefficient, k , as a function of temperature as shown in Figure 10.

The efficiency of various adsorbents has been investigated using this same technique. In Figure 11 activated charcoal is compared with silica gel, activated alumina, and porous glass. Various grades of activated charcoal are compared in Figure 12. Columbia Grade G is slightly superior; however, the samples are all about the same. Figure 13 shows that the performance of Linde Air Products Molecular Sieve materials is not as good as that of charcoal but where the combustibility of charcoal is a hazard, these materials are the best choice if moisture can be excluded. Sieve Material 5A is superior to others. 4A has too

small a pore size to admit krypton. Columbia Grade G activated charcoal has been used for most of the tests described below.

Figure 14 shows the effects of various sweep gases. Krypton is retained more efficiently in the presence of helium than with other gases, because helium is adsorbed to a lesser degree. Freon-12 is not suggested as a reactor sweep gas but it is used as a strongly adsorbed gas to illustrate the effect.

Figure 15 shows the effect of krypton partial pressure in oxygen with the total pressure constant at one atmosphere. In practical off-gas systems, the fission gas partial pressure does not ordinarily exceed 1 millimeter of mercury and the efficiency is not significantly affected. Figure 16 shows that small amounts of CO_2 cause a substantial reduction in the retention time. This effect can be quite important in the event of an accidental fire. One of our charcoal adsorbers serving a 5 MW reactor was ignited by an explosion of radiolytic deuterium in oxygen. Although the fire was put out without interrupting reactor operation, the combustion product, CO_2 , reduced the retention time by approximately a factor of two, temporarily, and resulted in a measurable but not hazardous release of radioactive gases. The adsorber was restored to normal service after the CO_2 had been displaced.

Water vapor has an effect similar to that of CO_2 . Figure 17 shows that the efficiency of charcoal may be reduced to one-half by adsorbed moisture. Charcoal in equilibrium with air at 50% relative humidity contains 5% water by weight.

The adsorption coefficient, k , shown in the preceding figures, is used for evaluating the average retention time, t_{max} . This quantity may be converted to the more useful breakthrough time, t_b , by the data contained in Figure 18, which applies to a 0.75 inch diameter charcoal adsorber in O_2 at 250 cc/min. The ratio of t_b to t_{max} approaches 90% for very long adsorbers. At 10 or 20 feet this ratio runs from 75 to 80%. For less than 5 feet ratios are less than 50%.

The adsorber must be fully packed. Figure 19 shows the performance of a horizontal adsorber which has only 3% of void space. The resulting tunnel provides a "short circuit" for the gas and the breakthrough time was only one-tenth of that ordinarily expected. It is desirable to install adsorbers vertically.

The adsorption coefficients for xenon have been determined for only a few conditions because only short-lived tracers are available. Xenon is retained about 10 to 30 times longer than krypton, depending upon temperature, as shown in Figure 20.

Design of Practical Adsorbers

With the theoretical results previously described and with the experimental data, we are now prepared to design a practical adsorber for krypton and xenon fission gases. In some applications it is useful to stop the flow of gas as soon as radioactivity is detected; and the retention time required is the time necessary to stop the flow of gas. In other cases it is necessary to remove fission gases from a continuously flowing gas stream. The adsorber delays the passage of radioactive krypton and xenon until they have decayed sufficiently except for Kr-85. The retention time required depends upon the concentration of fission gases entering the adsorber and upon the allowable concentration leaving. The production of the various isotopes of krypton and xenon may be evaluated using ORNL-2127 (3) by Blomeke and Todd. The allowable emission of fission gas isotopes depends upon the location of the plant and the environmental conditions. It may be limited by the permissible external or internal doses to personnel in the plant or in the surrounding community. Interference with sensitive radiation detection instruments may be the limiting factor. The report CF-58-12-10 (4) will assist in evaluating the allowable and initial emission rates.

From the ratio of input concentration to allowable output concentration and the half-life we calculate the decay time needed for each isotope and select the longest decay time required for krypton and xenon. From the last expression in Figure 4, using the approximation $(N - 1)/N = 1$, which is valid for large N , we obtain the amount of adsorbent required.

$$m = \frac{Ft_b}{kt_b/t_{\max}}$$

m = amount of adsorbent needed to provide the required breakthrough time (gm).

F = flow rate of sweep gas (cc/min).

k = dynamic adsorption coefficient obtained by retention time experiments under the conditions for which the adsorber is being designed $\left(\frac{\text{cc atm}}{\text{gm atm}}\right)$ or $\left(\frac{\text{cc STP}}{\text{gm atm}}\right)$.

t_b = breakthrough time. Selected to provide time for decay of radioactive gases or for emergency shutdown of gas flow (min).

t_{\max} = average retention time for fission gas atoms passing through adsorber. This quantity is strictly speaking the retention time for the greatest proportion of fission gas atoms and is very nearly identical to the average retention time (min).

Heat of radioactive decay may be removed by using small diameter pipes to contain the adsorbent and by immersing the pipes in cooling water. The pipes should be smaller near the entrance to the adsorber where the heat load is

greatest and may be larger in diameter near the outlet. High temperatures interfere with adsorption of the fission gases and in the presence of an oxidizing gas, can cause a fire hazard. We have observed ignition temperatures for charcoal in oxygen as low as 290°C. It is necessary to provide for measurement of the temperature of the adsorber so that if undesirable conditions occur, corrective action may be taken. A fire may be controlled safely by diverting the flow of the oxidizing gas to an alternate adsorber. This procedure has been tested experimentally as reported in CF-58-6-6 (5), and was applied successfully to a fire which occurred accidentally in a charcoal adsorber which was handling the fission gases in oxygen from a 5 MW homogeneous reactor.

Refrigerated adsorbers may be used to advantage where the size of the adsorber is important as with mobile reactors; however, it is necessary to take several precautions in their use. Failure of the refrigeration system produces an unsafe condition in which radioactive gases are desorbed as the adsorber warms up. In the applications where such a release cannot be tolerated, several alternatives are available, most of which require stopping the flow of radioactive gas from the reactor. The adsorber system may be sealed off in an emergency and designed for the very high pressures which will result or provided with an expansion volume in the adsorber system inside the shielding enclosure. Emergency stand-by refrigeration systems with an emergency power source may be provided or an auxiliary adsorber system into which the gas from the main adsorber can be allowed to expand may be used. Another hazard is the possibility of explosion in the adsorber if oxidizing gases, even in trace concentrations, are exposed to the charcoal at very low temperatures. Allowance must be made for the fact that the heat released by radioactive beta decay of the fission gases will cause the central temperature of

the adsorber to be higher than the wall temperature, and the refrigeration system must have capacity to carry away this heat of beta decay.

Applications

We have applied this information to the design of several adsorber beds. In a fused salt in-pile loop experiment we used a 34 pound charcoal bed with a gas flow rate of 20 scfm of air at 26°C. It provided a one minute retention time for krypton, sufficient for scrambling the reactor and for stopping the flow of air, leaving the krypton and xenon in the charcoal bed to decay. The procedures for designing a refrigerated adsorber for a similar application are given in CF-58-7-71 (6).

We have also applied this information to an adsorber system used on a 5 MW homogeneous reactor (7). Figure 21 shows a model of one of the three adsorbers used. Each one contains 520 pounds of charcoal in pipes ranging from 1/2 to 6 inches in diameter. Two of the adsorbers are normally used in parallel and carry a total of 2500 cc/min of oxygen contaminated with fission gases. At 26°C the retention time for krypton is six days and xenon is sixty days, sufficient for virtually complete decay of all the fission gases except krypton-85 which is vented through a small stack. This system has performed satisfactorily for more than a year.

Conclusion

The physical adsorption of noble gases upon adsorbents such as activated charcoal may be used to delay the release of gaseous fission products from nuclear reactors. Two design philosophies have been used. The first employs an adsorber

to provide a delay time during which the flow of the off-gas stream from a source of fission gases may be halted following an accidental release of radioactive gases. The second philosophy of design utilizes an adsorber to delay the radioactive fission gases for a time long compared to their half-lives so that they decay virtually completely in the adsorber. Only the ten year krypton-85 emerges from an adsorber of this type. This isotope may be discharged safely to the atmosphere under certain conditions in the case of low or medium power level reactors. High power reactors, especially near the heavily populated areas, will require an adsorber system of the first type for extracting krypton-85.

A theoretical analysis of the process whereby the fission gases are retained by an adsorber in a flowing gas stream provides the basis for design of practical units. Experimental data are provided which may be used under a wide variety of conditions in the design of adsorbers for the off-gas systems of nuclear reactors and of in-pile experiments.

The principal advantages of the dynamic adsorption process for controlling fission products are its simplicity and freedom from maintenance or replacement requirements. The entire fission gas processing unit is contained in simple metal pipes immersed in coolant water at room temperature, buried underground to confine the gamma radiation. The only requirement for instrumentation is thermocouples at a few representative points to verify the results of heat transfer calculations in the design of the adsorber and equipment for controlling or metering flow.

Acknowledgements

We acknowledge ideas contributed by D. E. Guss, C. C. Bolta, and T. W. Leland.

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9. Meyer Steinberg and Bernard Manowitz, "Recovery of Fission Product Noble Gases", Industrial and Engineering Chemistry, 51, No. 1, 47, January 1959.

