Conjugation of aminoadamantanes by copper-catalyzed alkyne-azide 1,3-dipolar cycloaddition

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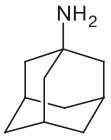
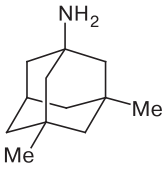
Four variants of conjugation of aminoadamantanes with 1,2,3-triazole- and ditriazole-containing spacers by copper-catalyzed alkyne-azide 1,3-dipolar cycloaddition of azido- and propargyl-containing aminoadamantanes were suggested.

Key words: 1-aminoadamantanes, *N*-propynylaminoadamantanes, *N*-(adamantan-1-yl)-2-azidoacetamides, terminal diazidoalkanes, 1,4-dipropynylpiperazine, 1,4-substituted 1,2,3-triazoles, 1,3-dipolar cycloaddition, conjugates.

The rational design of known pharmaceutical drugs is one of the possible approaches to the development of drugs affecting several targets.[1] From this perspective, in this work we present the data on synthetic approaches to the combination of adamantan-1-ylamines in one molecule. The study was prompted by the data on the biological activity of aminoadamantanes and their derivatives. Thus, adamantanylamine (amantadine) is used as an antiviral[2] and antiparkinsonian dopaminergic agent,[3] 3,5-dimethyladamantan-1-ylamine (memantine) is one of the main drugs used in the treatment of Alzheimer's disease,[4] and its derivatives modified at both the amino group[5—8] and the adamantane core[9] are also intensively studied in the last years.



Amantadine

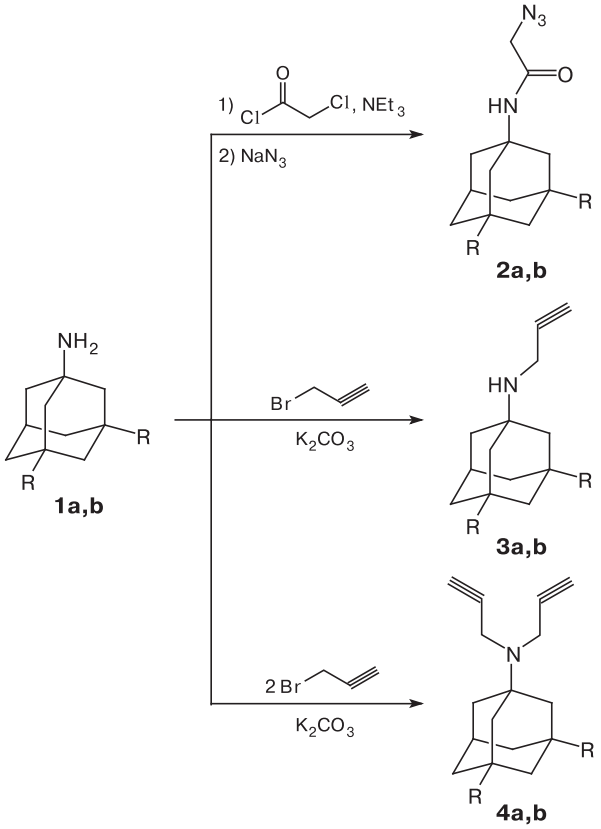


Memantine

The purpose of the present work is the studies of synthetic potential of copper-catalyzed alkyne-azide cycloaddition, which is one of the click-chemistry standards,[10,11] for the combination of azido- and propargyl-containing aminoadamantanes by triazole- and ditriazole-containing spacers.

The starting objects of the transformations, *N*-(adamantan-1-yl)-2-azidoacetamides (**2a**,**b**), were obtained using a *one-pot* method, namely, by sequential addition to a solution of aminoadamantanes **1a**,**b** and Et_3N in DMF of an equimolar amount of chloroacetyl chloride and, once the exothermic reaction reached completion, sodium azide was added with further heating at 50 °C for 30 min to complete the reaction. *N*-(Prop-2-yn-1-yl)adamantan-1-amines **3a**,**b** and *N*,*N*-di(prop-2-yn-1-yl)adamantan-1-amines **4a**,**b** were formed in 74—84% yield upon heating aminoadamantanes **1a**,**b** and propargyl bromide in Pr^iOH in the presence of three equivalents of K_2CO_3 (Scheme 1).

