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A THERMOBALANCE FOR STUDYING THE CHLORINATION
OF PLUTONIUM DIOXIDE WITH PHOSGENE

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ABSTRACT

An automatic recording thermobalance for studying the chlorination of plutonium dioxide with phosgene is described. The highly toxic and corrosive nature of these materials imposed restrictions on the construction and operation of the thermobalance.

The instrument operates on the deflection balance principle in which the extension of a precision quartz helix spring is measured by a linear variable differential transformer. Experimental results from the gas-solid reaction between phosgene and plutonium dioxide are discussed.

A THERMOBALANCE FOR STUDYING THE CHLORINATION OF PLUTONIUM DIOXIDE WITH PHOSGENE

INTRODUCTION

A process for the preparation of metallic plutonium using a chloride intermediate is being developed at Hanford.^(1, 2, 3) The proposed process would include calcination of plutonium nitrate to PuO_2 , chlorination of PuO_2 to PuCl_3 , and electrolytic reduction of PuCl_3 to the metallic plutonium.

An automatic recording thermobalance was built to study the chlorination of PuO_2 with phosgene (carbonyl chloride). The data obtained were used to determine calcination and chlorination conditions which affect the extent of chlorination of PuO_2 .

The instrument operates on the deflection balance principle in which the extension of a precision quartz helix spring is measured by a linear variable differential transformer. The highly toxic and corrosive nature of these compounds imposed restrictions on the materials of construction and on the operation of the instrument; that is, the toxicity of the alpha-emitting plutonium compounds and the toxicity and corrosiveness of phosgene. No commercial instrument that was readily adaptable to these severe conditions could be obtained. Therefore, a thermobalance was constructed and installed in a glove box where plutonium compounds and phosgene were kept under controlled conditions.

APPARATUS

The thermobalance operates on the deflection balance principle in which the extension of a precision quartz helix spring is measured by a linear variable differential transformer. Because of the highly toxic nature of plutonium compounds and phosgene, the instrument was installed in a standard 4 x 6 x 8 ft Hanford glove box which opened into a fume hood (see Figure 1). The associated electronic equipment was placed outside the glove box. To assure that no phosgene was released to the room atmosphere*, the

* The maximum permissible concentration for prolonged exposure to phosgene is about 1 ppm in air.

supply cylinder was kept in the fume hood, and the cylinder, valve, and flow meters were also placed in small ventilated enclosures.

FIGURE 1
Gloved Box

Balance System

The weighing system consisted of a quartz helix spring and a linear variable differential transformer (LVDT). An LVDT is an electro-mechanical transducer that produces an electrical output that is proportional to the displacement of a movable iron core. A rod-shaped magnetic core is positioned axially inside the cylindrical transducer coil and provides a path for magnetic flux linking the coils.

A quartz helical spring and the iron core (armature) of the LVDT (transducer coil) were suspended from a ground glass stopper. The extension of the spring causes the core to move; the amount of movement is proportional to the load applied. To protect the iron core from the corrosive atmosphere, it was enclosed in Pyrex. This operation was very critical, since heating the core will cause a change in its magnetic properties. Quartz links were used below the core to connect it to a quartz sample boat. Platinum could not be used because it is readily attacked by phosgene. To reduce the possibility of vibrational nodes in the suspension chain, the quartz links were constructed of various lengths. The spring did not require a thermostat to prevent elongation with respect to an increase in temperature, since the purge gas and heat reflecting material on the baffles prevented any excess heating of the spring. The balance mechanism as shown in Figure 2 was then suspended inside the thermobalance glassware as shown in Figures 3 and 4.

As shown in Figure 3, the thermobalance glassware was composed of two portions: the Pyrex unit which is above the heating zone, and the quartz reaction tube in the heating zone. The two units are connected by a 65/40 ball and socket of their respective materials.

The Pyrex unit consists of three sections. The upper portion houses the quartz helix spring (12 g load capacity, 6 cm extensions, 0.005 mm/mg extension) which responds to the change in sample weight. The center portion surrounded the iron core and was itself surrounded by the LVDT (linear range 0.100 in.). There was also an adjustable platform for positioning the transducer coil. The base of the platform rests on the shoulder of the lower portion of the Pyrex tube. This lower portion has an argon purge inlet near the top and four baffles below it to keep the sensing unit free of corrosive atmosphere. This system prevented any reacting gas from contacting the balance mechanism.