Figure Captions

- Figure 1. ORNL-LR-DWG-34426, Fission Products Important in Reactor Off-Gas Streams.
- Figure 2. ORNL-LR-DWG-23006, Krypton-Xenon Adsorption.
- Figure 3. ORNL-LR-DWG-21919, Theoretical Equation for Transport of Fission Gases Through an Adsorber.
- Figure 4. ORNL-LR-DWG-21918, Theoretical Plate Equations for Fission Gas Transport.
- Figure 5. ORNL-LR-DWG-36556, Typical Distribution of Fission Gas Activity in Experiment Effluent Gas Stream.
- Figure 6. ORNL-LR-DWG-33280, Schematic Diagram of Equipment for Measuring Retention Times.
- Figure 7. ORNL-PHOTO-44658, Equipment for Measuring Retentions.
- Figure 8. ORNL-PHOTO-43100, End Window GM Tube Equipped with Gas Cell for Measuring Kr-85.
- Figure 9. ORNL-LR-DWG-23007, Effect of Charcoal Bed Temperature.
- Figure 10. ORNL-LR-DWG-23005, Effect of Temperature on k.
- Figure 11. ORNL-LR-DWG-33283, Dynamic Adsorption Coefficients for Miscellaneous Adsorbents.
- Figure 12. ORNL-LR-DWG-33281, Dynamic Adsorption Coefficients for Various Activated Charcoals.
- Figure 13. ORNL-LR-DWG-33282, Dynamic Adsorption Coefficients for Linde Molecular Sieves.
- Figure 14. ORNL-LR-DWG-33284, Dynamic Adsorption Coefficient for Columbia G Charcoal with Various Sweep Gases.
- Figure 15. ORNL-LR-DWG-33285, Effect of Krypton Partial Pressure on Dynamic Adsorption Coefficient.

Figure Captions, Continued

- Figure 16. ORNL-LR-DWG-34418, Effect of CO_2 in the Sweep Gas on the Dynamic Adsorption Coefficient k .
- Figure 17. ORNL-LR-DWG-36554, Effect of Moisture on Dynamic Adsorption Coefficient.
- Figure 18. ORNL-LR-DWG-34446, Ratio (Breakthrough Time/Average Holdup Time) as a Function of Adsorber Bed Length.
- Figure 19. ORNL-LR-DWG-23009, Effect of Void Space in Horizontal Charcoal Bed.
- Figure 20. ORNL-LR-DWG-36555, Adsorption Coefficients for Krypton and Xenon on Charcoal with Oxygen or Nitrogen Sweep Gas.
- Figure 21. ORNL-PHOTO-45639, Model of Fission Gas Adsorption System Used on 5 MW Fluid Fuel Reactor.

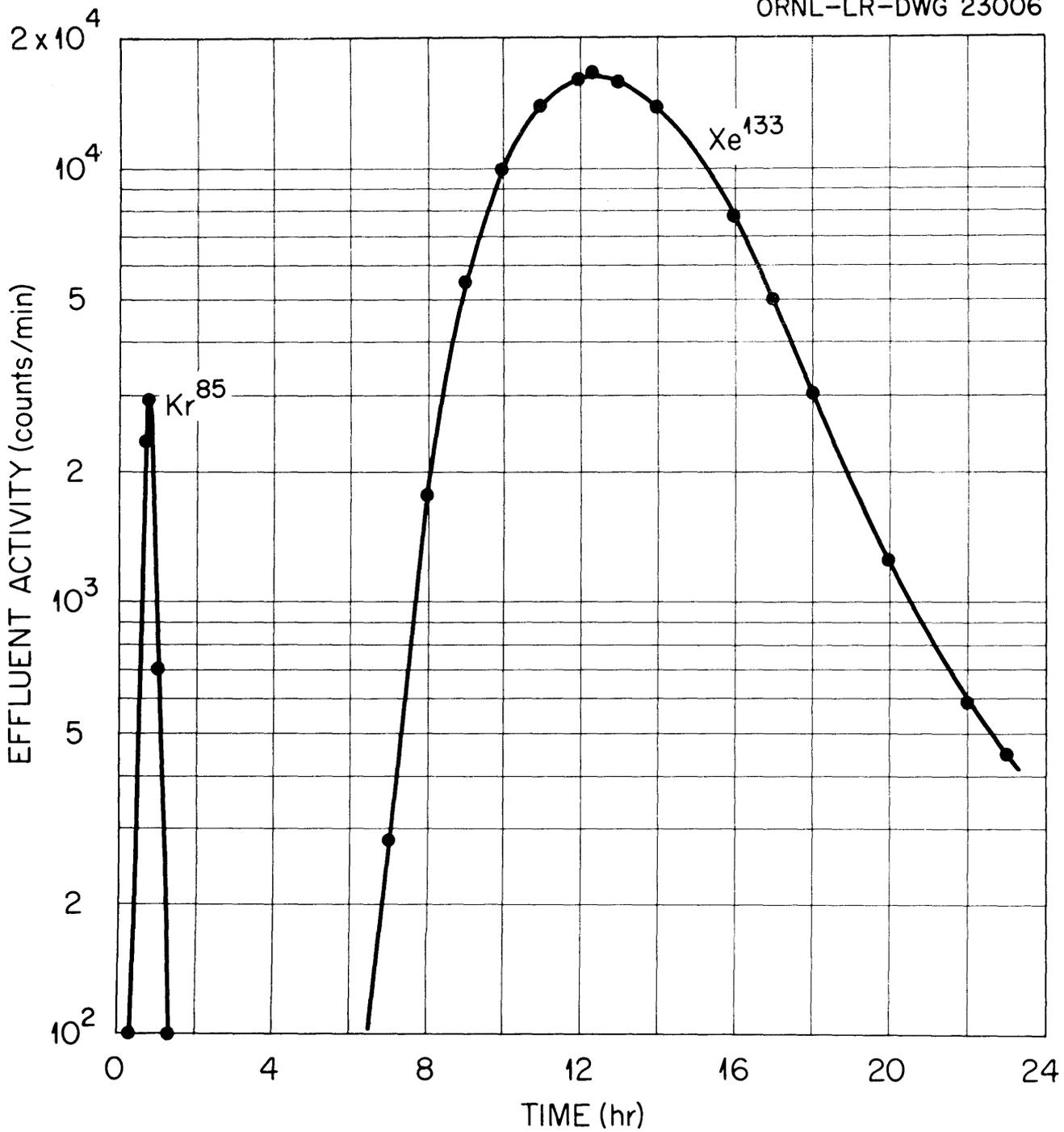
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FISSION PRODUCTS IMPORTANT IN REACTOR OFF-GAS STREAMS

ISOTOPE	HALF-LIFE	ISOTOPE	HALF-LIFE
KRYPTON		XENON	
Kr ^{83m}	114 MIN	Xe ¹³⁵	9.13 HR
Kr ^{85m}	4.36 HR	Xe ¹³⁷	3.9 MIN
Kr ⁸⁵	10.27 YR	Xe ¹³⁸	17 MIN
Kr ⁸⁷	78 MIN	IODINE	
Kr ⁸⁸	2.77 HR	I ¹³⁰	12.6 HR
Kr ⁸⁹	3.18 MIN	I ¹³¹	8.05 DAYS
XENON		I ¹³²	2.4 HR
Xe ^{131m}	12 DAYS	I ¹³³	20.8 HR
Xe ^{133m}	2.3 DAYS	I ¹³⁴	52.5 MIN
Xe ¹³³	5.27 DAYS	I ¹³⁵	6.68 HR
Xe ^{135m}	15.6 MIN	I ¹³⁶	1.43 MIN

Figure 1



Kr-Xe Adsorption

Figure 2

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$$\frac{\partial P}{\partial t} = - \frac{FL}{km} \frac{\partial P}{\partial y}$$

