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Azido Derivatives of Geminal Bis(alkoxy-NNO-azoxy)compounds

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Abstract—Mesylate of 2,2-bis(methoxy-NNO-azoxy)ethanol readily reacts with sodium azide without addition of bases affording 1-azido-2,2-bis(methoxy-NNO-azoxy)ethane. In reactions of bistriflates of 2,2-bis-(methoxy- and ethoxy-NNO-azoxy)propane-1,3-diols with sodium azide in DMSO 1,3-diazido-2,2-bis-(methoxy- and ethoxy-NNO-azoxy)propanes form in a high yield. These diazides react with phenylacetylene to give 2,2-bis(methoxy-NNO-azoxy)-1,3-bis(4-phenyl-1*H*-1,2,3-triazol-1-yl)propane and 2,2-bis-(ethoxy-NNO-azoxy)-1,3-bis(4-phenyl-1*H*-1,2,3-triazol-1-yl)-propane. The molecules of the obtained azides are prone to association in $CDCl_3$ by alkoxyazoxy groups.

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Alkoxy-NNO-azoxy compounds are regarded as promising components of energetic materials since they have the same elemental composition as secondary nitramines and a higher enthalpy of formation [1]. Bis-(methoxy-NNO-azoxy)methane and its homologs are among the best known and accessible specimens of this class of compounds, and their hydroxymethyl derivatives are the most available functionally-substituted alkoxyazoxy compounds [2–4]. The latter underlie the synthesis of several polyfunctional compounds with the other explosophoric groups [ONO_2, NNO_2, $C(NO_2)_3$, energy-rich heterocycles] [2, 5–7], however, no alkoxyazoxy compound with azide groups has been described.

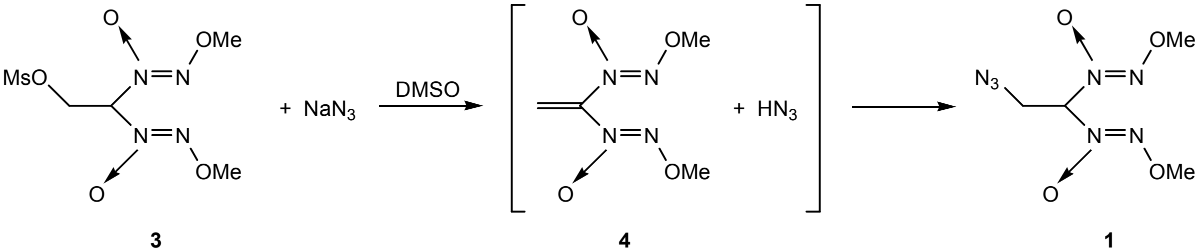
In this study formerly unknown 1-azido-2,2-bis-(methoxy-NNO-azoxy)ethane **1**, 1,3-diazido-2,2-bis-(methoxy- and ethoxy-NNO-azoxy)propanes **2a** and **2b** has been synthesized and characterized.

Monoazide **1** is readily obtained by the reaction of mesylate **3** [3] with sodium azide in DMSO. According to TLC monitoring the reaction was completed within 10 min with a virtually quantitative yield (96%) (Scheme 1).

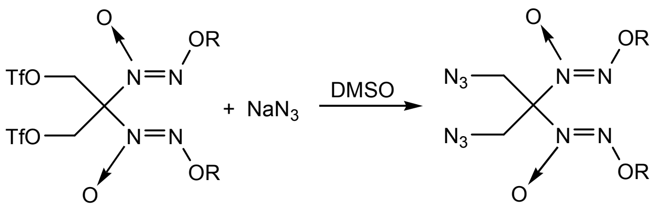
It was shown lately that the formally nucleophilic substitution in mesylate **3** [3, 4, 6–8] proceeded along the elimination–addition mechanism, and we succeeded to isolate in a pure state olefin **4** formed in the first stage [3]. In some reactions of the formal substitution olefin **4** was detected in the reaction mixtures by TLC. It was not found in the course of the reaction with sodium azide. The low stationary concentration of olefin **4** may be due to the fact that N_3^- anion is a strong nucleophile and a relatively weak base.

Diazides **2a** and **2b** were successfully prepared by the reaction of bistriflates **5a** and **5b** [9] with sodium azide in DMSO.

Scheme 1.



