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ORGANIC SYNTHESIS AND INDUSTRIAL ORGANIC CHEMISTRY

Synthesis of Amino Derivatives of 1,3,5-Triazines Containing Hetero- and Carbocyclic Moieties

S. A. Timofeeva[a], G. Z. Raskil'dina[b], L. V. Spirikhin[c], and S. S. Zlotskii[d]

[a]*Ufa State Academy of Economics and Services, Ufa, Bashkortostan, Russia*
[b]*Research Institute of Herbicides and Plant Growth Regulators, with a Pilot Plant, Academy of Sciences of the Republic of Bashkortostan, Ufa, Bashkortostan, Russia*
[c]*Institute of Organic Chemistry, Ufa Scientific Center, Russian Academy of Sciences, Ufa, Bashkortostan, Russia*
[d]*Ufa State Technical University of Oil, Ufa, Bashkortostan, Russia*
e-mail: Timofeeva_s_a@mail.ru

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Abstract—Reactions of chloro-sim-triazines with secondary amines containing 1,3-dioxolane and hemdichlorocyclopropane moieties were studied. Structures of amino derivatives of 1,3,5-triazines containing cyclic acetal and cyclopropane moieties were examined.

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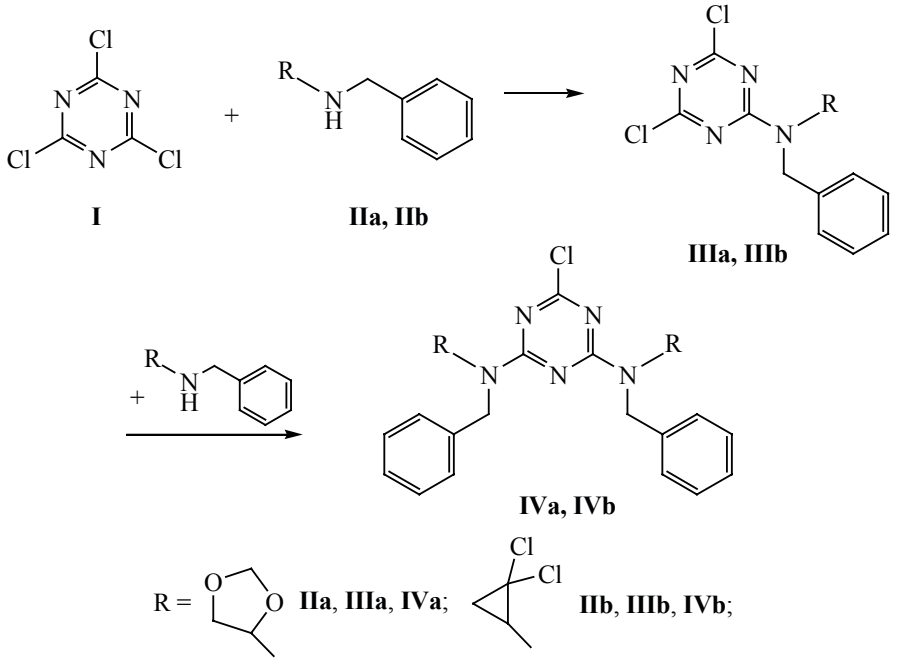
Cyanuric chloride (**I**) and other chloro-sim-triazines are widely used to manufacture biologically active substances, additives, and various small-tonnage products with wide spectrum of useful action [1, 2]. It is known that substitution of a chlorine atom in a trichloride with amino groups occurs in steps [3], with mono- and disubstituted derivatives formed.

To obtain hetero- and carbocyclic amino derivatives of triazines, we studied the reaction of cyanuric chloride (**I**) with secondary amines containing 1,3-dioxolane

Scheme.



(**IIa**) and hem-dichlorocyclopropane (**IIb**) moieties. As a result, the corresponding mono- and disubstituted derivatives **IIIa, IIIb** and **IVa, IVb** were obtained according to the scheme.