$$P = f_0 \left(t - \frac{km}{F} \right)$$

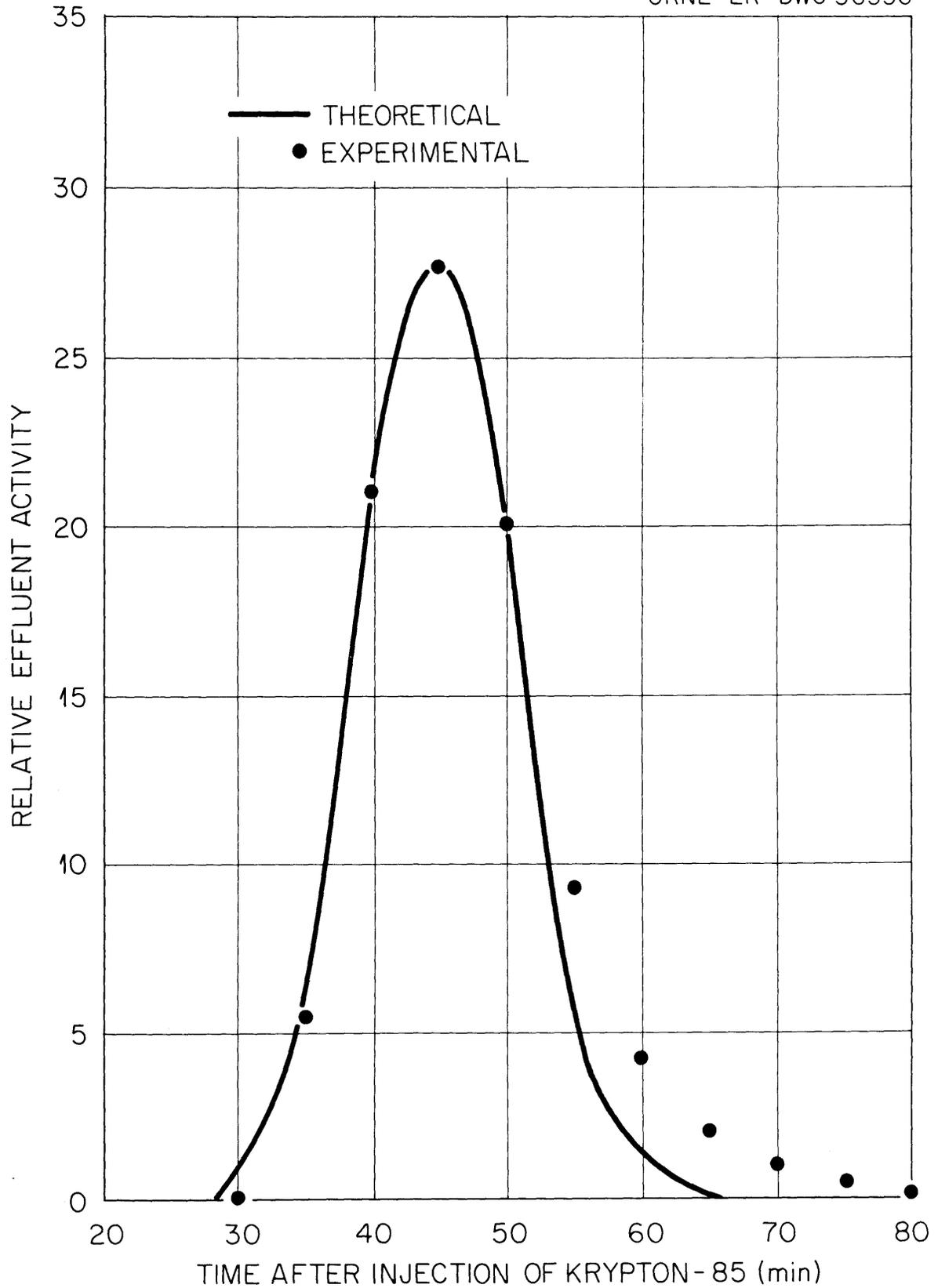
Figure 3

$$\frac{dP}{dt} = - \frac{FN}{km} P$$

$$P = \frac{N^N A F^{(N-1)} t^{(N-1)}}{(N-1)! (km)^N} e^{-NFt/km}$$

$$t_{\max} = \frac{(N-1)km}{NF}$$

Figure 4



Typical Distribution of Fission Gas Activity in Experiment Effluent Gas Stream.

Figure 5

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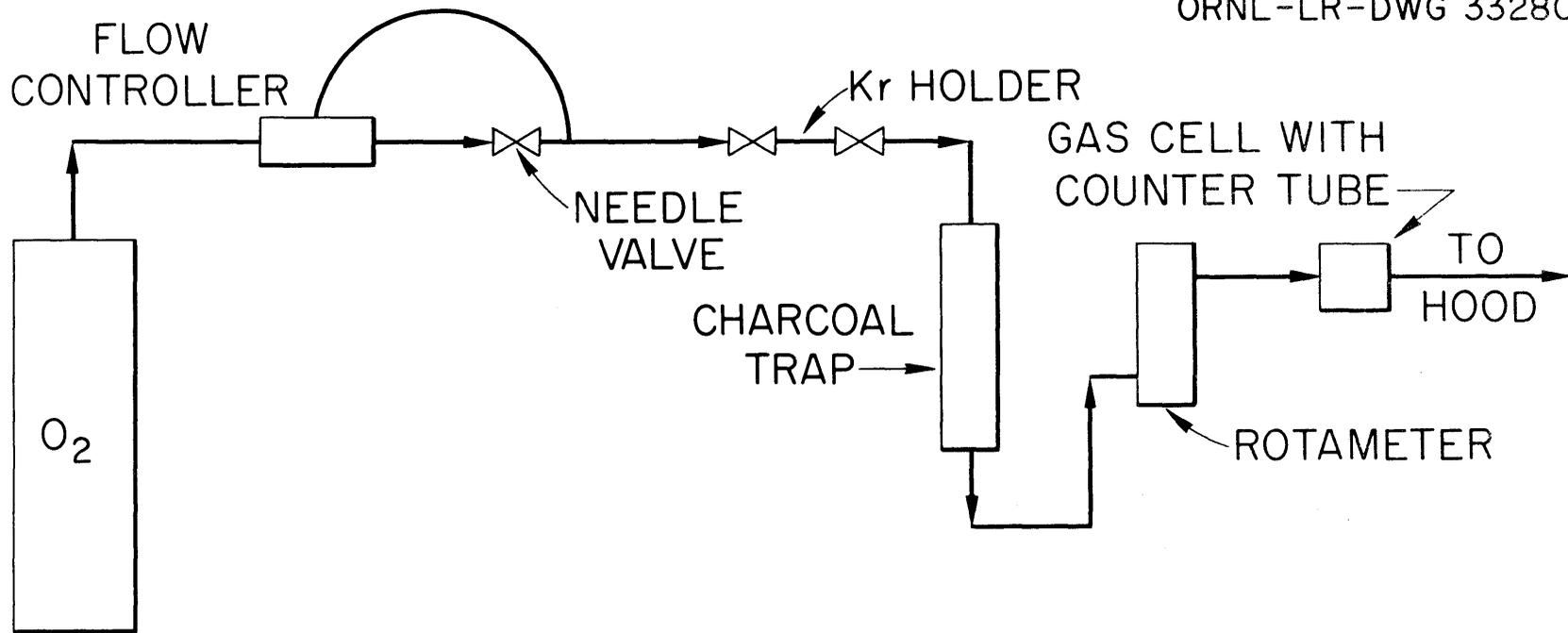
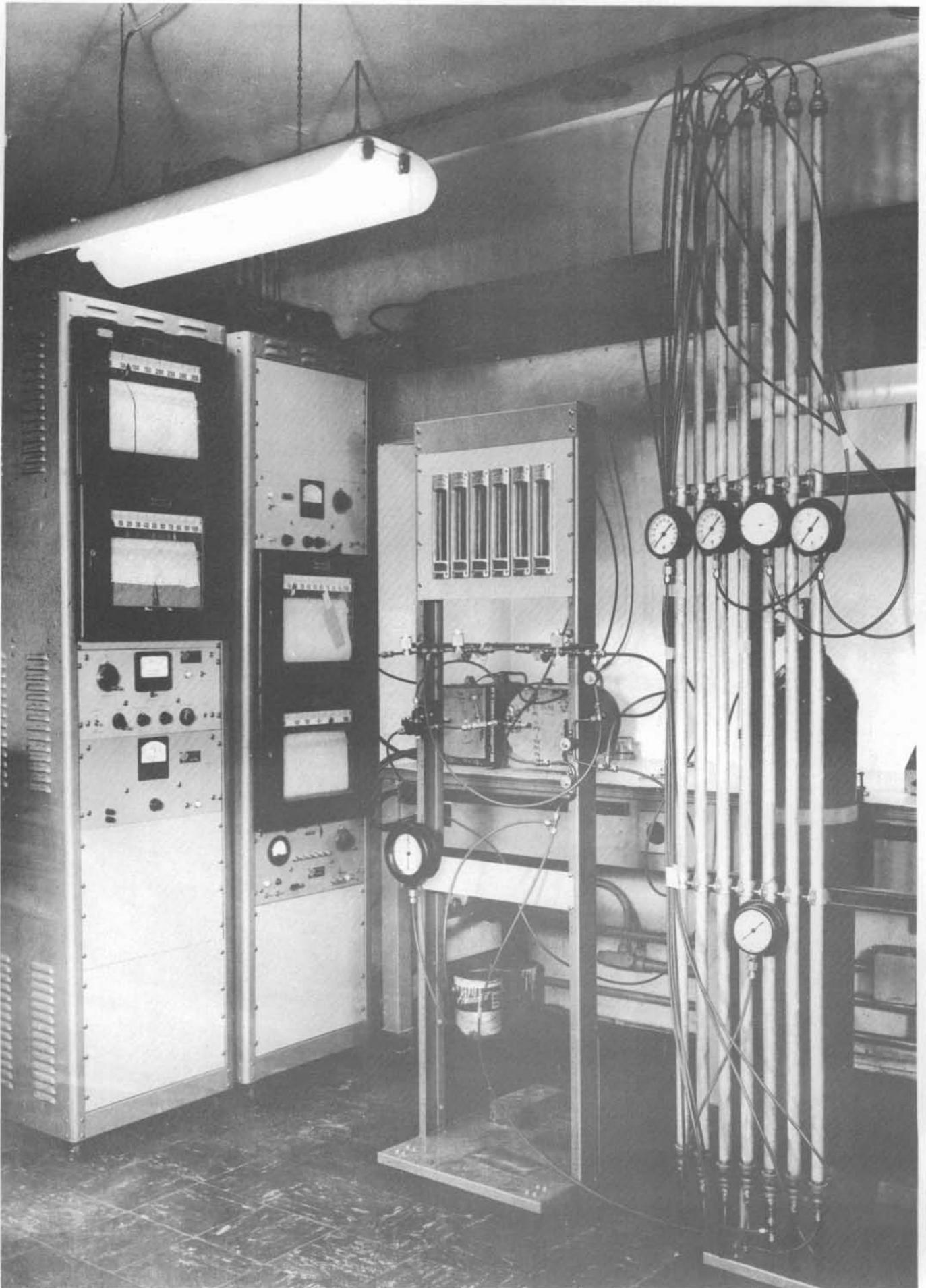


