INTERNATIONAL MULTIDISCIPLINARY JOURNAL FOR RESEARCH & DEVELOPMENT
SJIF 2019: 5.222 2020: 5.552 2021: 5.637 2022:5.479 2023:6.563
eISSN 2394-6334 https://www.ijmrd.in/index.php/imjrd Volume 10, issue 11 (2023)

METHOD OF PREPARATION OF N-VINYL BENZOTRIAZOLE

Karimova Sidora Bakhtyorovna,

Masharipova Azizakhan,

Otanazarov Behruzbek,

Torakulov Avazbek,

Vapoev Israel

Jizzakh Polytechnic Institute

Annotation: The cited article provides information on vinylation of benzotriazole, one of the organic compounds, in a homogeneous system and finding alternative conditions.

Key words: Benzotriazol, homogeneous system, vinyl group, heterocyclic compounds, N-vinylbenzotriazole, high-base system.

Nitrogenous organic compounds are important substances and have a wide range of applications, as biologically active substances such as defoliants, plant growth regulators, medicinal preparations, antihypertensives, etc.

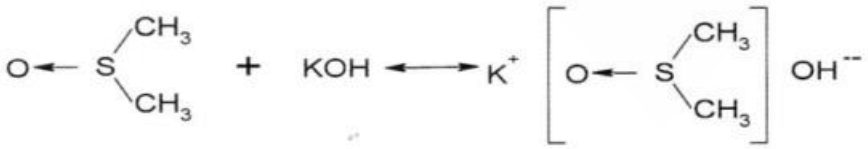
On the basis of heterocyclic compounds, including nitrogen-containing heterocyclic compounds, it is possible to perform various chemical changes and synthesis of various derivatives at the expense of an electron-donating nitrogen atom or at the expense of active hydrogen in cases where the nitrogen atom contains a hydrogen atom.

Benzotriazole, one of the important representatives of triazoles, has been studied to some extent in the literature. Its complex compounds, acids and other derivatives were obtained, but the N-vinyl derivative was not synthesized. For this reason, studying and scientifically analyzing the benzotriazole vinylation reaction is one of the urgent tasks.

Corresponding N-vinylamines are obtained with sufficiently high yields as a result of vinylation reactions of nitrogen-containing organic substances with acetylene compounds at high pressure. It should be noted that rare, expensive equipment is required to carry out such processes. Due to the fact that acetylene and its derivatives have explosive and flammable properties at high pressure, it is also necessary to strictly observe the safety of equipment.

Finding alternative conditions for vinylation of a selected substance in a homogeneous way, studying factors such as the nature of the catalyst and the effect of temperature on the vinylation process are important in explaining and developing the laws of catalytic homogeneous reactions in modern organic chemistry.

In addition, most of the substances used in the process are easily available. In particular, acetylene, which is used as a vinylizing agent, is considered an industrial product and is produced at the Navoi-Azot plant. It is planned to further increase its annual production in the future.



It is known that substances containing active hydrogen atoms, including primary and secondary amino compounds, are used for the synthesis of N-vinyl compounds. Based on these, catalytic vinylation reactions of pyridine, piperidine, morpholines in the presence of acetylene at atmospheric pressure were systematically studied.

The effect of factors such as the nature of the solvent, duration of the reaction, the amount of catalyst-KON and temperature on the progress of the reaction was studied and alternative conditions of the process were found; the amount of catalyst-KOH is 15% (in relation to the mass of pyridine), the solvent is DMSO, the temperature in the homogeneous method is 90 °C, and in the heterogeneous method it is 180 °C. The yield of 3-vinylpyridine was 35% and 45.1%, respectively.

Benzotriazole - S6N5N3 is a brown needle-like crystalline substance with a white base. Molecular mass is 119.12. Liquid = 97-98.5°C, boiling temperature 350°C, self-ignition at 210°C. Density 1.36 g/cm3, soluble in alcohol, benzene, DMFA, DMSO and toluene, practically insoluble in water. Purity 99.5-99.8%.

In the synthesis of N-vinylbenztriazole, benzotriazole was used as a starting material, and acetylene was used as a vinylizing agent.

