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Chemistry

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structural formulas I-IV

Figure 1: structural formulas I-IV

Abstract**Full Text***Chemistry*

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ON THE INTERACTION OF NAPHTHALENE WITH SULFUR

In 1911 M. M. Langry, passing vapors of naphthalene and sulfur through a red-hot iron tube, obtained a small amount of a ruby-red substance of composition $C_{10}H_6S_2$, m.p. 118.5° . On the basis of qualitative reactions and the empirical formula he assigned to this compound the structure of thienothiophthene I or II (¹). Later the opinion was expressed (²) that this substance should be assigned the structure of thienothionaphthene III.

However, in 1957 Indian investigators (³), who synthesized compounds I, II, and III by a method leaving no doubt as to their structures, established that none of them can be identical with the substance formed in the interaction of naphthalene with sulfur. Thus, the question of the structure of the interesting product obtained by M. M. Langry has remained open up to the present time. In reviewing the literature we noted that 1,8-naphthylenedisulfide (IV), synthesized in 1928 by S. Smiles and V. B. Price (⁴), having the same empirical formula $C_{10}H_6S_2$, is extremely close in physical properties (a red substance with m.p. 116° , and according to data (⁵) 119°) to the substance isolated by M. M. Langry.

Since the formation of 1,8-naphthylenedisulfide in the interaction of naphthalene with sulfur seemed to us more probable than the formation of thienothionaphthene, we repeated M. M. Langry's work in order to compare these two compounds directly. It should be noted that M. M. Langry, in his article, gives no experimental details (the ratio of the starting naphthalene and sulfur, the reaction temperature, and the method of isolating the substance from the resulting mixture). We passed vapors of naphthalene and sulfur in a ratio of 1 : 3 through a red-hot iron tube ($600-700^\circ$). As a result, a condensate was obtained containing a large amount of unchanged naphthalene, which was removed by steam distillation. From the residue, by repeated crystallization from alcohol and subsequent sublimation in vacuum (55° at 2 mm Hg), the expected red crystalline product with m.p. $116-117^\circ$ was obtained. 1,8-Naphthylenedisulfide was synthesized by us by the method described in the literature (⁴⁻⁶), starting from

Fig. 1. IR absorption spectrum of the substance obtained according to M. M. Lanfry (A), and of 1,8-naphthylenedisulfide (B)

Figure 2: Fig. 1. IR absorption spectrum of the substance obtained according to M. M. Lanfry (A), and of 1,8-naphthylenedisulfide (B)

Fig. 2. UV absorption spectrum of the substance obtained according to M. M. Lanfry (A), and of 1,8-naphthylenedisulfide (B)

Figure 3: Fig. 2. UV absorption spectrum of the substance obtained according to M. M. Lanfry (A), and of 1,8-naphthylenedisulfide (B)

1,8-naphthylaminesulfonic acid, and had m.p. 118—119°. Comparison of these two compounds showed that they do not give a depression of the melting point of a mixed sample, and their infrared and ultraviolet absorption spectra (see Figs. 1 and 2) are completely identical.

Thus, it may be considered established that, when vapors of naphthalene and sulfur are passed through a red-hot iron tube, 1,8-naphthylenedisulfide is formed.

An interesting property of 1,8-naphthylenedisulfide is its ability to give distinct positive reactions for a thiophene ring (the indophenine reaction with isatin and the Liebermann test with phenanthrenequinone), which led M. M. Lanfry to an erroneous conclusion concerning the structure of the compound he had obtained.

Fig. 1. IR absorption spectrum of the substance obtained according to M. M. Lanfry (A), and of 1,8-naphthylenedisulfide (B)

Fig. 2. UV absorption spectrum of the substance obtained according to M. M. Lanfry (A), and of 1,8-naphthylenedisulfide (B)

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