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Synthesis of Polycyclic Functionally-substituted Triazole- and Tetrazole-containing Systems*

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Abstract—Cycloaddition of organic mono- and diazides to the triple bond of propargyl esters of mono- and dicarboxylic acids gave rise to polycyclic 1,2,3-triazole-substituted carboxylates. Dipropargyl dicarboxylates with diazides afforded oligomeric products with alternating triazole and carboxylate fragments. Reaction of a dipropargyl ester with 5-azidomethyltetrazole furnished a tetracyclic system with alternating triazole and tetrazole rings.

We reported in [1] on procedures for building up chains of polycyclic unfused triazole- and tetrazole-containing systems. In extension of these studies we describe in this article the building up of functionally-substituted triazole-containing compounds, among them polymers, where beside heterocycles carboxylates serve as linking elements.

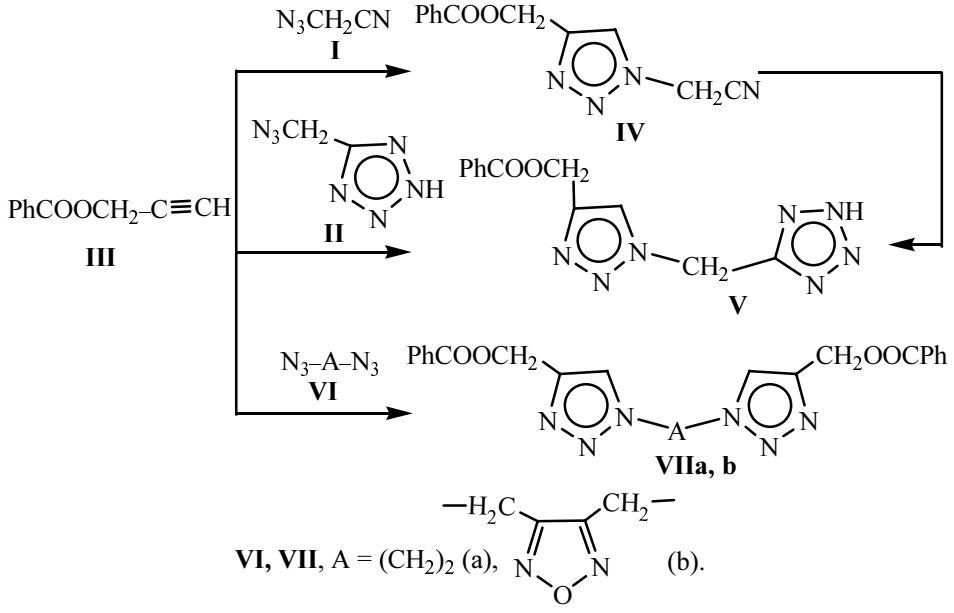
It was shown before that one way of building up a new ring in a polycyclic system consisted in incorporating a cyanomethyl group into the heterocyclic molecule thus leading to formation of a tetrazole fragment. Yet in the assembling of the functionally-substituted triazole-tetrazole blocks we stumbled upon difficulties in performing azidoacetonitrile cycloaddition to propargyl alcohol. Under the reaction conditions a secondary reaction presumably occurred between the cyano and hydroxy groups. At the same time the hydroxy group is a candidate for further building up of heterocycles. In this connection we applied as starting compounds those containing a hydroxy group protected by a benzoyl rest. It turned out that the benzoyl group considerably stabilized the acetylene molecule. For instance, cycloaddition of azidoacetonitrile (**I**) and 5-azidomethyltetrazole (**II**) to propargyl benzoate (**III**) occurred without tarring affording respectively 4-hydroxymethyl-1-cyanomethyl-1,2,3-triazole benzoate (**IV**) and (4-hydroxymethyl-1,2,3-triazol-1-yl)(tetrazol-5-yl)methane benzoate (**V**). Bicyclic compound **V** was also obtained from triazole (**IV**) and ammonium azide.

* For preceding communication see [1].

In cycloaddition of azides to the triple bond of acetylene compounds a formation of isomers is always possible. However as a rule reactions give rise predominantly to one of the isomers that can be successfully isolated by a fractional crystallization of the products.