Scheme 2.



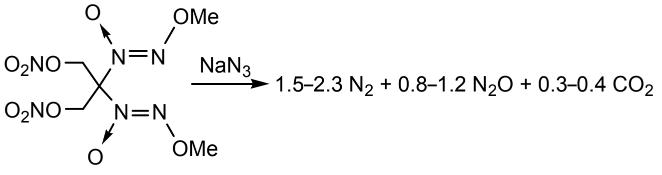
5a, **5b** **2a**, **2b**

R = Me (**a**), Et (**b**).

Alcohol triflates are very efficient in the synthesis of azides in the cases complicated with steric hindrances and acceptor β□substituents [10–14]. However, the trifluoromethanesulfonic anhydride is difficultly available, therefore even the sterically hindered polyazides are commonly synthesized from the respective mesylates, tosylates, and halides [15–22]. 1,3-Dihalo-2,2-bis(alkoxy-NNO-azoxy)propanes are unknown, the attempt to prepare 1,3-dichlorides by the reaction of 2,2-bis(alkoxy-NNO-azoxy)propane-1,3-diols with thionyl chloride yielded only the corresponding cyclic sulfites [23]. The attempts to synthesize bisazides **2a** and **2b** by the reaction of the corresponding mesylates, tosylates, and benzenesulfonates [9] with sodium azide in DMSO at room temperature failed, and at 60–70°C tarring occurred. The failure may be ascribed to low reaction rate of the S_N2-reactions of these sulfonates being by several orders of magnitude less than that of triflates. For instance, the acetolysis rate of ethyl triflate at 25°C is 30000 times faster than that of ethyl tosylate [24]. Apparently the competing S_N2-attack of the N_3^- anion on the sulfur atom of the non-fluorinated sulfonates became the main reaction with these substrates.

In the synthesis of aliphatic azides with other explosophoric groups also alkyl nitrates [25–34] are used, since they are more available and are between chlorides and bromides by the azidation rate [35]. The application of a known bisnitrate **6** [2] seemed attractive for the synthesis of diazide **2a**. Bisnitrate **6** readily reacted with sodium azide in DMSO at room temperature (Scheme 3).

Scheme 3.



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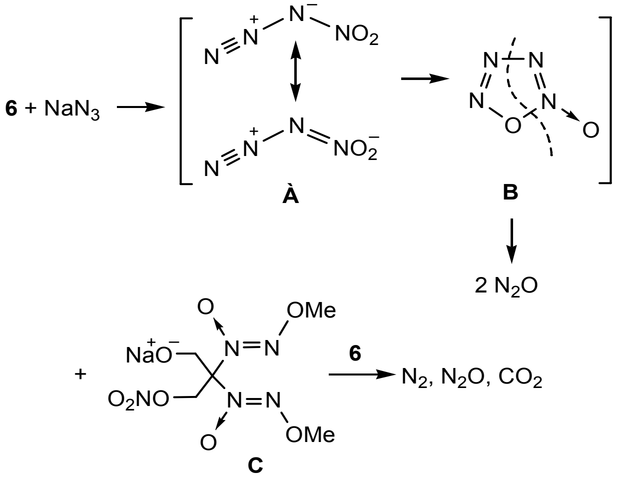
However the reaction was accompanied by voluminous gas evolution, and the target diazide **2a** was not detected by TLC even in trace amounts. The composition of gaseous products was analyzed by GC in two separate experiments.

At a longer reaction time the yields of N_2, CO_2, and N_2O proved to be ~1.5 times higher, but their ratio did not change. One of the probable reaction mechanisms involves in the first stage the nitration of the azide-anion with nitrate **6**. Intermediate **A** via oxatetrazole 2-oxide **B** decomposed into two N_2O molecules [36–38], and strong base **C** not only decomposed itself giving the observed gases, but also catalyzed the decomposition of the initial bisnitrate **6**. A similar decomposition of alkoxy-NNO-azoxy-compounds under the action of bases was described earlier [39–42] (Scheme 4).

Scheme 4.

