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A THERMODYNAMIC DATA PROGRAM INVOLVING PLUTONIA AND URANIA AT HIGH TEMPERATURES

MASTER

QUARTERLY REPORT NO. 11
FEBRUARY 1 TO APRIL 30, 1970

E. A. AITKEN
S. K. EVANS

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Quarterly Report No. 11 – February 1 to April 30, 1970

E. A. Aitken
S. K. Evans

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**A THERMODYNAMIC DATA PROGRAM INVOLVING
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Quarterly Report No. 11 — February 1 to April 30, 1970

E. A. Aitken
S. K. Evans**ABSTRACT**

In the tenth quarterly report (GEAP-12066) the continuing scope of work related to vaporization of urania, oxygen redistribution in a temperature gradient, and sodium fuel interactions was presented. Work progress is presented on these tasks.

In addition, the scope of new experiments to extend the study of oxygen activity in UO_2 - PuO_2 systems to higher temperatures is described.

1. INTRODUCTION

This quarterly report is eleventh in a series which describes experiments directed toward understanding plutonium segregation in urania-plutonia solid solutions and in fast reactor environments. In the tenth quarterly report (GEAP-12066) the progress of several experiments which have a bearing on plutonium segregation was described. Continued progress in these areas is described in this report. New experiments to study reactions in the Na-U-O system are discussed.

2. DETERMINATION OF COMPONENT ACTIVITIES

The determination of uranium activity in UO_2 - PuO_2 solid solution is being studied by means of a transpiration technique using air and CO/CO_2 as the carrier gases. Experiments have been undertaken to determine the system response to uranium-bearing gas pressures in the temperature range of 1300 to 1500°C using the carrier gases 10 CO_2/CO and $CO_2/10 CO$. Figure 1 shows results of experiments conducted to date in the 10 CO_2/CO carrier gas as a function of temperature. Also included on the graph is a representation of Alexander's data.¹ The data reported here are 30 to 40% lower than those reported by Alexander, but are quite self-consistent and in line with comparisons made with Alexander's work in the ninth quarterly report (GEAP-12047).

An experiment at 1500°C using $CO_2/10 CO$ as a carrier gas was not successful because a small amount of air was introduced into the furnace during the heatup period and resulted in an abnormally high value for the UO_3 vapor pressure. The source of this problem has been detected and steps have been undertaken to correct it.

Preparations are in progress to install the transpiration equipment in an alpha enclosure for commencement of experiments with UO_2 - PuO_2 compositions. During the next report period, this installation will be completed and 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.

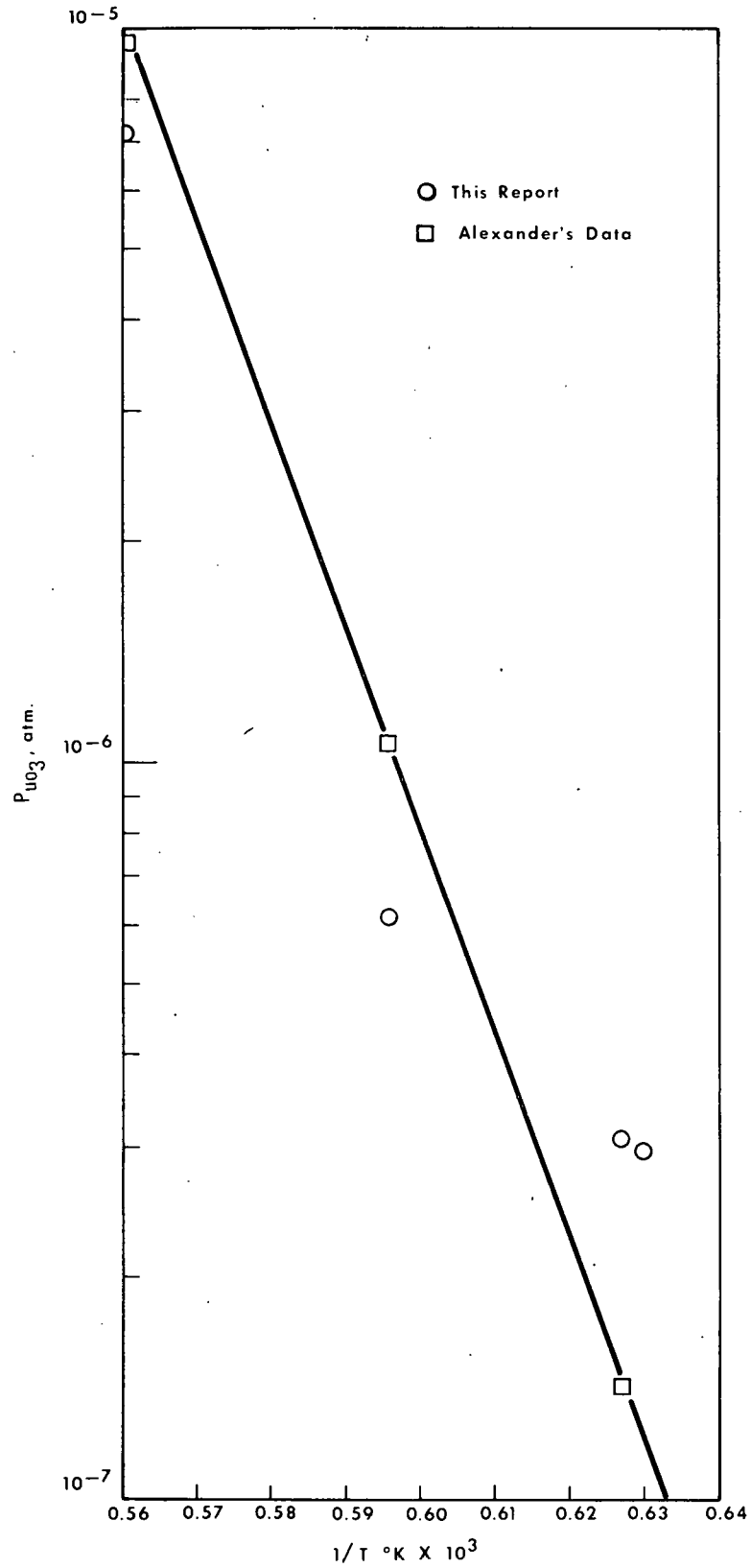


FIGURE 1. COMPARISON OF P_{UO_3} VERSUS $1/T$ WITH ALEXANDER'S DATA

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

Interpretations of previous data based on Aitken's² one component cyclic model were presented in the ninth quarterly report (GEAP-12047). To investigate the validity of these interpretations, thermal gradient experiments have been designed using three different fuel compositions: (U_{0.8}Pu_{0.2})O_{1.93}, (U_{0.8}Pu_{0.2})O_{1.995}, and (U_{0.6}Pu_{0.4})O_{1.95}. The last two of these capsules have undergone 1000-hour anneals in a temperature gradient from 900°C to 1600°C. The first composition will begin a 1000-hour anneal during the next quarter.

