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Synthesis of Some 1-Alkyl 3,5-Bis(ω-haloalkyl) Isocyanurates

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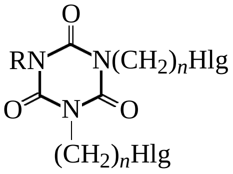
Abstract — 1-Methyl or 1-benzyl 3,5-bis(ω-bromoalkyl) isocyanurates with the alkyl chain comprising 3 through 6 methylene units were synthesized by the reaction of disodium methyl and benzyl isocyanurates with α,ω-dibromoalkanes. The reaction of disodium methyl and benzyl isocyanurates with ethylene chlorohydrin was used to obtain 1-methyl or 1-benzyl 3,5-bis(2-hydroxyethyl) isocyanurates whose treatment with PBr_3 or $SOCl_2$ gave the corresponding 1-alkyl 3,5-bis(2-haloethyl) isocyanurates. 1-Methyl and 1-benzyl 3,5-bis(chloromethyl) isocyanurates were prepared by treatment with $SOCl_2$ of 1-methyl or 1-benzyl 3,5-bis(hydroxymethyl) isocyanurates obtained, in their turn, by condensation of methyl and benzyl isocyanurates with formaldehyde.

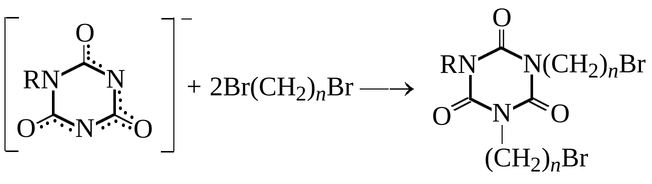
The present work reports on the synthesis of 1-methyl and 1-benzyl 3,5-bis(ω-haloalkyl) isocyanurates **Ia**–**If** and **IIa**–**IIf**, which are of interest as starting materials for preparing a variety of compounds, including practically valuable ones.



Ia–If, IIa–IIf

R = CH_3 (**I**), $C_6H_5CH_2$ (**II**); Hlg = Cl (**Ia, IIa**) or Br (**Ib–If, IIb–IIf**); *n* = 1 (**a**), 2 (**b**), 3 (**c**), 4 (**d**), 5 (**e**), 6 (**f**).

No similar structures have yet been reported. As follows from the available literature on related compounds, such as 1,3-diallyl 5-(ω-haloalkyl) and 1,3,5-tris(ω-haloalkyl) isocyanurates [1–10], as well as 1,3-bis(ω-haloalkyl)uracils [11, 12], 1-alkyl 3,5-bis(ω-haloalkyl) isocyanurates are most expedient to prepare by alkylation of disodium alkyl(aryl) isocyanurates with α,ω-dihaloalkanes. It was found that these reactions are best performed with anhydrous salts, in anhydrous DMF, and with a 5-fold molar excess of α,ω-dibromoalkane at 30–50°C. Under these conditions we prepared 1-methyl and 1-benzyl 3,5-bis(ω-bromoalkyl) isocyanurates **Ic**–**If** and **IIc**–**IIf** in yields of 24–82% (Table 1).



Ic–If, IIc–IIf

n = 3–6.

Along with the target 1-alkyl 3,5-bis(ω-bromoalkyl) isocyanurates, we always isolated oligomeric products. The reaction of disodium methyl isocyanurate with 1,2-dibromomethane under these conditions results almost exclusively in oligomer formation, and the yield of 1-methyl 3,5-bis(2-bromomethyl) isocyanurate (**Ib**) is as low as 2%. This fact is nicely consistent with the data on alkylation of disodium salts of uracils with α,ω-dibromoalkanes [11, 12] and can be explained by increased activity of the bromine atom in the *N*-alkyl chain of isocyanurate compared with 1,2-dibromomethane, which results in preferential oligomer formation.