At an equimolar ratio between the reagents (secondary amine : triazine = 1 : 1), the corresponding mono amino derivatives are formed in anhydrous 1,4-dioxane. As the molar ratio between a secondary amine and triazine increases from 1 : 1 to 2 : 1, disubstituted triazines **IVa, IVb** become the main reaction products (see the table).

It is noteworthy that, judging from the yields of the triazines formed, secondary amines **IIa** and **IIb** are close in activity.

The structure of the synthesized and isolated amino substituted triazines was confirmed by ^{1}H and ^{13}C NMR spectroscopic and chromato-mass-spectrometric data.

An analysis of the ^{1}H NMR spectrum of compound **IIIa** demonstrated that $H_b^{1'}$ and H_b^5 protons in the *trans* position with respect to the proton of the C^4H group of the dioxolane ring have a chemical shift (CS) at 3.50 and 3.56 ppm in the form of doublet-doublets with spin-spin coupling constants (SSCCs) (Hz): $^2J_{1'} = 14.4$, $^2J_5 = 8.4$, $^3J_{1'b\text{-}4} = 8.1$, and $^3J_{5b\text{-}4} = 5.7$. The protons H_a^5 and $H_a^{1'}$ in the *cis*-position to the C^4H proton are observed with CS 3.80 and 3.88 ppm (SSCC, Hz: $^2J_{1'} = 14.4$, $^2J_4 = 8.4$, $^3J_{1'a\text{-}4} = 3.1$, $^3J_{5a\text{-}4} = 6.2$). The proton of the CH group of the cyclic acetal appears as four doublets at 4.36 ppm, with SSCCs (Hz): $^3J_{1'b\text{-}4} = 8.1$, $^3J_{1'a\text{-}4}$, $3J_{5a\text{-}4} = 6.2$, $^3J_{5b\text{-}4} = 5.7$. Atoms of the CH_2 group of the second atom have CS = 4.85 and 5.06 ppm in the form of two high-intensity singlets. Signals of methylene protons of the benzyl group form two doublets with CS = 4.7 and 5.53 ppm (SSCC $2J = 15.4$ Hz) and a multiplet in the range 7.2–7.4 ppm from protons of the aromatic ring. The structure of compound **IVa** was determined in a similar way.

The ^{1}H NMR spectrum of compound **IIIb** demonstrated that the proton of the CH group of the cyclopropane ring appears at CS = 1.9 ppm as four doublets with SSCCs (Hz): $^3J_{2a\text{-}3} = 10.7$, $^3J_{2b\text{-}3} = 7.6$, $3J_{1'a\text{-}3} = 7.7$, $^3J_{1'b\text{-}1} = 5.8$. Protons of the CH_2 groups bonded to the nitrogen atom are characterized by the presence of a doublet-doublet signal with CS = 3.5 ppm (SSCC, Hz: $^2J = 14.6$, $^3J_{1'a\text{-}1} = 7.7$) for $C_1{}'H_a$ and 4.00 ppm ($^2J = 14.7$ Hz, $^3J_{1'b\text{-}1} = 5.8$ Hz) for $C_1{}'H_b$. As also in compound **IIIa**, to the benzyl group belong two doublets with CS = 4.8 and 5.3 ppm (SSCC $^2J = 15.4$ Hz), which correspond to signals from the CH_2 group, and a multiplet at 7.2–7.4 ppm from protons of the aromatic ring.

A characteristic feature of ^{13}C NMR spectra of the synthesized compounds **IIIa, IIIb** and **IVa, IVb** is the presence of signals in the weak-field part of the spectrum, which correspond to carbon atoms of the triazine ring. The chemical shifts of 165.49 (**IIIa**), 165.42 (**IIIb**), 165.09 (**IVb**), 165.39 and 165.61 ppm (**IVa**) correspond to carbons C–N bonded to the amine, and those of 170.59 (**IIIb**), 167.7 (**IVa**), 170.16 (**IVb**), 170.32 and 170.49 ppm (**IIIa**), to carbon atoms of the triazine ring with a C–Cl bond.