The results of the two experiments just completed are shown in Figures 2 and 3. Figure 2 indicates the results of the capsule experiment comprising (U_{0.8}Pu_{0.2})O_{1.995}. Within the ability to measure changes in oxygen-to-uranium ratio, no gradient in oxygen activity was observed as a result of the temperature gradient. The observed post-test oxygen-to-metal ratio in this experiment was 1.996, a possible indication that the oxygen-to-metal numbers may be biased slightly below the stoichiometric value.

Figure 3 shows the result of the capsule experiment containing (U_{0.6}Pu_{0.4})O_{1.95}. For comparison, the results of two earlier 1000-hour anneals consisting of PuO_{1.82} and (U_{0.8}Pu_{0.2})O_{1.972} are also shown in Figure 3. These data have been analyzed according to the procedure outlined in the ninth quarterly report (GEAP-12047) where the heats of transport experimentally determined for the compositions described were compared to Aitken's one component cyclic model according to the equation

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

where Q^*_{eff} is the heat of transport measured from Arrhenius plots of stoichiometry versus temperature data; Q^* is the heat of transport which would apply if the H₂/H₂O pressure in the capsule were sufficiently high that no oxygen pressure gradient existed as a function of temperature; L_s and L_g are related to the oxygen diffusion coefficients in the gas and solid phases, respectively.

Since the oxygen-to-plutonium ratios in the three experiments described were similar, not much change is expected in L_g , thus for calculational purposes it is held constant. On the other hand, L_s is assumed to be proportional to the oxygen vacancy concentration as suggested by Prigogine.³ For a constant oxygen-to-plutonium ratio, the oxygen vacancy concentration increases as the plutonium content increases. Thus the value for Q^*_{eff} should be altered accordingly as the plutonium content is changed. The calculations are made in the following manner. Since L_g is assumed to be constant, and L_s is proportional to the vacancy concentration, then one can write

$$Q^*_{\text{eff}} = \frac{Q^*}{1 + KX}$$

where K is a constant to be determined and X is the deviation from stoichiometry (example, if O:M = 1.97, then $X = 0.03$) and Q^*_{eff} and Q^* are defined above. Calculations were made using the Q^*_{eff} value for PuO_{2-x} as a reference. The results of these calculations are:

Pu Content (%)	O:M	X	Q* (kcal/mole)	Q* _{eff} Calculation (kcal/mole)	Q* _{meas} 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