Scheme 1

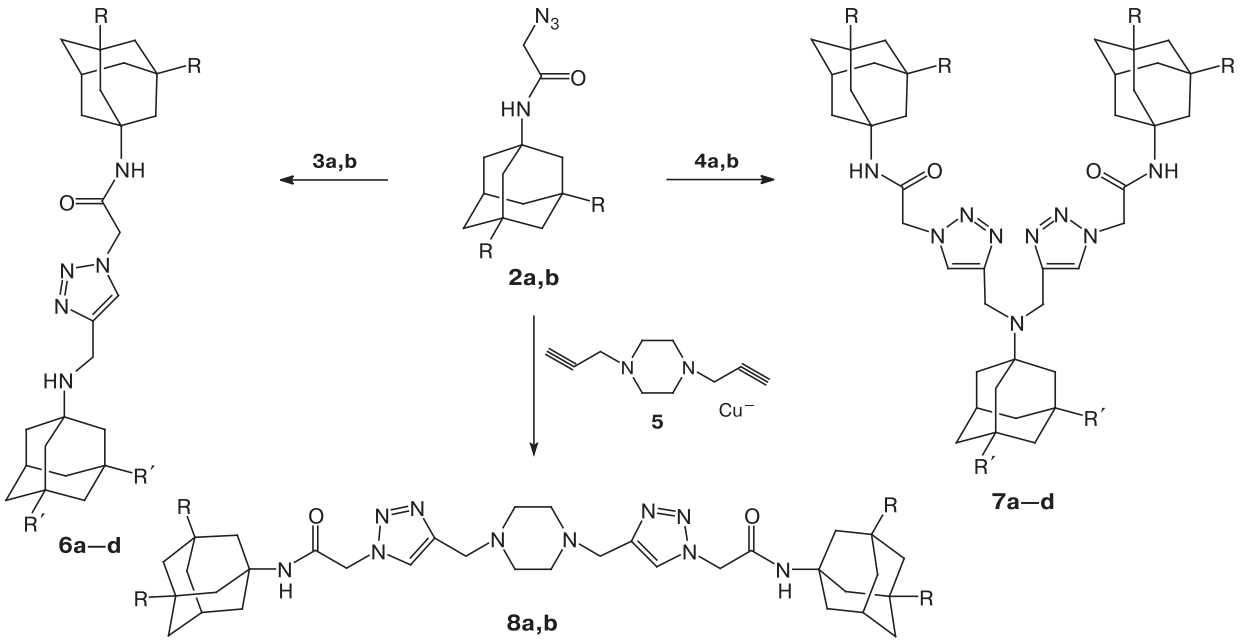


1—4: R = H (**a**), Me (**b**)

Published in Russian in *Izvestiya Akademii Nauk. Seriya Khimicheskaya,* No. 8, pp. 1401—1405, August, 2018.

1066-5285/18/6708-1401

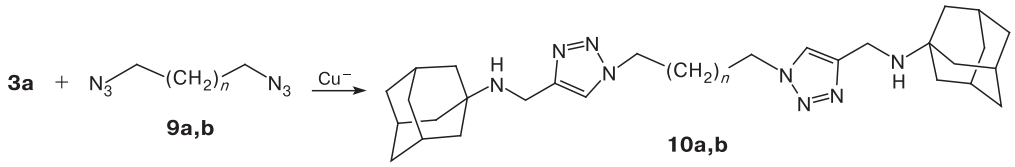
Scheme 2



6, 7: R = H, R′ = H (**a**); Me (**b**); R = Me, R′ = H (**c**); Me (**d**)
8: R = H (**a**), Me (**b**)

Conditions: $CuSO_4 \cdot 5\ H_2O$, CH_2Cl_2, sodium ascorbate, 40 °C.

