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ALKYLATION OF 4′,4′′-DINITRODIBENZO-18-CROWN-6

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Abstract

This article presents the results of alkylation of 4′,4′′-dinitrodibenzo-18-crown-6 with C_1-C_5 alkyl bromides of normal structure, isomers (*n-, sec-, iso-, tert-*) butyl bromide and unsaturated allyl bromide in ether media and under phase-transfer catalysis conditions. The alkylation of 4′,4′′-dinitrodibenzo-18-crown-6 via the Grignard reaction occurs with the parallel reduction of the nitro group to the nitroso group. In benzene, the alkylating group occupies the 3′,3′′-position. The branched structure of the alkylating radical affects the yield of the product. When carrying out the Grignard reaction in a diethyl ether medium, alkylation occurs at the 5′,5′′-position; branching of the radical does not affect the quantitative yield of the target product.

Keywords: *alkylation, 4′,4′′-dinitrozodibenzo-18-crown-6, diethyl ether, benzene, Grignard reaction*

Introdaction

Crown ethers are extremely interesting and important in various fields of science and industry. Crown ethers and their derivatives are widely used in drug delivery (Chehardoli G., 2019), and crown ethers cross-linked with quinazoline are used as epidermal growth factor receptor (EGFR) inhibitors (Hu S., 2012). A study of the effect of substituent length in diacyl derivatives of dibenzo-18-crown-6 (Komilova N. R., 2024) showed an increase in energy from 4′,4′′-diacetyl-DB18C6 to 4′,4′′(5′′)-divaleryl-DB18C6 with an increase in Ca^{2+} ionophore activity. Both 4′,4′′(5′′)-dibutyryl-DB18C6 and 4′,4′′(5′′)-divaleryl-DB18C6 effectively promote Ca^{2+} transport across fibroblast cell membranes in cellular processes determining Parkinson's disease (Komilova N. R., 2024).

Olefinic chiral frameworks containing 18-crown-6 and benzo-15-crown-5 inclusions exhibit high enantioselectivity when used as fluorescence sensors for the detection of chiral amino alcohols (Yuan C., 2021). Unique chemosensors (Zhang J., 2020) for determining the fluorescence of various metal ions in non-aqueous media have been created based on crown ether acylhydrazone, and crown-substituted coumarins have been used as chemosensors (Tkachenko N. V., 2019), as well as selective sorbents for the extraction of cations (Qutlimurotova N. K., 2023).

Nitrogen-containing crown ether ligands exhibit hCA (human carbonic anhydrase) enzymatic activity and inhibit enzyme-substrate interactions (Akkemik E., 2018).

(ϰ-arylpyridino)-dibenzoaza-14-crown-4 ethers exhibit activity against tumor cell membranes, exhibiting high antineurotoxic, antitumor, and cardioprotective activity (Cai X. J., 2017). The Grignard reaction can be used to obtain derivatives of organic compounds belonging to various classes of compounds (Kozinskaya L., 2022). Grignard reagents are capable of reacting with weakly acidic atomic groups, such as derivatives of alcohols (Roy D., 2018), acetylenes (Ryu T., 2017), and primary and secondary amines (Pearce N. A., 2019).

In 2021, chemists (Posevins D., 2021) presented a study of the cross-coupling of unactivated alkyl halides and a tosylate, transferring the alkyl and phenyl functional group using Grignard reagents. A classic example of the Grignard reaction involving benzo-18-crown-6 is described in the literature (Tatematsu Y., 2018). We previously demonstrated the possibility of obtaining tertiary alcohols, tertiary acetylenic alcohols, carbonyl compounds, and the dicarboxylic acid dibenzo-18-crown-6 via the Grignard reaction.

The aim of this study is to investigate the alkylation of 4′,4′′-dinitrodibenzo-18-crown-6 in ether and non-ether media.