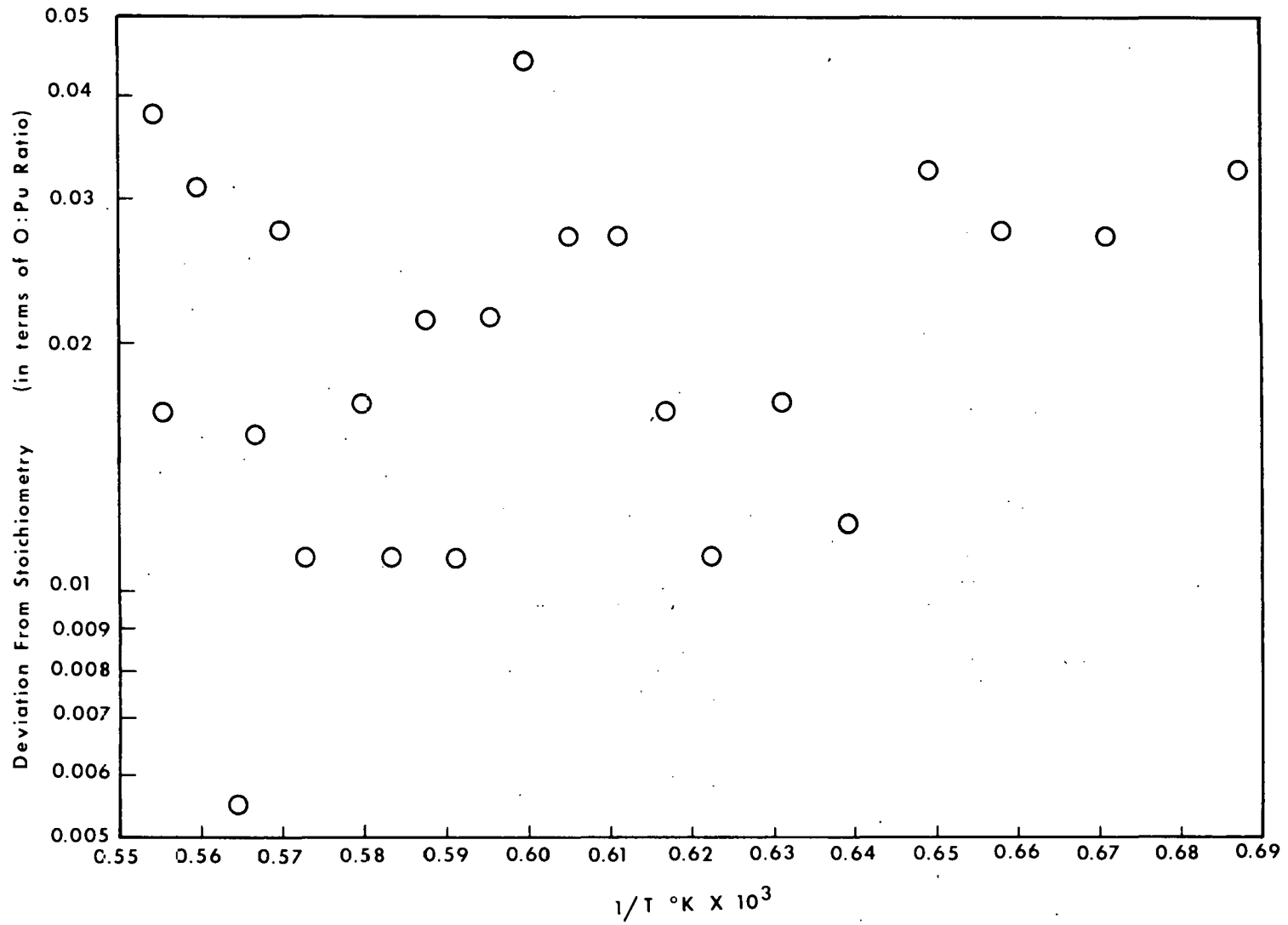


FIGURE 2. RESULTS OF 1000 HOUR THERMAL GRADIENT ANNEAL OF (U_{0.8}Pu_{0.2})O_{1.999}

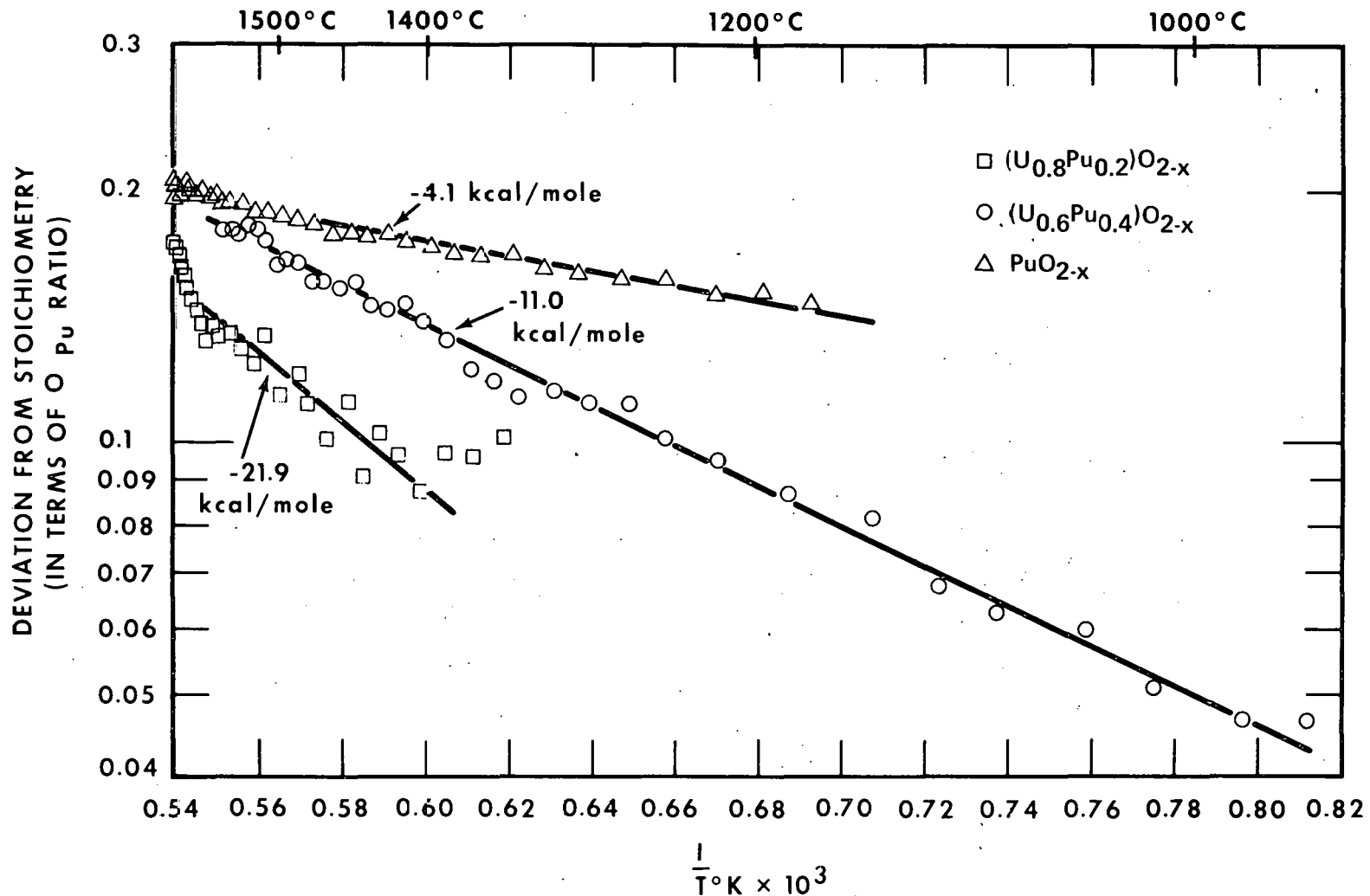


FIGURE 3. ARRHENIUS PLOTS OF 1000-HOUR THERMAL GRADIENT ANNEALS

Thermal gradient experiments in the fuel-stainless steel-cesium system carried out under other programs⁴ have led to the result that cesium enhances the rate of oxygen transport in a thermal gradient environment. Molybdenum capsules were loaded with UO_2 -25% PuO_2 of 1.965 stoichiometry along with a 1-inch section of Type-316 stainless steel placed at the cool end of the fuel and enough Cs_2MoO_4 to approximate 2% burnup irradiation conditions. Oxygen-to-metal ratio determinations made following a 100-hour thermal gradient anneal from 1550 to 870°C indicated that oxygen migration had taken place throughout the temperature gradient and resulted in a heat of transport value of about -7 kcal/mole. A knee in the Arrhenius plot at about 980°C resulted in a higher heat of transport (-18 kcal/mole) below that temperature. The validity of the knee of the curve and low temperature slope may be open to question if cesium contamination was present in those pellets. It is significant, however, that the existence of two apparent mechanisms for oxygen transport corresponds to similar data reported for higher temperatures without cesium.