Scheme 3



10: *n* = 0 (**a**), 2 (**b**)

The thus obtained azidoacetamides **2a,b** were studied in the cycloaddition reaction with propargyladamantanes **3a,b** and **4a,b** and 1,4-di(prop-2-yn-1-yl)piperazine (**5**) (Scheme 2). It was shown that azidoacetamides **2a,b** in the presence of catalytic amounts of Cu^I under phase-transfer conditions (dichloromethane—water) react with compounds **3a,b**, **4a,b**, and **5** to form the corresponding 1,2,3-triazoles **6a—d**, **7a—d**, and **8a,b** in high yield. In the ^{1}H NMR spectra of triazoles **6a—d**, **7a—d**, and **8a,b**, representing the superposition of the aminoadamantane fragments, characteristic singlets for the proton of the triazole ring are observed in the region of δ 6.8—7.3.

Under similar conditions, *N*-(prop-2-yn-1-yl)adamantan-1-amine (**3a**) reacted with terminal diazidoalkanes **9a**,**b** to form the corresponding conjugates **10a**,**b** in 83 and 87% yield (Scheme 3).

The ^{1}H NMR spectra of conjugates **10a**,**b** contain the singlets for the proton of the triazole ring in the region of δ 6.8—7.3 and the multiplets for the protons of the spacer methylene groups.

In conclusion, we suggested four variants for the combination of aminoadamantanes in one molecule by triazole- and ditriazole-containing spacers, using the click-reaction of 1,3-dipolar cycloaddition, allowing obtaining a variety of potential multitarget neuroprotectors.

Experimental

^{1}H NMR spectra were recorded on a Bruker DPX 200 spectrometer at 200.13 MHz relative to $SiMe_4$ (an internal standard). Melting points were measured in a glass capillary tube. 1,4-Di-(prop-2-yn-1-yl)piperazine (**5**) was obtained according to the

described procedure,[12] diazidoalkanes **9a**,**b** (see Ref. 13), aminoadamantanes **1a**,**b**, copper sulfate and sodium ascorbate (Aldrich) were used without additional purification.

Synthesis of *N*-(adamantan-1-yl)-2-azidoacetamide (2a) and 2-azido-*N*-(3,5-dimethyladamantan-1-yl)acetamide (2b) (general procedure). Chloroacetyl chloride (0.2 mmol) was added to a solution of aminoadamantane **1a,b** (0.2 mmol) and Et_3N (0.2 mmol) in DMF (30 mL) with stirring, the reaction mixture was stirred for 30 min, followed by the addition of NaN_3 (0.21 mmol), stirring for 30 min at 50 °C, and pouring into H_2O (100 mL). The precipitate formed was collected by filtration and recrystallized from 50% ethanol.

Synthesis of *N*-(prop-2-yn-1-yl)adamantan-1-amine (3a) and 3,5-dimethyl-*N*-(prop-2-yn-1-yl)adamantan-1-amine (3b) (general procedure). Potassium carbonate (0.6 mmol) was added to a solution of aminoadamantane **1a**,**b** (0.2 mmol) and propargyl bromide (0.2 mmol) in Pr^iOH (30 mL). The mixture was stirred for 5 h at 70 °C, cooled, poured into H_2O (100 mL), extracted with chloroform, and dried with Na_2SO_4. The solvent was evaporated *in vacuo*, the residue was purified by column chromatography on silica gel (chloroform—hexane, 5 : 1).

***N*,*N*-Di(prop-2-yn-1-yl)adamantan-1-amine (4a) and 3,5-dimethyl-*N*,*N*-di(prop-2-yn-1-yl)adamantan-1-amine (4b)** were obtained similarly to **3a**,**b** from aminoadamantane **1a**,**b** (0.2 mmol) and propargyl bromide (0.4 mmol).