The reaction of propargyl benzoate (**III**) with 1,2-diazidoethane (**VIa**) and 3,4-bis(azidomethyl)-1,2,5-oxadiazole (**VIb**) is also successful. This reaction affords functionally-substituted bistriazoles **VIIa, b**. In the ^{1}H NMR spectra of compounds **IV–VII** appear characteristic signals of protons attached to the heterocycle at 8.2–8.4 ppm, of protons in CH_2O groups at 5.4–5.5 ppm, protons of the phenyl moiety at 7.5–8.1 ppm, and also of the methylene protons at the nitrogen atom of the triazole ring whose chemical shifts vary in a wide range 4.95–6.15 ppm depending on the character of the adjacent group. The IR spectra of these compounds contain strong absorption bands in the region 1745–1710 cm^{-1} characteristic of ester groups, and also at 3140–3110 cm^{-1} corresponding to vibrations of the C–H bond of triazole rings.

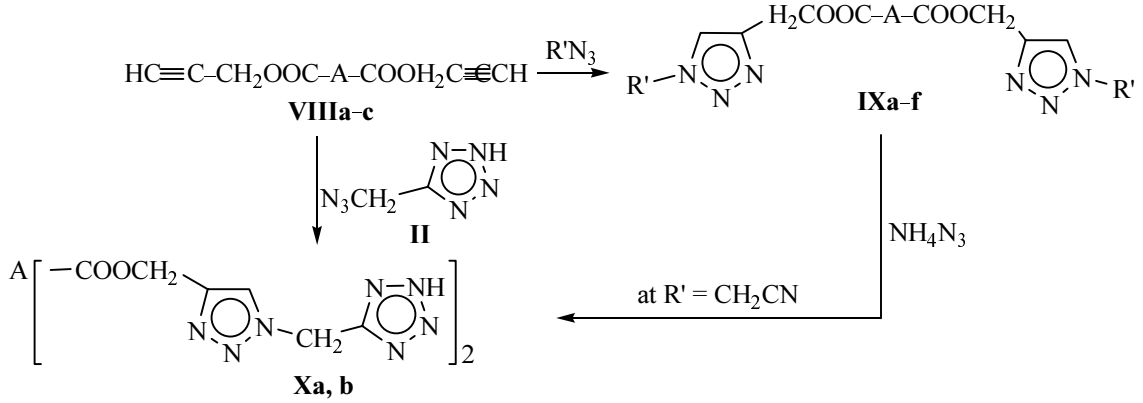
Preparation of polycyclic 1,2,3-triazoles **IX** and **X** is performed by reaction of azides with dipropargyl tetrphthalates, malonates, and oxalates (**VIIIa–c**). The reaction of benzyl azide and azidoacetonitrile with these diacetylene esters afforded esters of bis-4-hydroxymethyl-1(2)-substituted 1,2,3-triazoles (**IXa–f**). Thus obtained cyanomethyl-substituted bistriazole **IXc** by treatment with ammonium azide was converted into a tetracyclic compound **Xb** containing simultaneously



1,2,3-triazole and tetrazole rings. A similar compound **Xa** was prepared by adding 5-azidomethyltetrazole (**II**) to dipropargyl terephthalate (**VIIIa**). In the ^{1}H NMR spectra of compounds **IXb, c, e** only singlet signals were present corresponding to symmetric isomers. The chemical shifts of protons belonging to triazole ring were 8.2–8.4ppm, of protons from CH_2CN group 5.3–5.5 ppm, and of protons from CH_2O groups, 5.6–5.9 ppm. Besides in the spectrum of compound **IXb** a singlet is observed at 3.7 ppm corresponding to the protons of $CH_2(COO)_2$ moiety, and in the spectrum of compound **IXe** appeared a complex multiplet from the phenyl group protons at 7.15–7.25 ppm. The structure of compounds **IXa** and **Xa** is confirmed by the ^{13}C NMR spectra. As has been mentioned before [1], in the IR spectra of all *N*-cyanomethyl-substituted azoles obtained lacks the absorption band of the cyanomethyl group attached to the nitrogen of the triazole ring. The origin of this phenomenon is not yet clear.

