

from a chloroform solution with petroleum ether. The product thus obtained decomposed at about 278°. The nitrogen determinations, which were made on different preparations, were between 0.5 per cent. and 1 per cent. below the theoretical in each case.

Calculated for $C_{12}H_{18}O_4N_6$: C, 46.45; H, 5.85; N, 27.10.
Found: C, 46.22; H, 5.90; N, 26.36, 26.16, 26.60.

1-Azo-5-dimethyl-3-phenylhydantoin was prepared and purified similarly to the foregoing tetrazone. It decomposes to a brown liquid at 270° with gas evolution. The product analyzed had the external appearance of purity, but the analytical data do not conform closely to the formula given this substance.

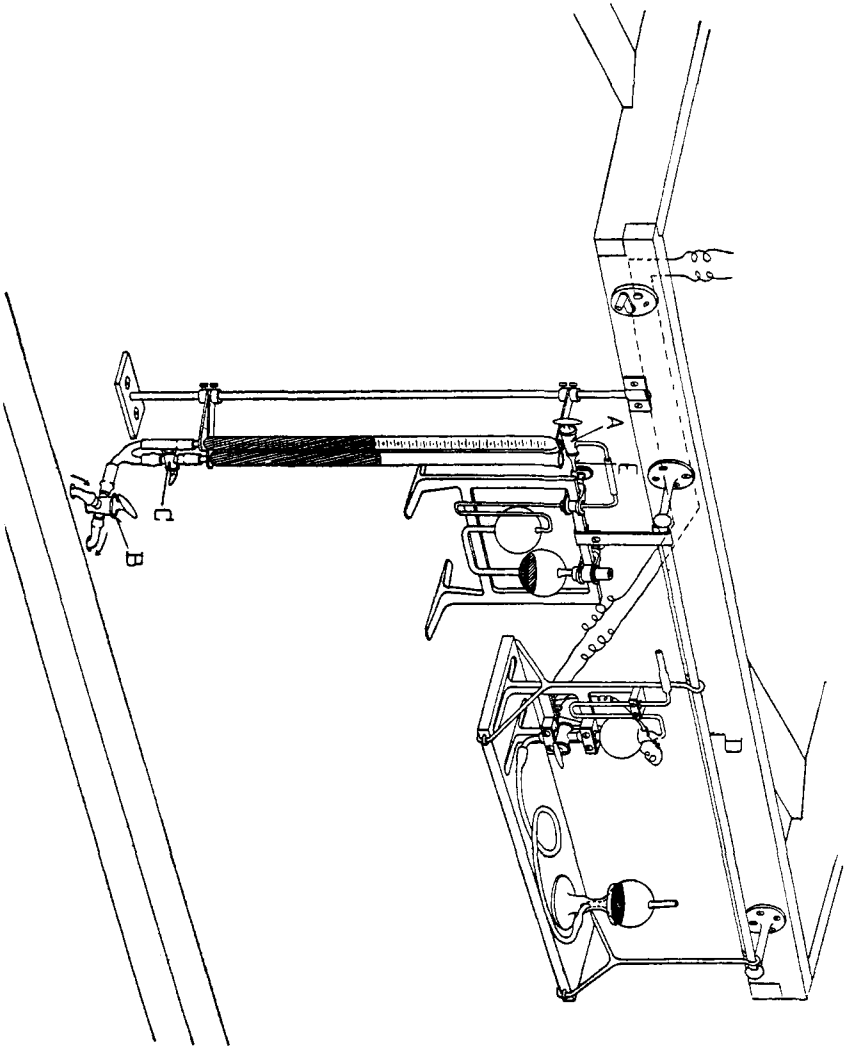
Calculated for $C_{22}H_{22}O_4N_6$: C, 60.83; H, 5.07; N, 19.35.
Found: C, 61.85; H, 5.26; N, 18.71, 18.90.

NOTES.

Stationary Hempel Apparatus.—The apparatus shown in the accompanying sketch was devised by Mr. W. J. Knox, of Pittsburg, and has been in constant use for a number of years with no radical modifications.

To a horizontal frame, near to and parallel to the edge of the working-bench, is attached a horizontal rod, D, about $\frac{5}{8}$ inch in diameter, on which the pipettes may be suspended from appropriate hooks attached to the frame of the pipette, while to a vertical rod, secured to frame and bench, are fastened the burette and leveling tube in such a manner as to permit free lateral motion. The prolong of the burette is bent at right angles to the burette and parallel to the graduations. The capillary tubes of the pipettes are also bent to 90°, so that only a single rubber connection is necessary to attach the pipette to the burette. At the top of the burette is a 3-way cock, A, the second outlet, E, being the stem of the cock. At the bottom of the leveling tube is a cock, C. Burette and leveling tube are connected by a Y-tube to one branch of a 3-way cock, B, the other branches of which are connected, respectively, to an overhead reservoir and drain.

The *modus operandi* is as follows: With C closed, open A to burette and fill burette with water by opening B to supply. Close A and attach sample bottle to prolong of burette. Pass enough



gas through E to insure complete elimination of air. Open A to burette and draw in sample by opening B to drain. To obtain the volume at atmospheric pressure: With A closed, open C and, by admitting or withdrawing water, by means of B, the level of burette and leveling tube is easily obtained.

With A still closed, attach the appropriate pipette to the prolong of burette; open A to burette and force gas into pipette by filling burette with water by means of B. When absorption is complete, return gas to burette by opening B to drain; then level, as described above. If it is desired to shake the pipette, a swinging motion may be given to it without disconnecting from the burette. The rod D is long enough so that the hanging shelf, on which the explosion pipette and mercury reservoir are placed, may be thrown back out of the way when not in use. The ordinary absorbents are used.

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Note on the Standardization of Iodine Solutions.—During the course of some extensive investigations upon sodium thiosulphate, the thought suggested itself that very possibly the anhydrous salt might be readily prepared in a state of sufficient purity to serve as a reagent for the standardization of the iodine solutions used in the work. This has been found to be the case, and a very simple and practical, as well as exact, method has been devised for accomplishing this.

The anhydrous thiosulphate is best prepared by recrystallizing the chemically pure salt of commerce from warm solutions (saturated at from 30° to 35° C.) by cooling and constant stirring. The salt thus obtained is of about the grain of granulated sugar, and is to be dried on filter-paper at room temperature. It is then dehydrated over sulphuric acid until it has fallen to a powder, and a portion in a test-tube shows no sign of fusion when heated to 50°. The final dehydration is carried on in an air-bath at about 80°, with repeated stirring of the powder. For relatively small quantities two hours' heating will usually be sufficient. The salt is then to be placed in a tight-stoppered weighing-bottle, from which samples are taken as desired.

Following are some results obtained by the use of this salt. The method was simply to take a weighed sample, dissolve in water and titrate with the iodine solution to the appearance of the