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on
THE EVALUATION OF URANIUM TETRACHLORIDE
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on

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THE EVALUATION OF URANIUM TETRACHLORIDE

Abstract

An apparatus has been devised which permits the determination of the volatility characteristics of a sample of uranium tetrachloride as related to temperature. The device provides a vertical sample chamber the temperature of which can be controlled, a horizontal tube the temperature of which can be maintained constant, and a cold chamber for the collection of the sublimate. Measurement of the rate of volatilization of the uranium tetrachloride is made by means of a pendulum suspended in the horizontal tube. The deflection of the pendulum is taken as a measure of the rate of volatilization.

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THE EVALUATION OF URANIUM TETRACHLORIDE

An instrument which may be used for the determination of the volatility characteristics of a sample of uranium tetrachloride as related to the temperature is described. The instrument is applicable to the problem of determining the effect of impurities (a) on the temperature at which volatilization of the sample begins and (b) on the variation in temperature necessary to produce a uniform rate of volatilization. Furthermore, the percent of non-volatile material in a given sample may be determined. It has been suggested that the instrument may also be of use in determining the nature of the impurities which may be in a given sample of uranium tetrachloride.

The time necessary for the complete volatilization of a sample of uranium tetrachloride (5-10 g) is about one hour. The time necessary for a run including loading, pumping, preheating and clean-up of the apparatus following the run is three to four hours.

Apparatus

Figure 1 shows the essential features of the apparatus. Of several different designs tried, the one shown gave the best results.

Volatilization Chamber. The volatilization chamber is constructed of 14 mm pyrex tubing and is about 15 cm in length. Temperature control of the chamber is provided by a small tubular heater. The heating element is wound on a glass tube covered with asbestos sheeting. Diametrically opposed longitudinal slits in the asbestos covering

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UCl₄ EVALUATION APPARATUS

Figure 1

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permit observation of the sample during volatilization. A chromel-alumel thermocouple is inserted between the heater and the volatilization chamber. A 110 v, 5 amp variac is used to control the voltage applied to the heater.

Horizontal Section. The horizontal section consists of pyrex tubing of the dimensions shown. It is surrounded entirely by a heater which consists of #23 Chromel-A wire wound over an asbestos-covered copper form. The heating element is covered by a layer of asbestos sheeting and a layer of asbestos cord. A small window, about two inches by three-fourths inch is cut in the heater to permit observation of the pendulum. A section of glass cut from 65 mm tubing is placed over the window to prevent cooling of that area and consequent condensation of the uranium tetrachloride on it. A chromel-alumel thermocouple placed between the heater and the pyrex tubing provides for temperature measurement.

Condensation Chamber. This chamber consists of a spherical pyrex flask three inches in diameter. The vacuum lead is from the top of the flask and the connection to the vacuum pump is by a 28/15 ball joint. A condensation chamber of this size permits several runs to be made before clean-up of the entire apparatus becomes necessary.

Pendulum. The pendulum stand is of pyrex glass of the dimensions shown. It is surrounded as shown by a heater constructed in the same manner as that for the horizontal section. A chromel-alumel thermocouple is inserted between the heater and the pendulum stand.

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Closure of the top of the stand is by a 34/25 ground joint. The pendulum bob is made of platinum 0.025 inches thick. The bob is suspended on two #40 platinum wires eleven inches long which are anchored above to an aluminum disc one-fourth inch thick. The pointer, a short piece of #22 platinum wire, is silver-soldered to the bob. After assembly of the bob, the wires were annealed by heating to redness for a short time.

Loading-Inlet Tube. The loading-inlet tube consists of a 10 cm length of 14 mm pyrex tubing. It is surrounded by a tubular heater made by winding #23 Chromel-A resistance wire over an asbestos-covered glass-tubing form. A chromel-alumel thermocouple is inserted between the heater and the loading tube. Sealed into the top of the loading-inlet tube is a piece of 4 mm pyrex rod. The rod is of such length as to extend about 6 cm into the sample chamber. On the lower end of the rod are five cone-shaped discs of aluminum spaced at intervals of 1 cm on the rod. This assembly serves as a baffle to prevent expulsion of the sample from the sample tube in case extreme outgassing of the sample occurs.

