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A THERMODYNAMIC DATA PROGRAM INVOLVING PLUTONIA AND URANIA AT HIGH TEMPERATURES

QUARTERLY REPORT NO. 12
MAY 1 TO JULY 31, 1970

MASTER

E. A. AITKEN
S. K. EVANS

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AND URANIA AT HIGH TEMPERATURES**

Quarterly Report No. 12 – May 1 to July 31, 1970

E. A. Aitken
S. K. Evans

Approved:



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ABSTRACT

In the eleventh quarterly report (GEAP-12099) the continuing scope of work related to vaporization of urania, oxygen activity at high temperatures, oxygen redistribution in a temperature gradient, and sodium fuel interactions was presented. Work progress is presented on these tasks.

1. INTRODUCTION

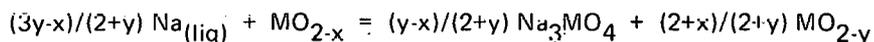
This quarterly report is twelfth in a series which describes experiments directed toward understanding plutonium segregation in urania-plutonia solid solutions and in fast reactor environments through a knowledge of its thermodynamic behavior. In the eleventh quarterly (GEAP-12099) the progress of several experiments which have a bearing on plutonium segregation was described. Continued progress in these areas is described in this report.

2. THERMODYNAMIC BEHAVIOR OF SODIUM-FUEL REACTION PRODUCTS

In the last quarterly report several methods for producing a sodium-fuel reaction product were described. Work has continued along three of these approaches to derive some thermodynamic data on the sodium-fuel reaction product. These approaches are to determine (a) the O:M threshold of the fuel necessary for formation of a sodium-fuel compound, (b) the oxygen content of sodium in the presence of fuel and sodium-fuel compound, and (c) the stability of compound by direct synthesis.

THERMAL GRADIENT EXPERIMENTS WITH EXCESS SODIUM

This experiment resembles closely the situation in failed irradiated fuel where sodium is in contact with fuel. Since the radial temperature gradient in the fuel ranges from about 700°C at the periphery to 2000°C at the center, sodium vapor will contact the fuel everywhere. Formation of a reaction product, however, most likely will occur in low-temperature regions where the oxygen content is higher and the sodium fuel compound is more stable. The extent of reaction will be governed by the amount of oxygen available for reaction since there is an excess of sodium. The oxygen available for reaction arises from (a) the oxygen shift as it responds to the temperature gradient and (b) the oxygen released from burnup. The over-all reaction may be described as follows:



where it is assumed that the fuel compound is $\text{Na}_3(\text{U,Pu})\text{O}_4$. The extent of reaction depends on the difference in the mean values of x and y before and after ingress of sodium. The value of y is important since it represents the threshold for formation of a sodium-fuel compound. From phase rule considerations, equilibrium value for y for this pseudo 3-component system is a function of the temperature and activity of sodium. Under isothermal conditions with an excess of sodium liquid, it is difficult to determine y since the presence of sodium interferes with oxygen-to-metal ratio determination. In the presence of a temperature gradient, the excess sodium tends to concentrate at the low temperature end and the sodium-fuel reaction product tends to form in a distinct region on the low-temperature side.

The fuel pellets at higher temperatures will be generally free of sodium and will give a representation of the O:M behavior along the temperature gradient. By following the O:M as a function of temperature along the gradient to the point where sodium fuel product occurs, the threshold O:M for formation of the compound can be determined.

This result represents a steady-state condition in which compound formation is carried out between low-temperature sodium and oxygen which drifts down from the high-temperature side. The position of the reaction along the temperature gradient is set by a combination of activity gradients in the nonisothermal system, a condition which parallels closely the actual situation in an irradiated fuel element.

A preliminary experiment was reported in the last period which demonstrated a reaction tendency between sodium and 80% UO_2 -20% PuO_2 fuel when heated in a thermal gradient. The fuel pellets and sodium were sealed in a Type-316 stainless steel container and heated for 100 hours. The temperature along the 6-inch container ranged from 550 to 1150°C. The pellet which was located at a temperature of 750°C showed a distinct reaction band at its cold end which extended over about one-third of the pellet length. The fuel pellets located above this temperature showed no evidence of reaction except that their oxygen-to-metal ratios decreased from 1.965 at 750°C to 1.955 at 1150°C. The fuel pellets below 750°C showed no gross reaction, however, they were contaminated with unreacted sodium which condensed at the low-temperature end of the capsule.

