may be determined by displacing the block along the plane until the spring-balance force is large enough to slide the block backwards upon removal of the displacing force. A reading of the spring balance should be taken at this point. Another reading is taken after the displacing force is removed and the block has stopped sliding. (The maximum restoring force should not be so great as to give a minimum restoring force of less than zero.) The arithmetical mean of the two corrected spring-balance readings is equal to the magnitude of the average restoring force and hence will be equal to the magnitude of the sliding frictional force. In determining the coefficient of sliding friction where the change in the frictional force for a given change in normal force is used, it is not necessary to correct the spring-balance readings, since the corrections would subtract to zero.

This method may also be applied in studying the friction of a body moving on an inclined plane. In this case, the average spring-balance reading would include the component of the weight of the body along the plane.

Thermostated Cell Compartment for the Beckman Spectrophotometer

PAUL H. BELL and C. R. STRYKER

American Cyanamid Company, Stamford, Connecticut

The Beckman Spectrophotometer, as furnished by the manufacturer, is quite useful for the study of any rate process involving a spectral change. However, since most mechanism studies depend on quantitative reaction-rate measurements, thermostating of the reacting solutions in the instrument becomes essential.

Preliminary research on penicillin had shown that marked changes of ultraviolet absorption took place during its chemical degradation. These experiments indicated clearly that the acid degradation forming penillic acid from penicillin was quite complicated, with one or more conjugated intermediates existing in the solution during the reactions. Because of the large amount of effort being spent on determining the structure of penillic acid, a careful study of the mechanism of its formation from penicillin was made.[1] The temperature control necessary for this complex study was obtained by the thermostated cell compartment described below.

The construction and outward physical appearance of the Beckman Spectrophotometer should be familiar to anyone interested in this report, and therefore detailed description is not necessary. The compartment described is designed to replace the sample holders furnished with the instrument. The solvent balancing feature of the instrument requires that a solvent cell as well as the sample cell be moved into the light beam. This is accomplished by moving the thermostating jacket containing the cells back and forth inside the light-tight compartment which is rigidly attached to the spectrophotometer.

The actual compartment in a partly dismantled condition is shown in Fig. 1. In Fig. 2, scale drawings are shown, with an accompanying legend giving the essential details of the construction. The main frame, thermostating jacket, pipes, guides, cell holder, and screws are all of brass. (The most important of these are indicated by crosshatching.) Other parts, such as the inside and outside plates forming the dead-air spaces in top, bottom, and ends, plates on both sides, slide handle, and the light-tight sliding door in the top, are constructed from bakelite.

The optical system of the spectrophotometer uses a spherical mirror in an off-axis position to focus the monochromatic light on the exit slit. This arrangement gives a divergent exit beam, the dimensions of which are shown by (1) and (13). The position for the absorption cells was chosen where this beam was nearly square in shape, making it possible to use cells with the same sample thickness but completely blocking the beam with only 0.5–0.75 cc. of solution. The space occupied by the cells (8) is located at one side of the water jacket to keep as small as possible the distance between the cells and the photocell detector. This feature minimizes the errors due to light scattering from the cells and solutions.

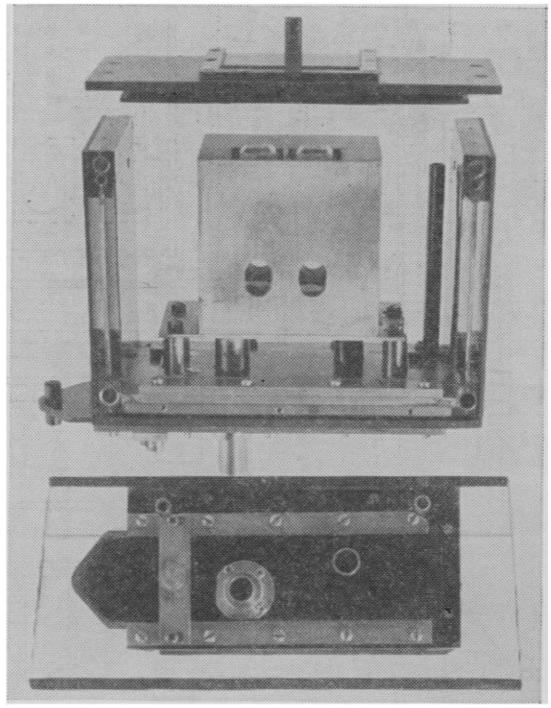

FIG. 1. Thermostated cell compartment with sides removed and the top in an exploded position, and a mirrored view of the bottom.

Since this equipment was designed to be useful several degrees above or below room temperature, certain insulating features were necessary. By the use of multiple walls, the dead-air spaces (5) were created. Also, the compartment was insulated from the spectrophotometer and the phototube compartment by bakelite plates with only small openings for the light beam. If the cell compartment was being maintained at a temperature much below that of the room, the problem of frosting of the absorption cell windows had to be overcome. To do this, space was allowed for desiccant, and drying gas

[1] The results of this work were reported at the Atlantic City meeting of the American Chemical Society, April 8-12, 1946, and will be published shortly.

ports (4) were provided. In order to keep the compartment from taking in damp air when the cells were being withdrawn or inserted through (6), the clearance between the movable water jacket and the top was kept as small as possible.

The water jacket, inside sliding plate (14), and the sliding handle (10) are held together by the liquid tubes (2 and 3).