Scale. The scale consists of a piece of ruled milk glass two inches long and one-fourth inch wide supported in aluminum clamps. Each scale division is approximately 0.36 mm. A short section of a closely-ruled thermometer stem would serve quite well as a scale.

Vacuum System. A pressure of 10^{-4} to 10^{-5} mm Hg is maintained in the apparatus during a run by a liquid-air-trapped mercury diffusion pump with a Cenco Hivac as the fore-pump.

Auxiliary Apparatus. Temperature control on the various heaters

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is provided by variacs. A Leeds and Northrup potentiometer is used in connection with the thermocouples mentioned to determine the temperature in the various parts of the apparatus. A short-focal-length telescope is used to observe the motion of the pendulum.

Operational Details

For the transfer of a sample of tuballoy tetrachloride from a dry box to the apparatus, a loading tube 16 inches long and 8 mm in diameter is used. The sample is placed in the tube while in the dry box. A glass wool plug at each end of the column of sample protects it from moisture during the transfer to the apparatus.

The tip of the loading-inlet tube is broken off the apparatus and a stream of dried air is passed through the apparatus. The loading tube is inserted into the sample chamber and the sample is pushed out of the loading tube with a glass rod. After the loading tube is removed, the tip of the loading-inlet tube is resealed to the apparatus and the apparatus is evacuated. The temperature of the loading-inlet tube, the horizontal section and the pendulum stand is then raised to 500°C. The temperature of the sample tube is then controlled in accordance with the behavior of the sample.

After the volatilization of the sample has been completed, the tip of the sample chamber is removed and stoppered. The amount of unvolatilized residue is determined by the difference in weight of the sample-chamber tip before and after cleaning. The same tip may be resealed to the sample chamber. Clean-up of the entire apparatus,

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when necessary, is accomplished by filling it with 3 N nitric acid, draining, and rinsing twice with distilled water. The apparatus is then dried by being heated while a stream of air is passed through it.

Results

All of the test runs made in this laboratory were for the purpose of determining whether or not the results obtained were reproducible. Consequently, all runs were made with portions of the same uranium tetrachloride product, namely, Sample P-63. Six such runs have been made. For the first three, an aluminum pendulum bob was used and for the last three the platinum bob described in this report was used. In each case the data obtained included temperature, deflection of the pendulum, and time. For these runs an effort was made to control the temperature in such a range as to produce a uniform volatilization rate.

For each run, a graph was made in which temperature and deflection of the pendulum were plotted against time. One of the graphs is shown in Figure 2. The area under the curve for deflection plotted against time can be expressed mathematically as

$$\int_0^T M dt$$

where T is the total time during which deflection occurs and M is the reading of the pendulum in scale divisions. The units for the quantity so obtained would be scale divisions-minutes. If one divides this quantity by the grams volatilized during the run, the result is in scale divisions-minutes per gram. The latter value

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8 fig. 2

Figure 2
DEFLECTION, SCALE MARKS

TIME-TEMPERATURE-DEFLECTION DATA FOR RUN 9

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should be constant for the instrument regardless of the size of the sample used so long as the same pendulum is used and the angle of deflection of the pendulum is small. In practice, the area under the curve was determined by counting squares.

The results of treating the data obtained in the six runs by the method described above are shown in Table 1. The constancy of the value for scale-divisions per gram is taken as a measure of the reproducibility of the response of the pendulum to a given amount of uranium tetrachloride passing through the apparatus in a given time. It should be mentioned that for Run 8 the horizontal tube was connected to the condensation chamber by a ball joint. It was hoped that by using a Silicone lubricant in the joint no loss of vacuum would occur even though the temperature of the joint was quite high. However, the lubricant did soften during the run and a noticeable increase in pressure did occur. This may account for the difference between the value for scale divisions-minutes per gram for this run and those for the other two runs in which the same pendulum was used.

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Table 1

SUMMARY OF TEST RUNS

Run	Grams Sample	Grams Residue	Grams Volatilized	Scale Divisions Minutes per Gram	Pendulum
5	8.20	0.14	8.06	83.5	A1
6	6.48	0.26	6.22	81.9	A1
7	11.50	0.25	11.25	83.4	A1
8	6.93	0.12	6.81	84.3	Pt
9	9.59	0.13	9.46	112	Pt
10	12.43	0.11	12.32	118	Pt

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