This experiment was extended further by using sealed molybdenum containers instead of stainless steel so that higher temperatures could be used to increase the amount of oxygen available for reaction with sodium and the fuel. About thirty-seven 75 UO_2 -25 PuO_2 pellets at an initial oxygen-to-metal ratio of 1.965 were sealed in molybdenum. One rod contained about 32 mg of sodium and another contained about 85 mg of sodium. The sodium was placed initially in the hot end to require vapor transport over the entire fuel column before condensation at the low-temperature end. The two rods were heated in a common temperature gradient which ranged from 900 to 1450°C for 300 hours. After heat treatment, the pellets were removed and examined for evidence of reaction. Figures 1 and 2 give a description of the appearance of the pellets in the two rods. Rod 1, which contained only 32 mg of sodium, had only a small residue of metallic sodium in the crevice of the end plug. A reaction zone over one-third of the pellet from the cold side was evident in several pellets at the cold end of the capsule. This reaction was nearly identical in appearance to the reaction zone detected in the stainless steel capsule experiment with sodium. Rod 2 showed a similar phenomenon in the last six pellets at the cold end. This rod had more residual sodium which deposited on the last two pellets than Rod 1, apparently because of the larger initial inventory of sodium. The appearance of a reaction product occurred at the same temperature in the two rods and all pellets above this temperature showed no visual indication of attack by the sodium. The reaction zone was easily identified by a distinct diametral swelling of several mils. The pellets which contained metallic sodium showed immediate deterioration on exposure to air. The pellets which showed a reaction zone did not deteriorate, however, some moisture pickup was observed after a few days storage in a plastic vial.

The pellets which were at a temperature higher than the reaction zone were subjected to thermogravimetric analysis for O:M determination. A summary of these results is given in Figure 3 where the deviation from stoichiometry is plotted against temperature according to an Arrhenius equation. The two rods give almost identical heats of transport. It is believed that steady-state conditions have been established in 300 hours, however, further tests are probably necessary to assure this claim. The observed heat of transport of -5.7 kcal/mole is somewhat less than that observed in comparable thermal gradient tests without sodium and may result from the influence of sodium vapor in transporting oxygen along the gradient.

The O:M threshold appears from the results in Figure 2 to be between 1.970 and 1.975 and occurred at a temperature of 1000°C under a condition where the condensed sodium phase was present at a temperature near 900°C. In the stainless steel capsule the reaction temperature occurred at 750°C under conditions where the sodium temperature was 550°C. Some of the pellets near the reaction zone gave unusually low O:M values which appear to result from sodium contamination. Subsequent heat treatment at 1650°C in wet hydrogen showed a small weight loss in the pellets from vaporization of residual sodium.

The formation of reaction zones at the cold end of the pellets below 1000°C is somewhat surprising and needs further investigation. It would appear that the kinetic factors are influencing the way in which nucleation and growth of the reaction phase occur.

KEY: N_S = No Swelling Of Pellet Detected
 S = Swelling Of Pellet Detected
 * = Pellet Absorbed Moisture After 3 Days Storage

