ИЗВЕСТИЯ ВЫСШИХ УЧЕБНЫХ ЗАВЕДЕНИЙ.
Т 64 (11) Серия «**ХИМИЯ И ХИМИЧЕСКАЯ ТЕХНОЛОГИЯ**» 2021
V 64 (11) **ChemChemTech** 2021

DOI: 10.6060/ivkkt.20216411.6376

УДК: 547.791

СИНТЕЗ 4-ФЕНИЛ-1Н-1,2,3-ТРИАЗОЛ-5-КАРБАЛЬДЕГИДА

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В данной работе впервые реакцией α-бромкоричного альдегида с азидом натрия выделен и описан 4-фенил-1Н-1,2,3-триазол-5-карбальдегид с выходом 87%. Бромированием 3-фенил-2-пропеналя молекулярным бромом с последующим дегидробромированием 2,3-дибром-3-фенил-2-пропеналя получен 2-бром-3-фенил-2-пропеналь. Строение 2-бром-3-фенил-2-пропеналя доказано методами ИК и ЯМР спектроскопии на ядрах ^{1}H и ^{13}C. Реакцией 2-бром-3-фенил-2-пропеналя с азидом натрия при комнатной температуре получен только 4-фенил-1Н-1,2,3-триазол-5-карбальдегид с выходом 87%. Строение 4-фенил-1Н-1,2,3-триазол-5-карбальдегида доказано методами ИК и ЯМР спектроскопии. В ИК спектре присутствуют полосы поглощения при 1694 см$^{-1}$ и 1562 см$^{-1}$, характерные для связи >C=O, >C=C< соответственно, а полосы поглощения фенильного кольца регистрируются в области 1584 и 1109 см$^{-1}$. В спектре ^{1}H ЯМР (DMSO$_{d6}$) характеристичным является сигнал альдегидной группы при 10,17 м.д., фенильному заместителю соответствуют две группы мультиплетов 7,52 и 7,95 м.д. В спектре ^{13}C ЯМР (DMSO$_{d6}$) присутствуют сигналы углерода триазольного кольца и альдегидной группы при 144,04 м.д. и 187,79 м.д. соответственно. Сигналы в области 128,91-134,20 м.д. соответствуют атомам углерода фенильного радикала. Очевидно, что реакционноспособная альдегидная группа в 4-фенил-1Н-1,2,3-триазол-5-карбальдегиде может легко окисляться, а также вступать в реакции нуклеофильного присоединения и присоединения-отщепления, и, тем самым, приводить к производным 1,2,3-триазола различной структуры. Получение 4-фенил-1Н-1,2,3-триазол-5-карбальдегида осуществляется в мягких условиях и без использования токсичных растворителей и катализаторов. Это означает, что предлагаемый метод синтеза 4-фенил-1Н-1,2,3-триазол-5-карбальдегида соответствует принципам «зеленой химии» и может использоваться в качестве строительных блоков при создании биологически активных соединений и лекарственных препаратов.

Ключевые слова: 3-фенил-2-пропеналь, азид натрия, замещение, 2-азидо-3-фенил-2-пропеналь, 4-фенил-1Н-1,2,3-триазол-5-карбальдегид, спектроскопия

Для цитирования:
Новокшонов В.В., Шаглаева Н.С., Подгорбунская Т.А., Цырендоржиева И.П., Айзина Ю.А. Синтез 4-фенил-1Н-1,2,3-триазол-5-карбальдегида. *Изв. вузов. Химия и хим. технология*. 2021. Т. 64. Вып. 11. С. 30–34

For citation:
Novokshonov V.V., Shaglaeva N.S., Podgorbunskaya T.A., Tsyrendorzhieva I.P., Aizina Yu.A. Synthesis of 4-phenyl-1H-1,2,3-triazole-5-carbaldehyde. *ChemChemTech* [*Izv. Vyssh. Uchebn. Zaved. Khim. Khim. Tekhnol.*]. 2021. V. 64. N 11. P. 30–34