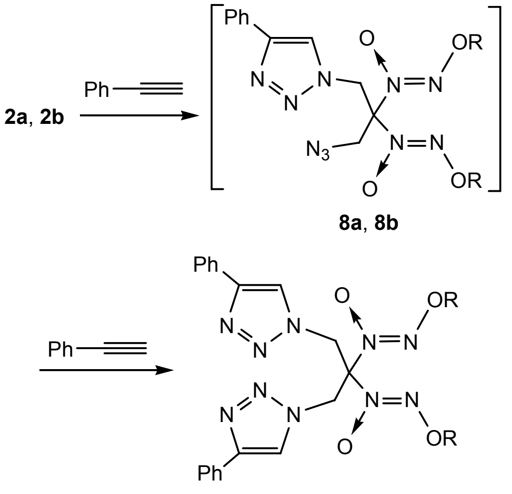


Bisazides **2a** and **2b** reacted with phenylacetylene with the formation of bistriazoles **7a** and **7b**; TLC made it possible to observe the intermediate compounds **8a** and **8b** (Scheme 5).

This reaction demonstrates the principal opportunity to synthesize alkoxyazoxy polymers from bisazides **2a** and **2b** and bisacetylene. A similar procedure for preparation of energetic polymers was formerly published [43, 44].

Azides **1**, **2a**, and **2b** are colorless or slightly yellowish oils, triazoles **7a** and **7b** are colorless high melting crystalline compounds. The composition and structure of compounds **1**, **2a**, **2b**, **7a**, and **7b** were established from the data of elemental analysis and ^{1}H, ^{13}C NMR spectra, and for azides **1**, **2a**, and **2b** they were confirmed by the data of ^{15}N NMR spectroscopy.

Scheme 5.

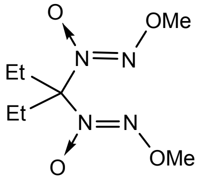


Signals of the groups N(O)=NOR [41] and N_3 [45] in the ^{15}N NMR spectra were assigned by comparison with the published data for compounds **9**–**11** and **12a**–**12c** (see the table).

All signals in the ^{15}N NMR spectrum of compound **2b** in $CDCl_3$ are shifted downfield as compared with the spectrum of compound **2a** in CD_3CN. In the chain of bonds from the group RON= to atoms N^{γ} the shift value grows from 1.0 to 4.6 ppm. It may be due to stronger association of **2a** and **2b** molecules in low polar $CDCl_3$ compared with CD_3CN, therewith the molecules in the associates exist with polar alkoxyazoxy groups turned inside, [46], and lipophilic azidomethyl groups, outside. A similar phenomenon was formerly observed with compound **9** in the ^{13}C NMR spectra in $CDCl_3$ depending on the concentration [41].

EXPERIMENTAL

^1H, ^{13}C, and ^{15}N NMR spectra were registered on a spectrometer Avance III 500 Bruker (at 500.1, 125.8, and 50.7 MHz respectively) at 22±1°C. Internal references: TMS for ^1H and ^{13}C, for ^{15}N spectrum of compounds **2a** and **2b** nitromethane, for compound **1** solvent CD_3CN (–136.30 ppm, the position of the signal was taken from the spectrum of compound **2a**). Spin-spin coupling constants ^1H–^{13}C in the ^1H NMR spectra were measured from the satellites of the main signals. The elemental analysis was carried out on a CHNS analyzer vario MICRO cube. The qualitative TLC analysis was performed on Silufol UV-254 plates, development under UV irradiation. The gaseous reaction products were analyzed on a chromatograph LKhM-80 (model 5), detector katharometer, carrier gas helium, flow rate 40 mL/min, steel columns 6 m × 3 mm, phase polysorb-1 (0.2–0.3 mm), column temperature 20°C. Methanesulfonate of 2,2-bis-(methoxy-NNO-azoxy)-



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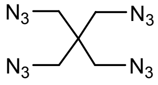

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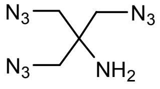

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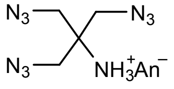

12a–**12c**

An = Cl (**a**), NO_3 (**b**), ClO_4 (**c**).