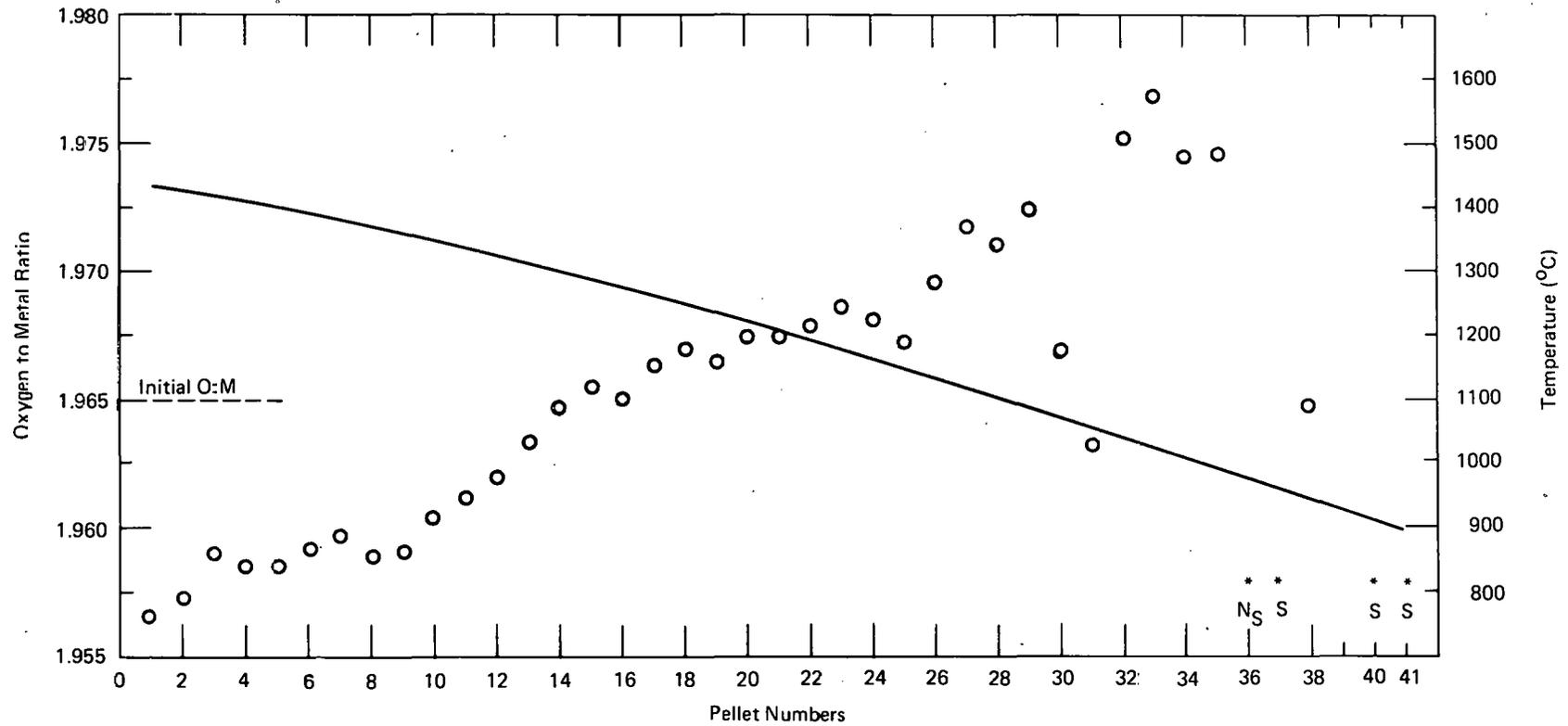


FIGURE 1. OXYGEN TO METAL RATIO AND TEMPERATURE AS A FUNCTION OF PELLET NUMBER FOR CAPSULE HEATED 300 HOURS WITH 32 mg Na

KEY: N_S = No Swelling Of Pellet Detected
 S = Swelling Of Pellet Detected
 * = Pellet Absorbed Moisture After 3 Days Storage