Figure 6



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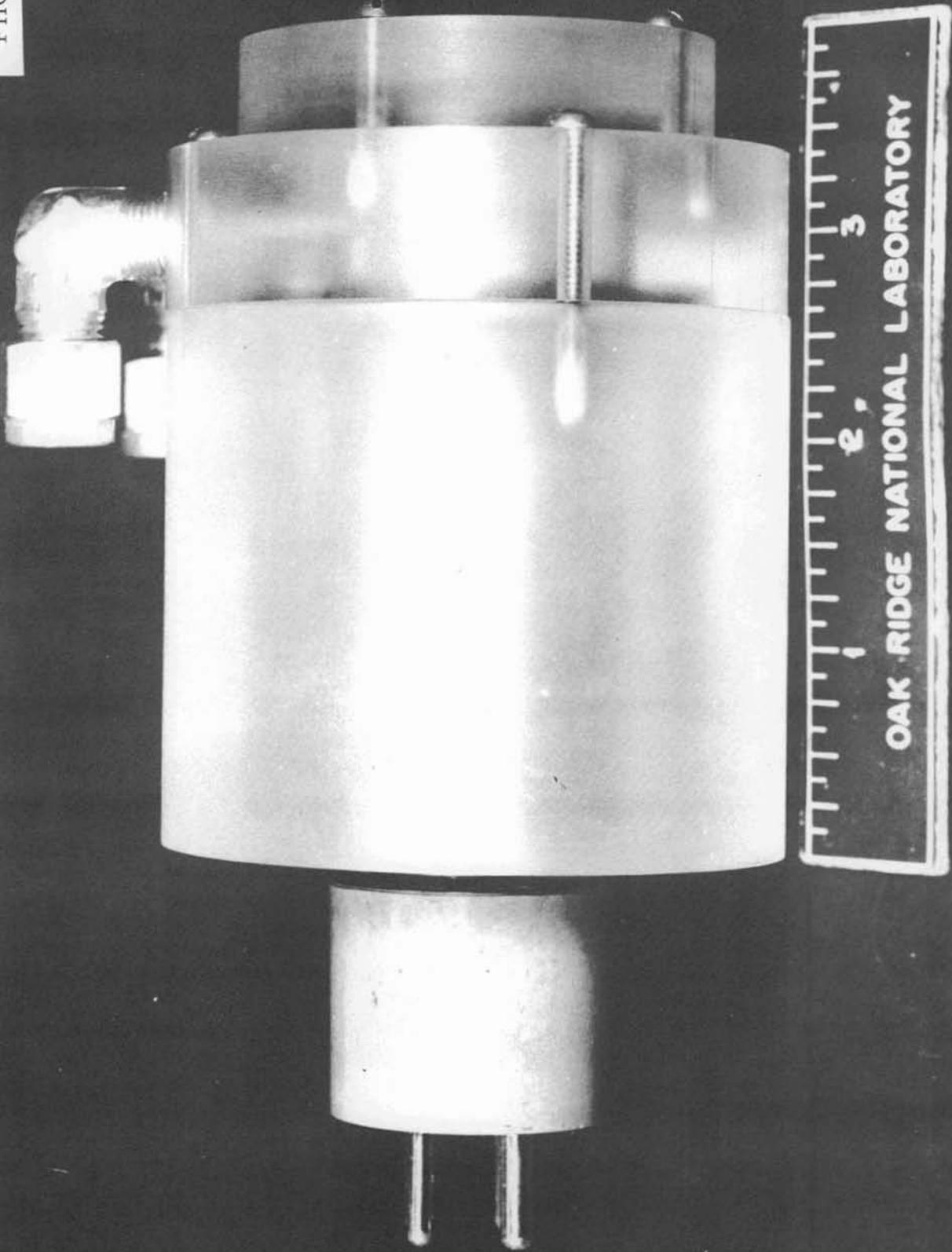
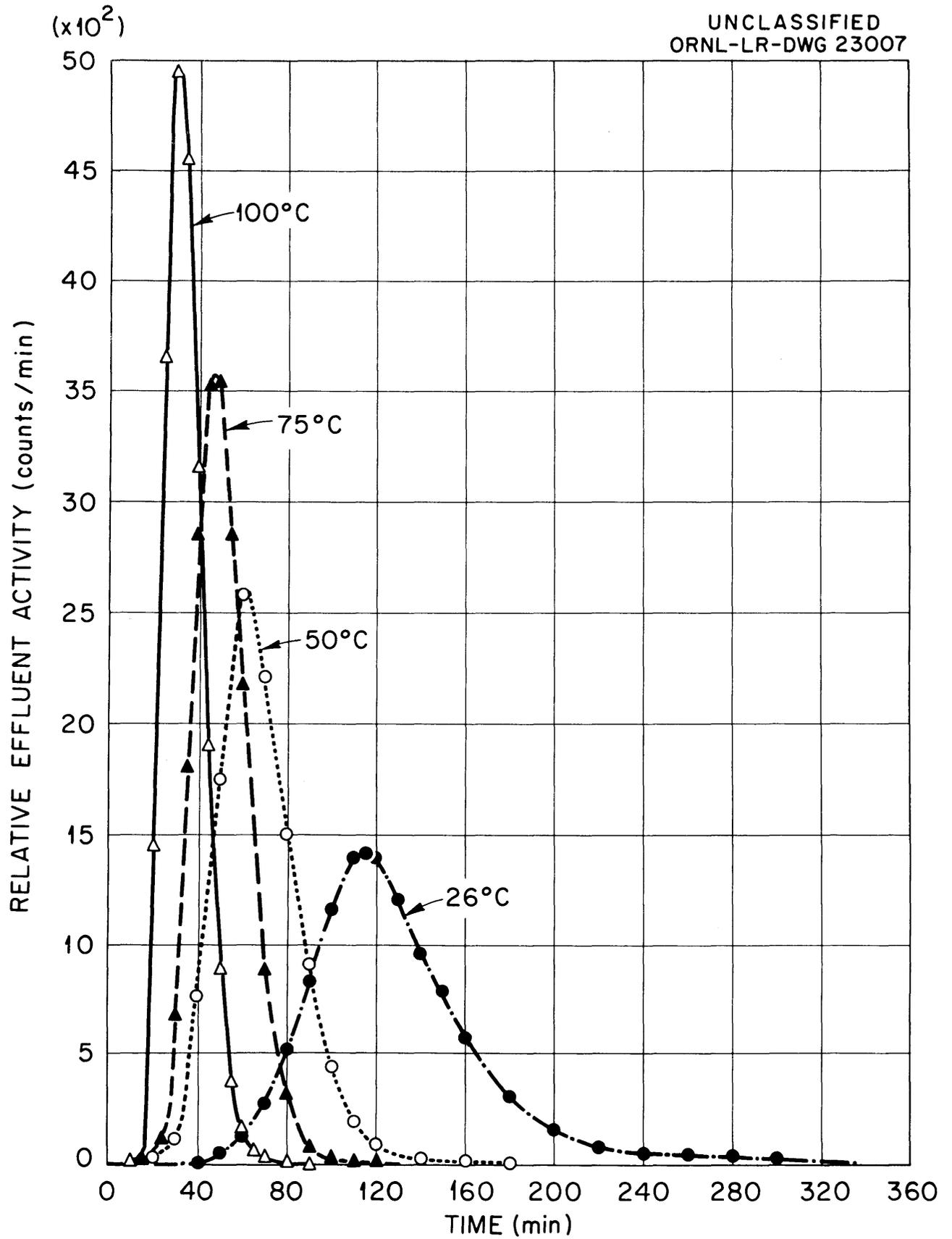
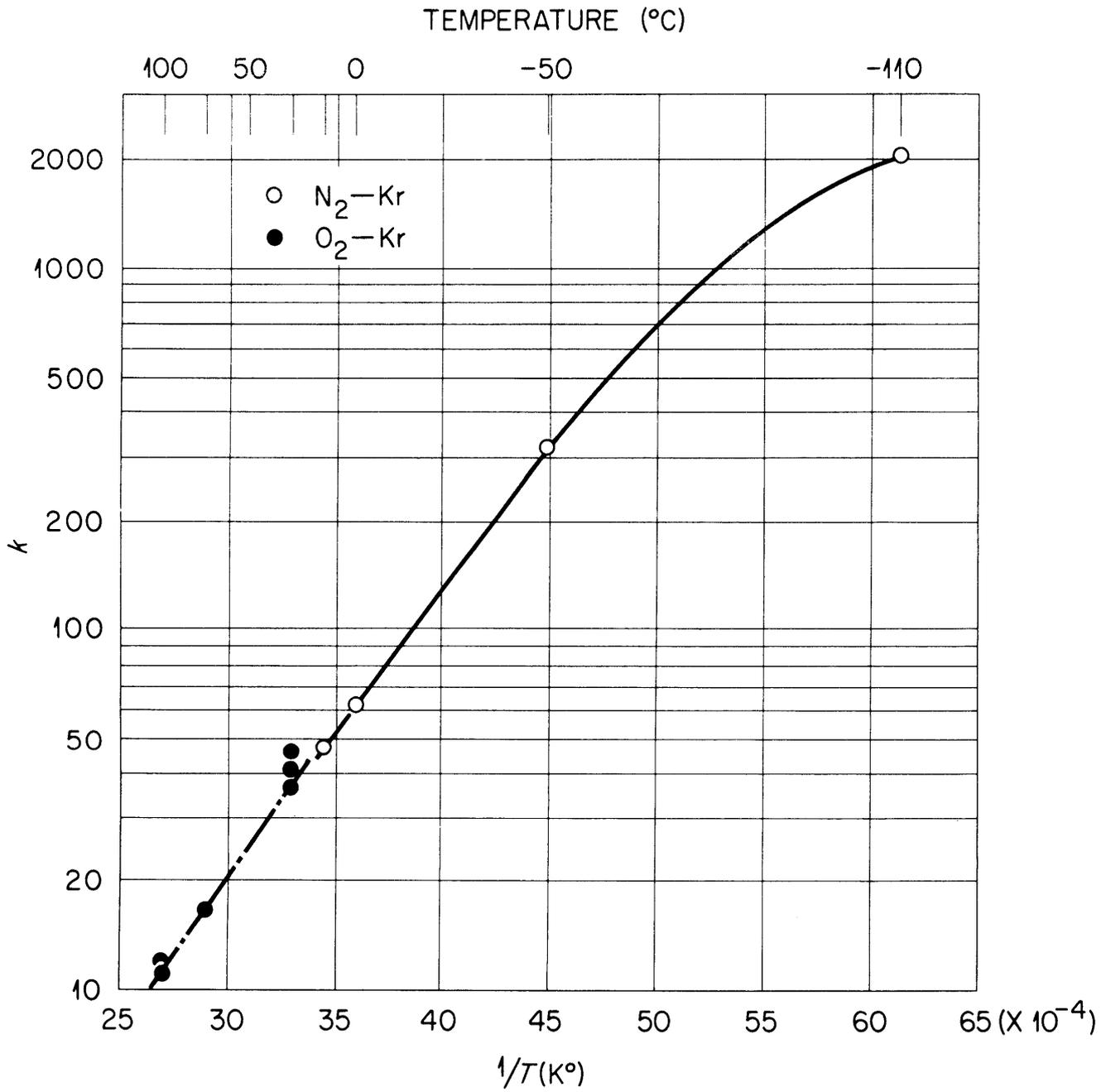


Figure 8



Effect of Charcoal Bed Temperature.