The composition of the products was proved by elemental analysis (Table 1). 1-Methyl and 1-benzyl 3,5-bis(ω-bromoalkyl) isocyanurates **Ic**–**If** and **IIc**–**IIf** are transparent thick oils readily soluble in benzene and acetone. In agreement with the proposed structure, the IR spectra of compounds **Ic**–**If** and **IIc**–**IIf** (Table 2) contain characteristic bands of the isocyanurate ring at 750–760, 1440–1460, and 1670–1720 cm^{-1} [ν(C=O)] [13]. In the 3200–3400 cm^{-1} region, no ν(NH) absorption is observed.

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Table 1. Yields, R_f values, melting points, and elemental analyses of 1-alkyl 3,5-bis(ω-haloalkyl) isocyanurates **Ia**–**If** and **IIa**–**IIf**

Comp. no.	Yield[a], %	R_f (diethyl ether–hexane, 1:1)	mp, °C	Found, %				Formula	Calculated, %			
				C	H	Br (Cl)	N		C	H	Br (Cl)	N
Ia	34	0.24	128	29.87	2.60	(29.14)	17.63	$C_6H_7Cl_2N_3O_3$	30.02	2.94	(29.54)	17.51
Ib	20	0.33	125	26.69	3.28	44.70	11.81	$C_8H_{11}Br_2N_3O_3$	26.91	3.11	44.77	11.77
Ic	24	0.28[b]		31.34	4.31	40.80	10.97	$C_{10}H_{15}Br_2N_3O_3$	31.18	3.92	41.50	10.91
Id	53	0.33		35.38	4.49	39.61	10.16	$C_{12}H_{19}Br_2N_3O_3$	34.88	4.63	39.68	10.17
Ie	63	0.53[c]		38.02	5.25	37.42	9.64	$C_{14}H_{23}Br_2N_3O_3$	38.09	5.26	37.24	9.51
If	40	0.41		41.33	6.28	34.12	9.21	$C_{16}H_{27}Br_2N_3O_3$	40.93	5.80	34.08	8.95
IIa	33	0.46	174	45.06	3.67	(22.57)	13.07	$C_{12}H_{11}Cl_2N_3O_3$	45.55	3.50	(22.41)	13.28
IIb	29	0.66	77	38.74	3.49	36.76	9.73	$C_{14}H_{15}Br_2N_3O_3$	38.82	3.49	36.90	9.70
IIb'	79	0.52	81	48.54	4.31	(20.03)	11.94	$C_{14}H_{15}Cl_2N_3O_3$	48.85	4.39	(20.60)	12.21
IIc	41	0.36		41.30	4.73	33.79	8.90	$C_{16}H_{19}Br_2N_3O_3$	41.67	4.53	34.66	9.11
IId	82	0.34		44.03	5.10	32.96	9.09	$C_{18}H_{23}Br_2N_3O_3$	44.19	4.73	33.16	8.58
IIe	76	0.47		46.85	5.33	30.68	7.94	$C_{20}H_{27}Br_2N_3O_3$	46.44	5.26	30.90	8.12
IIf	47	0.52		48.36	5.81	29.30	7.51	$C_{22}H_{31}Br_2N_3O_3$	48.45	5.73	29.31	7.71

[a] Per starting methyl or benzyl isocyanurate. [b] Diethyl ether–pentane, 1:1. [c] Benzene–ethyl acetate, 10:1.

Table 2. IR spectra and ^1H NMR spectra of 1-alkyl 3,5-bis(ω-haloalkyl) isocyanurates **Ia**–**If** and **IIa**–**IIf**