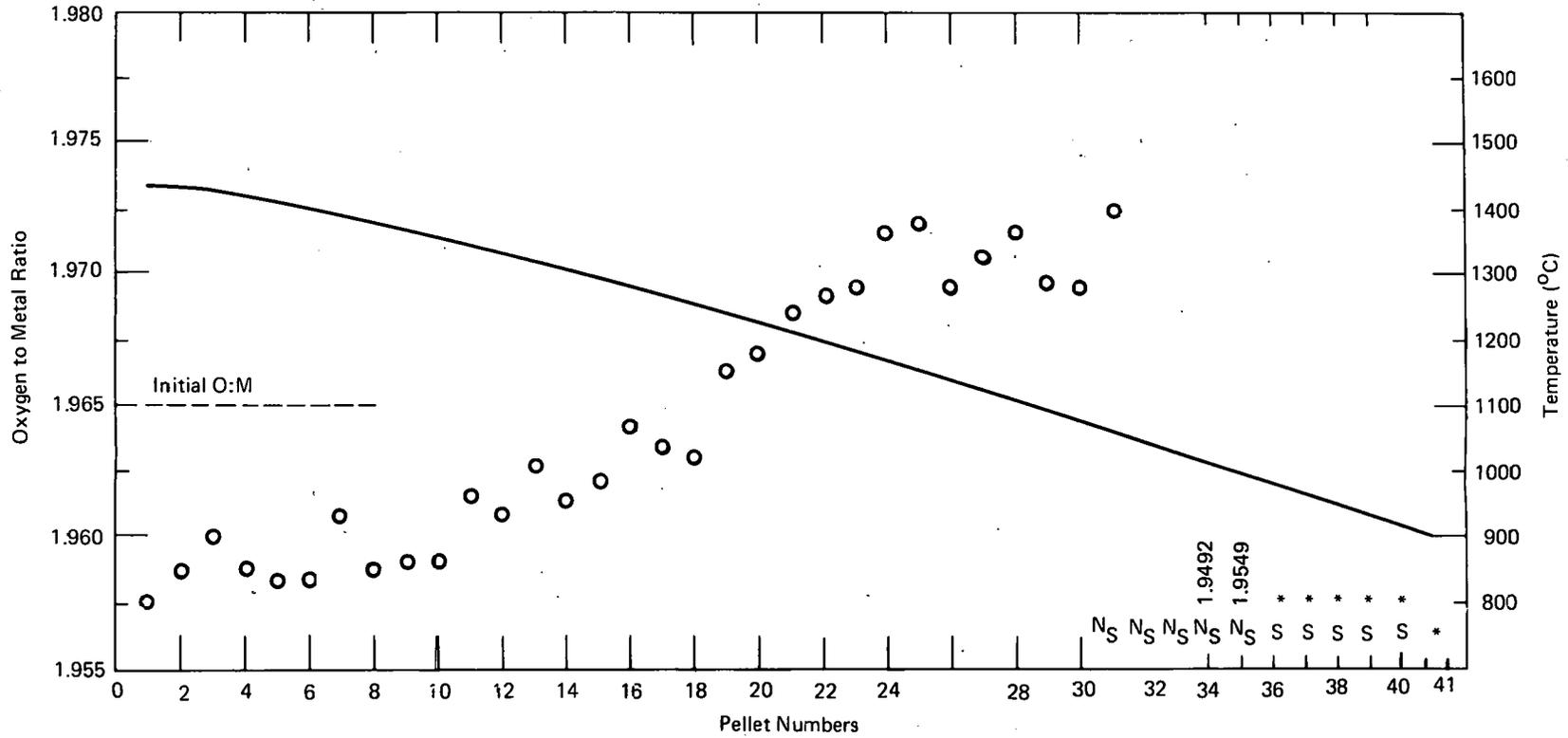


FIGURE 2. OXYGEN TO METAL RATIO AND TEMPERATURE AS A FUNCTION OF PELLET NUMBER FOR CAPSULE HEATED 300 HOURS WITH 85 mg Na