FIGURE 2
Balance Mechanism

FIGURE 3
Thermobalance Glassware

FIGURE 4
Quartz Furnace Tube and Sample Boat

The quartz unit was half filled with quartz chips to preheat the entering gas and diffuse the gas to prevent streaming. The two small tubes at the bottom of the reaction tube protrude from the bottom of the furnace. One contains a ceramic-covered Pt/Pt-10% Rh thermocouple which protrudes approximately $\frac{1}{2}$ in. above the quartz chips. The other is the reaction gas inlet. Near the top of the reaction tube is the exhaust outlet for both the inert and reacting gases. Figure 4 shows the quartz unit, including the thin quartz sheath.

The Pyrex unit was raised and lowered with a stainless steel rack and gear assembly. This was done because existing materials already in the glove box were used to construct the instrument. The entire unit is shock-mounted with lead bricks.

Heating and Control System

A low voltage, high amperage tube furnace was used to heat the sample. The furnace temperature was controlled with a time-proportional controller. The furnace was supplied with a sensing thermocouple, and a motor-driven programmer was connected to the controller via a step-down transformer. This arrangement is shown in the lower half of Figure 5, except for the transformer which was mounted on the end of the glove box.

The sample temperature was measured by the thermocouple in the quartz reaction tube and was recorded. The thermocouples in the quartz reaction tube and in the furnace were made of Pt/Pt-10% Rh.

In this system, the controller was actually controlling a 20 amp relay which is connected to the transformer via the programmer. This heavy relay caused transients to be introduced in the weight change sensing system. Although the effect was not too serious (it could be corrected), the equipment became a maintenance problem and is being replaced by a silicon-controlled rectifier power controller and a cam-driven programmer.

FIGURE 5

Heating and Control System Instrumentation

Recording System

If the sample gains or loses weight, the corresponding elongation or contraction of the quartz helix spring moves the iron core with respect to the transducer coil. This movement causes an electrical output that is directly proportional to the weight change. With the iron core in the center of the coil, there is no signal; but if it moves in either direction, a signal is generated, allowing the measurement of both weight gains and weight losses.

The electronic system was composed of the transducer, a converter, a damper, a controller, a programmer, and recorders. The converter reduced 110 to 6.3 Vac and converted the alternating current output from the transducer coil to direct current for a millivolt recorder. Since there was no mechanical damper in the system, it was desirable to electronically dampen (using capacitors) any eccentricities or oscillations. The recorder used was a standard -12 to 0 to +12 mv recording potentiometer that was calibrated with a linear scale in milligrams (see Figure 5). A timer was used to operate the instrument a predetermined time.

Gases

Argon was used as the inert gas for purging the Pyrex unit and the furnace tube. Phosgene, which was obtained in 10 lb cylinders, was placed inside a Lucite enclosure in the fume hood adjacent to the glove box. The gas was transferred from the hood to the glove box via a valve and flow meter. Figure 6 shows the valve, flow meter, and connections inside the Lucite enclosures. The enclosures were kept under slightly negative pressures. Since phosgene is denser than air, the vacuum was applied at the bottom of these enclosures.

After the PuO_2 was chlorinated, it was converted back to the oxide with air. It was not necessary to accurately meter the air, so a simple flow indicator was used. All the gases were transported by flexible polyethylene tubing inside the glove box; stainless steel tubing was used outside.