High-temperature thermal gradient experiments (2000°C maximum) reported by Evans, Aitken, and Craig⁵ resulted in heats of transport for UO_2 -20% PuO_2 of the order of -7 to -10 kcal/mole. However, data for lower temperature experiments (1580°C maximum reported in Quarterly Report Number 8 (GEAP-12032) indicate a heat of transport for the same composition of -21.9 kcal/mole. The findings of the cesium-bearing experiments show similar effects with the changes in mechanism appearing at about 980°C as compared to between 1550 and 1600°C in the tests without cesium.

Future experiments are designed to investigate these phenomena further. The capsules for the two experiments described in Quarterly Report Number 9 (GEAP-12047) have been fabricated and the thermal gradient anneals will begin during the next quarter. Two additional experiments have been planned to investigate further the effects of alkali metals on the mechanisms for oxygen migration. The capsules will contain (UO_2 -25% PuO_2) with two different amounts of sodium metal added to measure the influence of sodium activity on the transport mechanisms. These capsules will be annealed concurrently with the first two described.

4. THERMODYNAMIC BEHAVIOR OF SODIUM-FUEL REACTION PRODUCTS

Irradiated fuels which have been exposed to sodium through cladding failure show evidence of a reaction phase around the periphery. Microprobe examination has indicated that sodium is present although not always at the same amount. Direct verification of a reaction phase in the irradiated fuel by x-ray diffraction has been inconclusive. Several compounds are known to exist in the Na-U-O system, however it is recognized that a radiation environment or a particular chemical environment may influence the type of compound formed. We have undertaken a study of the thermodynamic stability of these compounds. The initial step is to determine the various conditions in which reaction will occur, then prepare compounds for thermodynamic stability measurement.

The first method of synthesis was to add a mixture of (U,Pu) O_2 granules and Na_2O to a large excess of sodium and heat treat at 850°C in a sealed nickel capsule. There was an apparent reaction product; however, the capsule ruptured prematurely and the sodium was lost. A second set of capsules has been made and is awaiting to be rerun. This experiment will enable us to make a direct comparison between the stability of the sodium fuel compound and the oxygen content of the sodium. The reacted mixture contains three phases [Na liquid, Na fuel compound, and unreacted (U,Pu) O_{2+x}]. These three phases are sufficient to establish equilibrium at a given temperature. Since the routine product is difficult to extract, other methods of synthesis are being considered.

The second method of synthesis is by direct reaction of the oxide fuel with a sodium salt (sodium oxide or sodium carbonate). This method allows us to make compacts of nearly pure compound, however, the principal drawback is that it is difficult to control the oxygen and sodium activity of the environment. Therefore, the product may not be equivalent to that which is found in a failed sodium fuel pin. The ability to make solid compacts, however, is important since characterization is easier and thermodynamic stability can be measured with an emf cell. Before making reacted compacts for thermodynamic measurements, we have attempted to characterize the reaction by thermogravimetric analysis (TGA) and by differential thermal analysis. The TGA work was done on 20% Pu-80% U coprecipitated powder with an oxygen-to-metal ratio of 2.25. The differential thermal analysis (DTA) work was done with UO_2 powder initially; later the apparatus will be transferred to a glove box for work with plutonium.

The third method of synthesis is to react sodium with fuel pellets in a sealed container in a temperature gradient. Sodium prefers to react with the fuel at the lower temperature end. Under a temperature gradient, oxygen in the fuel moves preferentially to the cold end. At some critical point along the gradient the oxygen content of the fuel becomes high enough to react with sodium to form a sodium fuel compound. Thus there should be a boundary temperature below which sodium fuel compound will form. Above this boundary no sodium fuel compound is formed. The fuel pellet immediately above the boundary should be near equilibrium with the sodium compound and its oxygen-to-metal ratio should be representative of the thermodynamic stability of the sodium fuel compound.

An initial attempt to determine the boundary fuel composition in contact with Na-fuel compound was made during this period. A sealed Type-316 stainless steel capsule containing sintered oxide pellets with an initial oxygen-to-metal ratio of 1.965 was heated for 100 hours in a thermal gradient ranging from 1150 to 550°C over a length of about 6 inches. The capsule contained about 65 milligrams of sodium metal which was initially situated at the high temperature side. During the heat treatment the sodium was expected to vaporize and condense at the low temperature side. In moving down the gradient, the sodium vapor would interact with fuel and permitted adjustments in the oxygen-to-metal ratio. The capsule was opened by making two slots along the length so that the cladding could be peeled away from the pellets as shown in Figure 4. The first sixteen pellets from the hot end were unchanged from visual inspection and the internal cladding surface opposite them was bright. The seventeenth pellet, however, was significantly etched and showed signs of deterioration along one-third of its length. This observation has been made before when sodium reacts with a fuel pellet. The remaining pellets at the cold side were essentially unchanged except for a layer of sodium metal which oxidized on exposure to air. The oxygen-to-metal ratio on the first sixteen pellets was determined, the seventeenth pellet which had undergone an apparent change will be examined metallographically for evidence of sodium-fuel reaction product. The oxygen-to-metal ratio as a function of position is shown in Figure 5 together with temperature profile. The results indicate a general decrease in the oxygen-to-metal ratio of the pellets at the hot end which suggests a net movement of oxygen to the cold end. The oxygen-to-metal ratio increases as one proceeds toward the cold end and approaches a value of about 1.965 at the "apparent" reaction interface of pellet 17 which corresponds to a temperature of about 750°C. This tentative result suggests that the sodium-fuel compound at 750°C will coexist with fuel at an oxygen-to-metal ratio of 1.965 in excess sodium.

Further experiments are planned to cover a wider range of temperatures to induce a larger amount of oxygen transport down the gradient to produce a reaction product. The amount of oxygen transported determines the amount of reaction product as long as the sodium is in excess.