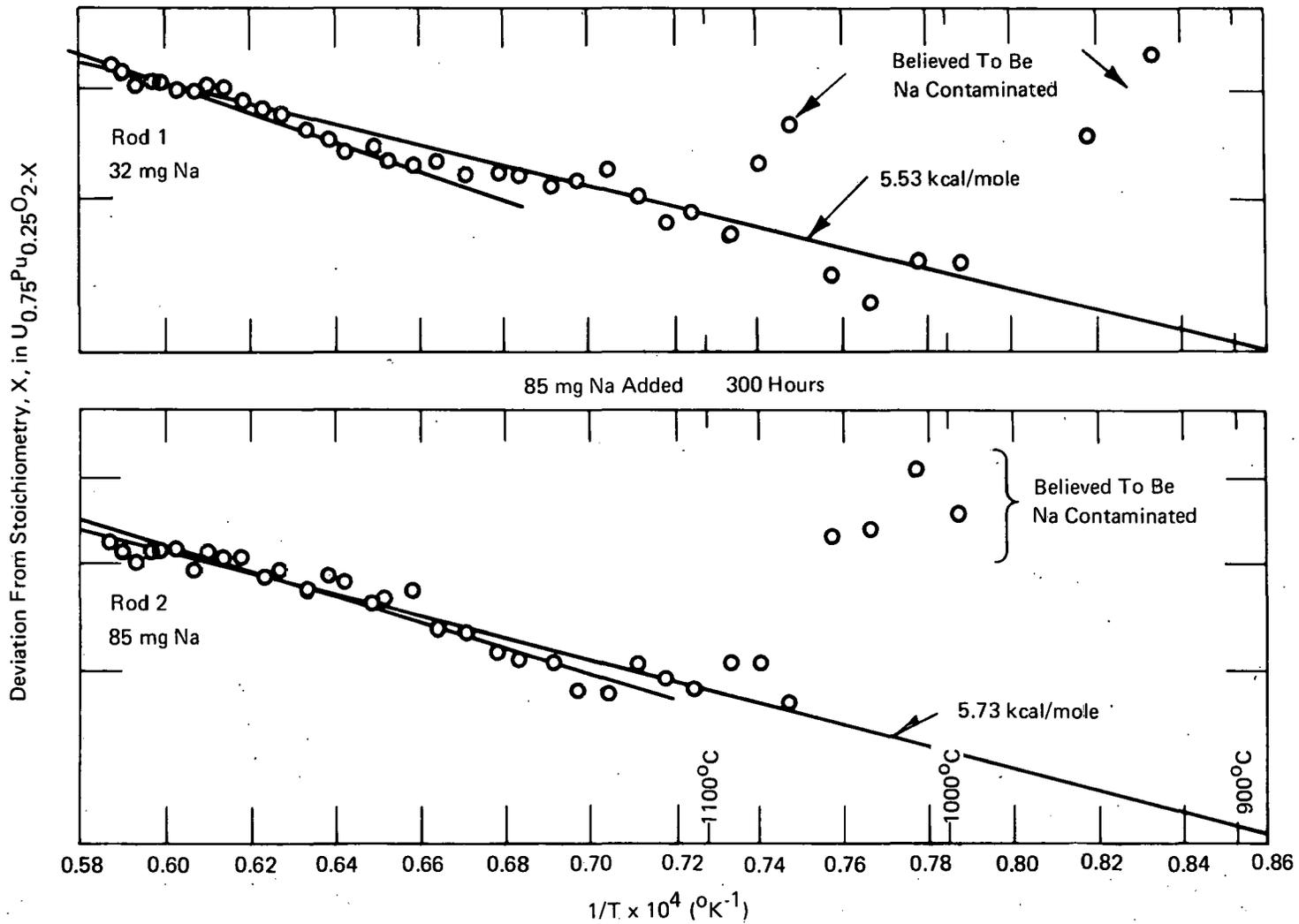


FIGURE 3. ARRHENIUS PLOT OF O:M VARIATION WITH TEMPERATURE AFTER 300 HOURS IN A TEMPERATURE GRADIENT

ISOTHERMAL EXPERIMENTS IN EXCESS SODIUM

In this experimental approach, the oxygen content of the sodium is equilibrated with sodium fuel compound and the fuel phase. The first set of capsules was lost due to rupture since the electrolytic nickel capsules were too soft at 850°C to resist the internal pressure. The second set was heated for 60 hours at 750°C and no swelling was apparent, however, some leakage occurred through pin holes in the welded capsules during the heat treatment in a vacuum vessel. Sufficient sodium however, was retained to provide a means for analysis of oxygen in the sodium. A sodium distillation apparatus for oxygen analysis in a glove box has been designed and will be set up in the next report period.

SODIUM-FUEL COMPOUNDS BY DIRECT SYNTHESIS

The stability of the sodium-fuel compound can be measured during direct synthesis by using differential thermal analysis (DTA) techniques. Initial runs were reported last quarter on UO_2 powder mixed with either Na_2CO_3 or Na_2O . Exothermic reactions were observed in a flowing 6% H_2 -He mixture or pure helium. The products formed were either NaUO_3 in a hydrogen atmosphere or $\text{Na}_2\text{U}_2\text{O}_7$ in a helium atmosphere. These observations are consistent with the different oxygen activity levels of these two gaseous environments. Several large exothermic peaks were observed between 300 and 600°C when Na_2CO_3 was used. It was found that these peaks were associated with decomposition of Na_2CO_3 and some of the heat evolved was probably due to the catalytic reaction between $\text{CO}_2 + \text{H}_2$ at the surface of a nickel boat.

The exotherms from reaction of Na_2O and UO_2 were observed in the temperature region 450 to 520°C. The DTA runs in 6% H_2 -94% He with identical mixtures gave nonreproducible exotherms, an indication that other reactions were occurring.

3. DETERMINATION OF COMPONENT ACTIVITIES

The determination of uranium activity in UO_2 - PuO_2 solid solutions is being studied by means of a transpiration technique using air and CO/CO_2 as the carrier gases. Installation of the transpiration equipment in an alpha enclosure for commencement of experiments with UO_2 - PuO_2 compositions is nearly complete. During the next report period experiments will be conducted in the 1300 to 1500°C range with UO_2 -40% PuO_2 using 10 CO_2/CO as the carrier gas.

4. OXYGEN MIGRATION IN (U,Pu)O₂ SYSTEMS

Continuing experiments to examine the validity of Aitken's¹ one-component cyclic model for UO_2 - PuO_2 oxygen migration in a temperature gradient have been carried out.

