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## An Apparatus for the Study of the Kinetics of Thermal Decompositions

R. B. Anderson  
*State University of Iowa*

H. H. Rowley  
*State University of Iowa*

R. F. Makens  
*State University of Iowa*

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## AN APPARATUS FOR THE STUDY OF THE KINETICS OF THERMAL DECOMPOSITIONS

R. B. ANDERSON, H. H. ROWLEY, AND R. F. MAKENS

For kinetic studies accurate control of the temperature of the reaction flask is required. The reaction flask was placed in a furnace which was constructed in a large metal container A (Fig. 1). Forty-four turns of #22 nichrome wire were wound about a 12" x 4 1/2" refractory cement core. To insure uniform distribution of heat, the flask was immersed in a bath of molten tin. The tin was contained in an iron crucible C constructed by welding a bottom on a large iron pipe. The temperature of the molten tin was determined with a chromel-alumel thermocouple E of twenty alternate hot and cold junctions. Each hot junction was enclosed in a Pyrex tube which was held in place by a metal clip G. A sturdy clamp H was required to hold the reaction flask in the molten tin. For temperature regulation of the furnace, the control of Zazel and Hancox, 1934, was used. Variations of the E. M. F. of the thermocouple produce deflections of a mirror galvanometer. This causes a beam of light from a spotlight to move off or onto a photo-electric cell producing phase variations which control a Thyatron tube. In this manner the current of the furnace circuit can be changed by 0.7 ampere, and a temperature control of  $\pm 0.3^{\circ}\text{C}$ . can be obtained in the range 300-400°C.

The reaction system was a variation of a general type for studying a decomposition involving gases which are noncorrosive to mercury as described by Thompson and Frewing, 1935. It consisted of a reaction flask R (Fig. 2) which communicates by capillary tubing to a capillary mercury manometer M, and to the remainder of the system by stopcock A. Since the compounds studied were liquids at room temperature, the capillary manometer, capillary tubing, and manifold were heated to 95°C. to prevent condensation in the tubing or stopcocks; this is indicated by sectioning in Fig. 2. For this purpose the tubing was wound with nichrome wire, and the stopcocks were provided with mercury seals to prevent leakage at this temperature.

The heated capillary manometer M was connected to an open end differential manometer D. The levels of the capillary manometer were equalized, and the pressure was read on the manometer D.