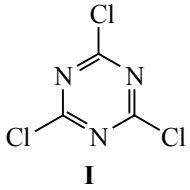
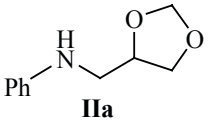
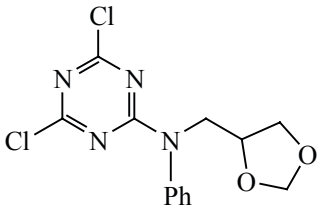
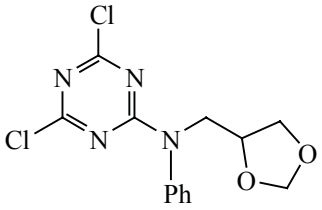
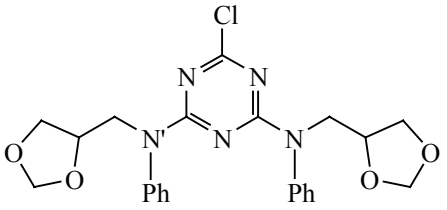
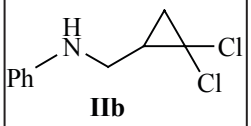
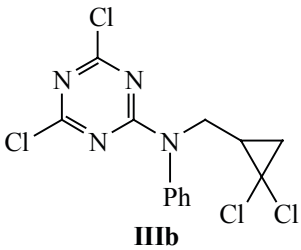
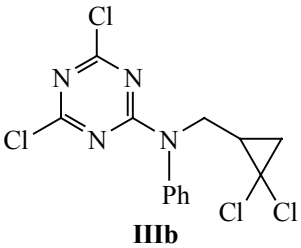
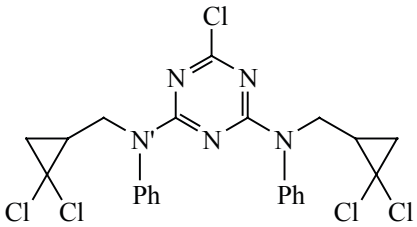
In the mass spectra of the synthesized compounds **IIIa**, **IIIb** and **IVa**, **IVb** , molecular ions are characterized by low-intensity peaks (<<1%). In all the spectra, the maximum intensity is observed for the $[ArCH_2]^+$. ion with $m/z = 91$ (100%). Under the electron impact, molecular ions of amino derivatives of triazine form high-intensity ions of *N*-benzyl-*N*-(1,3-dioxolan-4-yl)methylamine or *N*-benzyl-4,6-dichloro-*N*-[(2,2-dichlorocyclopropyl)methylamine substituents, which further decompose.

The content of ions corresponding to the presence of a triazine ring in the spectrum does not exceed 10% for **IIIa, IIIb** and 5% for **IVa**, **IVb**. Destruction of the triazine ring in the molecular ion $M^{+\cdot}$ of the compounds occurs with loss of HNCCl and formation of $[C_4H_7O_2NHCNCH_2Cl]^+$ moiety (m/z = 176/178) for **IIIa** and **IVa** and $[ArCH_2C_4H_7O_2NCNCCl]^{+\cdot}$ (m/z = 266/268) for (**IVa**).

EXPERIMENTAL

^{1}H and ^{13}C NMR spectra were recorded with a Bruker AM-300 spectrometer (300.13 and 75.43MHz, respectively) in $CDCl_3$ with Me_4Si as internal standard; and chromato-mass spectra, with a Shimadzu GCMS-QP2010 Plus (electron impact, 70 eV, ion source temperature 200°C, direct input temperature 40–290°C, heating rate 12 deg min^{-1}). A GLC analysis was made on an LKhM-8MD chromatograph with a heat-conductivity detector, helium as carrier-gas with a flow rate of 1.5 l h^{-1}, and 2-m-long column packed with 5% SE-30 on Chromaton N-AW. A TLC analysis was made on alufol Merck chromatographic plates (20 : 1 benzene–methanol mixture as eluent). A preparative separation was performed by column chromatography on aluminum oxide, with the role of eluent played by benzene with a methanol content increasing from 5 to 100%.