Gloved Box

Hood

FIGURE 6
Gas Handling System

Exhaust Gases

The composition of the gas resulting when a stream of phosgene is heated above 500 C has been reported by Lust⁽⁴⁾ to be phosgene, carbon monoxide, chlorine, and hydrogen chloride. He reports that appreciable dissociation of phosgene was not observed below 500 C, but when PuO_2 was present, dissociation was observed at all temperatures investigated. The exhaust gases from the chlorination of PuO_2 at 425 C, after filtering and cooling were shown to be phosgene, carbon dioxide, carbon monoxide, chlorine, and hydrogen chloride. Other authors report that the dissociation of phosgene starts at 300 C, and is complete at 800 C⁽⁵⁾ and that phosgene is 21% dissociated at 400 C.⁽⁶⁾ The system could, therefore, be expected to contain phosgene, carbon monoxide, carbon dioxide, chlorine, hydrogen chloride, and volatile metallic chlorides from impurities.

Walker⁽⁷⁾ has shown that the system is extremely corrosive towards gold, platinum, etc. For example, he exposed samples of platinum and gold to a stream of phosgene at 500 C and found that they were completely destroyed in less than 70 hr.

During the chlorination, solid deposits were found on the upper portion of the quartz reaction tube, ball and socket joint, and on the lower portion of the Pyrex unit. They varied in color from light yellow to dark reddish-brown; the nearer the furnace, the darker the color. Lust⁽⁴⁾ has previously shown that these deposits are due to impurities in the PuO_2 , and that surfaces kept above 325 C showed no trace of these solids which are volatile metal chlorides.

Pressure surges due to bubbling the exhaust gas through a caustic solution decreased the sensitivity of the balance. Therefore, the exhaust gases were passed up through a continuous, gas-phase type, scrubbing tower. This apparatus was very efficient and kept the inside of the glove box almost corrosion free.

OPERATION AND PROCEDURE

A sample was placed in the tared quartz sample boat and weighed on the analytical balance. The boat was then suspended from the quartz chain, the Pyrex unit was lowered into position, and the instrumentation was started. Although larger samples could be used, most thermograms were run using approximately 200 mg of material. The bed depth was, therefore, kept to less than $\frac{1}{8}$ -in. and better resolution was obtained. After the system was cooled (the sample was usually cooled under an inert atmosphere), the sample boat was removed and reweighed.

Standardization

The instrument was standardized with standard platinum weights, calcium carbonate, and calcium oxalate monohydrate. The weight change "breaks" agreed with both theoretical and reported values for calcium oxalate monohydrate.^(8, 9, 10, 11) The standardization was begun as described above, with argon flowing at the rate of approximately 40 cc/min through the reaction gas inlet, and at least 70 cc/min through the purge gas inlet. The instrument was heated from room temperature to approximately 1050 C at less than 3 C°/min. At this slow rate of heating, there appeared to be very little thermal lag between the furnace temperature and the sample temperature as measured by the thermocouple enclosed in the thin quartz sheath.

Two types of experiments were made at constant weight to check the pen drift with respect to temperature and time. A 200.1 mg quartz weight was used. A slight decrease in apparent weight was observed upon heating from room temperature to 1050 C in approximately 5 hr. A further slight decrease was observed during the subsequent 2 hr at 1000 C. In a different test, the instrument was raised to 500 C, the quartz weight and sample boat were inserted in the instrument, and the temperature was held for approximately 7 hr. The general trend indicated a slight weight loss with time. In both experiments, the apparent weight loss was less than 5.0 mg. The capability of the thermobalance is thus limited by a 5% maximum error in the

determination of sample weights. It was necessary to restandardize the thermobalance and run blank checks rather frequently during the chlorination experiments, since the volatile metal chlorides sublime onto the quartz chain and increase the weight of the suspension system.

Chlorination

When a sample of PuO_2 was to be chlorinated, the instrument was heated to a predetermined temperature, usually 500 C. Phosgene was allowed to flow at approximately 45 cc/min for at least 15 min (at least three reaction tube volumes) before the sample was introduced. The caustic scrubber was always started simultaneously with the phosgene.

The Pyrex unit was then quickly raised, the preweighed sample boat was suspended from the quartz chain, and the Pyrex unit was lowered. This was only a momentary break in the system, and since phosgene is denser than air, very little would escape from the quartz furnace tube. Of course, the phosgene was still entering the bottom of the furnace tube.