Two molybdenum capsules containing $(\text{U}_{0.75}\text{Pu}_{0.25})\text{O}_{2-x}$ were annealed for 1000 hours in a temperature gradient from 1430 to 870°C. One capsule contained mixed oxide with an average oxygen-to-metal ratio of 1.929. The other capsule contained mixed oxide with an average oxygen-to-metal ratio of 1.969. Molybdenum spacers were placed between each of the pellets in this second capsule to prevent solid phase transport of oxygen between pellets.

The results of these two experiments are shown in Figures 4 and 5. The measured heat of transports are -1.7 kcal/mole for the 1.929 O:M capsule and -8.3 kcal/mole for the spacer experiment.

The heat of transport for the capsule containing mixed oxide of average composition $(\text{U}_{0.75}\text{Pu}_{0.25})\text{O}_{1.929}$ can be compared directly with previous uranium-plutonia thermal gradient experiments using Aitken's model. According to this model, the observed heat of transport (Q^*_{eff}) can be related to the relative mobilities of oxygen in the solid and gas phases by the following equation

$$Q^*_{\text{eff}} = \frac{Q^*}{1 + (L_s/L_g)} \quad (1)$$

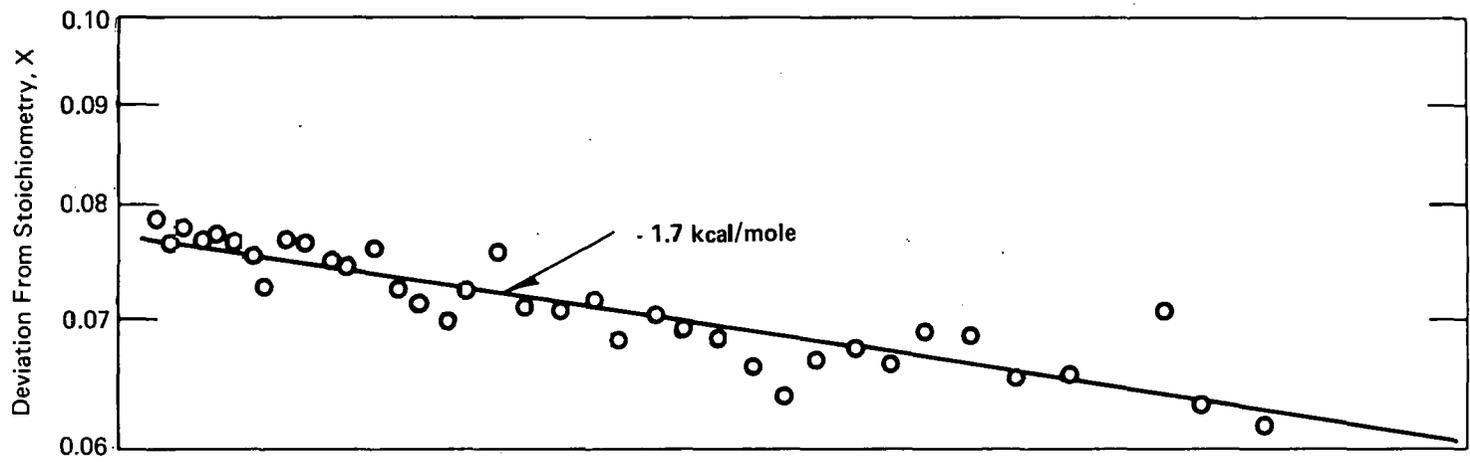


FIGURE 4. ARRHENIUS PLOT OF 1000 HOURS THERMAL GRADIENT ANNEAL OF $(U_{0.75}Pu_{0.25})O_{1.929}$

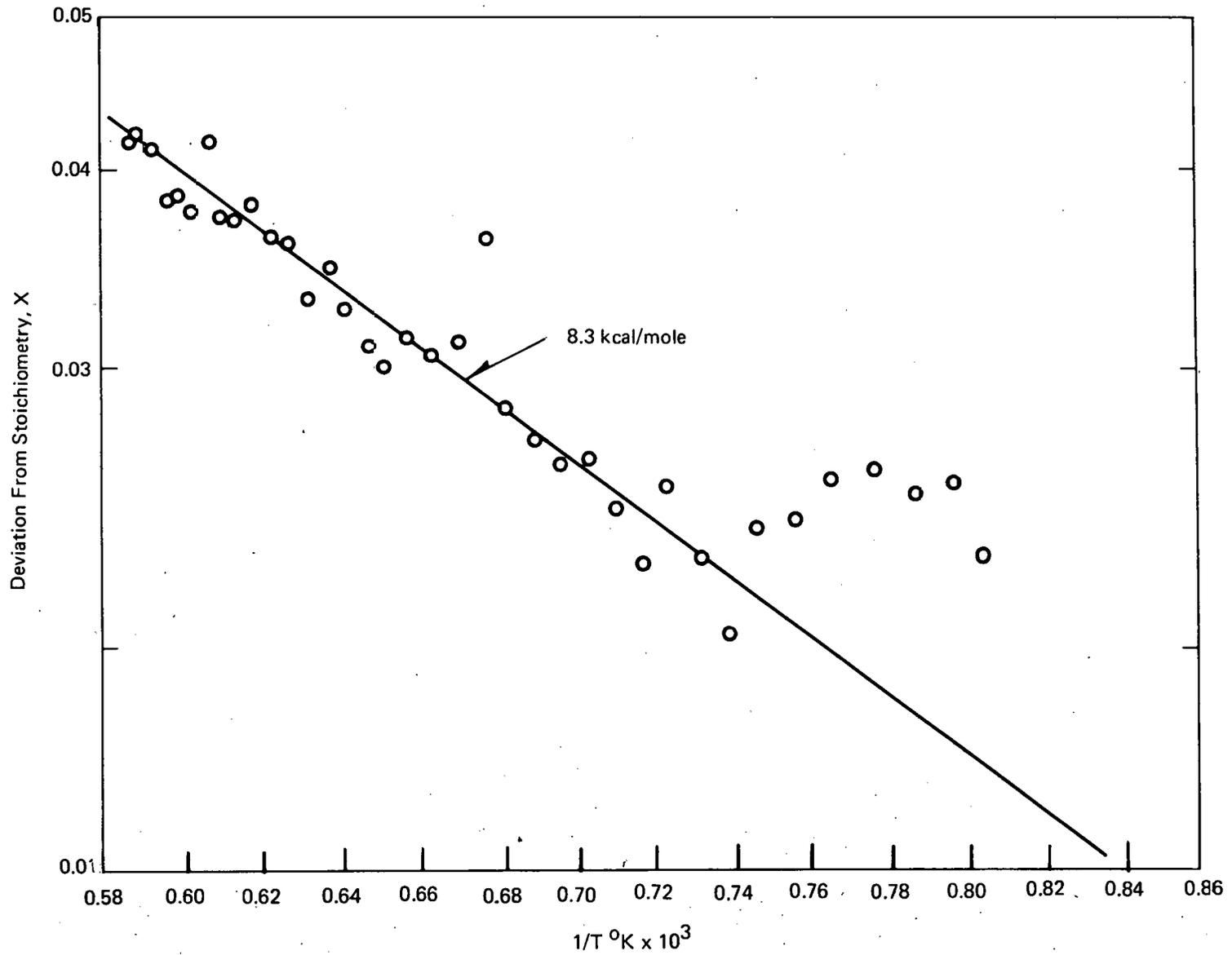


FIGURE 5. ARRHENIUS PLOT OF 1000 HOURS THERMAL GRADIENT ANNEAL OF $(U_{0.75}Pu_{0.25})O_{1.929}$ WITH Mo SPACER