SYNTHESIS OF 4-PHENYL-1H-1,2,3-TRIAZOLE-5-CARBALDEHYDE

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4-Phenyl-1H-1,2,3-triazole-5-carbaldehyde has been synthesized for the first time in 87% isolated yield by the reaction of α-bromocinnamaldehyde with sodium azide. Bromination of 3-phenyl-2-propenal with molecular bromine followed by dehydrobromination of 2,3-dibromo-3-phenyl-2-propenal affords 2-bromo-3-phenyl-2-propenal. The structure of the latter is proved by IR and ^{1}H and ^{13}C NMR techniques. The reaction of 2-bromo-3-phenyl-2-propenal with sodium azide proceeds at room temperature to deliver 4-phenyl-1H-1,2,3-triazole-5-carbaldehyde exclusively in 87% yield. The structure of 4-phenyl-1H-1,2,3-triazole-5-carbaldehyde is established by IR and NMR spectroscopies. The IR spectrum contains absorption bands at 1694 cm^{-1} and 1562 cm^{-1}, characteristic of the >C=O, >C=C< bonds, respectively, and the absorption bands of the phenyl ring are recorded in the range of 1584 and 1109 cm^{-1}. In the ^{1}H NMR spectrum ($DMSO_{d6}$), the characteristic signal of the aldehyde group is detected at 10.17 ppm, while two multiplets at 7.52 and 7.95 ppm are attributable to the phenyl substituent. The ^{13}C NMR spectrum (DMSOd6) shows the triazole ring signals and the aldehyde group of carbons at 144.04 ppm and 187.79 ppm, respectively. Signals in the range of 128.91-134.20 ppm correspond to the phenyl radical of carbons. The synthesis of 4-phenyl-1H-1,2,3-triazole-5-carbaldehyde is carried out under mild conditions and without toxic solvents and catalysts. It can be concluded that the developed method for the synthesis of 4-phenyl-1H-1,2,3-triazole-5-carbaldehyde meets the requirements of "green chemistry" and the target compounds can be employed as building blocks for the design of biologically active compounds and drugs.

Key words: 3-phenyl-2-propenal, sodium azide, substitution, 2-azido-3-phenyl-2-propenal, 4-phenyl-1H-1,2,3-triazole-5-carbaldehyde, spectroscopy

INTRODUCTION

The reaction of azide-alkyne cycloaddition (AAC) was pioneered by R. Huisgen, gave an impetus to the development of a wide variety of methods and approaches in organic synthesis. This transformation is a striking example of atom-economic reactions, which are accompanied by the formation of stable heterocyclic systems [1-5]. At the same time, the reaction has one significant drawback associated with low regioselectivity under the conditions of a thermally induced process.

K.B. Sharpless [6] and M. Meldal [7] proposed independently an alternative method for the AAS reaction implementation with copper salts. The proposed system distinctive feature is the high regioselectivity of the formation of substituted 1,2,3-triazoles. Later, it was revealed that the salts of other metals can also be used as catalysts [8-11]. Low cost and availability of the catalysts, the ease of the target product isolation made this transformation a bright illustration of the "click-chemistry" that found intensive application in organic synthesis, biochemistry, and polymer chemistry as well. However, toxicity the metal salts limits their use in drug design. Therefore, the search for regioselective metal catalyst-free methods for the 1,2,3-triazole preparation and its derivatives are carried out [12, 13].

1,2,3-Triazoles are being used extensively in the drugs production [14-19], new materials [20-22], the components of explosive mixtures [23], plant protection agents and plant growth regulators [24]. The catalytic 1,3-dipolar azides cycloaddition to alkynes is one of the main approaches to the 1,2,3-triazoles synthesis. However, direct preparation of some substituted 1,2,3-triazoles via this reaction is still problematic [25]. Consequently, the elaboration of novel "green" strategies for the synthesis of functional substituted

1,2,3-triazoles is under way [13, 26-30]. In the present work, we have implemented the regioselective synthesis of 4-phenyl-1*H*-1,2,3-triazole-5-carbaldehyde by the reaction of 2-bromo-3-phenyl-2-propenal with sodium azide at room temperature for the first time.