Table. Chemical shifts (δ, ppm) in ^{15}N NMR spectra of compounds **1**, **2a**, and **2b** in comparison with published data on compounds **9**–**11** and **12a**–**12c**

Compound no.	Solvent	RON=N(O)		$CH_2N^{\alpha}=N^{\beta+}=N^{\gamma-}$			References
		RON=	=N(O)	N^{α}	N^{β}	N^{γ}	
1	CD_3CN	7.56	–74.20	–316.73	–134.34	–166.17	–
2a	CD_3CN	6.52	–73.07	–317.18	–134.93	–167.04	–
2b	$CDCl_3$	7.54	–71.23	–313.91	–131.98	–162.44	–
9	$CDCl_3$	4.51	–65.75	–	–	–	41
10	$CDCl_3$	–	–	–314.5	–131.2	–167.8	45
11	$CDCl_3$	–	–	–313.4	–131.0	–168.3	45
12a	DMSO	–	–	–314.6	–131.8	–166.5	45
12b	DMSO	–	–	–314.6	–132.5	–169.6	45
12c	CD_3CN	–	–	–315.9	–133.1	–166.1	45

ethanol **3** [3], bistrifluoromethanesulfonates of 2,2-bis-(methoxy- and ethoxy-NNO-azoxy)propane-1,3-diols **5a** and **5b** [9], and 2,2-bis(methoxy-NNO-azoxy)propane-1,3-diol dinitrate **6** [2] were prepared by known procedures.

1-Azido-2,2-bis(methoxy-NNO-azoxy)ethane (1). To a solution of 545 mg (2 mmol) of compound **3** in 3 mL of DMSO was added at stirring 295 mg (3 mmol) of sodium azide. The mixture was stirred for 20 min at room temperature. The reaction course was monitored by TLC (eluent benzene–ethyl acetate, 1 : 2): R_f 0.42 (**1**), 0.18 (**3**). The reaction mixture was diluted with water (10 mL), extracted with CH_2Cl_2 (3 × 10 mL). The extract was washed with brine (20 mL) and chromatographed on silica gel Silpearl (40 mL), eluents $CH_2Cl_2 \rightarrow CH_2Cl_2$–ethyl acetate, 3 : 1 v/v. Yield 412 mg (94%), slightly yellowish oil. ^{1}H NMR spectrum, δ, ppm (22% in CD_3CN): 4.122 s (6H, MeO, J_{CH} 145.8 Hz), 4.214 d (2H, CHC$\underline{H}_2$, J 6.8 Hz), 6.156 t (1H, C$\underline{H}$CH$_2$, J 6.8, J_{CH} 167.7 Hz). ^{13}C NMR spectrum, δ, ppm (22% in CD_3CN): 48.40 (1C, CH_2N_3), 63.15 (2C, MeO), 91.32 (1C, CN_2). ^{15}N NMR spectrum, δ, ppm (22% in CD_3CN): –316.73 s (α-N_3), –166.17 s (γ-N_3), –134.34 s (β-N_3), –74.20 s [$\underline{N}$(O)=NO], 7.56 s [N(O)=$\underline{N}$O]. Found, %: C 22.29; H 4.62; N 44.25. $C_4H_9N_7O_4$. Calculated, %: C 21.92; H 4.14; N 44.74. After additional elution with a mixture CH_2Cl_2–ethyl acetate, 1 : 1, we isolated 9 mg (2.3%) of 2,2-bis(methoxy-NNO-azoxy)ethanol, mp 113.5–114.6°C (114.5–115.2 [3], 115.5–116.1°C [4]).

1,3-Diazido-2,2-bis(methoxy-NNO-azoxy)-propane (2a). To a solution of 488 mg (1 mmol) of compound **5a** in 5 mL of anhydrous DMSO was added at stirring 269 mg (4 mmol) of sodium azide. The mixture was stirred for 5 h at room temperature. The reaction course was monitored by TLC (eluent benzene–ethyl acetate, 1 : 5): R_f 0.37 (**2a**), 0.53 (**5a**). The reaction mixture was diluted with water (10 mL), extracted with CH_2Cl_2 (3 × 10 mL). The extract was washed with brine (20 mL), passed through a short (2 cm) column packed with silica gel (5 g), eluted with dichloromethane, the solvent was distilled off in a vacuum. Yield 263 mg (96%), colorless oil. ^{1}H NMR spectrum, δ, ppm (18% in CD_3CN): 4.147 s (6H, MeO, J_{CH} 148.0 Hz), 5.042 s (4H, CH_2N_3, J_{CH} 152.3 Hz). ^{13}C NMR spectrum, δ, ppm (18% in CD_3CN): 50.67 (2C, CH_2N_3, $\Delta\nu_{1/2}$ 1.7 Hz), 63.60 (2C, MeO, $\Delta\nu_{1/2}$ 1.6 Hz), 99.69 (0.7C, CN_2, $\Delta\nu_{1/2}$ 4.5 Hz). ^{15}N NMR spectrum, δ, ppm (18% in CD_3CN): –317.18 s (α-N_3), –167.04 s (γ-N_3), –134.93 s (β-N_3), –73.07 s [$\underline{N}$(O)=NO], 6.52 s [N(O)=$\underline{N}$O]. Found, %: C 21.53; H 3.87; N 50.35. $C_5H_{10}N_{10}O_4$. Calculated, %: C 21.90; H 3.68; N 51.08.