THERMAL GRAVIMETRIC ANALYSIS STUDY

The TGA runs were made on a Cahn microbalance in a quartz envelope to provide different atmospheres. During some preliminary runs it was found that moisture caused noticeable weight loss as a result of the formation of NaOH vapor. Runs with Na₂O in 6% H₂-94% He also showed significant vaporization probably because of the formation of NaOH vapor. Vaporization was less significant in argon. With Na₂CO₃ as a reactant, a noticeable reaction could be ascertained from the CO₂ loss. Figure 6 shows several TGA runs made in a dry argon atmosphere which was gettered by titanium chips at the bottom of the quartz envelope. The samples were blended as powders and a few milligrams of the powder mix was transferred to the platinum dish before heating. The heating was accomplished by raising the temperature at the rate of approximately 1000°C/h and then holding at 1000°C for times up to 2 hours. With Na₂CO₃ alone, the decomposition is slow and begins to appear at around 1000°C. The weight loss rate is constant with time at 1000°C. With the coprecipitated (U_{0.8}Pu_{0.2})O_{2.25} powder alone, there was about 1% loss between 200 and 500°C which can be attributed to reduction to a near stoichiometric composition. Run 1 which consisted of a Na₂CO₃ to (U,Pu)O₂ ratio of about 1.1 resulted in only a small loss until about 800°C. Run 2 which had a corresponding ratio of 1.75 showed similar results to Run 1 and extensive weight loss was observed after 50 minutes at 1000°C. The arrow mark identified with CO₂ indicates the expected loss for complete decomposition of the Na₂CO₃. It is evident that CO₂ loss occurs at a much faster rate when Na₂CO₃ is heated in a mixture with (U,Pu)O₂ compound, indicative of a reaction between the two oxides. Run 3 had the same powder mixture as Run 1 except a small amount of Na metal was added to see if the reaction could be accelerated. The amount of Na added increased the over-all Na/(U+Pu) to about 3.0. This run indicated no initial weight loss as with the other runs but the onset of CO₂ decomposition occurred around 800°C and

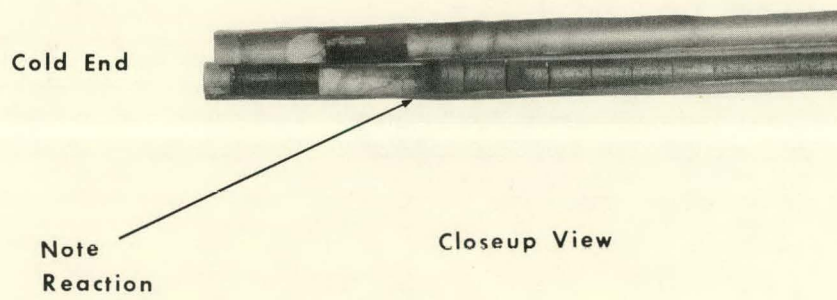
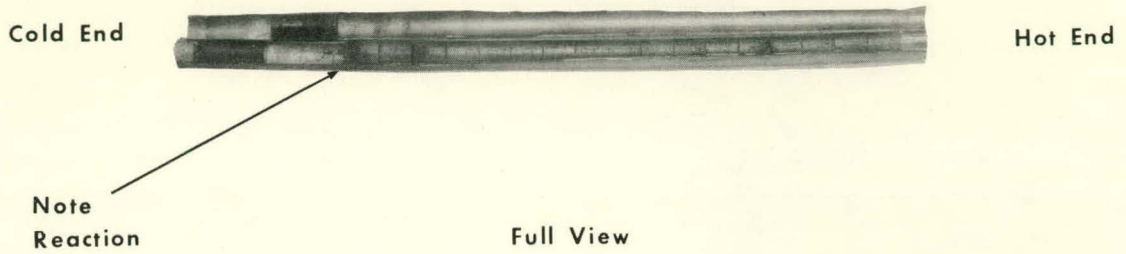


