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Note

THE THERMAL LIGHT EMISSION OF $NH_3 \cdot BH_3$

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The thermal decomposition of ammonia-borane, $NH_3 \cdot BH_3$, in a nitrogen or low-pressure atmosphere, results in the release of hydrogen and the formation of a mixture of products including poly(aminoborane), $(NH_2BH_2)_x$, borazine and other substances [1–3]. DTA studies of this compound [1,2] showed that the thermal dissociation reaction begins as an endothermic process which then becomes highly exothermic. During these processes, a voluminous, porous white solid is formed as a result of at least four distinct reactions [1].

We have recently observed that during the thermal decomposition reaction of $NH_3 \cdot BH_3$ in an oxygen atmosphere, a very unusual light emission was detected by the technique of emission thermophotometry (ETP) [4]. This report is concerned with the details of this phenomenon.

EXPERIMENTAL

The ETP apparatus used has been previously described [4]. This apparatus consists of a photomultiplier tube (PMT), a photometer, a modified DSC cell mounted on an Omnitherm controller and a microcomputer data collection and display system. The procedure employed was essentially identical to that previously used: sample size, 4–5 mg; dynamic oxygen furnace atmosphere at a flow rate of 40 ml min^{-1}; furnace heating rate, 20°C min^{-1}; photometer sensitivity, 30. Other samples were studied under identical conditions except that nitrogen was substituted for the oxygen as the furnace atmosphere.

The $NH_3 \cdot BH_3$ was obtained from the Callery Chemical Company and sublimed until a sample held in a closed capillary tube began to melt at not less than 115°C when determined on a Melt Temp heated block set at 40 V. *Caution*: melting point capillary tubes should be sealed with a plug of modeling clay or wax; those sealed with a torch may shatter under the pressure developed during the thermal decomposition reaction.

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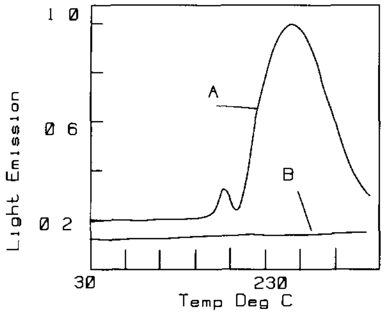

Fig. 1. ETP curves of $NH_3 \cdot BH_3$ in (A) oxygen and (B) nitrogen atmospheres.

RESULTS AND DISCUSSION

The ETP curves of $NH_3 \cdot BH_3$ in oxygen and nitrogen atmospheres are illustrated in Fig. 1. It is evident from the curves that essentially no light is detected with a nitrogen atmosphere over the sample (Fig. 1B) whereas copius light emission results when an oxygen atmosphere is present (Fig. 1A). The fact that light emission first appears at 180–190°C suggests that it is not $NH_3 \cdot BH_3$ itself, but rather one or more of its thermal decomposition products which is responsible for the light emission since $NH_3 \cdot BH_3$ decomposition begins near 120°C [1,2]. We believe that in the oxygen atmosphere an oxidation reaction of some decomposition product(s) occurs emitting the observed light.

Boranes, in general, have very exothermic heats of combustion [5] and pentaborane(9), one of the likely decomposition products, can inflame spontaneously upon contact with oxygen (eqn. 1). Mixtures of B_5H_9 and O_2 below the explosive ratio are reported to undergo a luminous reaction depositing a white solid on the container walls [6]:

$$2B_5H_9(g) + 12O_2(g) \rightarrow 5B_2O_3(s) + 9H_2O(g) \qquad (1)$$

Thus it seems reasonable to suppose that the observed thermal light emission from $NH_3 \cdot BH_3$ has its origin in oxidation reaction(s) involving the B–H bonds of one or more of its decomposition products.

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