where Q^* represents the heat of transport calculated on the basis of oxygen migration by a single component in the gas phase only; L_s is the phenomenological coefficient related to solid state oxygen diffusion and is shown to be proportional to the oxygen vacancy concentration; L_g is related to gas phase migration of oxygen. Prigogine² has shown that the phenomenological coefficient L_g is related to the chemical diffusion coefficient as follows

$$L_g = CD/R \quad (2)$$

where C is the concentration of the diffusing species and D is the chemical diffusion coefficient for the diffusing species in a gas mixture. The concentration C is equal to P_{H_2O} and the diffusion coefficient of H_2O varies inversely as P_{H_2O} . Therefore, L_g is invariant to changes in H_2O pressure. Thus, we can write Equation (1) in the following form

$$Q^*_{eff} = Q^*/1+kx \quad (3)$$

where Q^* is defined as before; k is a constant; x is the oxygen ion vacancy concentration. A comparison of the results reported in the last quarterly report (GEAP-12099) with the results of the $(U_{0.75}Pu_{0.25})O_{1.929}$ capsule introduced here is shown in Table 1. The heat of transport observed for the 25% Pu composition is a factor of 4 less negative than the calculated value. There is ample evidence³ that as the oxygen activity is reduced, the kinetics of the approach to steady state in thermal gradient experiments such as these become less rapid. Thus, there is a strong possibility that 1000 hours is not long enough to reach a steady-state condition. Additional experiments are being made up to anneal the same composition for 100 and 300 hours to study the time dependence of the phenomena observed.