Interaction of secondary amines containing 1,3-dioxolane and hem-dichlorocyclopropane moieties with chloro-sim-triazines. A : B molar ratio 0.007 : 0.007, 20 ml of 1,4-dioxane. T = 80–90°C, 2 h

Starting compound		Reactants ratio	Product		Yield, % (C:D)
A	B		C	D	
 **I**	 **IIa**	1 : 1	 **IIIa**		73
		1 : 2	 **IIIa**	 **IVa**	86 (1 : 8)
	 **IIb**	1 : 1	 **IIIb**		80
		1 : 2	 **IIIb**	 **IVb**	85 (1 : 16)

***N*-Benzyl-(1,3-dioxolan-4-yl)methylamine (IIa).** A 50-ml reactor equipped with a mechanical stirrer, reflux condenser, and thermometer was charged with 0.08 mol (8.56 g) of benzylamine, 0.12 mol (6.72 g) of solid KOH, 0.05 mol (6.15 g) of 4-chloromethyl-1,3-dioxolane, 0.24 g of TEBAC, and 20 ml of DMSO. The reaction mixture was agitated and irradiated in a Sanyo EM-S1073W microwave oven (power 250 W) for 0.5 h. The temperature was 20–25°C at the beginning of a run and increased in the process to 35–40°C (outside the microwave irradiation zone).After the reaction was complete, the mixture was cooled to 20–25°C, washed with a twofold amount of water, and twice extracted with diethyl ether. The ether extract was shaken with a 10% solution of NaOH, washed with water to neutral reaction, dried with K_2CO_3, and ether was evaporated in a rotor evaporator. The residue was subjected to vacuum distillation in the atmosphere of nitrogen.

Mass spectrum (electron impact, 70 eV), m/z (J_{rel}, %): 193 [M^+] (0), 192 (0.5), 162 (2), 148 (7), 120 (100),

91 (100), 77 (3), 65 (10). ^{1}H NMR spectrum ($CDCl_3$, δ, ppm, *J*, Hz): 2.75 s (2N, $C^{1'}N_2$), 3.63 t (1N, C^5N_b, ^2J 9.0, ^3J 9.0), 3.84 s (1N, $C^{1''}N_a$), 3.95 t (1N, C^5N_b, ^2J 9.0, ^3J 9.0), 4.20 kV (1H, C^5H, ^3J 9.0), 4.86 s (1H, C^2H_a), 5.01 s (1H, C^2H_b), 7.20–7.40 m (5H, Ph–).

***N*-Benzyl-1,1-(2,2-dichlorocyclopropyl)methylamine (IIb).** To a mixture of 0.1 mol (10.7 g) of benzylamine, 0.025 mol of 2-chloromethyl-1,1-dichlorocyclopropane was poured 30 ml of DMSO. The reaction mixture was agitated for 4 h at 70–75°C. On being cooled, it was washed with a 20% NaOH solution and extracted with ether; the extract was washed with water to neutral reaction and dried with K_2CO_3. After the solvent, ether, was evaporated, the residue was distilled in a vacuum in the atmosphere of nitrogen. Mass spectrum (electron impact, 70 eV), *m/z* (J_{rel}, %): 228/230/232 [M^+] (12/8/3), 194/196 (5/1), 165 (2), 151 (6), 147 (8), 146 (68), 133 (14), 132 (37), 120 (42), 118 (7), 104/106/108 (18/14/3), 91 (43), 90/92 (100/25), 89 (11), 77 (6), 65 (30). ^{1}H NMR spectrum ($CDCl_3$, δ, ppm, *J*, Hz): 1.17 t (1H, C^2H_b , ^2J 7.4, ^3J 7.4), 1.65 d.d (1H, C^2H_a, ^3J 7.4, ^2J 10.4), 1.85 d.d.d.d (1H, C^3H, ^3J 7.4, 10.4, 8.3, 5.6), 2.75 d.d (1H, $C^{1'}H_a$, ^2J 12.8, ^3J 8.3), 2.9 d.d (1H, $C^{1'}H_b$, ^2J 12.8, ^3J 5.6), 3.85 s (2H, $C^{1''}H_2$), 7.20–7.40 m (5H, Ph–).