EXPERIMENTAL PART

^{1}H and ^{13}C NMR spectra were recorded on a Bruker DPX-400 spectrometer in chloroform ($CDCl_3$) and dimethyl sulfoxide ($DMCOd_6$) solutions. Hexamethyldisiloxane (HMDS) was used as an external standard.

The IR spectrum was run on a Bruker Vertex70 spectrometer in KBr pellets and in vaseline oil.

2-Bromo-3-phenyl-2-propenal (3)

In a three-necked flask equipped with a thermometer, a reflux condenser and a dropping funnel was placed acetic acid (260 ml), cinnamaldehyde (116.15 g, 0.88 mol). Then Br_2 (140.62 g, 0.88 mol) was added dropwise upon stirring at 7-8 °C. After addition of the total amount of molecular bromine (Br_2), the mixture was stirred with a magnetic stirrer for 3 h at a room temperature. Afterwards K_2CO_3 (60.65 g, 0.44 mol) was added in small portions and the reaction mixture was being heated up to 80-90 °C for 2 h, then cooled to a room temperature and poured into cold water (700 ml). The formed precipitate was filtered off and recrystallized from aqueous ethanol (2 ml of 96% EtOH x 1 ml of H_2O) and dried to give 162.04 g (90%) of needle-like, yellowish-white crystals, rapidly yellowing in the air.

IR: 1685, 1600, 1580, 1116, 1082;

^{1}H ($CDCl_3$) σ: 7.50-7.52(m, 3H, *n*-Ph and *o*-Ph), 7.91(s, 1H, Ph-CH), 8.01-8.03(m, 2H, *m*-Ph), 9.36(s, 1H, CH=O);

^{1}H ($DMSO_{d6}$) σ: 7.55-7.58(m, 3H, *n*-Ph and *o*-Ph), 8.02-8.05(m, 2H, *m*-Ph), 8.43(s, 1H, Ph-CH), 9.41(s, 1H, CH=O);

^{13}C($CDCl_3$) σ: 124.38, 128.89, 137.07, 131.71, 133.04, 149.27, 187.17.

4-Phenyl-1H-1,2,3-triazole-5-carbaldehyde (4)

2-Bromo-3-phenyl-2-propenal (**3**) (0.21 g, 0.001 mol) and sodium azide (0.13 g, 0.002 mol, 2 equiv.) a three-necked flask equipped with a thermometer, a reflux condenser and a dropping funnel was placed. Then the solution of Na_2CO_3 (0.21 g, 0.002 mol) in DMSO (1 ml) was added. The reaction mixture was being stirred in a magnetic stirrer at a room temperature for 20 h. Water (10 ml) was added. The mixture was extracted with ethyl acetate (5×10 ml) and the combined organic phase was washed with a saturated aqueous solution of NaCl (5×5 ml) and dried over calcined $MgSO_4$, the salt was filtered off, the solvent was distilled off in the vacuum of a water-jet pump at a temperature not higher than 35 °C. As result, 0.165 g (87%) of a brown-orange product was obtained.

IR: 1677, 1565, 2135, 2094;

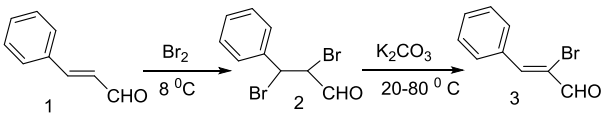
^{1}H ($CDCl_3$) σ: 6.43(s, 1H, Ph-CH), 7.36-7.43(m, 3H, *n*-Ph and *o*-Ph), 7.84-7.86(d, 2H, *m*-Ph), 9.46(s, 1H, CH=O);

^{1}H ($DMSO_{d6}$) σ: 6.96(s, 1H, Ph-CH), 7.46-7.48(m, 2H, *n*-Ph and *o*-Ph), 7.89-7.91(d, 2H, *m*-Ph), 9.52(s, 1H, CH=O);

^{13}C ($CDCl_3$) σ: 128.91, 130.57, 131.06, 133.19, 134.20, 135.13, 144.04, 187.79.