TABLE 1. Thermal Gradient Experiment Results for Various Plutonium Compositions

Pu Content (%)	O:M	X	Q^* kcal/mole	Q^*_{eff} Calculation (kcal/mole)	Q^*_{eff} Measurement (kcal/mole)
100	1.817	0.183	-43	- 4.1	- 4.1
40	1.950	0.050	-49	-13.7	-11.0
20	1.973	0.027	-48	-20.2	-21.9
25	1.929	0.071	-32	- 6.8	- 1.7

The molybdenum spacer experiment was designed to study the effect of negating any solid state diffusion in the thermal gradient experiment. According to Aitken's model, as L_s goes to zero, an indication there is no contribution to the oxygen flux from solid state diffusion, then the observed heat of transport should become equal to that calculated for one-component gas phase transport (approximately 50 kcal/mole). The observed -8.3 kcal/mole heat of transport is not in agreement with that calculation. The difference may be caused by the inability of the system to reach steady state in 1000 hours.

The experimental arrangement deserves some comment. Each experiment to date has involved a horizontal stack of right circular cylindrical pellets laid end to end in a sealed molybdenum tube. No attempt was made to improve solid-to-solid contact between pellets by altering the pellet surfaces in some way. Therefore, we cannot be sure how efficient oxygen transfer between pellet interfaces has been. The statement that solid-state diffusion is a viable mechanism all along the pellet column implies that oxygen transfer from pellet to pellet at the interface junctions is rapid compared to the solid-state diffusion itself. By placing spacers between the pellets, we may have merely provided a more tortuous path for oxygen in the gas phase and thereby slowed down the gas phase kinetics at the interfaces.

To define the role of solid state diffusion in the experimental model better, three capsule experiments will be fabricated and annealed for 2000 hours in a temperature gradient from 1500 to 900°C. The capsules will contain UO_2 -25% PuO_2 of identical stoichiometry (approximately 1.97 O:M). One capsule will consist of a column of 1/4-inch-long pellets in a sealed molybdenum container. The second capsule will consist of the same pellet arrangement and capsule configuration as the first except that molybdenum spacers will be placed between each of the pellets. The third capsule will consist of three or four 2-1/2 to 3-inch-long rods sealed in a molybdenum container. The length of these rods is limited primarily by fabrication restraints.

5. OXYGEN ACTIVITY STUDIES

The basis for carrying out oxygen activity studies in the UO_2 - PuO_2 system was described in the tenth quarterly report (GEAP-12066). Tantalum capsules were used as oxygen permeable containers for $(\text{U}_{0.75}\text{Pu}_{0.25})\text{O}_2$ which were heated in dry He-6% H_2 at 1700°C. Results of oxygen-to-metal ratio determinations on these oxide materials indicated that the tantalum reduced the oxide to a lower oxygen activity than that dictated by the atmosphere. Bureau of Mines data⁴ indicate that Ta_2O_5 should be unstable in dry He-6% H_2 above about 1700°C; therefore, a number of further experiments were carried out at 2000 and 2200°C in dry He-6% H_2 and dry He-6% H_2 diluted with helium. The results of these tests are given in Table 2. The lack of over-all variability in the results indicates that even at the higher temperatures, the tantalum reduces the oxide to the boundary of the single-phase region; no further reduction is possible unless the oxygen activity is decreased considerably.

TABLE 2. Results of High Temperature Oxygen Activity Studies

Temperature (°C)	Time at Temperature (hours)	Furnace Gas	O:M Ratio
1700	3	15 cfh He-6% H_2	1.915
1700	10		1.913
1700	10		1.922
2000	3		1.908
2000	3		1.911
2230	1		1.921
2000	5	40 cfh He + 10 cfh He-6% H_2	1.901
2200	3		1.908
2200	1		1.922
2250	2		1.919
2100	3.5	50 cfh He + 5 cfh He-6% H_2	1.909
2200	2		1.908
2250	1		1.924

In future experiments, molybdenum will be used as a capsule material. Molybdenum has been shown to be permeable to oxygen at temperatures above 2000°C and will not reduce the oxide material as the tantalum appears to do.

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