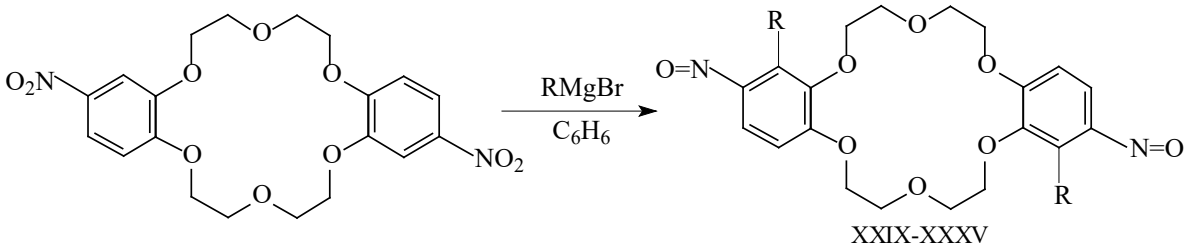
Subjects and Methods

Grignard reagents with different radical lengths, C_1-C_5 and -CH_2-CH=CH_2, were chosen as the study subjects. Particular attention was paid to butyl radicals in ether and non-ether media.

Results and discussion

The action of any type of Grignard reagents on mononitroarenes leads exclusively to N-alkylation products due to 1,2-addition to the nitro group. Examples of conjugated interactions also include polynitro compounds. The interaction of mononitroarenes with alkyl magnesium halides leads to nucleophilic alkylation at the ortho- or para-positions. Thus, the reactivity of alkyl and aryl reagents is now differentiated. This interaction is believed to proceed through the conjugated state (1,4 or 1,6). Italian chemists (G. Bartoli, 2014) proposed a simple method for introducing a substituent into a nitro-substituted benzene ring using the Grignard reaction.

The structure of dibenzo-18-crown-6 contains two benzene rings, which participate in all electrophilic substitution reactions, taking into account the existing pyrotechol substituents. Similar to this report, the reaction of 4′,4′′- dinitrodibenzo-18-crown-6 with Grignard reagents based on normal alkyl bromides was carried out:



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R= –CH_3, –C_2H_5, –C_3H_7, –C_4H_9, –C_5H_{11}, –CH_2–CH=CH_2

The replacement of the hydrogen atom in aromatic compounds by nucleophilic reagents can only be carried out if these compounds contain strong electron-withdrawing groups, and the substituent is in the ortho and para positions with respect to them.

Alkylation of aromatic nitro derivatives, as well as DBCE nitro derivatives, takes place in the form of two parallel processes:

– alkylation of the benzene ring to reduce the nitro to nitroso group.

All reactions, as before, proceeded in 2 visible stages: the formation of a flocculent complex 4′,4′′-dinitro-DB18C6 with magnesium and the formation of the final product. The obtained compounds are light green amorphous powders of 3′,3′′-dialkyl-4′,4′′-dinitroso-DB18C6, with a yield of 65–78%. The structures of the obtained compounds were proved using elemental analysis, IR -,^{1}H- and^{13}C-NMR spectroscopy, to determine the double bond in the allyl radical,

a qualitative reaction was used to decolorize a solution of potassium permanganate.

Bartoli and coworkers studied the interaction of nitroarenes and substituted naphthenic derivatives in classical ether media of the Grignard reaction. The study revealed the regioselectivity of the introduction of a newly introduced substituent, depending on the electron nature of the *o*- and *p*-positions. The authors also proposed a reaction mechanism in which the nitro group exhibits the properties of a carboxyl group and, due to the partial negative charge of the oxygen atoms, the addition of a positive^{+}MgBr species occurs, while the alkyl chain, with its partial negative charge, interacts with the α-carbon of the benzene ring. We have already considered (Kozinskaya L., 2012) the possibility of conducting Grignard reactions in non-ether media through the formation of an intermediate magnesium complex with the oxygen atoms of the macrocycle.

Each experiment was conducted using the same experimental procedure as for ethyl bromide, using dilute hydrochloric acid during hydrolysis to decompose the reaction mixture. The reactions are often accompanied by the formation of resins and trace amounts of byproducts, primarily arising from the decomposition of nitroso derivatives (azoxy, nitro, etc.).