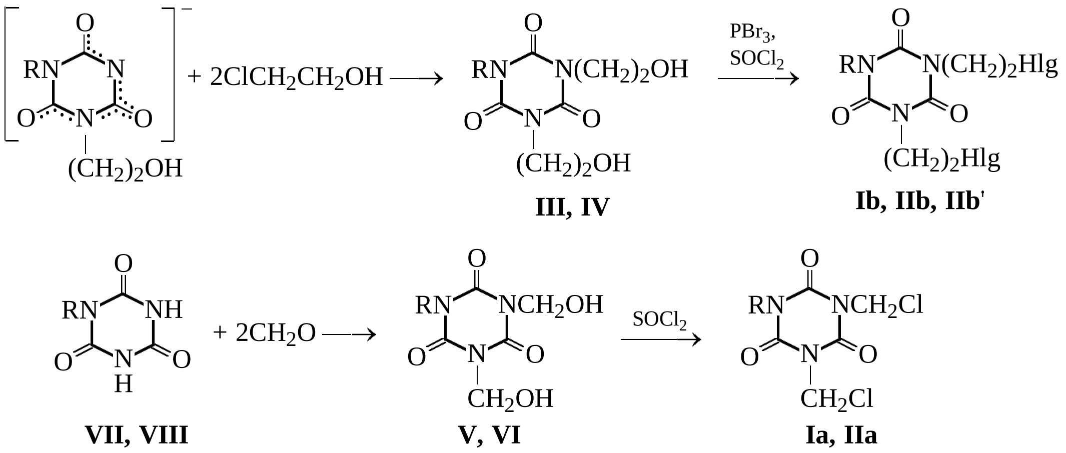
Comp. no.	IR spectrum[a], ν, cm^{-1}			^1H NMR spectrum (CCl_4)[b], δ, ppm					
	isocyanurate ring	C=O	C–Br	C_6H_5	$C_6H_5CH_2$	CH_3	NCH_2	CH_2Br	CH_2
Ia	766 s	1686 v.s, 1720 v.s	–	–	–	3.40 s	5.67 s		–
Ib	760 s	1650 v.s, 1720 v.s	650 w	–	–	3.35 s	4.29 t	3.55 t	–
Ic	760 s, 1446 v.s	1686 v.s	650 w, 554 m	–	–	3.25 s	3.95 t	3.35 t	2.15 m
Id	760 s, 1446 v.s	1686 v.s	650 w, 554 m	–	–	3.29 s	3.88 t	3.43 t	1.88 m
Ie	760 s, 1450 v.s	1686 v.s	650 w, 554 m	–	–	3.28 s	3.81 t	3.40 t	1.68 m
If	767 s, 1460 v.s	1690 v.s	650 w, 567 m	–	–	3.22 s	3.78 t	3.35 t	1.45 m
IIa	760 s	1700 v.s	–	7.23 m	5.03 s	–	5.62 s		–
IIb	760 s, 1460 v.s	1686 v.s, 1700 v.s	560 sl	7.22 m	4.95 s	–	4.20 t	3.47 t	–
IIb'	760 s, 1460 v.s	1680 v.s, 1700 v.s	–	7.23 m	4.97 s	–	4.19 t	3.67 t	–
IIc	760 s, 1446 v.s	1686 v.s	642 w, 560 m	7.23 m	4.95 s	–	3.95 t	3.37 t	2.17 m
IId	750 s, 1460 v.s	1670 v.s	644 w, 560 w	7.22 m	4.90 s	–	3.83 t	3.35 t	1.80 m
IIe	760 s, 1440 v.s	1670 v.s	644 m, 560 m	7.27 m	4.90 s	–	3.80 t	3.31 t	1.65 m
IIf	760 s, 1440 v.s	1670 v.s	634 m, 547 m	7.19 m	4.92 s	–	3.78 t	3.32 t	1.46 m

[a] The IR spectra of compounds **Ia, Ib**, and **IIa** were obtained in Vaseline, and those of compounds **Ic**–**If** and **IIb**–**IIf**, in thin films.
[b] The ^1H NMR spectra of compounds **Ia, Ib**, and **IIa** were obtained in $CDCl_3$.

In addition, the IR spectra of 1-benzyl 3,5-bis(ω-bromoalkyl)isocyanurates contain characteristic absorption bands of the benzene ring [14]. In the 550–650 cm^{-1} region, there are two medium or weak bands assignable to ν(C–Br) [14].