Synthesis of *N*-(adamantan-1-yl)-2-{4-[(adamantan-1-ylamino)methyl]-1*H*-1,2,3-triazol-1-yl}acetamide (6a), *N*-(adamantan-1-yl)-2-{4-[(3,5-dimethyladamantan-1-ylamino)methyl]-1*H*-1,2,3-triazol-1-yl}acetamide (6b), 2-{4-[(adamantan-1-ylamino)methyl]-1*H*-1,2,3-triazol-1-yl}-*N*-(3,5-dimethyladamantan-1-yl)acetamide (6c), *N*-(3,5)-dimethyladamantan-1-yl)-2-{4-[(3,5-dimethyladamantan-1-ylamino)methyl]-1*H*-1,2,3-triazol-1-yl}acetamide (6d) (general procedure). The corresponding *N*-propenyaminoadamantane **3a,b** (0.1 mmol), $CuSO_4 \cdot 5H_2O$

Table 1. Yields, melting points, and elemental analysis data for compounds **2a**,**b**, **3a**,**b**, **4a**,**b**, **6a—d**, **7a—d**, **8a**,**b**, and **10a**,**b**

Compound	Yield (%)	M.p./°C	Found / Calculated (%)			Molecular formula
			C	H	N	
2a	91	96—98	61.31 61.52	7.93 7.74	24.12 23.91	$C_{12}H_{18}N_4O$
2b	88	83—85	64.28 64.09	8.66 8.45	21.07 21.36	$C_{14}H_{22}N_4O$
3a	76	56—57	82.27 82.48	10.30 10.12	7.22 7.40	$C_{13}H_{19}N$
3b	74	An oil	83.11 82.89	10.48 10.67	6.29 6.44	$C_{15}H_{23}N$
4a	82	85—84	84.71 84.53	9.50 9.31	6.05 6.16	$C_{16}H_{21}N$
4b	84	An oil	84.82 84.65	10.05 9.87	5.31 5.48	$C_{18}H_{25}N$
6a	80	118—120	70.71 70.89	8.68 8.80	6.72 6.53	$C_{25}H_{37}N_5O$
6b	81	111—113	72.03 71.80	8.99 9.15	15.72 15.51	$C_{27}H_{41}N_5O$
6c	85	102—103	71.63 71.80	9.33 9.15	15.33 15.51	$C_{27}H_{41}N_5O$
6d	86	95—97	72.43 72.61	9.62 9.46	14.45 14.60	$C_{29}H_{45}N_5O$
7a	81	267—269	68.82 69.03	8.06 8.26	18.30 18.11	$C_{40}H_{57}N_9O_2$
7b	88	251—252	69.49 69.68	8.27 8.49	17.63 17.41	$C_{42}H_{61}N_9O_2$
7c	91	244—245	70.11 70.27	8.56 8.71	16.52 16.76	$C_{44}H_{65}N_9O_2$
7d	87	233—235	71.08 70.82	9.11 8.92	16.34 16.16	$C_{46}H_{69}N_9O_2$
8a	86	162—164	64.92 64.73	8.17 7.99	22.41 22.20	$C_{34}H_{50}N_{10}O_2$
8b	85	137—139	66.22 66.44	8.70 8.51	20.21 20.39	$C_{38}H_{58}N_{10}O_2$
10a	83	140—142	68.69 68.54	8.84 8.63	22.66 22.84	$C_{28}H_{42}N_8$
10b	87	146—147	69.27 69.46	9.15 8.94	21.79 21.60	$C_{30}H_{46}N_8$

Table 2. ^{1}H NMR spectra of compounds **2a**,**b**, **3a**,**b**, **4a**,**b**, **5a—d**, **6a—d**, **8a**,**b**, and **10a**,**b**