Interaction of secondary amines with chlorosim-triazine (1 : 1). A mixture of 0.007 mol (1.29 g) of cyanuric chloride (**Ia**) and 0.007 mol of amine **IIa, IIb** [1.35 g of *N*-benzyl-(1,3-dioxolan-4-yl)methylamine or 1.61 g of *N*-benzyl-1-1-(2,2-dichlorocyclopropyl) methylamine] in 20 ml of dioxane was boiled under agitation for 3 h, then cooled to 20°C, a solution of 0.39 g (0.007 g-mol) of KOH in 5 ml of water was added, and the mixture was boiled under agitation for 1 h. The mixture was cooled to 10–15°C, 100 ml of icy water was added, and the resulting mixture was washed with benzene (3 Ч 30 ml). The extract was dried over Na_2SO_4, concentrated to a volume of 5 ml, and chromatographed on a column with Al_2O_3 (*H* = 20 cm, *d* = 4.5 cm), with elution by a 20 : 1 mixture of benzene and methanol. This procedure was used to obtain compounds **IIIa** and **IIIb**.

***N*-Benzyl-4,6-dichloro-*N*-(1,3-dioxolan-4-ylmethyl)-1,3,5-triazine-2-amine (IIIa).** Yield 73%, isolated by column chromatography (R_f = 0.65) (20 : 1 benzene–methanol as eluent). Mass spectrum (electron impact, 70 eV), *m/z* (J_{rel}, %): 340/342/343 [M^+] (0), 254/256/258 (24/14/3), 252/254/256 (10/7/4), 219/221 (3/1), 211 (5), 207 (4), 182 (7), 183 (3) , 176/178/180 (6/3/0.5), 157 (11), 150 (11), 148 (7), 132 (4), 131 (42), 125 (13), 116 (8), 104 (57), 91 (100) , 79 (35), 65 (50), 62/64 (42/20.5). ^{1}H NMR spectrum ($CDCl_3$, δ, ppm, *J*, Hz): 3.50 d.d (1H, $C^{1'}H_b$, ^2J 14.4, ^3J 8.1), 3.56 d.d (1H, C^5H_b, ^2J 8.4, ^3J 5.7), 3.80 d.d (1H, $C^{1'}H_a$, ^2J 14.4, ^3J 3.1), 3.98 d.d (1H, C^5H_b, ^2J 8.4, ^3J 6.2), 4.36 d.d.d.d (1H, C^4H, ^3J 8.1, 3.1, 5.7, 6.2), 4.85 s (1H, C^2H_a), 5.06 s (1H, C^2H_b), 4.79 d (1H, $C^{1''}H_a$, ^2J 15.1), 5.3 d (1H, $C^{1''}H_b$, ^2J 15.1), 7.20–7.40 m (5H, Ph–). ^{13}C NMR spectrum ($CDCl_3$, δ, ppm): 48.39 ($^{1'}$C); 51.54 ($^{1''}$C); 67.54 (^{5}C); 73.92 (^{4}C); 95.15 (^{2}C); 128.07 ($^{6'''}$C); 128.02 ($^{3'''}$C, $^{5'''}$C); 128.89 ($^{2'''}$C, $^{4'''}$C); 135.63($^{1'''}$C); 165.49 ($^{2''''}$C); 170.32, 170.49 ($^{5''''}$C, $^{4''''}$C).