1,3-Diazido-2,2-bis(ethoxy-NNO-azoxy)-propane (2b) was obtained similarly from 516 mg (1 mmol) of compound (**5b**). TLC (benzene–ethyl acetate, 1 : 5): R_f 0.41 (**2b**), 0.57 (**5b**). Yield 284 mg (94%), colorless oil. ^{1}H NMR spectrum, δ, ppm (14% in $CDCl_3$): 1.423 t (6H, C$\underline{H}_3$CH$_2$O, J 7.1, J_{CH} 127.6 Hz), 4.455 s (4H, CH_2N_3, J_{CH} 151.5 Hz), 4.477 q (4H, CH$_3$C$\underline{H}_2$O, J 7.1 Hz). ^{13}C NMR spectrum, δ, ppm (14% in $CDCl_3$): 14.35 (2C, $\underline{C}$H$_3$CH$_2$O), 49.71 (2C, CH_2N_3), 71.96 (2C, CH$_3\underline{C}$H$_2$O), 98.37 (1C, CN_2). ^{15}N NMR spectrum, δ, ppm (14% in $CDCl_3$): –313.91 s (α-N_3), –162.44 s (γ-N_3), –131.98 s (β-N_3), –71.23 s [$\underline{N}$(O)=NO], 7.54 s [N(O)=$\underline{N}$O]. Found, %: C 27.52; H 4.96; N 45.72. $C_7H_{14}N_{10}O_4$. Calculated, %: C 27.82; H 4.67; N 46.34.

1,3-Bis(4-phenyl-1*H*-1,2,3-triazol-1-yl)-2,2-bis(methoxy-NNO-azoxy)-propane (7a). A mixture of 91 mg (0.33 mmol) of compound (**2a**), 85 mg (0.83 mmol) of phenylacetylene, 3.7 mg (0.03 mmol) of 2-amino-phenol, 14 mg (0.07 mmol) of copper acetate mono-hydrate, and 1 mL of acetonitrile was stirred for 17 h at room temperature. The reaction course was monitored by TLC (eluent benzene–ethyl acetate, 1 : 3): R_f 0.15 (**7a**), 0.25 (**8a**), 0.37 (**2a**). The reaction mixture was diluted with 5% aqueous ammonia (4 mL), extracted with $CHCl_3$ (3 × 10 mL), the extract was chroma-tographed on silica gel Silpearl (20 mL), eluent $CHCl_3 \rightarrow CHCl_3$–ethyl acetate, 3 : 1 v/v. The fraction containing compound (**7a**) (128 mg) was recrystallized from ethanol. Yield 117 mg (74%), mp 220–221°C (decomp.). ^{1}H NMR spectrum, δ, ppm (1.3% in $CDCl_3$): 4.057 s (6H, CH_3O), 5.462 s (4H, CH_2), 7.382 t.d (2H, H^{4}_{Ph}, J 7.5, 1 Hz), 7.460 deformed t (4H, $H_{Ph}^{3,5}$, J 7.5 Hz), 7.871 deformed d (4H, $H_{Ph}^{2,6}$, J 7.5 Hz), 8.221 s (2H, triazole). ^{13}C NMR spectrum, δ, ppm (1.3% in $CDCl_3$): 48.45 (2C, CH_2), 63.00 (2C, CH_3O), 98.11 (0.6C, CN_2), 122.21 (2C, C^{4}_{Ph}), 125.85 (4C, $C_{Ph}^{2,6}$), 128.76 (2C, $C^{5}_{triazole}$), 129.01 (4C, $C_{Ph}^{3,5}$), 129.62 (0.4 × 2C, C^{1}_{Ph}), 148.29 (0.4 × 2C, $C^{4}_{triazole}$). Found, %: C 52.34; H 4.91; N 28.86. $C_{21}H_{22}N_{10}O_4$. Calculated, %: C 52.72; H 4.63; N 29.27.

