N-PHENYLISONAPHTHALIMIDE.

STRUCTURE OF THE PRODUCT OF THE REACTION OF 1,8-NAPHTHALOYL CHLORIDE WITH ANILINE

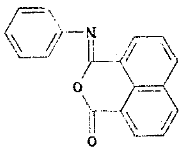
É. V. Ganin, V. F. Makarov, and
V. I. Nikitin

UDC 547.657'836'816.07

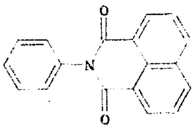
It is shown that N-phenylisonaphthalimide rather than N-phenylnaphthalimide, as previously assumed, is formed in the reaction of 1,8-naphthaloyl chloride with aniline.

According to the data in [1], a yellow compound, for which the N-phenylnaphthalimide structure (I) was proposed, is formed in the reaction of 1,8-naphthaloyl chloride with aniline. Considering the data on the products of the reaction of phthaloyl chloride with amines that we previously obtained [2], one may possibly also expect the formation of N-phenylisonaphthalimide (II) in the case under consideration.

II



I



In order to ascertain the true structure of the product of the reaction of 1,8-naphthaloyl chloride with aniline we reproduced the experiment in [1] and also obtained imide I by alternative synthesis from naphthalic anhydride and aniline by the method in [3]. The substances obtained by the methods in [1] and [3] have identical empirical formulas corresponding to structures I and II but different physicochemical characteristics. Thus the compound obtained in accordance with [3] was a white substance with mp 201-202°C, and two intense absorption bands at 1705 and 1665 cm^{-1} are present in the IR spectrum. The substances synthesized by the method in [1] was yellow and had mp 162-163°C, and its IR spectrum contained two intense absorption bands at 1770 and 1680 cm^{-1}. In accordance with the general rule [4], the shift of the absorption bands in the IR spectrum of this compound to the shorter-wave region makes it possible to assign it to isoimide structure II and to assign the compound obtained by the method in [3] to structure I. In analogy with N-phenylisophthalimide and N-phenylphthalimide [5], the difference in the melting points and colors of the compounds under examination is in agreement with the proposed assignment. The isomerization of the compound synthesized in accordance with [1] to a compound identical to that formed by the method in [3], i.e., the isomerization of isonaphthalimide II to naphthalimide I, additionally confirms the correctness of the assignment of N-phenylisonaphthalimide structure II to the compound obtained by the method in [1], since similar isomerization is observed for N-phenylisophthalimide [4].

EXPERIMENTAL

The IR spectra of KBr pellets of the compounds were recorded with a IKS-29 spectrometer. The mass spectra were obtained with an MAT-112 spectrometer at an ionizing voltage of 70 eV with direct introduction of the samples into the source. Thin-layer chromatography (TLC) was carried out on Silufol UV-254 plates with elution by benzene, chloroform, and acetone–hexane (1:2-1:5) and development in UV light (imide I luminesces, whereas isoimide II extinguishes) and by means of ninhydrin (imide I does not give any color, whereas isoimide II gives red spots).

1,8-Naphthaloyl chloride was obtained by the method in [1].

All-Union Scientific-Research and Design Institute of Monomers, Tula 300600. I. I. Mechnikov Odessa State University, Odessa 270000. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 8, pp. 1114-1115, August, 1985. Original article submitted July 13, 1984.

0009-3122/85/2108-0932$09.50

N-Phenylnaphthalimide (I). This compound was obtained in 78% yield by the method in [3] with subsequent crystallization from methanol. The white crystals had mp 201-202°C. Found: N 5.2%. $C_{18}H_{11}NO_2$. Calculated: N 5.1%. IR spectrum: 1705, 1665 cm^{-1} (imide C=O, symmetrical and asymmetrical). Mass spectrum: M^+ 273.

N-Phenylisonaphthalimide (II). This compound was obtained in 73% yield by the method in [1]. The yellow crystals had mp 162-163°C. Found: N 5.2%. $C_{18}H_{11}NO_2$. Calculated: N 5.1%. IR spectrum: 1770 (C=O), 1680 cm^{-1} (C=N). Mass spectrum: M^+ 273. Isonaphthalimide II was thermally stable and sublimed without isomerization upon heating to 500°C.

Isomerization of N-Phenylisonaphthalimide (II) to N-Phenylnaphthalimide (I). A 0.2-g sample of isomide II was dissolved in 5 ml of benzene, and 3 ml of triethylamine was added. After 1.5 months, 1.8 g of imide I, which was identical to a genuine sample obtained by the method in [3], precipitated.

LITERATURE CITED

1. F. L. Mason, J. Chem. Soc., 125, 2116 (1924).
2. É. V. Ganin, V. F. Anikin, B. V. Rozynov, V. F. Makarov, and G. L. Kamalov, Khim. Geterotsikl. Soedin., No. 9, 1280 (1984).
3. B. A. Porai-Koshits, Zh. Obshch. Khim., 7, 604 (1937).
4. M. K. Hargreaves, J. G. Pritchard, and H. R. Dave, Chem. Rev., 70, 439 (1970).
5. M. L. Sherrill, F. L. Shaeffer, and E. P. Shover, J. Am. Chem. Soc. 50, 474 (1928).

CYCLIZATIONS OF N-ALKYLAZINIUM CATIONS WITH BIFUNCTIONAL NUCLEOPHILES.

18.* SYNTHESIS AND STRUCTURE OF HETEROCYCLIC SYSTEMS BASED ON QUINOXALINE

V. N. Charushin, M. G. Ponizovskii, O. N. Chupakhin,
A. I. Chernyshev, N. A. Klyuev, T. I. Malinovskii,
V. Kh. Kravtsov, and V. N. Biyushkin

UDC 547.863.1'7.04:543.422.25

N-Methylquinoxalinium iodide reacts with ethylenediamine, o-phenylenediamine, and o-aminophenol to give cyclization products of the type involving [2,3-b] annelation of the six-membered heteroring. Two molecules of the quinoxaline participate in the cyclization with 2-aminoethanol to give a complex polycyclic compound.

One-step methods for the synthesis of heterocyclic compounds based on the cyclizations of azines with bifunctional nucleophiles have undergone significant development in recent years [2, 3]. They have found greatest application in series of azine derivatives containing a pyrazine ring, the pronounced capacity of which for diaddition or disubstitution reactions serves as the basis for various sorts of cyclizations with dinucleophiles. The development of research in this field has also been stimulated by the fact that substances with various forms of biological activity have been discovered among pyrazine and quinoxaline derivatives condensed with five- and six-membered heterorings [2, 4-6].

We have previously studied the cyclizations of pyrazinium salts with 1,3-dinucleophiles, which lead to substances with a hydrogenated pyrazine ring fused with five-membered heterorings [3]. In the present research we studied the peculiarities of the annelation of six-membered heterorings to quinoxalines. With this in kind, we investigated the reactions of

---

*See [1] for communication 17.

S. M. Kirov Ural Polytechnic Institute, Sverdlovsk 620002. Institute of Applied Physics, Academy of Sciences of the Moldavian SSR, Kishinev 277028. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 8, pp. 1116-1124, August, 1985. Original article submitted July 4, 1984.

0009-3122/85/2108-0933$09.50