***N*-Benzyl-4,6-dichloro-*N*-[(2,2-dichlorocyclopropyl)methyl]-1,3,5-triazine-2-amine (IIIb).** Yield 80%, isolated by column chromatography (R_f = 0.78, 20 : 1 benzene–methanol as eluent). Mass spectrum, *m/z* (J_{rel}, %): 376/378/380/382 [M^+] (0), 268/270 (0.3/0.2), 242/244/246 (3.5/1.8/0.3), 207 (1), 192/194 (0.8/0.4), 132 (2), 104 (5), 92 (3), 91 (100) , 87 (8.5), 65 (11). ^{1}H NMR spectrum ($CDCl_3$, δ, ppm, *J*, Hz): 1.2 t (1H, C^2H_b, ^2J 7.6, ^3J 7.6), 1.7 d.d (1H, C^2H_a, ^2J 7.6, ^3J 10.7), 1.9 d.d.d.d (1H, C^3H, ^3J 7.6, 10.7, 7.7, 5.8), 3.50 d.d (1H, $C^{1'}H_a$, ^2J 14.6, ^3J 7.7), 4.00 d.d (1H, $C^{1'}H_b$, ^2J 14.6, ^3J 5.8), 4.80 d (1H, $C^{1''}H_a$, ^2J 15.3), 5.30 d (1H, $C^{1''}H_b$, ^2J 15.3), 7.20–7.40 m (5H, Ph–). ^{13}C NMR spectrum ($CDCl_3$, δ, ppm, *J*, Hz): 25.71 (^{2}C), 28.52 (^{3}C), 47.10 ($^{1'}$C), 50.55 ($^{1''}$C), 59.80 (^{1}C), 128.31 ($^{4'''}$C), 127.76 ($^{3'''}$C, $^{5'''}$C), 128.97 ($^{2'''}$C, $^{4'''}$C), 135.52 ($^{1'''}$C), 165.42 ($^{2''''}$C), 170.59 ($^{4''''}$C, $^{6''''}$C).

Interaction of secondary amines with chlorosim-triazines (2 : 1). A mixture of 0.007 mol (1.29 g) of cyanuric chloride **Ia** and 0.014 g-mol of amine **IIa, IIb** [3.1 g of *N*-benzyl-(1,3-dioxolan-4-yl)methylamine or 3.22 g of *N*-benzyl-1-1-(2,2-dichlorocyclopropyl) methylamine] in 20 mL of 1,4-dioxane was boiled under agitation for 3 h, then it was cooled to 20°C, a solution of 0.78 g (0.014 g-mol) of KOH in 5 mL of water was added, and the mixture was boiled under agitation for 1 h. Then the mixture was cooled to 10–15°C, 100 ml of icy water was added, and washed the resulting mixture with benzene (3 Ч 30 ml). The extract was dried over Na_2SO_4, concentrated to a volume of 5 ml, and chromatographed on a column with Al_2O_3 (*H* = 20 cm, *d* = 4.5 cm), with elution by a 40 : 1 benzene–ethanol mixture. This procedure was used to obtain compounds **IVa** and **IVb**.

***N,N'*-Dibenzyl-6-chloro-*N,N'*-bis(1,3-dioxolan-4-ylmethyl)-1,3,5-triazine-2,4-diamine (IVa).** Yield 76%, isolated by column chromatography (R_f = 0.55, 40 : 1 benzene–methanol mixture as eluent. Mass spectrum (electron impact, 70 eV), *m/z* (J_{rel}, %): 496/498 [M^+] (0), 266/268 (1.4/0.9), 218/220 (0.4/9.2), 207 (4), 190 (0.3), 176/178 (0,5/0.3), 131 (0.9), 104/106 (1.9/1), 91 (100), 92 (7), 87 (1.7), 65 (11), 62/64 (2/1.1). ^{1}H NMR spectrum ($CDCl_3$, δ, ppm, *J*, Hz): 3.20–4.10 m (8H, $C^{1''}H_2$, $C^{1'''}H_2$, C^5H_2, $C^{5'}H_2$); 4.35 m (2H, C^4H, $C^{4'}H$), 4.85 s (1H, C^2H_a, s, 1H, $C^{2'}H_a$); 5.02 s (1H, C^2H_b, s, 1H, $C^{2'}H_b$); 4.79 d (2H, $C^{1'''}H_a$, $C^{1''''}H_a$, ^2J 15.6); 5.15, 5.20 d (1H, $C^{1''''}H_b$, d, 1H, $C^{1'''}H_b$, ^2J 13.5, ^2J 15.1); 7.20–7.40 m (10H, Ph–). ^{13}C NMR spectrum ($CDCl_3$, δ, ppm, *J*, Hz): 48.21, 48.79 ($^{1'}$C, $^{1'''}$C); 50.86, 51.32 ($^{1'''}$C, $^{1''''}$C); 67.32, 67.63 (^{5}C, $^{5'}$C); 74.91, 75.03 (^{4}C, $^{4'}$C); 94.85, 95.00 (^{2}C, $^{2'}$C); 128.09 ($^{4''''}$C, $^{4'''''}$C); 127.59, 127.48 ($^{3''''}$C, $^{3'''''}$C, $^{5''''}$C, $^{5'''''}$C); 127.70, 128.61 ($^{2''''}$C, $^{2'''''}$C, $^{6''''}$C, $^{6'''''}$C), 137.44, 137.59 ($^{1''''}$C, $^{1'''''}$C); 165.39, 165.61 ($^{2'''''}$C, $^{4'''''}$C); 167.71 ($^{6'''''}$C).

