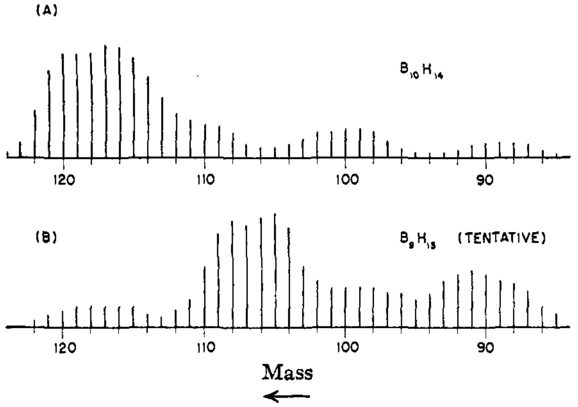

Fig. 1.

Wiberg[2] has proposed the type formula B_nH_{n+4} which includes the more stable known hydrides: B_2H_6, B_5H_9, B_6H_{10} and $B_{10}H_{14}$. The less stable B_4H_{10} and B_5H_{11} belong to the series B_nH_{n+6}. The composition B_9H_{13} fits into the more stable series formula. However, the composition B_9H_{15} is not excluded by the mass spectrum observed.

The group of peaks in Fig. 1(B) centering about masses 105 to 108 is obviously from some new material in addition to the $B_{10}H_{14}$ present. The position and distribution of these new peaks are not inconsistent with the spectrum which such a material as B_9H_{13} would be apt to furnish. Peaks of double ionization also were observed in the region 50–53, which indicates resemblance to $B_{10}H_{14}$ in its ionization characteristics, and the volatility of these two materials is of the same order.

Higher boron hydrides (above $B_{10}H_{14}$) were sought up to mass 400 but none was found of sufficient volatility to enter the mass spectrometer.

[2] E. Wiberg, *Ber.*, **69B**, 2816 (1936).

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Redetermination of the Heat of Combustion of Diphenylmethane

BY GEORGE S. PARKS AND JOHN R. MOSLEY

Parks and co-workers[1] reported 9822.2 cal./g. for the heat of combustion of diphenylmethane under the conditions of their calorimetric bomb process. They ascribed their value to the liquid state, as they had been able to undercool the liquid, in a test-tube, several degrees below 25.1°, m. p. of crystalline diphenylmethane. Recently, however, we have tried to burn undercooled liquid cyclohexanol (m. p. 25.2°) in our bomb in an analogous fashion, but this material, although liquid at 23° in a small glass bulb exposed to atmospheric pressure, invariably crystallized within the bomb on the imposition of 30 atm. pressure at 24°. Such an experience led to doubt concerning the state of diphenylmethane in the previous work and accordingly we have now made a redetermination of the heat of combustion under unambiguous conditions.

Our material was an Eastman sample which was further purified by nine fractional crystallizations to a final m. p. of 25.1°. It was utilized in the present study immediately after preparation, whereas the sample employed by Parks and co-workers had stood in a bottle of transparent glass for almost two months prior to their combustions.

In our study three combustion determinations were first made upon crystalline diphenylmethane, obtained by cooling to 0°. The resulting mean was 9799.9 ± 1.0 cal./g. for the bomb process. Then three crystallization tests, all negative, were made by exposing liquid samples at 23° to a pressure of more than 30 atm. within the bomb. Thereupon we proceeded to combustion measurements on this liquid with a mean result of 9825.8 ± 1.1 cal./g. from five determinations.

These data demonstrate that Parks and co-workers were previously dealing with liquid diphenylmethane. However, their material had probably deteriorated with time in the manner described by De Vries and Strow[2] and thus produced a result about 0.04% lower than our present one.

Our new, and more reliable, value yields $\Delta H_R = -1653.83 \pm 0.32$ kcal. for the molal heat of combustion of the liquid at 25° and 1 atm. constant pressure. The corresponding heat of formation from the elements is $\Delta H_f^0 = 21.25$ kcal.

[1] G. S. Parks, T. J. West, B. F. Naylor, P. S. Fujii and L. A. McClaine, THIS JOURNAL, **68**, 2524 (1946).

[2] T. De Vries and H. A. Strow, THIS JOURNAL, **61**, 1796 (1939).

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Catalytic Hydrogenation of Some 2- and 4-Stilbazole Methiodides

BY ARTHUR P. PHILLIPS

Recently the author has reported the preparation of a series of 2- and 4-stilbazole methiodides.[1] It was considered that powerful pharmacological activities could reasonably be anticipated in the stilbazoline hydrogenation products of these stilbazoles by virtue of their relationship to such alkaloidal types as coniine, lobeline and laudanosine. Spasmolytic, analgesic or curare-like activities were specifically sought. This paper deals solely with the chemical aspects of these substances.

The desired compounds, salts of 1-methyl-2- or 4-substituted phenethylpiperidines, were readily obtained by the catalytic hydrogenation of the corresponding 2- or 4-stilbazole methiodides using Adams catalyst in methanol solution. In general

[1] Phillips, *J. Org. Chem.*, **12**, 333 (1947); **14**, 302 (1949).