Compound	δ (*J*/Hz)
2a	1.69 (s, 6 H, $C_{Ad}H_2$); 2.01 (s, 6 H, $C_{Ad}H_2$); 2.09 (br.s, 3 H, $C_{Ad}H$); 3.87 (s, 2 H, CH_2N); 5.94 (s, 1 H, NH)
2b	0.86 (s, 6 H, Me); 1.16 (s, 2 H, $C_{Ad}H_2$); 1.22—1.48 (m, 4 H, $C_{Ad}H_2$); 1.55—1.76 (m, 4 H, $C_{Ad}H_2$); 1.84 (s, 2 H, CH_2); 2.16 (septet, 1 H, $C_{Ad}H$, $J_{H,H}$ = 3.0); 3.85 (s, 2 H, CH_2N); 5.78 (s, 1 H, NH)
3a	1.52—1.78 (m,13 H, NH + $C_{Ad}H_2$); 2.01—2.17 (m, 3 H, $C_{Ad}H$); 2.19 (t, 1 H, CH, $J_{H,H}$ = 2.5); 3.41 (d, 2 H, CH_2N, $J_{H,H}$ = 2.5)
3b	0.84 (s, 6 H, Me); 1.08—1.15 (m, 2 H, $C_{Ad}H_2$); 1.21—1.33 (m, 4 H, $C_{Ad}H_2$); 1.35 (s, 1 H, NH); 1.46—1.52 (m, 4 H, $C_{Ad}H_2$); 1.80 (s, 2 H, CH_2); 2.14 (septet, 1 H, $C_{Ad}H$, $J_{H,H}$ = 3.1); 2.19 (t, 1 H, CH, $J_{H,H}$ = 2.5); 3.41 (d, 2 H, CH_2N, $J_{H,H}$ = 2.5)
4a	1.63 (s, 6 H, $C_{Ad}H_2$); 1.83 (s, 6 H, $C_{Ad}H_2$); 2.08 (br.s, 3 H, $C_{Ad}H$); 2.21 (t, 2 H, CH, $J_{H,H}$ = 2.3); 3.69 (d, 4 H, CH_2N, $J_{H,H}$ = 2.3)
4b	0.87 (s, 6 H, Me); 1.08—1.17 (m, 2 H, $C_{Ad}H_2$); 1.25—1.35 (m, 4 H, $C_{Ad}H_2$); 1.36—1.56 (m, 4 H, $C_{Ad}H_2$); 1.8—34 (s, 2 H, CH_2); 2.17 (septet, 1 H, $C_{Ad}H$, $J_{H,H}$ = 3.2); 2.22 (t, 2 H, CH, $J_{H,H}$ = 2.0); 3.70 (d, 4 H, CH_2N, $J_{H,H}$ = 2.0)
6a	1.60—1.75 (m, 18 H, $C_{Ad}H_2$); 1.98 (s, 6 H, $C_{Ad}H_2$); 2.05—2.20 (m, 7 H, $C_{Ad}H$ + NH); 3.99 (s, 2 H, $CH_2C(O)$); 4.97 (s, 2 H, $\underline{CH_2}NH$); 5.91 (s, 1 H, NHC(O)); 7.71 (s, 1 H, CH=)
6b	0.86 (s, 6 H, Me); 1.14 (s, 2 H, $C_{Ad}H_2$); 1.24—1.47 (m, 8 H, $C_{Ad}H_2$); 1.52—1.72 (m, 8 H, $C_{Ad}H_2$); 1.90—2.10 (m, 11 H, $C_{Ad}H_2$ + $C_{Ad}H$); 3.93 (s, 2 H, $CH_2C(O)$); 4.96 (s, 2 H, $\underline{CH_2}NH$); 6.30 (s, 1 H, NHC(O)); 7.71 (s, 1 H, CH=)
6c	0.83 (s, 6 H, Me); 1.13 (s, 2 H, $C_{Ad}H_2$); 1.20—1.41 (m, 4 H, $C_{Ad}H_2$); 1.47—1.84 (m, 18 H, $C_{Ad}H$ + $C_{Ad}H_2$); 2.05—2.18 (m, 4 H, $C_{Ad}H_2$); 2.27 (s, 1 H, NH); 3.95 (s, 2 H, $CH_2C(O)$); 4.92 (s, 2 H, $\underline{CH_2}NH$); 5.95 (s, 1 H, NHC(O)); 7.66 (s, 1 H, CH=)