Figure 9



Effect of Temperature on k

Figure 10

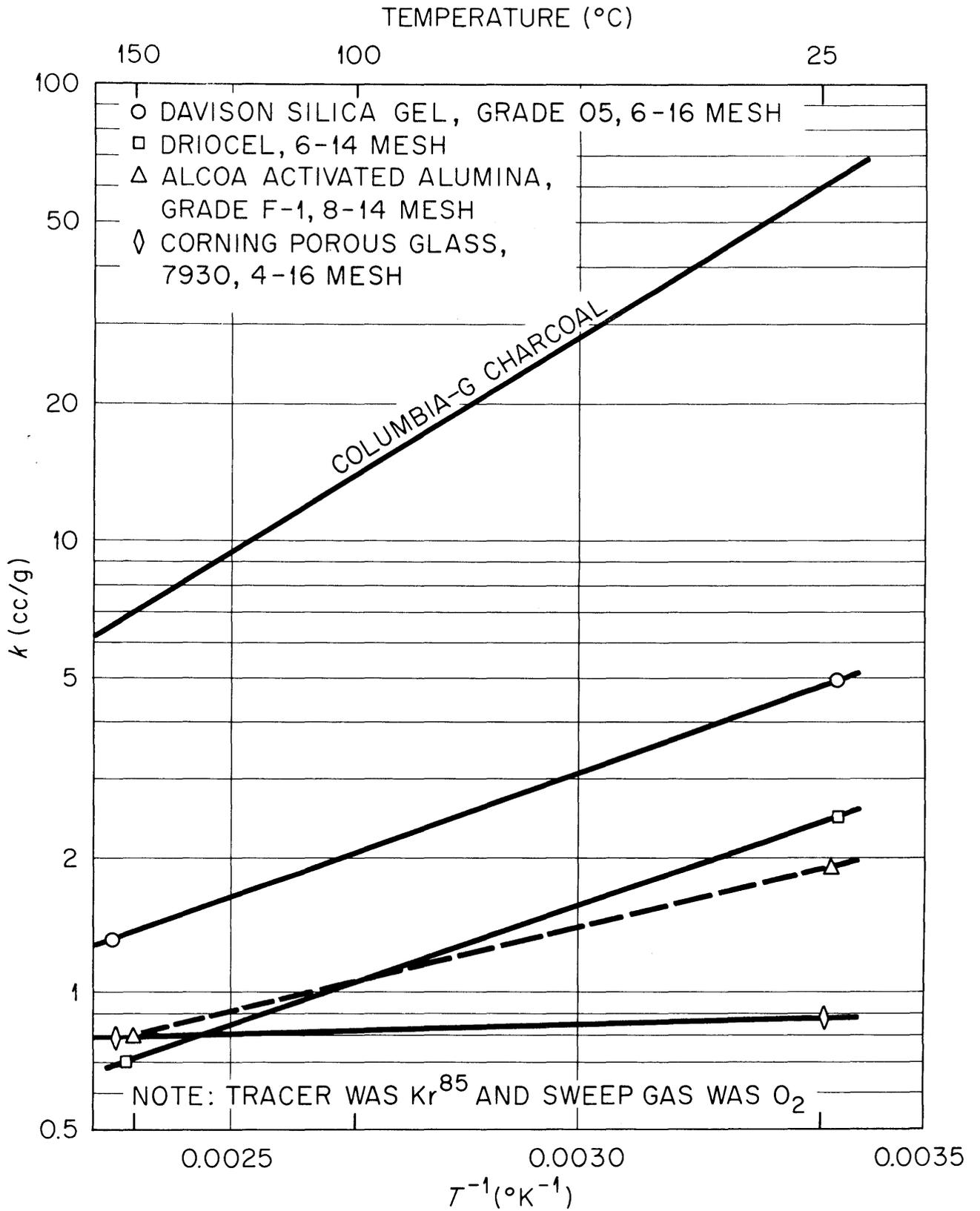


Figure 11

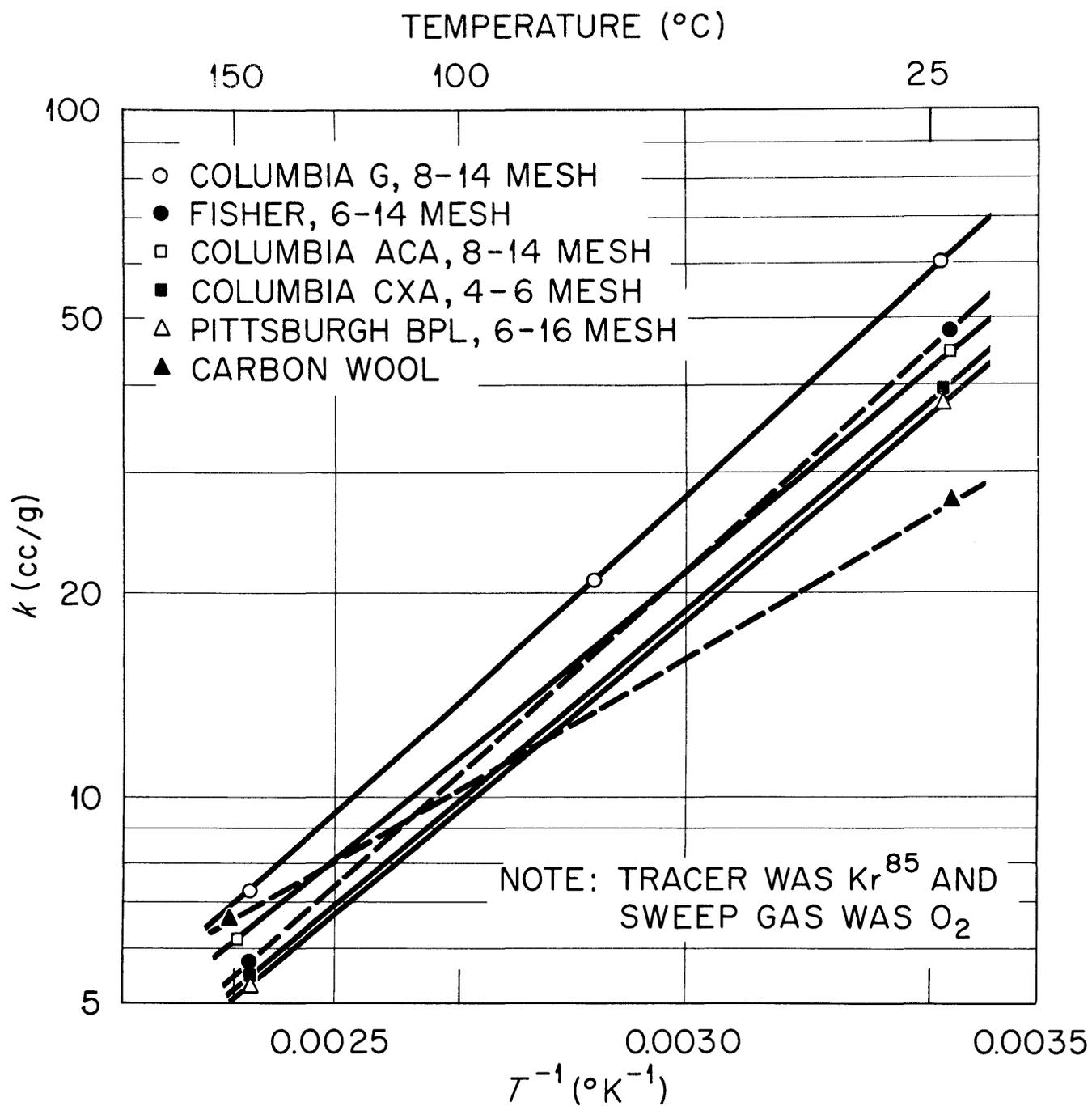


Figure 12