***N,N'*-Dibenzyl-6-chloro-*N.N'*-bis[(2,2-dichlorocyclopropyl)methyl]-1,3,5-triazine-2,4-diamine (IVb).** Yield 80%, isolated by column chromatography (R_f = 0.62, benzene as eluent. Mass spectrum, *m/z* (J_{rel}, %): 569/571/573/575/577/579 [M^+] (0), 280/282/284 (2.6/1.7/0.3), 278/280/282 (3.7/2.7/1.7), 252/254/256 (2.2/1.6/0.3), 207 (0.5), 188/190/192 (3.7/2.8/0.7), 131 (3.7), 128/130 (4/2.1), 125/127 (2.5/1.7), 104 (6), 92 (10), 91 (100) , 87 (7.3), 77 (3.4), 65 (12.5). ^{1}H NMR spectrum ($CDCl_3$, δ, ppm, *J*, Hz): 1.25 m (1H, C^2H_a, 1H, $C^{2'}H_a$), 1.7 m (1H, C^2H_b, m, 1H, $C^{2'}H_b$), 2.1 m (1H, C^3H, m, 1H, $C^{3'}H$), 4 m (1H, $C^{1''}H_a$, 1H, $C^{1'''}H_a$), 4.00 m (1H, $C^{1''}H_b$, 1H, $C^{1'''}H_b$), 4.80 d (1H, $C^{1''''}H_a$, ^2J 15.4, d, 1H, $C^{1''''}H_a$, ^2J 15.4), 5.30 d (1H, $C^{1''''}H_b$, ^2J 15.4, d, 1H, $C^{1''''}H_b$, ^2J 15.4), 7.20–7.40 m (5H, Ph''''–, 5H, Ph'''''–). ^{13}C NMR spectrum ($CDCl_3$, δ, ppm, *J*, Hz): 25.31, 25.03 (^{2}C, $^{2'}$C); 29.07, 28.61 (^{3}C, $^{2'}$C); 46.20, 47.14 ($^{1'}$C, $^{1'''}$C); 49.72, 50.49 ($^{1'''}$C, $^{1''''}$C); 60.05, 60.13 (^{1}C, $^{1'}$C); 127.01, 127.42 ($^{4''''}$C, $^{4'''''}$C); 127.49, 127.58 ($^{3''''}$C, $^{3'''''}$C, $^{5''''}$C, $^{5'''''}$C); 127.96, 128.33 ($^{2''''}$C, $^{2'''''}$C, $^{6''''}$C, $^{6'''''}$C), 136.97 ($^{1''''}$C, $^{1'''''}$C); 165.09 ($^{2'''''}$C, $^{4'''''}$C); 170.16 ($^{6'''''}$C).

CONCLUSIONS

(1) The reaction of chloro-sim-triazines with secondary amines containing 1,3-dioxolane and hemdichlorocyclopropane moieties was used to obtain the corresponding mono- and disubstituted derivatives.

(2) The structure of the synthesized amino derivatives of 1,3,5-triazines was confirmed by ^{1}H and ^{13}C NMR spectroscopy, and the disubstituted structure of the compounds, by chromato-mass spectrometry.

(3) It was demonstrated that reactions of amines with chloro-sim-triazines in anhydrous 1,4-dioxane are a convenient way to synthesize the corresponding amino derivatives of triazines.

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