6d	0.83 (s, 6 H, Me); 0.87 (s, 6 H, Me); 1.14 (s, 4 H, $C_{Ad}H_2$); 1.24—1.47 (m, 12 H, $C_{Ad}H_2$); 1.49—1.70 (m, 5 H, NH + $C_{Ad}H_2$); 1.74—1.82 (m, 2 H, $C_{Ad}H_2$); 2.09—2.20 (m, 4 H, $C_{Ad}H_2$ + $C_{Ad}H$); 3.94 (s, 2 H, $CH_2C(O)$); 4.92 (s, 2 H, $\underline{CH_2}NH$); 5.93 (s, 1 H, NHC(O)); 7.65 (s, 1 H, CH=)
7a	1.50—1.77 (m, 22 H, $C_{Ad}H_2$); 1.85—2.15 (m, 23 H, $C_{Ad}H_2$ + $C_{Ad}H$); 3.81 (s, 4 H, $CH_2C(O)$); 4.97 (s, 4 H, $\underline{CH_2}N$); 7.77 (s, 2 H, CH=); 7.86 (s, 2 H, NHC(O))
7b	0.84 (s, 6 H, Me); 1.14 (s, 2 H, $C_{Ad}H_2$); 1.21—1.53 (m, 8 H, $C_{Ad}H_2$); 1.57—1.73 (m, 21 H, $C_{Ad}H_2$); 1.85—2.15 (m, 12 H, $C_{Ad}H_2$ + $C_{Ad}H$); 3.97 (s, 4 H, $CH_2C(O)$); 4.92 (s, 4 H, $\underline{CH_2}N$); 5.82 (s, 2 H, NHC(O)); 7.65 (s, 2 H, CH=)
7c	0.81 (s, 12 H, Me); 1.10 (s, 4 H, $C_{Ad}H_2$); 1.18—1.39 (m, 8 H, $C_{Ad}H_2$); 1.57—1.80 (m, 23 H, $C_{Ad}H_2$); 1.95—2.13 (m, 6 H, $C_{Ad}H_2$ + $C_{Ad}H$); 3.62 (s, 4 H, $CH_2C(O)$); 4.65 (s, 4 H, $\underline{CH_2}N$); 7.77 (s, 2 H, CH=); 7.87 (s, 2 H, NHC(O))
7d	0.81 (s, 18 H, Me); 1.10 (s, 6 H, $C_{Ad}H_2$); 1.17—1.46 (m, 12 H, $C_{Ad}H_2$); 1.48—1.80 (m, 16 H, $C_{Ad}H_2$); 2.00—2.13 (m, 5 H, $C_{Ad}H_2$ + $C_{Ad}H$); 3.61 (s, 4 H, $CH_2C(O)$); 4.95 (s, 4 H, $\underline{CH_2}N$); 7.76 (s, 2 H, CH=); 7.88 (s, 2 H, NHC(O))
8a	1.51—1.70 (m, 12 H, $C_{Ad}H_2$); 1.80—2.10 (m, 18 H, $C_{Ad}H_2$ + $C_{Ad}H$); 2.20—2.56 (m, 8 H, $N\underline{CH_2}CH_2$); 3.50 (s, 4 H, $CH_2C(O)$); 4.94 (s, 4 H, $\underline{CH_2}N$); 7.83 (s, 4 H, CH= + NHC(O))
8b	0.81 (s, 12 H, Me); 1.10 (s, 4 H, $C_{Ad}H_2$); 1.17—1.46 (m, 8 H, $C_{Ad}H_2$); 1.48—1.80 (m, 12 H, $C_{Ad}H_2$); 2.00—2.13 (m, 2 H, $C_{Ad}H$); 2.18—2.54 (m, 8 H, $N\underline{CH_2}CH_2$); 3.50 (s, 4 H, $CH_2C(O)$); 4.93 (s, 4 H, $\underline{CH_2}N$); 7.78 (s, 2 H, CH=); 7.83 (s, 2 H, NHC(O))
10a	1.53—1.82 (m, 18 H, $C_{Ad}H_2$); 1.86—2.17 (m, 14 H, NH + $C_{Ad}H$ + $C_{Ad}H_2$); 3.85 (s, 4 H, CH_2CH_2); 4.86 (s, 4 H, $\underline{CH_2}NH$); 7.30 (s, 2 H, CH=)
10b	1.54—1.84 (m, 22 H, $NCH_2\underline{CH_2}$ + $C_{Ad}H_2$); 1.86—2.01 (m, 6 H, $C_{Ad}H_2$); 2.01—2.20 (m, 8 H, NH + $C_{Ad}H$); 3.85 (s, 4 H, $N\underline{CH_2}CH_2$); 4.86 (s, 4 H, $\underline{CH_2}NH$); 7.30 (s, 2 H, CH=)