1,3-Bis(4-phenyl-1*H*-1,2,3-triazol-1-yl)-2,2-bis(ethoxy-NNO-azoxy)propane (7b). A mixture of 92 mg (0.3 mmol) of compound **2b**, 94 mg (0.9 mmol) of phenylacetylene, 30 mg (0.47 mmol) of copper powder, 1 mL of *t*-BuOH, and 1 mL of water was stirred for 7 days at room temperature. The reaction course was monitored by TLC (eluent benzene–ethyl acetate, 1 : 3): R_f 0.36 (**7b**), 0.55 (**8b**), 0.72 (**2b**). The reaction mixture was diluted with water (4 mL), filtered, the precipitate was dissolved in $CHCl_3$ (10 mL), filtered through a short column with silica gel (5 mL), eluted with a mixture $CHCl_3$–ethyl acetate, 2 : 1 v/v, the solvent was distilled off in a vacuum. The residue (154 mg) was recrystallized from ethanol (4 mL). Yield 108 mg (70%), mp 203–204°C. The mother liquor (33 mg after removal of the solvent) contained compounds (**2b**, **7b**, and **8b**) in comparable quantities. ^1H NMR spectrum, δ, ppm (1.1% in $CDCl_3$): 1.232 t (6H, $C\underline{H}_3CH_2O$, *J* 7.1 Hz), 4.371 q (4H, $CH_3C\underline{H}_2O$, *J* 7.1 Hz), 5.473 s (4H, CH_2N), 7.382 deformed t (2H, H^{4}_{Ph}, *J* 7.5 Hz), 7.457 deformed t (4H, $H_{Ph}^{3,5}$, *J* 7.5 Hz), 7.862 deformed d (4H, $H_{Ph}^{2,6}$, *J* 7.5 Hz), 8.209 s (2H, triazole). ^{13}C NMR spectrum, δ, ppm (1.1% in $CDCl_3$): 14.29 (2C, $\underline{C}H_3CH_2O$), 48.60 (2C, CH_2N), 72.23 (2C, $CH_3\underline{C}H_2O$), 98.10 (0.7C, CN_2), 122.15 (2C, C^{4}_{Ph}), 125.84 (4C, $C_{Ph}^{2,6}$), 128.72 (2C, $C^{5}_{triazole}$), 128.99 (4C, $C_{Ph}^{3,5}$), 129.65 (0.5 × 2C, C^{1}_{Ph}), 148.28 (0.3 × 2C, $C^{4}_{triazole}$). Found, %: C 54.32; H 5.56; N 27.51. $C_{23}H_{26}N_{10}O_4$. Calculated, %: C 54.54; H 5.17; N 27.65.

Reaction of 2,2-bis(methoxy-NNO-azoxy)propane-1,3-diol dinitrate (6) with sodium azide *a*. To a solution of 314 mg (1 mmol) of compound **6** in 3.5 mL of DMSO was added at stirring 260 mg (4 mmol) of sodium azide, gas evolution was observed. The mixture was stirred at room temperature for 6 h till complete consumption of compound **6**. At TLC monitoring of the reaction compound **2a** was not found even in trace amounts (eluent benzene–ethyl acetate, 1 : 3): R_f 0.52 (**2a**), 0.62 (**6**).

b. A solution of 314 mg (1 mmol) of compound **6** in 4 mL of DMSO and 260 mg (4 mmol) of sodium azide were separately placed in a glass vessel (*V* 900 mL), the vessel was evacuated, filled with helium, and evacuated again. The reagents were mixed, and the reaction mixture was stirred for 2 days at room temperature and 3 h at 60°C. Gaseous products were analyzed by GC method. Yield N_2 2.31, CO_2 0.41, N_2O 1.20 mmol.

c. The reaction was carried out as described in experiment *b*, 0.3 h at 60°C. Yield N_2 1.48, CO_2 0.27, N_2O 0.84 mmol.

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