Potassium hydroxide, sodium hydroxide, lithium hydroxide as a catalyst.

Benzene, DMFA and DMSO were used as solvents.

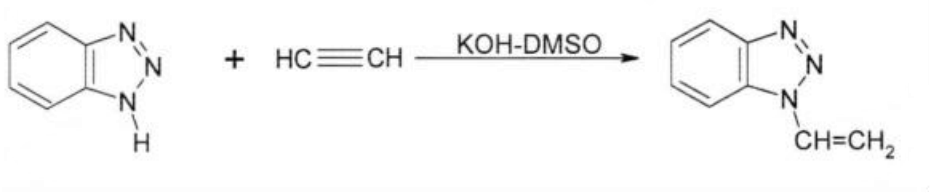
Another such substance is finding alternative conditions for the homogeneous vinylization process of benzotriazole, studying factors such as the nature of the catalyst and the effect of temperature on the vinylation process are important in explaining and developing the laws of catalytic homogeneous reactions in current organic chemistry.

For the synthesis of N-Vinylbenzotriazole, 300 ml of DMSO, 3.33 g of KON, and 0.2 g of hydroquinone were placed in a flask equipped with a three-necked flask, a thermometer, a mechanical stirrer, and an acetylene transfer nozzle. The resulting product was heated at 80 °C, then cooled, and then 35.7 g of benzotriazole was added. The reaction temperature increased to 80°C. Acetylene was added under constant stirring for 5 hours. After two hours, the reaction was stopped and after cooling, the reaction mass was extracted with ether and dried over sodium sulfate. Then the mixture with the presence of an inhibitor - hydroquinone is distilled and a fraction with a boiling temperature of 200-222 oC is obtained for use.

In this work, the mixture was then cooled to room temperature, neutralized with 5% sulfuric acid solution, and extracted with benzene (25 mL until the solution became colorless). The extract was dried over calcium chloride overnight, and the solvent was removed. The resulting product was driven under vacuum. N-vinylbenzotriazole is a thick oily substance, boiling point 10-13 mm s.u. 200-222°C under vacuum conditions. vinylation reaction of benzotriazole with acetylene at atmospheric pressure was carried out, it was found that N-vinylbenzotriazole was

formed in the process. The effect of various factors on the reaction progress and product yield was studied. The process was carried out in a homogeneous way.

The reaction goes according to the following scheme:



The obtained experimental results show that the maximum yield of the synthesized product was equal to 42.6% when the catalyst - KON - 10%, temperature - 120-1250C, time duration - 5 hours in DMSO medium. The purity and structure of the synthesized N-vinylbenzotriazole was determined using physicochemical research methods.

The purity of the starting materials and the obtained products, the composition of the compounds formed during the reaction was determined by gas-liquid chromatographic method. A gas-liquid chromatogram of the catalyst formed in the vinylation of benzotriazole with acetylene is presented. It can be seen from the picture that the catalyst (1) contains unreacted N-vinylbenzotriazole, benzotriazole (2), solvent DMSO (3) and a small amount of a substance whose structure has not been studied.