The results show that these reactions occur with a wide variety of substrates. All compounds studied lead to C-alkylation products via conjugate addition to the nitroarene system. The results provide some insight into the reaction mechanism and the selectivity of the attack.

Nitroarenes, as well as nitroalkanes, undergo 1,2-addition at the nitro group with Grignard reagents, followed by reduction of the resulting adducts. N-substituted hydroxylamines may form as byproducts.

The Grignard reaction proceeds via the conjugate addition of RMgX to 4′,4′′-dinitrodibenzo-18-crown-6. After the addition of Lewis acids and water, the reaction mixture decomposes to form amorphous powders of light green color, characteristic of nitrosoarenes: 3′, 3′′-dialkyl-4′,4′′-dinitrodibenzo-18-crown-6.

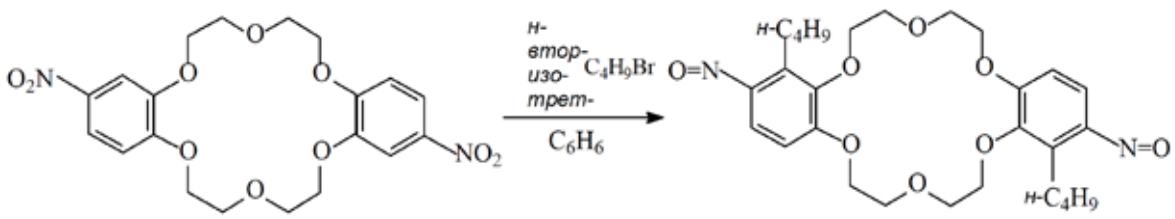
This reactivity, relative to normal 1,2-addition, may be due to the stabilizing effect of the fused ring on the adducts.

These results are consistent with the hypothesis that the formation of a cyclohexadiene-type adduct occurs via a two-step mechanism: in the first step, RMgX and the substrate react to form a 1:1 coordination complex, which readily undergoes subsequent attack by RMgX to form the adduct. The 1,2-addition competes with the 1,4-addition, with the para-position relative to the nitro group being accessible to nucleophilic attack by RMgX.

Since the proposed mechanism is nucleophilic for the incoming alkyl group, any of the ortho-positions relative to the nitro group should be equally open to the insertion of a substituent.

The next step in the study of 4′,4′′-dinitrodibenzo-18-crown-6 in the Guignard reaction was to examine the structure of the radical and the effect of carbon chain length on the rate of formation of the target product.

The most common and readily available isomers of various structures containing primary, secondary, tertiary, and quaternary carbon atoms are butyl isomers. Therefore, Grignard reagents based on butyl bromides of various structures were used:

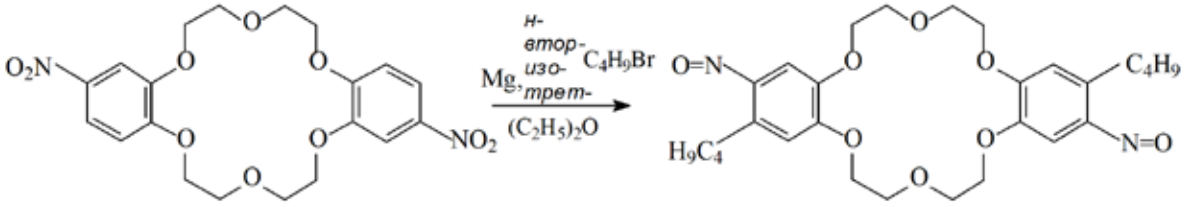


Visually, the Grignard reagent based on butyl bromide formed a complex with the polyether macrocycle faster than all the bromobutane isomers. Complexes formed in all four flasks. However, the formation of the target product with branched radicals did not occur.

Thus, Grignard reagents with branched substituents are less reactive than their normal-chain counterparts. The magnesium

complex of the alkyl halide with the macrocycle cavity is difficult to form, resulting in a reduced yield of the target products.