All polycyclic compounds obtained containing ester groups in the IR spectra as expected had strong absorption bands in the region 1740–1710 cm^{-1}, weak bands at 1600–1590 cm^{-1} in case of the phenyl group presence, and also absorption bands characteristic of vibrations of C–H bonds in the triazole rings.

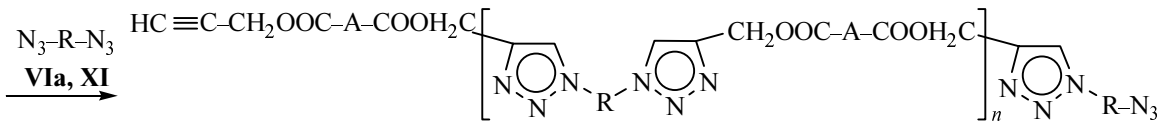
Reaction of diazides **VIa, c** with diacetylenes **VIIIa–c** involved polycycloaddition of diazides across the triple



VIII, A = 1,4-C_6H_4 (a), CH_2 (b), – (c); **IX**, A = 1,4-C_6H_4, R' = CH_2CN (**a**); A = CH_2, R' = CH_2CN (**b**); A = – , R' = CH_2CN, (**c**); A = 1,4-C_6H_4, R' = CH_2Ph (d); A = –, R' = CH_2Ph (**e**); A = CH_2, R' = CH_2Ph (f); R' = CH_2CN, A = 1,4-C_6H_4 (**a**), CH_2 (**b**), – (**c**); R' = CH_2Ph, A = 1,4-C_6H_4 (**d**), – (**e**), CH_2 (**f**); **X**, A = 1,4-C_6H_4 (**a**), – (**b**).

$$HC{\equiv}C{-}CH_2OOC{-}A{-}COOH_2C{-}C{\equiv}CH$$

VIIIa–c



XII

XI, R = (CH_2)–O–(CH_2); **XII**, A = –, R = $(CH_2)O(CH_2)$ (**a**); A = CH_2, R = $(CH_2)O(CH_2)$ (**b**); A = 1,4-C_6H_4, R = $(CH_2)_2$ (**c**); A = 1,4-CH_4, R = $(CH_2)O(CH_2)$ (**d**); n = 2–4.

bonds to afford triazole-containing oligomeric products in whose structure alongside triazole units were present carboxylate units and ether fragments.

In the IR spectra alongside strong absorption bands of carboxy groups in the region 1700 cm^{-1} and the band corresponding to triazole ring at 3100 cm^{-1} as weak absorp-tion is observed at 2100 cm^{-1} evidencing the presence in the polymers of a small quantity of terminal azide groups and acetylene bonds

Depending on the bridging fragment A the oligomers obtained are either viscous substances or yellow powders. The molecular weights of the oligomers measured by cryoscopy [2] (see table) show that the number of repeating structural units in the chain does not exceed four. The powdery compounds melt without decomposition and are soluble either in highly donor solvents (DMF) or highly acceptor ones (acetic acid).

EXPERIMENTAL

IR spectra of compounds synthesized were recorded on spectrophotometer Specord M-80 from mulls in mineral oil on CaF_2 plates. ^1H and ^{13}C NMR spectra of compounds dissolved in acetone-d_6 or DMSO-d_6 were registered on spectrometer Bruker VXR-500 S at operating frequencies 500 and 125.6 MHz respectively. The reaction progress was monitored by TLC on Silufol plates, eluent ethyl acetate–hexane, 2:3, spots visualized under UV irradiation or in iodine vapor.

Propargyl esters of dicarboxylic acids **VIIIa–c** were prepared by esterification of propargyl alcohol along known procedures..

1,2-Diazidoethane (**VIa**), 3,4-bis(azidomethyl)-1,2,5-oxadiazole (**VIb**), and 1,5-diazido-3-oxopentane (**XI**) were obtained by treating appropriate dichlorides with sodium azide under conditions of the phase-transfer catalysis with Et_3BnNCl as catalyst. Azidomethyltetrazole (**II**) was prepared by addition of sodium azide to azidoacetonitrile (**I**).