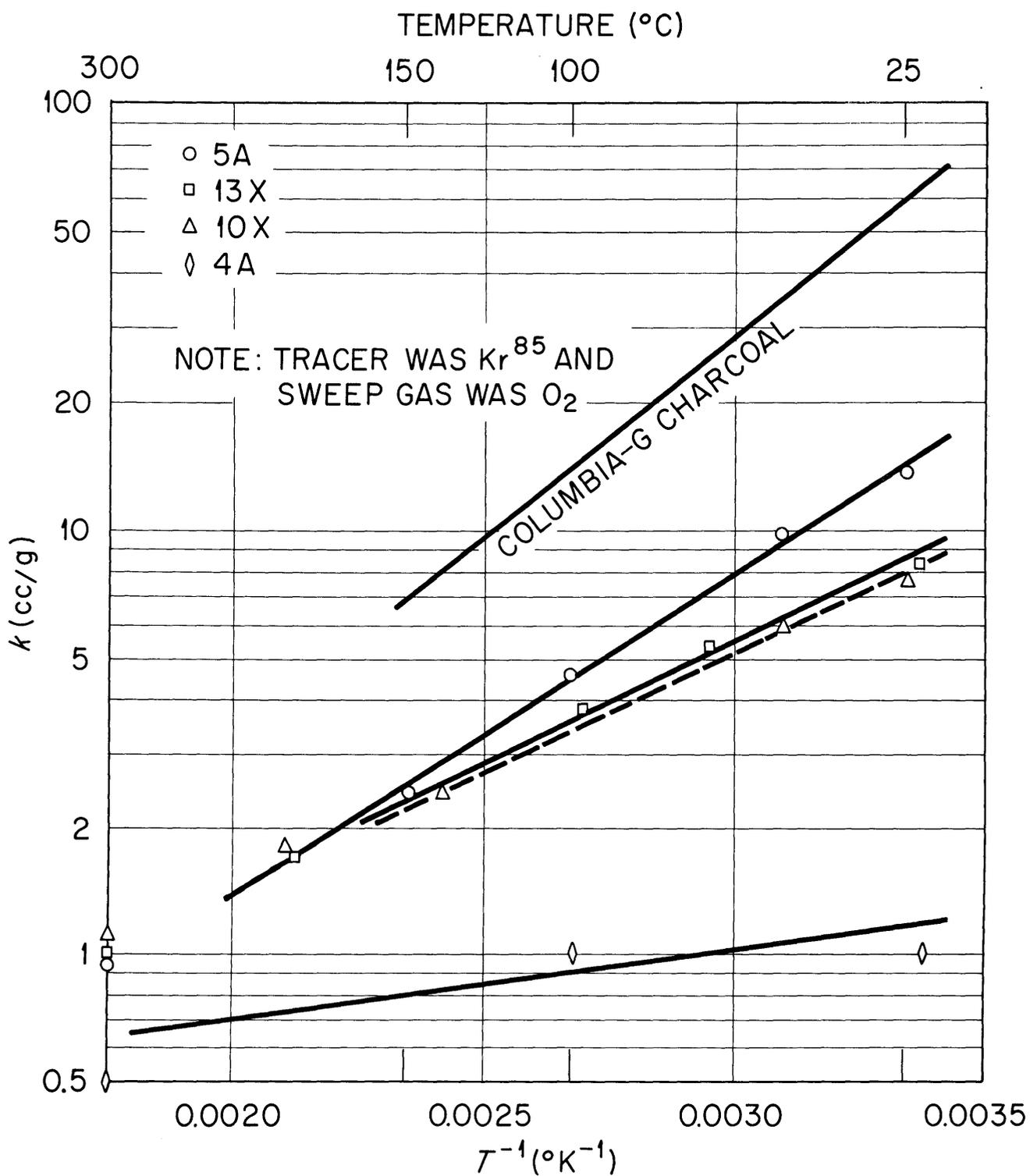


Figure 13

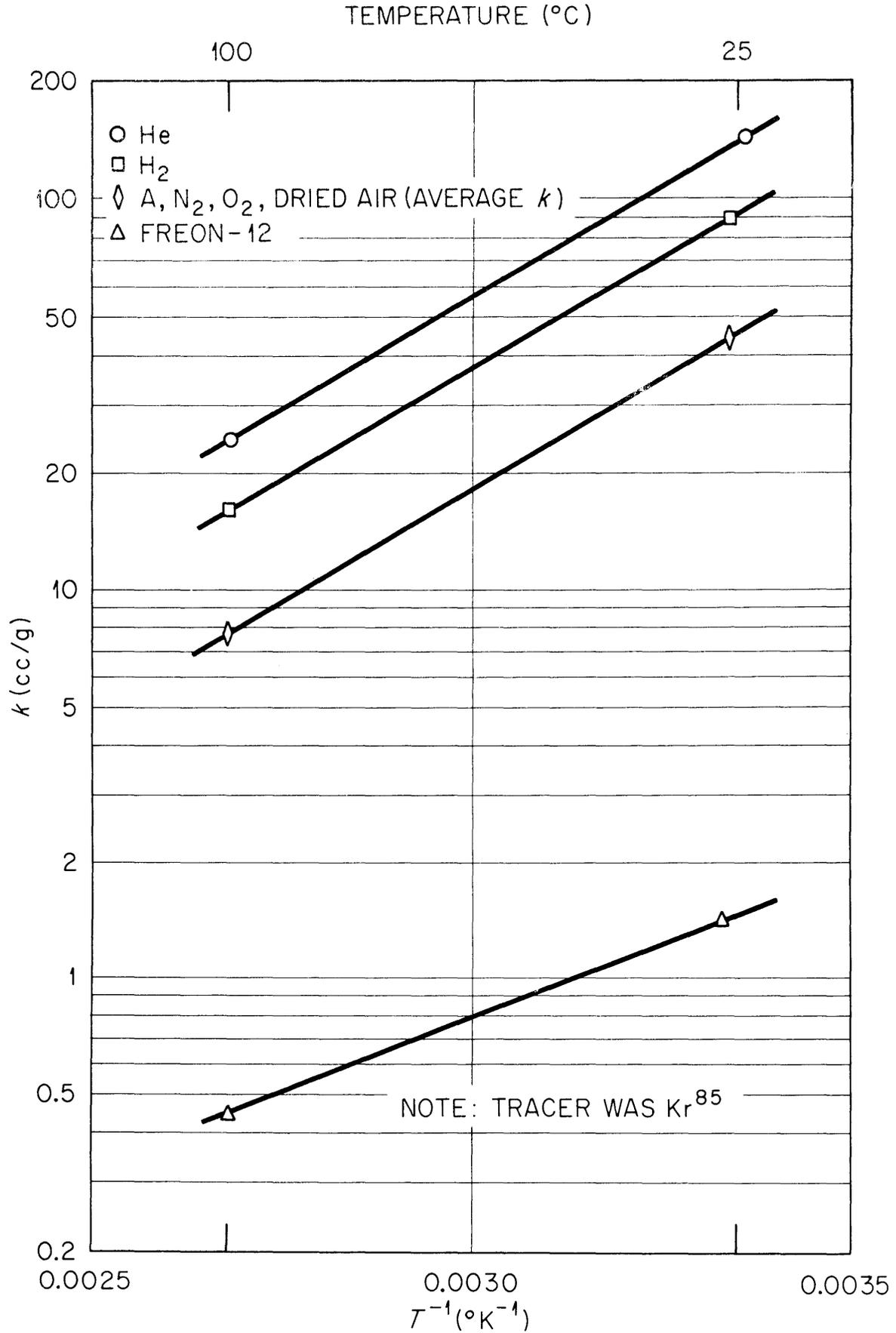
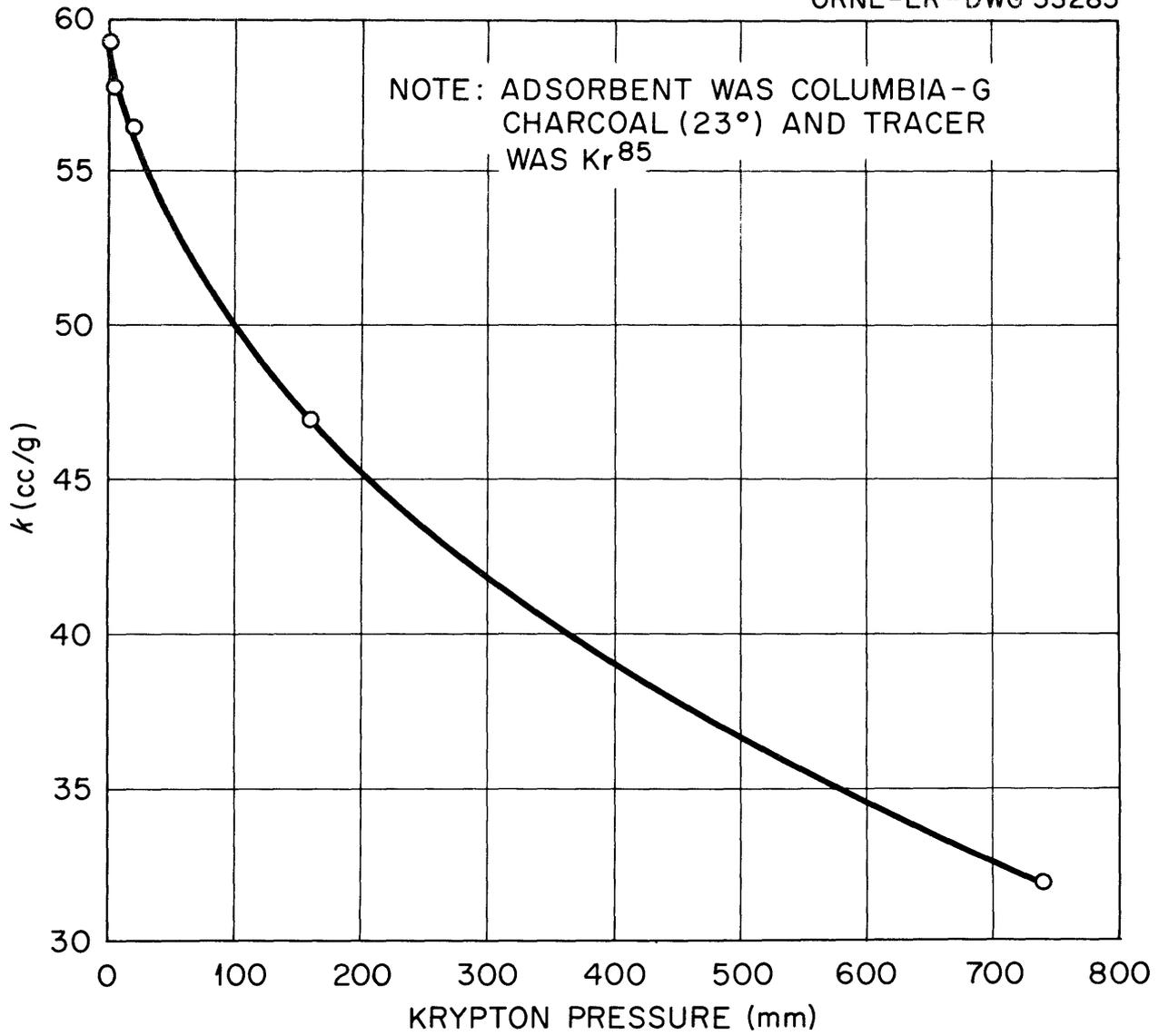
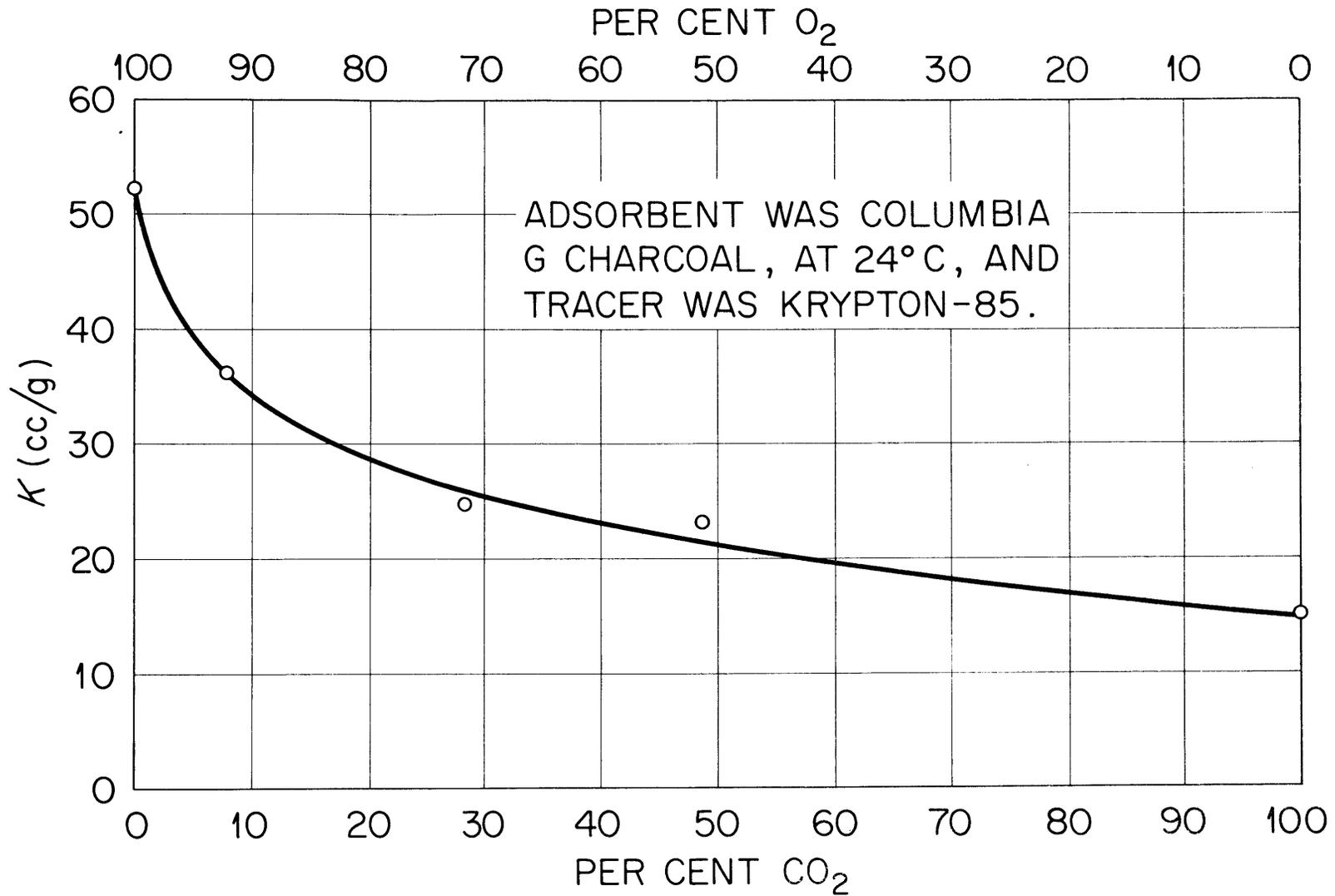