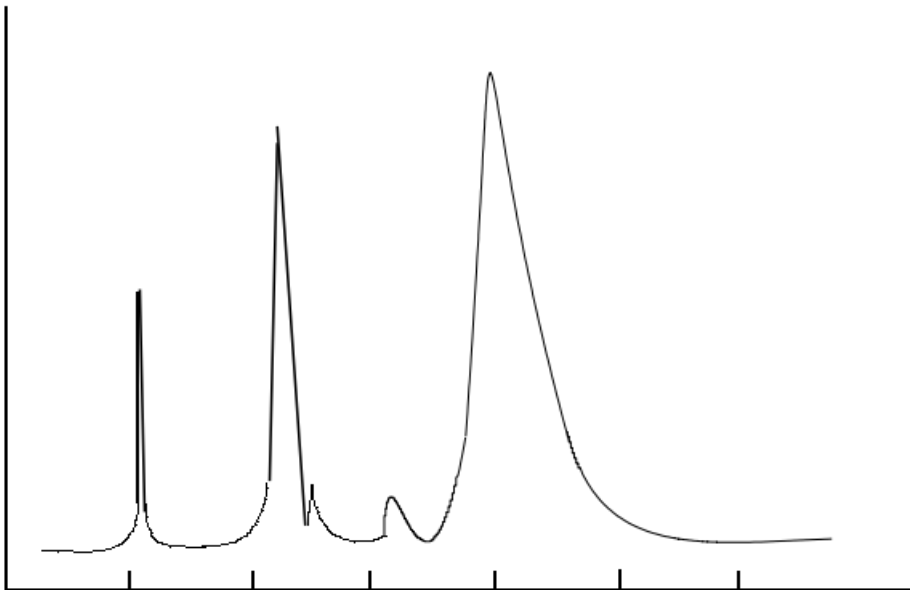


Figure 2.1. Gas-liquid chromatogram of benzotriazole vinylation reaction catalyst: 1-DMSO; 2-N-vinylbenzotriazole; 3-Benzotriazole.

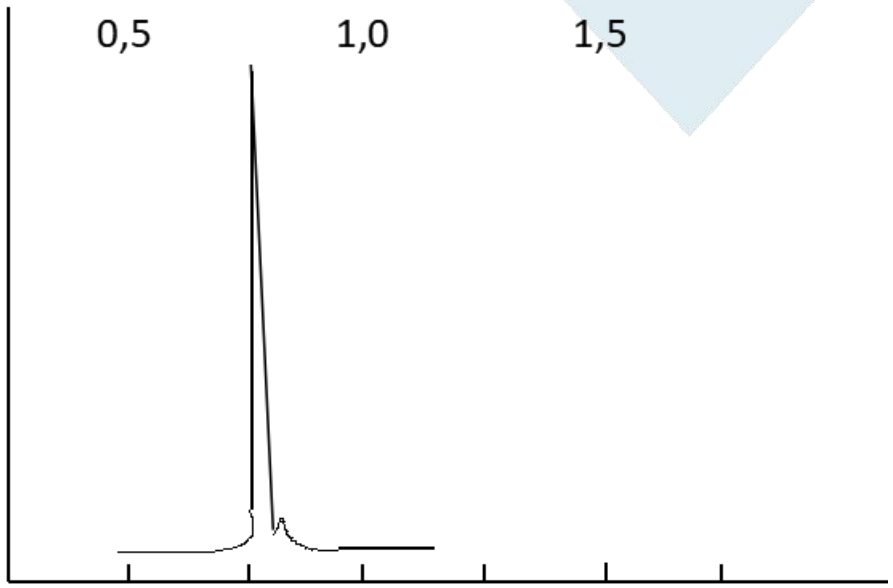


Figure 2.2. Gas-liquid chromatogram of benzotriazole.

The following systems were used for thin layer chromatography (TLC) of the obtained products on "Sulufol" plates (volume ratios):

1. Benzene:ether:methanol-10:5:2 (system #1)

2. Benzene:ether:ethanol-10:6:2 (system #2)

Of them, system No. 2 was found to be convenient.

In all cases, the chromatograms were opened with an iodine crystal.

The vinylation products in YuQX give a yellow spot characteristic of benzotriazole at Rf=0.573, and Rf= 0.769 is compatible. The IR-spectra of the obtained products were obtained in the 400-4000 cm-1 range on UR-70 spectrophotometers. In this case, the IR-spectra of liquid substances is in a thin-layer solution (or in an individual state).

The density of the synthesized substance was measured by a pycnometric method, and the refractive index was measured by a refractometer.

LIST OF REFERENCES USED

1. Nurmonov Suvon Erkhanovich, Karimova Sidora Bakhtiorovna, Isomiddinov Jawahir Qamariddin son Mamatkulov J.R. (2020) Study of the catalytic vinylation reaction of benztriazole in a homogeneous system based on acetylene gas #1 Journal of Natural ScienceS
2. Karimova, S. B., & Mullajonova, Z. S. Q. (2021). Study of catalytic vinylation reaction of benzotriazole in a homogeneous system based on acetylene gas. Science and Education, 2(1).
3. Neyland O.Ya. Organic chemistry.-Moscow: Vysshaya shkola, 1990, 751 p.; V.F. Traven, Organic chemistry, Moscow, "Akademkniga", 2004, t.1., -728 p.;