Azidoacetonitrile (I). To an emulsion of 53 g (0.7 mol) of chloroacetonitrile in 90 ml of water was added at vigorous stirring 52 g (0.8 mol) of sodium azide, and the mixture was slowly heated to 70°C; then the reaction mixture self-heated. It was maintained in the range 80–85°C by cooling for 2 h. The reaction mixture got dark. Then it was cooled to 20–25°C and poured into 50 ml of ethyl acetate. The organic layer was separated, the water layer was extracted with ethyl acetate, the extract was dried on $CaC1_2$. Vacuum distillation afforded 44.5 g (74%) of compound **I**, bp 70°C (17 mm Hg), n_d^{20} 1.4472.

5-Azidomethyltetrazole (II). A suspension of 1.95 g (30 mmol) of sodium azide and 3.3 g (30 mmol) of diethylamine hydrochloride in 10 ml of DMF was heated at stirring to 90°C. On cooling a solution of 1.96 g (24 mmol) of azidoacetonitrile (**I**) in 2 ml of DMF was added, and the reaction mixture was cautiously heated to 110°C and stirred at this temperature for 1 h. The precipitate was filtered off, the filtrate was evaporated

Properties of oligomers

Compd. no.	Yield, %	Appearance	mp, °C	[η], dl/g[a]	Molecular weight
XIIa	85	Viscous substance	–	0.02	–
XIIb	90	Viscous substance	–	0.01	600–800
XIIc	93	Powder	210–215	0.08	1200–1800
XIId	98	Powder	145–150	–	800–1000

[a] Intrinsic viscosity of oligomer solution in acetic acid at 20°C.

in a vacuum, the residue was poured into 60 ml of cold water, washed with ether, and acidified with hydrochloric acid to pH ~ 2. The acidified solution was extracted with ethyl acetate. The extract was evaporated in air, the residue was recrystallized from chloroform. Yield 2.5 g (89%), mp 56–57°C ($CHCl_3$). IR spectrum, cm^{-1}: 2120 (N_3). ^{13}C NMR spectrum, δ, ppm: 44.9 (1C, CH_2), 157.5 (1C, tetrazole NCN). ^1H NMR spectrum, δ, ppm: 4.91 s (2H, CH_2). Found, %: C 19.17; H 2.36; N 78.45. $C_2H_3N_7$. Calculated, %: C 19.20; H 2.40; N 78.40.

(1-Cyanomethyl-1,2,3-triazol-4-yl)methyl benzoate (IV). A mixture of 5 g (30 mmol) of propargyl benzoate (**III**) and 3.28 g (40 mmol) of azidoacetonitrile (**I**) in 10 ml of ethanol was stirred at reflux for 7 h, the solvent was removed in air. The residue was recrystallized from ethanol. Yield 7 g (93%), mp 104–105°C (EtOH). IR spectrum, cm^{-1}: 3150 (C–H of triazole), 1740 (OCO), 1590 (Ph). ^1H NMR spectrum, δ, ppm: 5.46 s (2H, CH_2CN), 5.77 s (2H, CH_2O), 7.50–8.02 m (5H, Ph), 8.33 s (1H, CH of triazole). Found, %: C 59.47; H 4.10; N 23.11. $C_{12}H_{10}O_2N_4$. Calculated, %: C 59.50; H 4.13; N 23.14.

[1-(2-Tetrazole-5-ylmethyl)triazole-4-yl]methyl benzoate (V). (a) A mixture of 1.32 g (80 mmol) of propargyl benzoate (**III**) and 1 g (80 mmol) of 5-azidomethyltetrazole (**II**) in 10 ml of ethanol was heated at reflux for 16.5 h. The reaction mixture was evaporated in air, and the residue was recrystallized from a mixture ethanol–water, 1:1 to get 2.3 g (98%) of lustrous plate-like crystals, mp 152–154°C (EtOH/H_2O). IR spectrum, cm^{-1}: 3150 (C–H of triazole), 1710 (OCO), 1590 (Ph). ^1H NMR spectrum, δ, ppm: 5.47 s (2H, CH_2O), 6.16 s (2H, NCH_2), 7.50–8.05 m (5H, Ph), 8.32 s (1H, CH of triazole). Found, %: C 50.47; H 4.00; N 34.32. $C_{12}H_{11}O_2N_7$. Calculated, %: C 50.52; H 3.86; N 34.39.