Figure 14



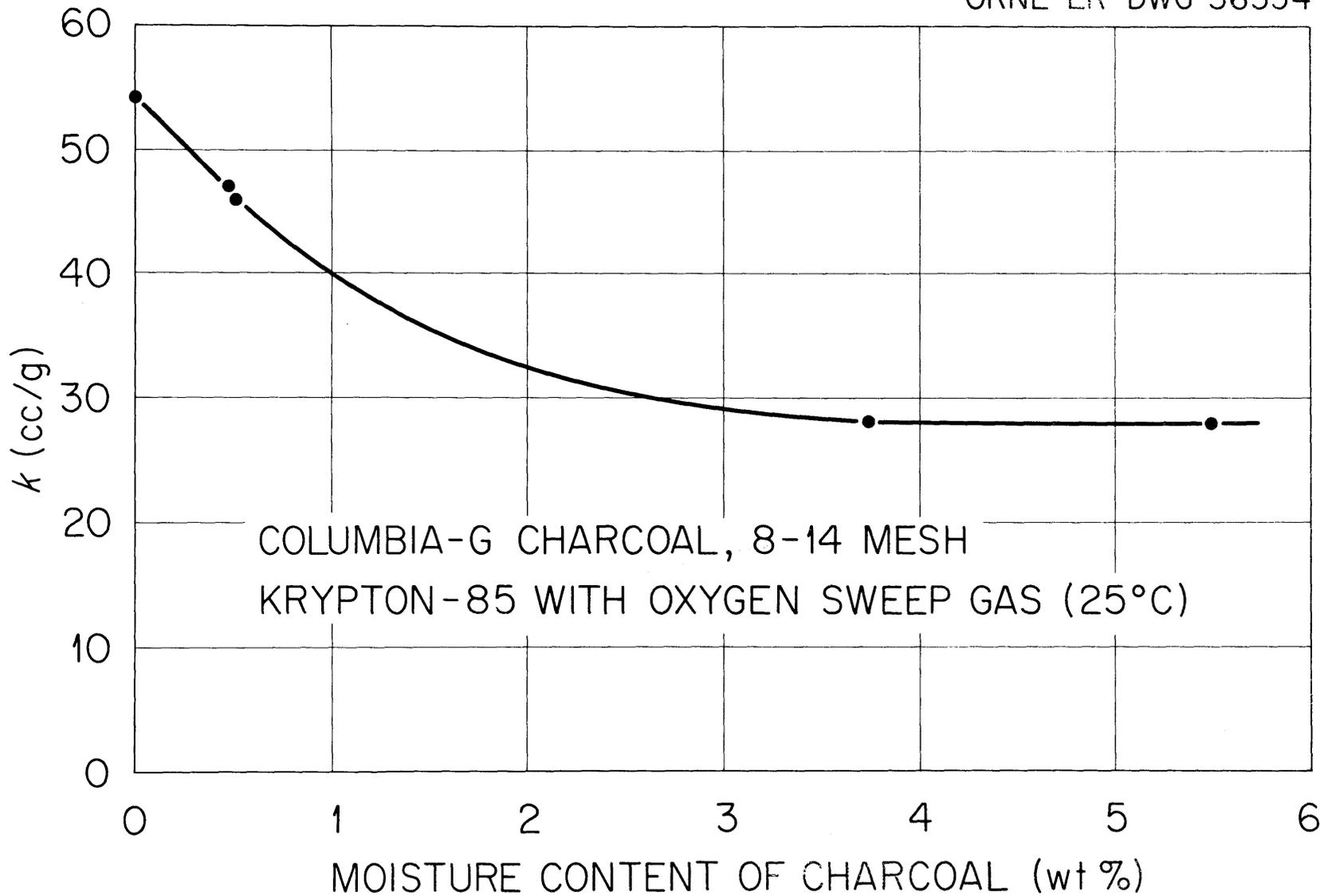
Effect of Krypton Partial Pressure on Dynamic Adsorption Coefficient.

Figure 15



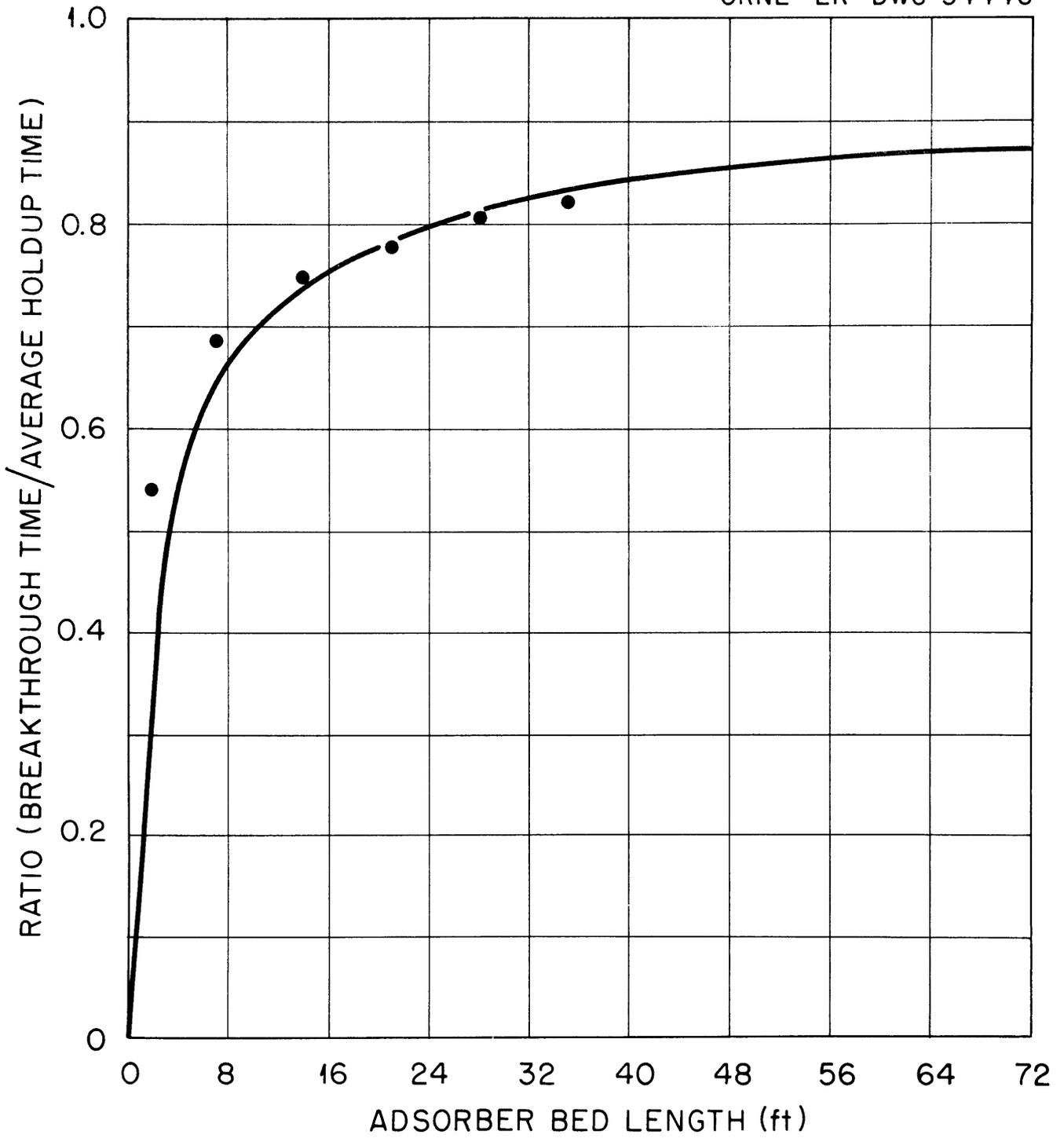
Effect of CO₂ in the Sweep Gas on the Dynamic Adsorption Coefficient K .