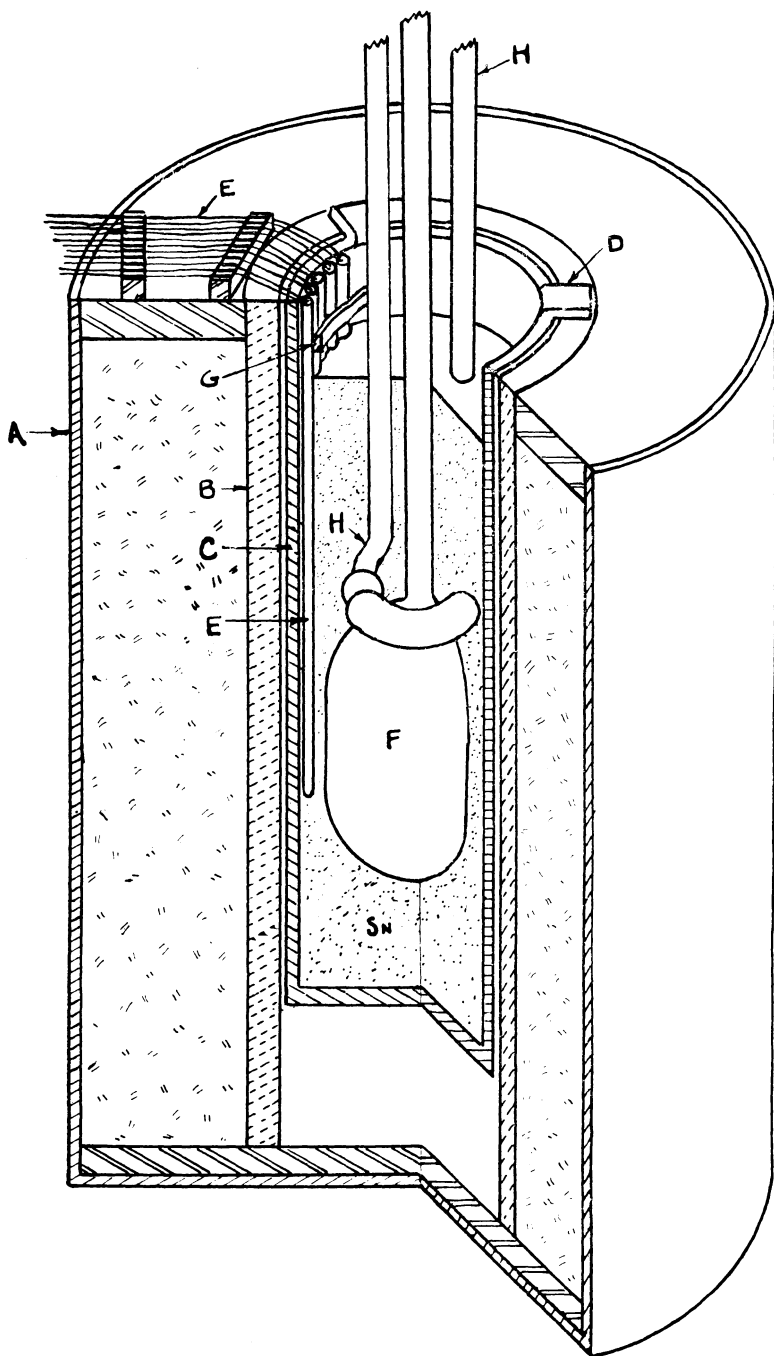


Fig. 1. The furnace and reaction flask

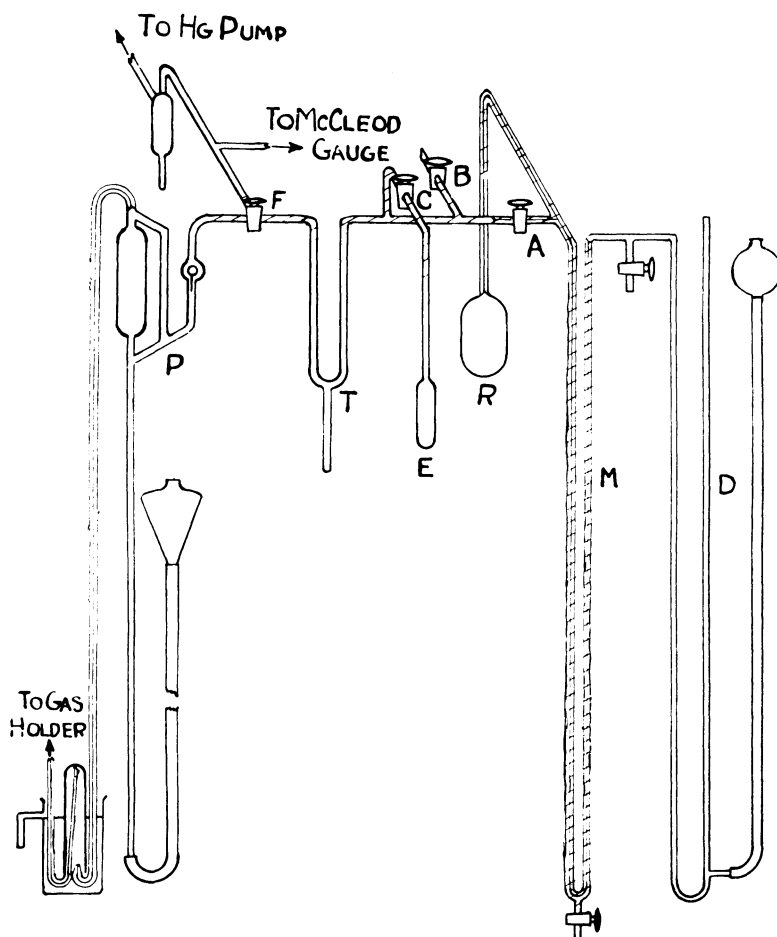


Fig. 2. The reaction system

For evacuation the system was provided with a large mercury diffusion pump backed by an oil pump. Bulb E, which contains the reactant was placed in an acetone-dry ice bath, and the system was evacuated with stopcocks C and A open until a pressure of less than 0.1 micron was attained. Stopcock F was turned to disconnect the part of the system to the right of this stopcock from the mercury and Toepler pumps. Bulb E and trap T were warmed, and the reactant was distilled into the reaction flask R to a desired pressure set upon the manometers. Stopcock A was immediately closed, and this was taken as zero time. The changes of pressure can be followed by the manometers.

The contents of the reaction flask may be removed at any desired time with the Toepler pump P, and transferred to a gas holder. Liquids and gases with high boiling points may be condensed in trap T by using suitable cooling mixtures.

STATE UNIVERSITY OF IOWA,  
IOWA CITY, IOWA

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