RESULTS AND DISCUSSION

2-Bromo-3-phenyl-2-propenal (**3**), used as the initial substrate in the reaction with sodium azide, was obtained by bromination of 3-phenyl-2-propenal (**1**) with molecular bromine in the presence of acetic acid, followed by dehydrobromination of the intermediate dibromo derivative **2** (Scheme 1). It was found that 2,3-dibromo-3-phenyl-2-propenal (**2**) was quantitatively converted to 2-bromo-3-phenyl-2-propenal (**3**) under the influence of K_2CO_3 at 80-90 °C for and the whole process is finished in 2 h.

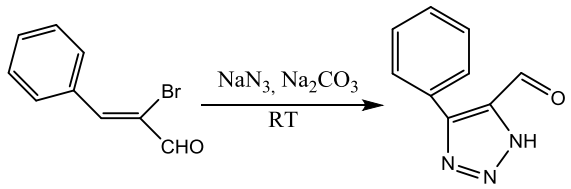


Scheme 1
Схема 1

The structure of 2-bromo-3-phenyl-2-propenal (**3**) has been established by IR, ^{1}H and ^{13}C NMR techniques. The IR spectrum contains an absorption band at 1685 cm^{-1}, which is attributable to the stretching vibrations of the C=O bond, and the characteristic absorption of the C=C bond at 1560-1585 cm^{-1}, hidden under the absorption bands of the phenyl ring at 1600, 1580, 1116, 1082 cm^{-1}. In the ^{1}H NMR spectrum ($DMSOd_6$), characteristic resonance signals are observed at 9.41 (CHO) and 8.43 (CH=C(Br) ppm. Two groups of multiplets (5H) at 7.55 and 8.05 ppm are assigned to the aromatic ring. The ^{13}C NMR ($DMSOd_6$) spectrum also confirms the compound structure (**3**). The IR-spectrum and NMR-spectroscopy obtained while researching the structure 2-bromo-3-phenyl-2-propenal (Scheme 1) are satisfactorily coordinated with the results of work [31], where on the basis of IR and NMR as well as Mass-spectroscopy the component structure has been proved (**3**).

2-Bromo-3-phenyl-2-propenal (**3**) is a light yellowish needle-like crystal that is unstable to light and requires storage at a low temperature.

The reaction of 2-bromo-3-phenyl-2-propenal (**3**) with sodium azide at a room temperature which 20 h took in DMSO in the presence of Na_2CO_3 afforded only 4-phenyl-1H-1,2,3-triazole-5-carbaldehyde (**4**) in 87% yield, no other reaction products were being detected (Scheme 2).



3 4

Scheme 2
Схема 2

In our opinion, the alkyne (3-phenylpropanal) obtained according to scheme 2 immediately, as usual, enters into condensation with the azide.

CONCLUSION

In conclusion, an efficient regioselective method for the synthesis of 4-phenyl-1H-1,2,3-triazole-5-carbaldehyde in 87% yield with the help of the reaction of α-bromocinnamaldehyde with sodium azide has been developed. The target product structure is established by IR and NMR spectroscopy. Further chemical modification of the aldehyde group will allow synthesizing a wide range of 1,2,3-triazole derivatives.

ACKNOWLEDGMENT

This work was financially supported by the Basic Research Russian Foundation (grant No. 19-08-00342).

ФОНДОВАЯ ПОДДЕРЖКА

Работа выполнена при финансовой поддержке Российского фонда фундаментальных исследований (договор № 19-08-00342).

The authors declare the absence a conflict of interest warranting disclosure in this article.

Авторы заявляют об отсутствии конфликта интересов, требующего раскрытия в данной статье.

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Поступила в редакцию 05.02.2021
Принята к опубликованию 30.09.2021

Received 05.02.2021
Accepted 30.09.2021