Figure 16



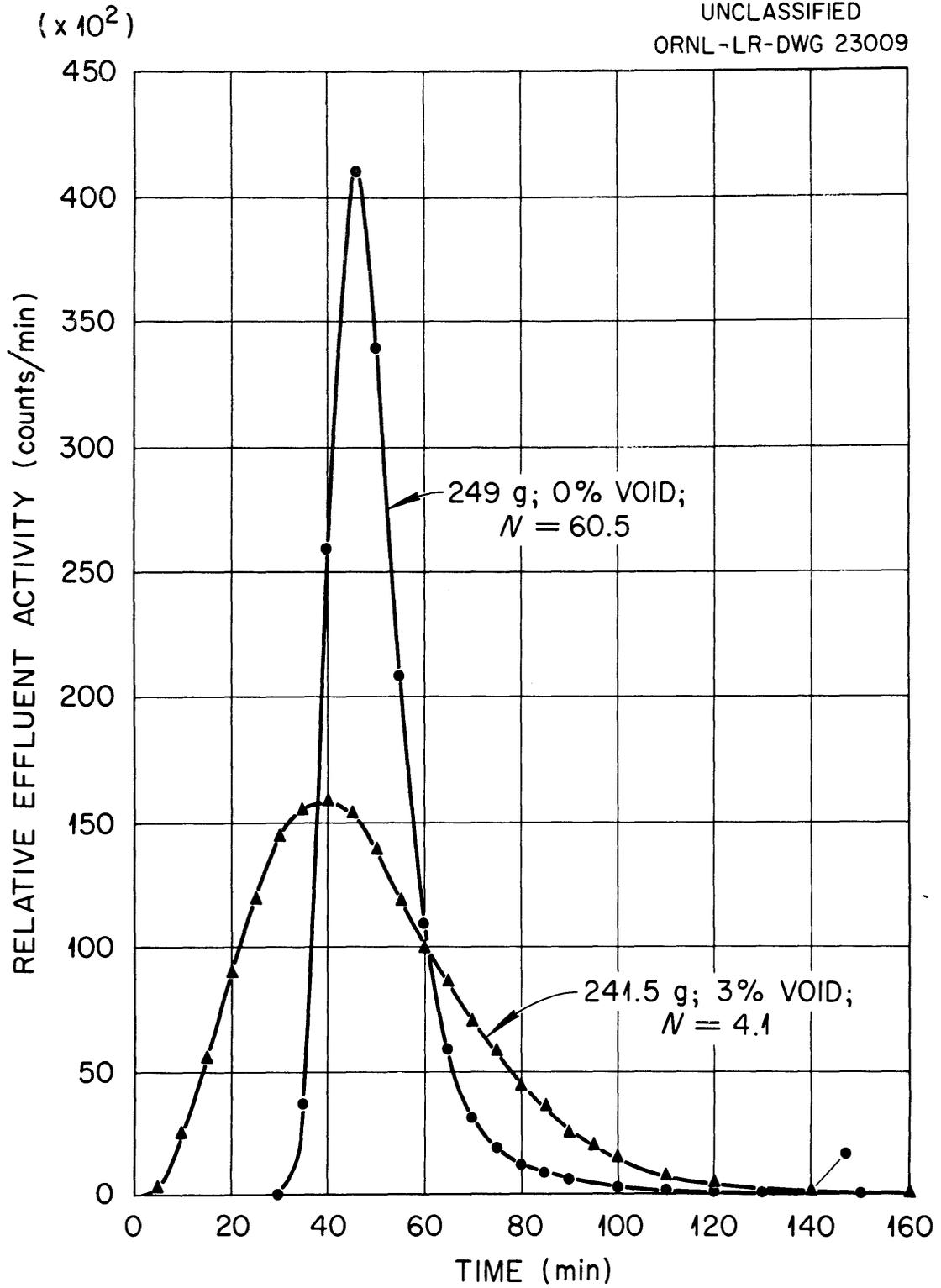
Effect of Moisture on the Dynamic Adsorption Coefficient

Figure 17



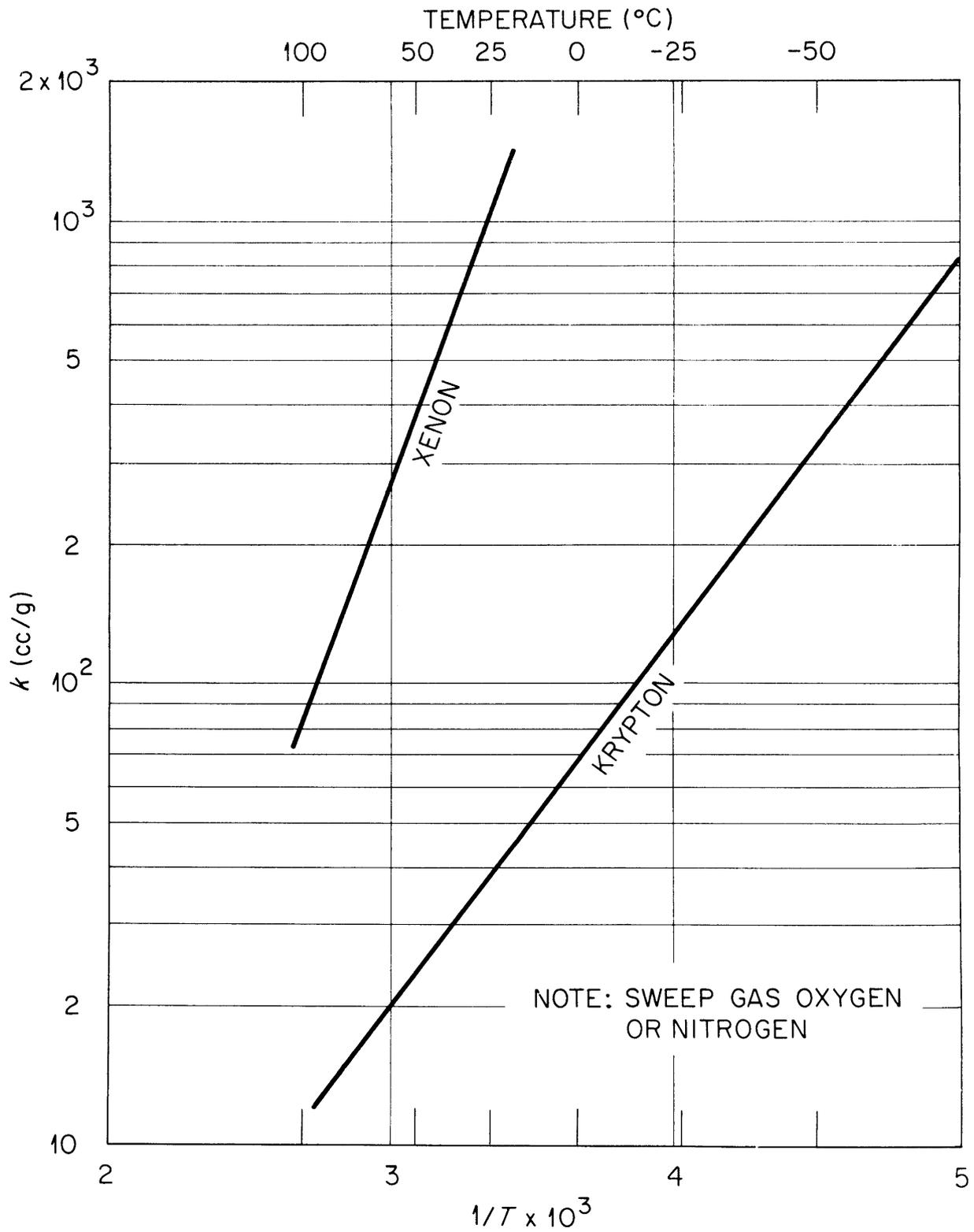
Ratio (Breakthrough Time/Average Holdup Time)
as a Function of Adsorber Bed Length.

Figure 18



Effect of Void Space in Horizontal Charcoal Bed.

Figure 19



Dynamic Adsorption Coefficients for Krypton and Xenon on Charcoal

Figure 20