FIGURE 4. HEAT TREATED FUEL CONTAINING SODIUM IN A TEMPERATURE GRADIENT

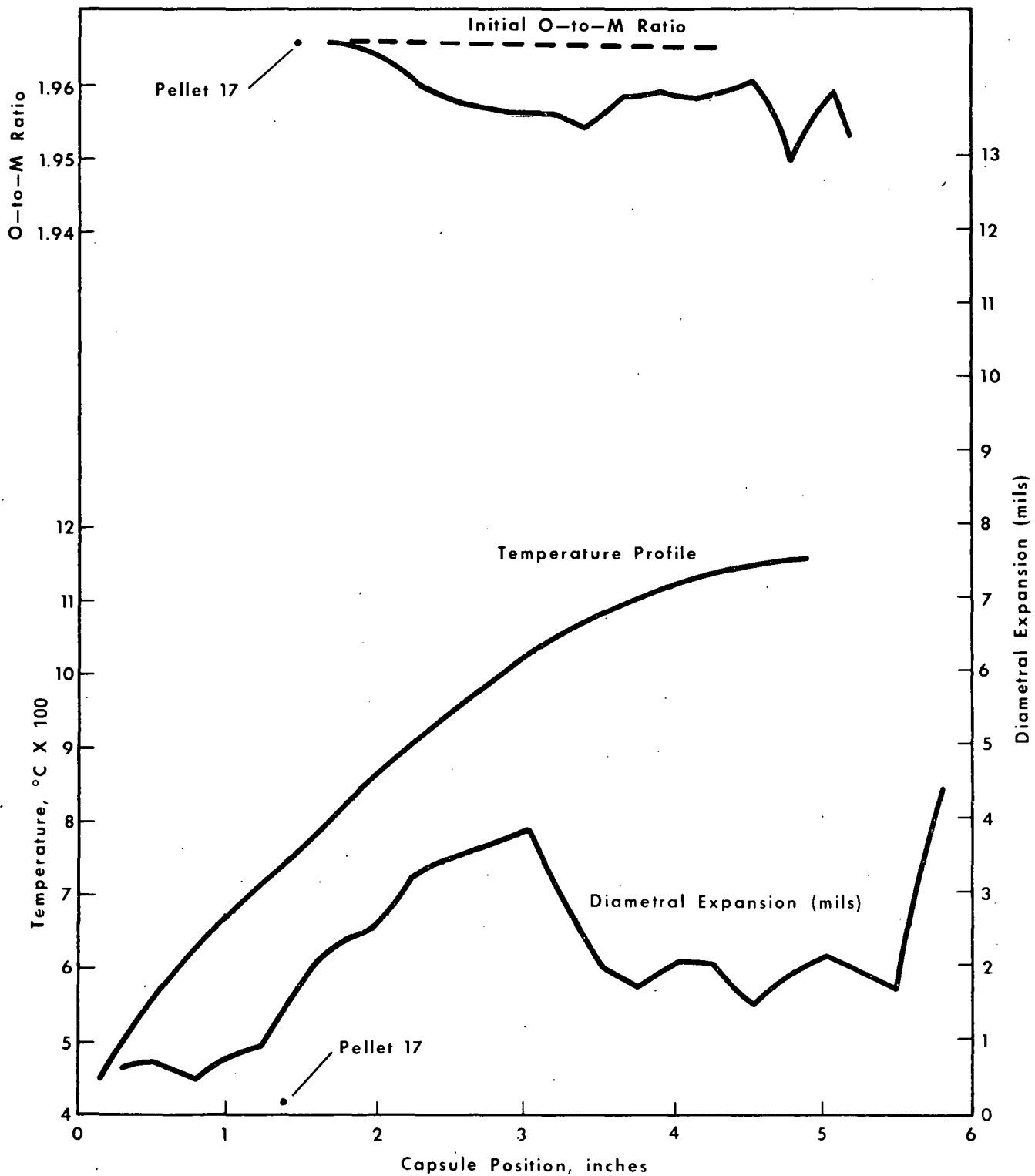


FIGURE 5. CHANGES IN O-TO-M AND DIAMETRAL EXPANSION OF FUEL CAPSULE AFTER 100 HOURS IN Na ATMOSPHERE IN A TEMPERATURE GRADIENT

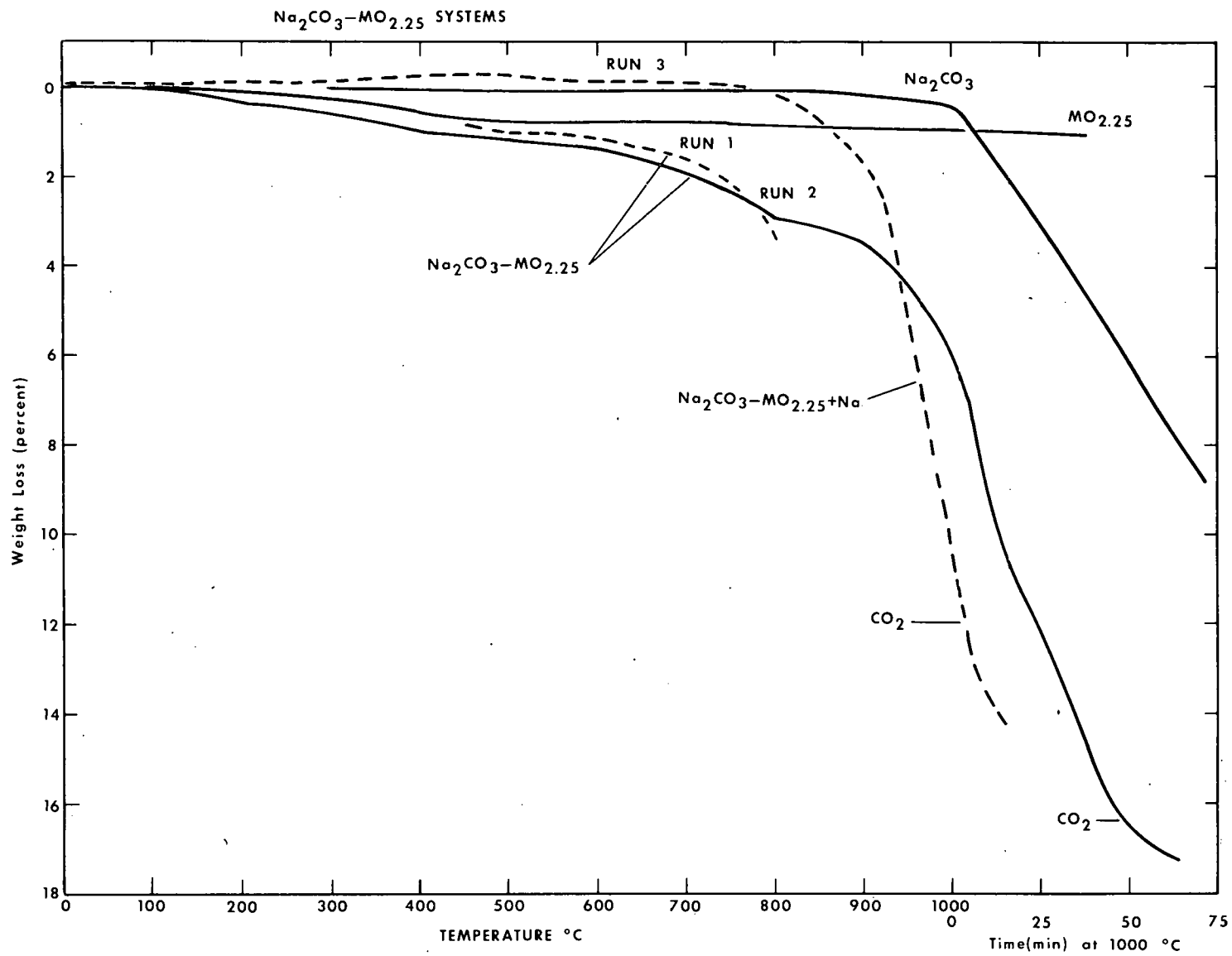


FIGURE 6. WEIGHT LOSS $\text{Na}_2\text{CO}_3\text{-MO}_{2.25}$ MIXTURE VERSUS TEMPERATURE + TIME at 1000°C

was nearly complete at 1000°C. The presence of molten sodium does indeed speed the reaction and apparently reduces the hyperstoichiometric fuel by forming Na₂O at around 200 to 400°C. After a few minutes at 1000°C, the weight loss continued beyond the CO₂ decomposition probably by vaporization of unreacted sodium.