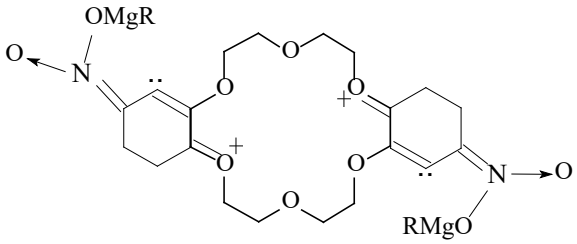
Polish scientists (Antoniak D., 2022) conducted a study of the influence of solvent and reaction temperature on the alkylation of nitropyridines. Based on the results of this work, alkylation of 4′,4′′- dinitrodibenzo-18-crown-6 isomers of bromobutane was carried out in a diethyl ether medium:



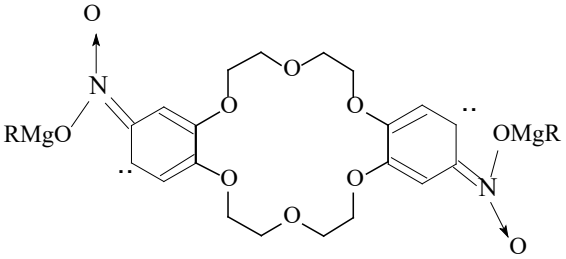
a= –*n*-C_4H_9, b= – *sec*- C_4H_9, c= – *izo*- C_4H_9, d= –*tert*- C_4H_9

In a diethyl ether medium, butybromide forms a complex with magnesium from all isomers, since the ether groups are not linked by a macrocycle as is the case with the crown ether macrocycle. The reaction with dinitrodibenzo-18-crown-6 occurs with butyls of various structures at the 5' position of the benzene ring of dibenzo-18-crown-6. This is easily explained by the spatial position of the –N=O group.

Due to the possibility of forming different structures upon solvent replacement, different intermediates are also possible during the formation of the Grignard reagent in different environments:



Intermediate state in benzene



and in diethyl ether

The structures of the synthesized compounds 3′,3′′-di-n-butyl-4′,4′′-dinitroso- and 4′,4′′-dinitroso-5′,5′′-di-(n-, sec-, iso-, tert-)butyldibenzo-18-crown-6 were confirmed using elemental analysis, IR, ^{1}H- and ^{13}C-NMR spectroscopy. The yields are 70–75%.

Conclusion

Alkylation of 4′,4′′-dinitrodibenzo-18-crown-6 via the Grignard reaction occurs with parallel reduction of the nitro group to the nitroso group. In benzene, magnesium forms a polyetherate with the macrocycle cavity, similar to magnesium diethyl etherate in Grignard reactions, and directs the alkylating group to the 3′,3′′-position. The branched structure of the alkylating radical affects the product yield. When the Grignard reaction is carried out in diethyl ether, a complex of magnesium etherate with diethyl ether is formed. Alkylation occurs at the 5′,5′′-position; branching of the radical does not affect the quantitative yield of the target product.

Experimental part

^{1}H- and ^{13}C-NMR spectra were obtained in a $CDCl_3$ solution on a Bruker VXR-400 spectrometer at an operating frequency of 400 MHz, using a TMS solvent as an internal standard (7.27 ppm by TMS)

Melting points were determined in the usual way in a metal block.