(0.002 g, 0.01 mmol) in water (0.5 mL), and sodium ascorbate (0.008 g, 0.04 mmol) in water (0.25 mL) were added to a solution of azidoacetamide **2a**,**b** (0.1 mmol) in dichloromethane (20 mL) with vigorous stirring. The reaction mixture was stirred for 7 h at 40 °C, cooled to room temperature, diluted with dichloromethane (10 mL), and washed with 2% aqueous ammonia (10 mL) and water. The organic layer was separated and dried with Na_2SO_4, The solvent was evaporated *in vacuo*, the residue was subjected to chromatography on silica gel (60 mesh, eluent methanol—chloroform, 1 : 10).

2,2′-{4,4′-[(Adamantan-1-ylazanediyl)bis(methylene)bis-(1*H*-1,2,3-triazole-4,1-diyl)]bis(*N*-adamantan-1-yl)acetamide} (7a), 2,2′-{4,4′-[(3,5-dimethyladamantan-1-ylazanediyl)bis-(methylene)bis(1*H*-1,2,3-triazole-4,1-diyl)]bis(*N*-adamantan-1-

yl)acetamide} (7b), 2,2′-{4,4′-[(adamantan-1-ylazanediyl)bis(methylene)bis(1*H*-1,2,3-triazole-4,1-diyl)]bis(*N*-3,5-dimethyladamantan-1-yl)acetamide} (7c), and 2,2′-{4,4′-[(3,5-dimethyladamantan-1-ylazanediyl)bis(methylene)bis(1*H*-1,2,3-triazole-4,1-diyl)]bis(*N*-3,5-dimethyladamantan-1-yl)acetamide} (7d) were obtained similarly to **6a**—**d** from azidoacetamide **2a**,**b** (0.1 mmol) and *N*.*N*-dipropenylaminoadamantane **4a,b** (0.2 mmol).

1,4-Bis({1-[2-(adamantan-1-ylamino)-2-oxoethyl]-1,2,3-triazol-4-yl}methyl)piperazine (8a) and 1,4-bis({1-[2-(3,5-dimethyladamantan-1-ylamino)-2-oxoethyl]-1,2,3-triazol-4-yl}methyl)piperazine (8b) were obtained similarly to **6a**—**d** from azidoacetamide **2a**,**b** (0.2 mmol) and 1,4-dipropenylpiperazine **5** (0.1 mmol).

***N*,*N*′-{[1,1′-(Ethane-1,2-diyl)bis(1*H*-1,2,3-triazole-4,1-diyl)]bis(methylene)}bis(adamantan-1-amine) (10a) and *N*,*N*′-{[1,1′-(butane-1,2-diyl)bis(1*H*-1,2,3-triazole-4,1-diyl)]bis(methylene)}bis(adamantan-1-amine) (10b)** were obtained similarly to **6a**—**d** from *N*-propenylaminoadamantane **3a** (0.2 mmol) and diazidoalkanes **9** (0.1 mmol).

Yields, melting points, elemental analysis data, and spectral characteristics of compounds **2a**,**b**, **3a**,**b**, **4a**,**b**, **6a**—**d**, **7a**—**d**, **8a**,**b**, and **10a**,**b** are given in Tables 1 and 2.

This work was financially supported by the Russian Science Foundation (Project No. 14-23-00160-P: synthesis of conjugates **6a**—**d**, **7a**—**d**, **8a**,**b**, and **10a**,**b**) and within the State Assignment for 2018 (Theme No. 0090-2017-0023: synthesis of compounds **2a**,**b**, **3a**,**b**, and **4a**,**b**).

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Received April 20, 2018;
in revised form May 29, 2018;
accepted May 31, 2018