DIFFERENTIAL THERMAL ANALYSIS STUDY

Experiments were planned for using the DTA technique to investigate the conditions and thermodynamics for the formation of NaUO₃ according to



Approximately 3-mg aliquots of a 0.96:1 (Na₂O:UO₂) mixture was heated in the DTA equipment at 10°C/min under 8.5% H₂/He atmosphere (50 ml/min) to 1000°C. The UO₂ used was analyzed for O:U ratio; found UO_{2.09}. All transfers and loadings were carried out in an inert atmosphere glove box. A typical run is shown in Figure 7 using 4.2 mg of the mixture with Al₂O₃ as the reference material. X-ray analysis after heating confirmed the formation of a well-crystallized NaUO₃ with a residual amount of UO₂. Inspection of the thermogram shows an exothermic reaction occurring at 450°C. The exotherm is very steep with multiple peaks. This phenomenon may be due to local environmental heating and fast reaction at a few isolated areas. The two solids were ground to a fine powder while mixing together. The endotherm at 425°C is typical of UO₂.

Because of the ease of manipulation, Na₂CO₃ was substituted for Na₂O. A mixture (3 to 5) containing UO₂:Na₂CO₃ ratio of 1.46:1 was used in a DTA run to 1000°C under 8.5% H₂/He atmosphere. A 6.5-mg aliquot run at the same conditions as before gave three exotherms as shown in Figure 8. X-ray analysis after heating confirmed the formation of extremely well-crystallized NaUO₃.

The presence of three exothermic peaks in Figure 8 was unexpected, since previously, only one had been seen during reaction (1). It was surmised that diffusion and local reaction of the H₂ with the Na₂CO₃ on the nickel surface had possibly generated compounds, such as CO, CO₂, H₂O, and CH₄ and may account for the other peaks. As a check, a 4.0-mg mixture of 3 to 5 was subjected to the same conditions as before except with only a helium atmosphere. X-ray analysis indicated well-crystallized Na₂U₂O₇. This experiment was repeated, except with a mixture (3 to 13) containing a mole ratio of UO₂:Na₂CO₃ of 2.75:1. X-ray analysis again confirmed Na₂U₂O₇ as the product. The thermogram from this last run showed no exothermic peaks until about 800°C and corresponds to reaction temperatures observed in the TGA experiments in Argon. Standard thermograms of Na₂CO₃, Na₂O, NaOH, and UO₂ were also run with 8.5% H₂/He atmosphere as well as Na₂U₂O₇ in air as comparisons.

5. OXYGEN ACTIVITY STUDIES

The basis for carrying out oxygen activity studies in the UO₂-PuO₂ system was described in the tenth quarterly report (GEAP-12066). The initial series of experiments was performed to determine equilibration times in the system. Capsules consisting of (U_{0.75}Pu_{0.25})O_{2.0} clad in tantalum, then annealed in dry He-6% H₂ for 3 and 10 hours at 1700°C. Oxygen-to-metal ratio determinations were made on the capsules following the anneals resulted in values of 1.915 and 1.913 for the 3 and 10 hour runs. Extrapolations of Markin and McIver's⁶ data indicate that pellets of this composition annealed at 1700°C in He-6% H₂ containing 100 ppm H₂O should have an equilibrium oxygen-to-metal value of 1.946. The extremely low value obtained indicates that the tantalum may be reducing the fuel to a lower oxygen activity than that dictated by the atmosphere. Bureau of Mines⁷ data on the free energy of formation of Ta₂O₅ indicate that Ta₂O₅ should be unstable in dry He-6% H₂ above about 1700°C, therefore, future tests will be conducted at higher temperatures.

Tantalum capsules have been fabricated for anneals at 2000 and 2200°C in dry He-6% H₂ and dilutions of this gas with high purity helium to increase the H₂O-to-H₂ ratio.