(b) A suspension of 0.56 g (9 mmol) of sodium azide and 0.48 g (9 mmol) of ammonium chloride was dissolved at stirring and heating in 10 ml of DMF, then the solution was cooled, and 2 g (8 mmol) of compound **IV** was added thereto. The mixture was stirred for 14 h at 120°C. The reaction mixture was cooled, the precipitate was filtered off, the filtrate was evaporated in a vacuum. The residue after removal of solvent was poured into water, washed with ether, acidified with hydrochloric acid till pH ~ 2, then the solution was extracted with ethyl acetate, and the extract was evaporated to dryness in a vacuum. The dry residue was twice recrystallized from ethanol. Yield 2.6 g (74%), all physical and spectral characteristics are the same as of compound **V** prepared by procedure (a).

1,2-Diazidoethane (VIa). A dispersion of 24.4 g (0.13 mol) of 1,2-dibromoethane, 17.5 g (0.27 mol) of sodium azide, and 0.5 g of Et_3BnNCl in 30 ml of water was vigorously stirred for 3 h at 90–95°C. The water lauer was extracted with ether, the extract was dried with $CaCl_2$, the ether was removed in air and then in a vacuum. The residue (10.3 g) was further used without distillation because of explosion hazard.. IR spectrum, cm^{-1}: 2145 (N_3).

1,2-Bis(4-benzoyloxymethyl-1,2,3-triazol-1-yl)ethane (VIIa). A mixture of 6.56 g (40 mmol) of propargyl benzoate (**III**), 2.24 g (20 mmol) of 1,2-diazidoethane (**VIa**) in 15 ml of ethanol was heated at reflux for 16 h. The solvent was filtered off from the crystallized reaction mixture, the residue was recrystallized from ethanol to isolate 2.1 g (22%) of compound **VIIa**, mp 139–140°C (EtOH). IR spectrum, cm^{-1}: 3140 (C–H of triazole), 1710 (OCO), 1590 (Ph). ^1H NMR spectrum, δ, ppm: 4.92 s (4H, $2CH_2O$), 5.34 s (4H, NCH_2CH_2N), 7.5–8.0 m (10H, 2Ph), 8.12 s (2H, 2CH of triazole). Found, %: C 61.14; H 4.60; N 19.48. $C_{23}H_{20}O_4N_6$. Calculated, % : C 61.11; H 4.63; N 19.44.

4,5-Bis(4-benzoyloxymethyl-1,2,3-triazol-1-ylmethyl)furazan (VIIb). To a solution of 13 g (0.1 mol) of 3,4-bis(hydroxymethyl)-1,2,5-oxadiazole in 20 ml of benzene was added dropwise 14 g (0.12 mol) of thionyl chloride in 10 ml of benzene. The reaction mixture was stirred for 3 h at 20–25°C, and then excess thionyl chloride and solvent were distilled off in a vacuum of a water-jet pump. The residue was added dropwise at vigorous stirring to a suspension 6.5 g (0.1 mol) of sodium azide and 0.3 g of Et_3BnNCl in 12 ml of water. The reaction mixture was stirred for 3 h at 45°C, then 12 ml of cold water was added, and the organic layer was treated with ether. The extract was evaporated in a vacuum to afford 8 g of dark-brown residue of diazide **VIb** that decomposed at attempted distillation in a vacuum. IR spectrum, cm^{-1}: 2140 (N_3). A solution of 6.56 g (40 mmol) of propargyl benzoate (**III**) and 2.24 g (20 mmol) of diazide **VIb** in 15 ml of ethanol was heated at reflux for 16 h. On cooling the reaction mixture was filtered, the residue was recrystallized from ethanol to isolate one isomer of compound **VIIb** in amount of 2.1 g (22%), mp 139–140°C (EtOH). IR spectrum, cm^{-1}: 3140 (C–H of triazole), 1710 (OCO), 1590 (Ph). ^1H NMR spectrum, δ, ppm: 4.92 s (4H, $2CH_2O$), 5.34 s (4H, NCH_2CH_2N), 7.5–8.0 m (10H, 2Ph), 8.12 s (2H, 2CH of

triazole). Found, %: C 61.14; H 4.60; N 19.48. $C_{22}H_{20}O_4N_6$. Calculated, %: C 61.11; H 4.63; N 19.44.