4. Darvishev O.K., Mirkhamitova D.Kh., Nurmanov S.E. Vinylirovanie azotosoderjashchikh heterocyclicheskikh soedineniy acetylenom. 14. Patent RF #1463228. N-vinilnyx proizvodnyx azolov // Trofimov B.A., Mikhaleva A.I. / B.I. -2002. - No. 27;
5. Нурмонов С. Е. и др. Синтез и свойства полиметиленнафталин сульфокислоты на основе вторичного продукта пиролиза углеводородов //Universum: химия и биология. – 2022. – №. 7-2 (97). – С. 31-36.
6. Mahramovich K. S., Khodiyevich K. S. Chemical structure and practical significance of resveratrol. – 2022.
7. Hakberdiev, S. M., Talipov, S. A., Dalimov, D. N., & Ibragimov, B. T. (2013). 2, 2′-Bis {8-[(benzylamino) methylidene]-1, 6-dihydroxy-5-isopropyl-3-methylnaphthalen-7 (8H)-one}. *Acta Crystallographica Section E: Structure Reports Online*, *69*(11), o1626-o1627.
8. Хакбердиев Ш. М., Тошов Х. С. Моделирование реакции конденсации госсипола с о-толуидином //ББК 74.58 G 54. – С. 257.
9. Khamza, Toshov, Khakberdiev Shukhrat, and Khaitbaev Alisher. "X-ray structural analysis of gossypol derivatives." *Journal of Critical Reviews* 7.11 (2020): 460-463.
10. Хамидов, С. Ҳ., & Хакбердиев, Ш. М. (2021). Бирламчи алифатик аминларнинг госсиполли ҳосилалари синтези. *Science and Education*, *2*(3), 113-118.
11. Khaitbaev A. K., Khakberdiev S. M., Toshov K. S. Isolation of Gossypol from the Bark of Cotton Roots //Annals of the Romanian Society for Cell Biology. – 2021. – С. 1069-1073.
12. Хакбердиев Ш. Синтез, строение и получение супрамолекулярных комплексов ароматических аминов с госсиполом //Журнал естественных наук. – 2021. – Т. 1. – №. 4.
13. Хакбердиев Ш. М. и др. Синтез госсипольных производных орто, мета, пара толуидина и их строение //Science and Education. – 2021. – Т. 2. – №. 10. – С. 195-200.
14. Khakberdiev, Sh M., et al. "Synthesis and structure of gossypol azomethine derivatives." *Young Scientist,(4)* (2015): 42-44.
15. Mahramovich, K. S., Sattarovna, K. F., & Farangiz, M. (2022). Synthesis of Gossipy Products of Pyrimidine Bases and Getting Their Water-Solved Complexes. *Eurasian Scientific Herald*, *8*, 118-121.
16. Mahramovich, K. S. (2022). Results of computer study of biological activity of gossipol products. *Web of Scientist: International Scientific Research Journal*, *3*(6), 1373-1378.
17. Хакбердиев, Ш., Муллажонова, З., & Маматова, Ф. (2022). Аденининг госсиполли ҳосиласи унинг металло ва супрамолекуляр комплексларини турли таҳлиллар асосида ўрганиш. *Журнал естественных наук*, *1*(2 (7)), 288-293.
18. Khakberdiyev Shukhrat Mahramovich, & Mamatova Farangiz Qodir qizi. (2022). Synthesis ofmetallocomplexes of schiff bases and their structural analysis.World Bulletin of Public Health,16, 173-177. Retrieved from.
19. Mahramovich, K. S. (2023). Structural analysis of supramolecular complexes of schiff bases. *American Journal of Interdisciplinary Research and Development*, *12*, 36-41.
20. Mahramovich, K. S., & Khodiyevich, K. S. (2023). Study of the practical significance of benzimidazole and some of its derivatives. *Open Access Repository*, *4*(02), 80-85.
21. Khakberdiev Shukhrat Mahramovich, Khamidov Sobir Khodiyevich. (2023). Chemical structure and practical significance of benzoxazole . *Ethiopian International Journal of Multidisciplinary Research*, *10*(09), 75–77.