1-Methyl and 1-benzyl 3,5-bis(chloromethyl) isocyanurates (**Ia, Ib**) and 1-methyl and 1-benzyl 3,5-bis(2-haloethyl) isocyanurates (**IIa, IIb, IIb**') were obtained by halogenation of the corresponding hydroxy derivatives **III**–**VI**.

The previously unknown 1-methyl and 1-benzyl 3,5-bis(2-hydroxyethyl) isocyanurates (**III, IV**) were prepared by the reactions of disodium methyl and disodium benzyl isocyanurates with a small excess of



R = CH_3 (**I, III, V, VII**), $C_6H_5CH_2$ (**II, IV, VI, VIII**); Hlg = Br (**Ib, IIb**), Cl (**IIb'**).

ethylene chlorohydrin in DMF at 90–100°C. Compounds **III** and **IV** are transparent resin-like solids soluble in methanol. Their IR spectra contain all characteristic bands of the isocyanurate ring, as well as a strong broad band near 3400 cm^{-1}, assignable to ν(OH).

Treatment of compounds **III** and **IV** with phosphorus tribromide gave respectively 1-methyl and 1-benzyl 3,5-bis(2-bromomethyl) isocyanurates (**Ib**, **IIb**). Treatment of compound **IV** with thionyl chloride gave 1-benzyl 3,5-bis(2-chloroethyl) isocyanurate (**IIb'**). The structure and composition of the products were confirmed by the elemental analyses and the ^{1}H NMR and IR spectra (Tables 1 and 2).

1-Methyl and 1-benzyl 3,5-bis(hydroxymethyl) isocyanurates (**V, VI**) were synthesized by the reactions of respectively methyl and benzyl isocyanurates (**VII, VIII**) with formaldehyde [7]. Methyl isocyanurate (**VII**) was dissolved in 34% formaldehyde on heating at 60°C. Unreacted methyl isocyanurate was then filtered off, and the solvent was removed to leave a transparent glassy product whose elemental analysis and IR spectrum corresponded to 1-methyl 3,5-bis-(hydroxymethyl)isocyanurate (**V**).

The yield of 1-benzyl 3,5-bis(hydroxymethyl) isocyanurate (**VI**) in the similar reaction was as low as 20%. This compound proved to be thermally unstable: It decomposed above 55°C with evolution of formaldehyde. To improve the yield of 1-benzyl 3,5-bis(hydroxymethyl) isocyanurate, the reaction was performed at 40°C in the presence of pyridine. After removal of the solvent, a white amorphous substance remained, whose elemental analysis and IR spectrum corresponded to 1-benzyl 3,5-bis(hydroxymethyl) isocyanurate (**VI**).

By treatment of compounds **V** and **VI** with thionyl chloride we obtained respectively 1-methyl and 1-benzyl 3,5-bis(chloromethyl) isocyanurates (**Ia, IIa**) whose composition and structure were confirmed by the elemental analyses and the ^{1}H NMR and IR spectra (Tables 1 and 2).

Methyl and benzyl isocyanurates (**VII, VIII**) were obtained by the known procedure [15] by condensation of *N*-substituted biurets with diethyl carbonate. It is known that *N*-substituted biurets are prepared by reactions of nitrobiuret with primary amines, but for a reasonable (50%) yield of *N*-methylbiuret (**IX**) the reaction should be performed at 100°C in a sealed ampule [16]. We propose a more convenient synthetic procedure for this compound (see Experimental).

EXPERIMENTAL

The IR spectra were recorded on a Specord IR-75 instrument in thin films or in suspensions in Vaseline oil. The ^{1}H NMR spectra were obtained on a Varian T-60 spectrometer (60 MHz), internal reference TMS.