Using thermocouples, immersed in the liquid contained in the absorption cells (4 cc. each), temperature measurements were made to test the efficiency of thermostating. The thermostating liquid was supplied by an American Instrument Company Refrigerated Constant Temperature Bath and passed through the compartment at a rate of 1 gallon/minute.

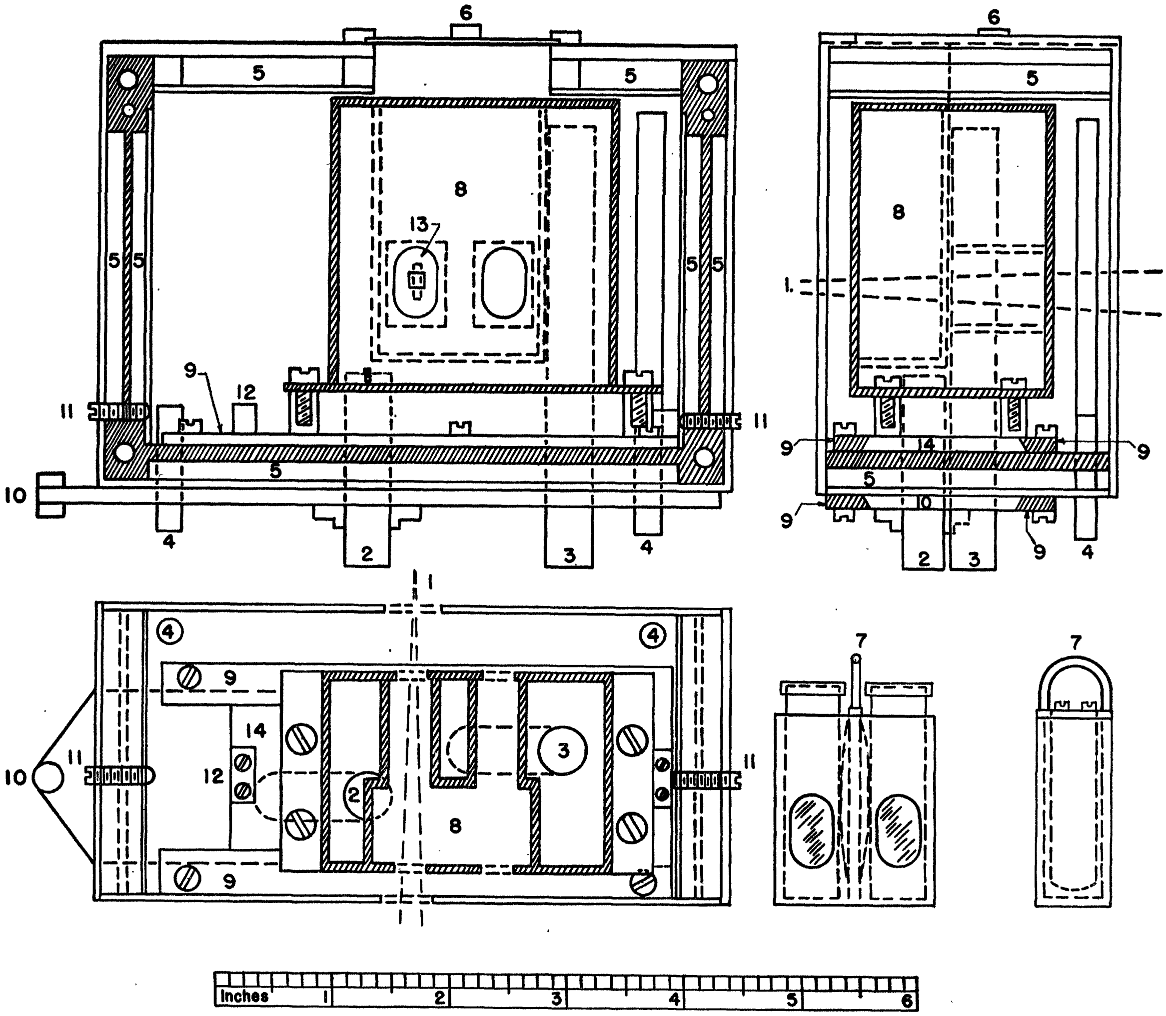

FIG. 2. Scale drawing of thermostated cell compartment.

(1) Light path from spectrophotometer
(2) Thermostating liquid inlet
(3) Thermostating liquid exit
(4) Drying gas ports
(5) Insulating dead-air spaces
(6) Sliding light-tight cover
(7) Cells and cell holder
(8) Space for cell holder in thermostat
(9) Guides for the movable plates carrying the thermostat
(10) Sliding handle
(11) Adjustment screws for limiting horizontal motion
(12) Bumper for adjustment screw
(13) Position and shape of light beam
(14) Sliding supporting plate

This unit can move within the guides (9), since openings are provided in the brass bottom and the bakelite plate forming the bottom air space, to accommodate the movement of the tubes (2 and 3). The supporting plate (14) and the sliding handle plate (10) cover these slots at all times, thereby maintaining the dead-air space (5) and keeping the entire compartment light tight.

Starting with the solutions at room temperature, approximately 10 minutes was necessary to obtain thermal equilibrium. At the highest temperature tested (76°C.), less than a 0.5°C. drop was observed between the bath and solutions in the thermostating compartment. This thermostating arrangement has been tested and used from 5°C. to 76°C. and found to give very satisfactory temperature control.