3′,3′′-dimethyl-4′,4′′-dinitrodibenzo-18-crown-6. To a solution of 5.0 g (11.1 mmol) of 4′,4′′- dinitrodibenzo-18-crown-6 in 250 ml of benzene with heating to 800C and constant stirring were added 2.66 g (111 mmol) of magnesium, within 30 min a voluminous flocculent complex was formed, then 3 ml (33.3 mmol) of methyl bromide were added. The light-brown benzene flocculent complex dissolved with a color change to light

green. The reaction progress was monitored by TLC on silufol in the system acetone: hexane = 2: 1. The completion of the reaction was judged by the disappearance of the starting 4′,4′′-dinitrozo-dibenzo-18-crown-6 in the reaction mixture. The reaction time was 3 h. The mixture was decomposed with water and washed until a neutral medium was obtained. Purification of the obtained product was carried out by column chromatography on aluminum oxide in the system acetone: hexane = 2:1. Yield 3.56 g (72%), mp 161–165^{0}C. IR spectrum (ν, cm-1): 3082, 2960, 2874, 1599,1580, 1524, 1442, 1402, 1380, 1347, 1282,1232,1141, 802.795, 731.^{1}H-NMR spectrum (δ, ppm): 3.28, 3.89–3.91, 4.13–4.17, 6.87–6.89, 7.10–7.12.^{13}C NMR (δ, ppm): 15.29, 69.35–71.14, 115.70–119.17, 120.45, 148.71–152.90, 157.75. Elemental analysis: Found, %: C 59.12, H 5.79. Calculated, %: C 59.19, H 5.83, N 6.28, O 28.70. Molecular formula: $C_{22}H_{26}N_2O_8$.

3′,3′′- diethyl-4′,4′′- dinitrosodibenzo-18-crown-6. To 5.0 g (11.11 mmol) of 4′,4′′- dinitrosodibenzo-18-crown-6 were added 2.66 g (110 mmol) of magnesium and 3.4 ml (33.3 mmol) of ethyl bromide. Yield 3.68 g (70%), mp 172–174^{0}C. IR spectrum (ν, cm-1): 3091, 2877, 2852, 1925, 1608, 1594, 1577, 1524, 1402, 1398, 1372, 1341, 1300, 1282,1231, 1139, 1104, 812,800, 788, 771.^{1}H-NMR spectrum (δ, ppm): 1.28–1.32, 2.51–2.55, 3.87–3.91, 4.08–4.12, 6.75, 7.20.^{13}C NMR (δ, ppm): 16.55, 17.55, 69.32–71.40, 114.75, 121.04, 150.94–152.46, 160.19. Elemental analysis: Found, %: C 61.02, H 6.68. Calculated, %: C 60.76, H 6.33, N 5.91, O 27.00. Molecular formula: $C_{24}H_{30}N_2O_8$

3′,3′′- di-n-propyl-4′,4′′-dinitrodibenzo-18-crown-6. To 5.0 g (11.11 mmol) of 4′,4′′- dinitrodibenzo-18-crown-6 were added 2.66 g (110 mmol) of magnesium and 3.7 ml (33.3 mmol) of n-propyl bromide. Yield 4.02 g (72%), mp 186–189^{0}C. IR spectrum (ν, cm-1): 3081, 2962, 2870, 1954, 1860, 1604, 1598, 1580, 1404, 1341, 1304, 1279,1235, 1138, 812,791, 737.^{1}H-NMR spectrum (δ, ppm): 0.9–0.93, 1.60–1.67, 2.41–2.45, 3.87–3.90, 4.08–4.14, 6.96–6.99, 7.09–7.11.^{13}C NMR (δ, ppm): 14.30, 20.90–24.16, 69.64–71.14, 115.22, 116.65, 123.52,149.48–156.67, 158.37. Elemental analysis: Found, %: C 62.94, H 7.06. Calculated, %: C 62.15, H 6.77, N 5.58, O 25.50. Molecular formula: $C_{26}H_{34}N_2O_8$.