Upon completion of the chlorination reaction, the entering gas was changed to argon and a PuCl_3 weight was obtained. Since PuCl_3 is hygroscopic and reactive, an air weight could not be obtained. The correction on the thermogram due to different gas densities was very small. Air was then used to oxidize the PuCl_3 to PuO_2 . The temperature of the system was raised to 1050 C, held for at least 1 hr in air, cooled, and the sample boat was reweighed—first in argon and then on the analytical balance. The weight of the high-fired oxide was used in all the calculations. This was necessary since the starting material contained 4 to 60 wt% impurities.

The 1050 C oxide material was considered to be stoichiometric PuO_2 . It has been reported that 1180 or 1250 C^(12, 13) is necessary to form the stoichiometric dioxide. Since it is generally accepted that quartz holds a constant weight to approximately 1035 C and that devitrification occurs above this temperature, the system was usually heated no higher than 1050 C and an occasional 1200 C. No significant weight changes were observed when several test oxides were heated from 1050 to 1200 C.

To correct the instrument for buoyance, convection currents, and viscous drag, a thermogravimetric blank for the empty crucible containing a 200 mg quartz weight was determined exactly as the sample was to be run. Usually the blank correction was approximately 5%. With increasing temperature, the blanks consistently showed a weight loss.

APPLICATIONS

The following data illustrate the application of this instrument. The data shown are preliminary and are presented mainly to illustrate rather than to inform.

Effect of Temperature

A 10 g sample of specially-prepared PuO_2 that was produced at HAPO and which was especially reactive was used as a standard. Samples of this standard were chlorinated at 25 C° temperature intervals between 425 and 525 C (isothermal). The first order (with respect to PuO_2) character of the reaction is indicated by the straight lines obtained on plotting the log of the concentration of unreacted PuO_2 versus time as shown in Figure 7. Approximately the same rate of chlorination is achieved at temperatures from 450 to 525 C, with 500 C giving a slightly greater rate. The volatile material by actual weight was approximately 5.3%.

Figure 8 shows a plot of the logarithm of the reaction rate constants versus the reciprocal temperature in Kelvin. From the slope of this line, the energy of activation (E^*) was calculated to be 17.2 kcal.

Effect of Particle Size

A sample of relatively unreactive PuO_2 which had been calcined at 300 C for 14 hr was analyzed for particle size. Samples of the original material which contained 80 wt% 105 to 250 micron and 10 wt% 49 to 105 micron particles; a 49 to 105 micron fraction of the original material, and a less than 49 micron fraction were chlorinated. As shown in Figure 9, the smaller the particle size, the greater the reaction rate.

FIGURE 7

Effect of Temperature on the Chlorination Rate

FIGURE 8
Energy of Activation
(Arrhenius)

FIGURE 9
Effect of Particle Size on the Chlorination Rate

Effect of Calcination Time

A sample of PuO_2 was calcined at 300 C for a prolonged time and small aliquots were taken after 0.1, 2.0, 6.0, and 20 hr. These samples were chlorinated in the thermobalance at 500 C. The results, shown in Figure 10, indicate that a short calcination time is desirable.

FIGURE 10

Effect of Plutonium Dioxide Calcination Time (300 C)
on the Chlorination Rate

The reaction rate is decreased as the calcination time is increased. The longer exposure causes a gradual perfection in the highly distorted and impurity-containing PuO_2 lattice obtained by shorter calcination times.

Effect of Additives

PuO_2 prepared by direct calcination from the nitrate was found to chlorinate slowly and incompletely. It has been assumed that this low reactivity is due, at least in part, to the relative impermeability of the

PuO_2 particles. This low reactivity has also been attributed to the high degree of crystallinity and the lack of impurities in the oxide.

Activating additives were investigated in an effort to obtain more reactive direct calcination oxides. They would aid by causing an open structure whereby gas access to the center of the particle is increased and by inhibiting crystal growth and perfection. Both oxalic acid and ammonium sulfate were tried and it was found that the latter was a more satisfactory activating agent when used in massive quantities. Figure 11 shows the effect of sulfate and oxalate additions for several mole ratios of plutonium to additive. The additive has been shown to be quantitatively removed during the chlorination.

FIGURE 11

Effect of Additives on the Chlorination Rate

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