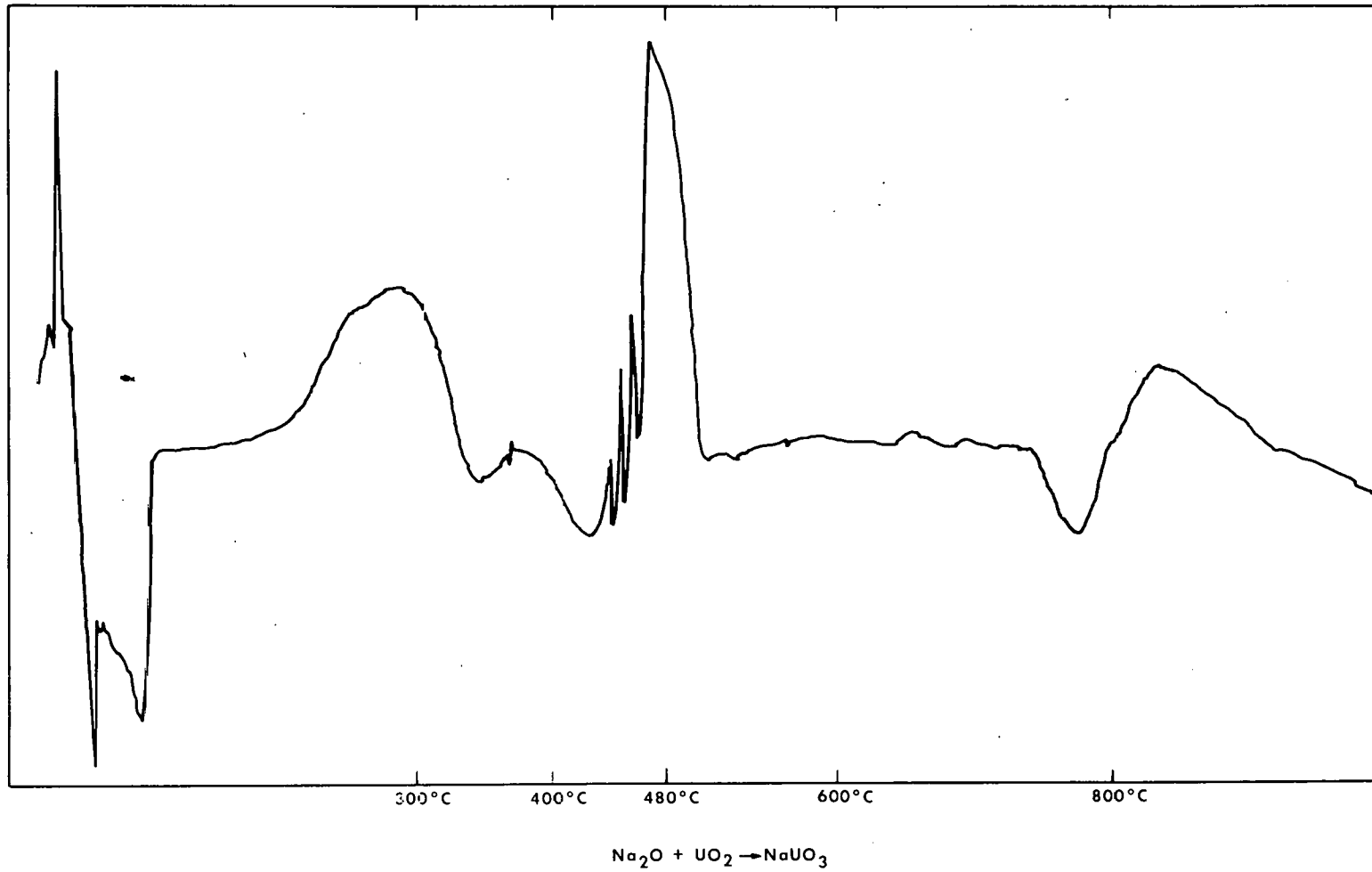


FIGURE 7. THERMOGRAM OF $\text{Na}_2\text{O}:\text{UO}_2$ MIXTURE AS A FUNCTION OF TEMPERATURE IN A HYDROGEN ATMOSPHERE

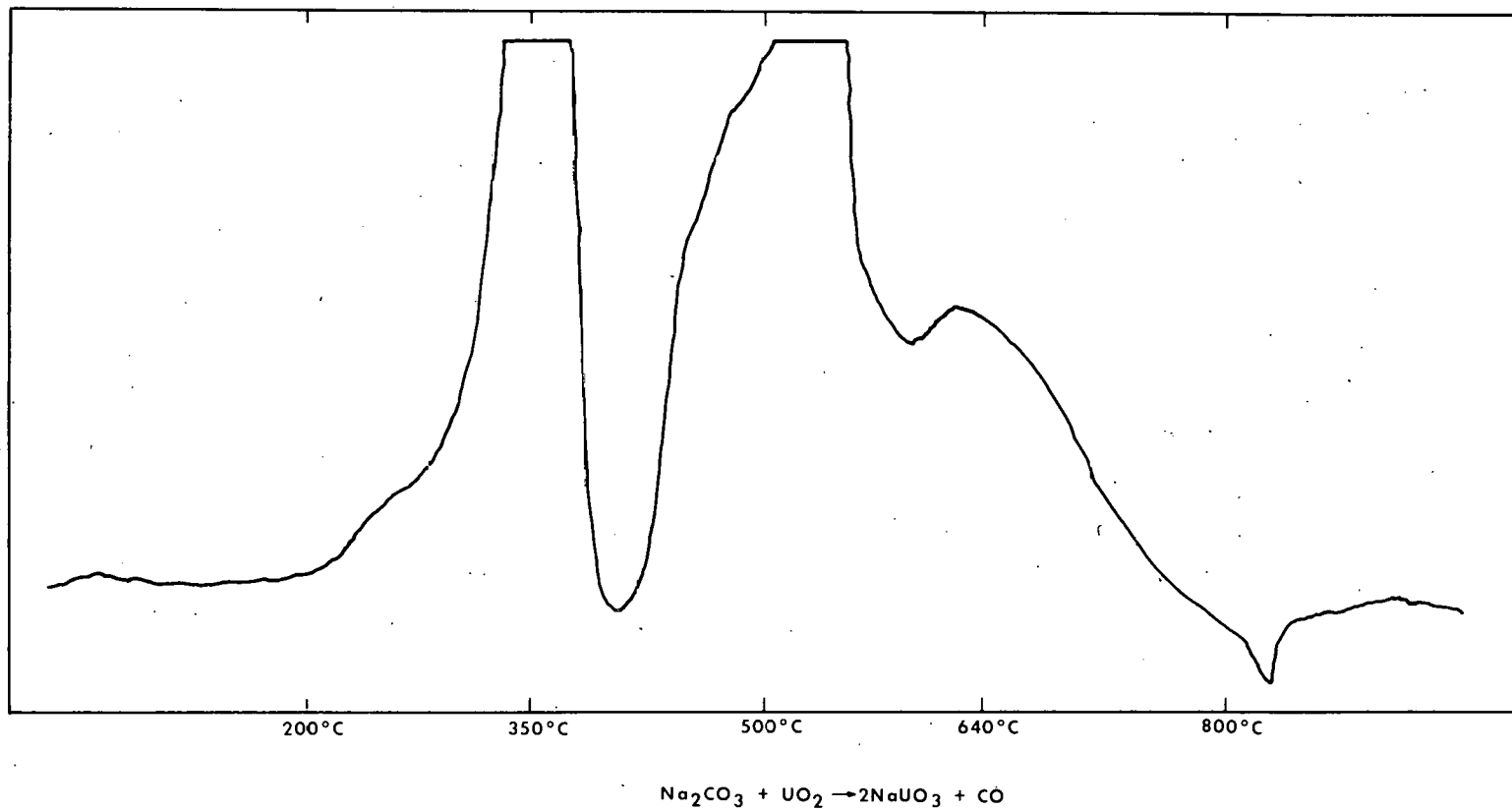


FIGURE 8. THERMOGRAM OF $\text{Na}_2\text{CO}_3:\text{UO}_2$ MIXTURE AS A FUNCTION OF TEMPERATURE IN A He HYDROGEN ATMOSPHERE

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