Bis[(1-cyanomethyl-1,2,3-triazol-4-yl)methyl] terephthalate (IXa). A mixture of 2 g (8 mmol) of propargyl terephthalate (**VIIIa**) and 1.47 g (18 mmol) of azidoacetonitrile (**I**) in 15 ml of ethanol was heated at reflux and stirring for 18–20 h. The separated precipitate was filtered off, and washed with cold ethanol. Yield 0.9 g (27%), mp 132°C (EtOH). IR spectrum, cm^{-1}: 3125 (C–H of triazole ring), 1720 (OCO), 1230, 1100, 1050 (triazole ring). ^{13}C NMR spectrum, δ, ppm: 36.6 (2C, CH_2 at CN), 57.3 (2C, CH_2 at OCO), 114.1 (2C, 2CN), 125.2 (2C, 2CH of triazole), 128.9 and 132.6 ($C^{1,2}$ arom), 141.6 (2C, 2C–CH_2 of triazole), 164.0 (2C, 2OCO). Found, %: C 53.17; H 3.47; N 27.61. $C_{18}H_{14}O_4N_8$. Calculated, %: C 53.20; H 3.45; N 27.59.

Likewise were synthesized compounds **IXb–f**.

Bis[(1-cyanomethyl-1,2,3-triazol-4-yl)methyl] malonate (IXb) was obtained from a mixture of 3 g (17 mmol) of propargyl malonate (**VIIIb**) and 3 g (37 mmol) of azidoacetonitrile (**I**) in 20 ml of ethanol. Yield 1.29 g (23%), mp 170–172°C (EtOH). IR spectrum, cm^{-1}: 3130 (C–H of triazole), 1725 (OCO). ^1H NMR spectrum, δ, ppm: 5.25 s (4H, $2CH_2CN$), 5.9 s (4H, $2CH_2OCO$), 3.65 s [2H, $CH_2(COO)_2$], 8.3 s (2H, 2CH of triazole). Found, %: C 45.87; H 3.44; N 32.02. $C_{13}H_{12}O_4N_8$. Calculated, %: C 45.35; H 3.49; N 32.56.

Bis[(1-cyanomethyl-1,2,3-triazol-4-yl)methyl oxalate (IXc) was obtained from a mixture of 3 g (18 mmol) of propargyl oxalate (**VIIIc**) and 3.3 g (39 mmol) of azidoacetonitrile (**I**) in 20 ml of ethanol. Yield 2.06 g (35%), mp 197–199°C (EtOH). IR spectrum, cm^{-1}: 3130 (C–H of triazole), 1745 (OCO). ^1H NMR spectrum, δ, ppm: 5.4 s (4H, $2CH_2CN$), 5.85 s (4H, $2CH_2OCO$), 8.4 s (2H, 2CH of triazole). Found, %: C 43.94; H 3.05; N 33.15. $C_{12}H_{10}O_4N_8$. Calculated, %: C 43.64; H 3.03; N 33.94.

Bis[(1-benzyl-1,2,3-triazol-4-yl)methyl] terephthalate (IXd) was obtained from a mixture of 1 g (4.1 mmol) of propargyl terephthalate (**VIIIa**) and 1.09 g (8.3 mmol) of benzyl azide in 10 ml of ethanol. Yield 0.83 g (39.7%), mp 170–172°C (EtOH), sparingly soluble in organic solvents.. IR spectrum, cm^{-1}: 3130 (C–H of triazole), 1710 (OCO), 1590 (Ph). Found, %: C 66.62; H 4.75; N 16.05. $C_{28}H_{24}O_4N_6$. Calculated, %: C 66.14; H 4.72; N 16.54.