3′,3′′- di-n-butyl-4′,4′′-dinitrodibenzo-18-crown-6. To 5.0 g (11.11 mmol) of 4′,4′′- dinitrodibenzo-18-crown-6 were added 2.66 g (110 mmol) of magnesium and 3.6 ml (33.3 mmol) of n-butyl bromide. Yield 4.40 g (75%), mp 198–206^{0}C. IR spectrum (ν, cm-1): 3079, 2968, 2890, 2861, 2264, 1938, 1926, 1857, 1602, 1598, 1581, 1408, 1360, 1337, 1292, 1274,1235, 1117, 812, 801, 738.^{1}H-NMR spectrum (δ, ppm): 0.91–0.94, 1.32–1.36, 1.58–1.65, 2.52–2.56, 3.88–3.92, 4.10–4.15, 6.86–6.88, 7.16–7.18.^{13}C NMR (δ, ppm): 13.94, 22.47–32.86, 68.14–72.08, 115.18, 119.10, 121.50, 148.14–152.82, 157.45. Elemental analysis: Found, %: C 63.54, H 7.48. Calculated, %: C 63.40, H 7.17, N 5.28, O 24.15. Molecular formula: $C_{28}H_{38}N_2O_8$.

3′,3′′- di-n-pentyl-4′,4′′-dinitrodibenzo-18-crown-6. To 5.0 g (11.11 mmol) of 4′,4′′- dinitrodibenzo-18-crown-6 were added 2.66 g (110 mmol) of magnesium and 4 ml (33.3 mmol) of n-pentyl bromide. Yield 4.46 g (72%), mp 215–219^{0}C. IR spectrum (ν, cm^{-1}): 3084, 3001, 2966, 2876, 2861, 2843, 1927, 1592, 1598, 1581, 1523, 1474, 1358, 1337, 1280, 1242, 1141, 1138, 816, 804, 747, 522.^{1}H-NMR spectrum (δ, ppm): 0.83–0.92, 1.34–1.40, 1.69–1.75, 2.42–2.51, 3.87–3.91, 4.08–4.16, 6.87–6.89, 7.07–7.09.^{13}C NMR (δ, ppm): 13.99, 24.34–32.40, 69.35–71.08, 116.97, 118.70, 117.99, 147.12–154.47, 157.51. Elemental analysis: Found, %: C 64.80, H 7.92. Calculated, %: C 64.51, H 7.53, N 5.02, O 22.94. Molecular formula: $C_{30}H_{42}N_2O_8$.

3′,3′′ –diallyl- 4′,4′′- dinitroso dibenzo-18-crown-6. To 5.0 g (11.11 mmol) of 4′,4′′- dinitrodibenzo-18-crown-6 were added 2.66 g (110 mmol) of magnesium and 3 ml (3.33 mmol) of allyl bromide. Yield 4.09 g (74%), mp 179–182^{0}C. IR spectrum (ν, cm^{-1}): 3086, 2876, 1865,1841, 1647, 1602, 1527, 1345, 1277, 1237, 1134, 990, 935, 868, 855, 800.^{1}H-NMR spectrum (δ, ppm): 2.89–2.91, 3.99–4.02, 4.27–4.80, 5.02–5.05, 6.92–6.98, 7.12–7.18.^{13}C NMR (δ, ppm): 28.56, 70.68–72.04, 115.81, 117.80, 119.47, 147.90, 154.20, 156.92. Elemental analysis: Found, %: C 63.08, H 6.87. Calcu-

lated, %: C 62.65, H 6.02, N 5.63, O 25.70. Molecular formula: $C_{26}H_{30}N_2O_8$.

3′,3′′(6′′)-di-n-butyl-4′,4′′-dinitrodibenzo-18-crown-6 in different solvents. 2.7 g (100 mmol) of magnesium turnings were added to 4.5 g (10 mmol) of 4′,4′′-dinitrodibenzo-18-crown-6 in 250 ml of different solvents (ether, THF, benzene, toluene, hexane) and the mixture was heated for 20–30 min, then 4 ml (30 mmol) of n-butyl bromide were added, and the mixture was refluxed for 3 h. The reaction progress was monitored by TLC on Silufol in an acetone: hexane, 2:1 system. Then hydrolysis was carried out in a weakly acidic medium, the precipitate was washed, purified by fractional crystallization from hexane, dried in a drying oven at 40 ˚C. Light yellow powder, yield in ether 45%, in THF – 49%, in benzene 67%, in toluene – 61%, in hexane 14%. mp 198–206 ˚C.

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