Bis[(1-benzyl-1,2,3-triazol-4-yl)methyl] oxalate (IXe) was obtained from a mixture of 3 g (18 mmol) of propargyl oxalate (**VIIIc**) and 4.8 g (36 mmol) of benzyl azide in 15 ml of ethanol. Yield 0.62 g (11%), mp 167–170°C (EtOH). IR spectrum, cm^{-1}: 3130 (C–H of triazole), 1745 (OCO), 1590 (Ph). ^1H NMR spectrum, δ, ppm: 5.37 s (4H, $2CH_2$–Ph), 5.6 s (2H, $2CH_2OCO$), 7.3–7.5 m (10H, 2Ph), 8.3 s (2H, 2CH of triazole). Found, %: C 60.74; H 4.54; N 19.71. $C_{22}H_{20}O_4N_6$. Calculated, %: C 61.11; H 4.63; N 19.44.

Bis[(1-benzyl-1,2,3-triazol-4-yl)methyl] malonate (IXf) was obtained from a mixture of 2 g (11 mmol) of propargyl malonate (**VIIIb**) and 2.93 g (22 mmol) of benzyl azide in 15 ml of ethanol. Yield 1.88 g (38%) of noncrystallizable oil.. IR spectrum, cm^{-1}: 3125 (C–H of triazole), 1745 (OCO), 1590 (Ph).

Bis{[1-(tetrazol-5-ylmethyl)-1,2,3-triazol-4-yl]methyl} terephthalate (Xa). A mixture of 0.97 g (4 mmol) of ester **VIIIa** and 1 g (8.5 mmol) of azide **II** in 10 ml of ethanol was heated at reflux for 10.5 h. The separated precipitate was filtered off and washed with cold ethanol. We obtained 0.8 g (41%) of light crystals of mp 239°C (EtOH). IR spectrum, cm^{-1}: 3120 (C–H of triazole), 1710 (OCO). ^{13}C NMR spectrum, δ, ppm: 42.2 (2C, $2CH_2N$), 57.5 (2C, $2CH_2OCO$), 125.3 (2C, 2CH of triazole), 129.0 and 132.7 ($C^{1,2}$ arom), 141.1 (2C, 2C–CH_2O of triazole), 152.9 (2C, 2NCN of tetrazole ring), 164.1 (2C, 2OCO). Found, %: C 43.65; H 3.38; N 39.98. $C_{18}H_{16}O_4N_{14}$. Calculated, %: C 43.90; H 3.25; N 39.84.

Bis{[1-(tetrazol-5-ylmethyl)-1,2,3-triazol-4-yl]-methyl} oxalate (Xb). A suspension of 0.8 g (12 mmol) of sodium azide and 0.64 g (12 mmol) of ammonium chloride was dissolved at stirring and heating to 90–95°C in 7 ml of DMF, then the solution was cooled, and 2 g (6 mmol) of compound **IXc** was added. The reaction mixture was stirred for 25 h at 120°C. The precipitate was filtered off, the filtrate was evaporated in a vacuum. The concentrated water solution was washed with ether, poured into 30 ml of cold water, acidified with hydrochloric acid to pH 2, the separated precipitate was filtered off and washed with ethanol. Yield 0.68 g (27%), mp 210°C (subl.). IR spectrum, cm^{-1}: 3110 (C–H of triazole), 1710 (OCO). ^1H NMR spectrum, δ, ppm: 5.7 dC (4H, $2CH_2$ between heterocycles), 5.8 dC (4H, $2CH_2O$), 8.4 dC (2H, CH of triazole). Found, %: C 34.64; H 3.94; N 47.17. $C_{12}H_{12}O_4N_{14}$. Calculated, %: C 34.62; H 2.88; N 47.12.

Polycycloaddition of diazides VIa and XI to diacetylene compounds VIIIa–c. A mixture of 8 mmol of diacetylene compound **VIIIa–c** and 8 mmol of diazide

VIa or **XI** in 10 ml of DMF was maintained for 16 h at 90–95°C. On cooling the reaction mixture was poured into 100 ml of ether. From the separated precipitate (viscous substance) the solvent was decanted, and the product was several times washed with ether. The isolated oligomer was dried in a vacuum till constant weight.

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