1,3-Bis(ω-bromoalkyl)-1-methyl- and 1-benzyl-3,5-bis(ω-bromoalkyl)-1,3,5-triazin-2(1*H*),4(3*H*),6(5*H*)-triones Ic–If and IIc–IIf. Methyl or benzyl isocyanurate (**VII, VIII**), 0.05 mol, was added with stirring to sodium butylate prepared from 0.1 mol of sodium in 300 ml of absolute butanol, and the mixture was stirred for 8 h, after which the butanol was removed by vacuum azeotropic distillation with toluene. Excess toluene was removed by vacuum distillation. The residual anhydrous disodium salt was treated with 300 ml of absolute DMF and 0.5 mol (5-fold excess) of corresponding α,ω-dibromoalkane. The mixture was stirred at 35–45°C for 2–5 h (to pH 7), the precipitate was filtered off, and the DMF and α,ω-dibromoalkane were distilled off in a vacuum. The residue was treated with toluene, the mixture was filtered, and the filtrate was evaporated in a vacuum. The product was purified by column chromatography on silica gel, eluents benzene–ethyl acetate, 20 : 1 (**Ic**–**Ie, IIc**), benzene–acetone, 100 : 1 (**IId, IIe**), or benzene–dioxane, 100 : 1 (**If, IIf**). Compounds **Ic**–**If** and **IIc**–**IIf** are colorless or yellowish thick oils; compound **IIc** crystallizes on standing, mp 61–63°C.

1,3-Bis(2-hydroxyethyl)-5-methyl-1,3,5-triazine-2(1*H*),4(3*H*),6(5*H*)-trione (III). Methyl isocyanurate, 7.15 g, was added with stirring to sodium butylate obtained from 2.3 g of sodium in 200 ml of absolute butanol, and the mixture was refluxed for 8 h. The butanol was distilled off in a vacuum and completely removed by azeotropic distillation with toluene. Excess toluene was removed, the residue was treated with 200 ml of absolute DMF, heated to 90°C, and 8.9 g of ethylene chlorohydrin was added dropwise to the solution. The reaction mixture was stirred at 95–100°C for 3 h (to pH 7), cooled, the precipitate was filtered off, the DMF and ethylene chlorohydrin were distilled off in a vacuum, and the residue was subjected to column chromatography on silica gel, eluents benzene–methanol, 10 : 1, to obtain 7.1 g (62%) of compound **III**. R_f 0.13 (benzene–methanol, 10 : 1). IR spectrum (thin film), ν, cm^{-1}: 3370–3200 s (OH), 1730 s, 1670 v.s (C=O), 1440 s, 760 m (triazine ring), 1040 s (C–O). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 3.33 s (3H, CH_3), 3.86 m (2H, CH_2O), 4.06 t (2H, NCH_2). Found, %: C 41.10; H 5.40; N 18.43. $C_8H_{13}N_3O_5$. Calculated, %: C 41.56; H 5.67; N 18.18.

1-Benzyl-3,5-bis(2-hydroxymethyl)-1,3,5-triazine-2(1*H*),4(3*H*),6(5*H*)-trione (IV) was obtained in a similar way and purified by column chromatography on silica gel, eluent benzene–methanol, 20 : 1. Yield 81%. R_f 0.26 (benzene–methanol, 10 : 1). IR spectrum (thin film), ν, cm^{-1}: 3470–3330 s (OH), 1700 s, 1680 v.s (C=O), 1440 s, 765 m (triazine ring), 1040 s (C–O). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 3.80 m (2H, CH_2O), 3.98 t (2H, NCH_2), 4.99 s (2H, C_6H_5–CH_2), 7.25 s (5H, C_6H_5). Found, %: C 55.35; H 5.37; N 10.58. $C_{14}H_{17}N_3O_5$. Calculated, %: C 55.24; H 5.41; N 10.74.

1,3-Bis(2-bromomethyl)-5-methyl-1,3,5-triazine-2(1H),4(3H),6(5H)-trione (Ib). A solution of 8.08 g of PBr_3 in 5 ml of 1,2-dichloroethane was added dropwise to a solution of 6.9 g of compound **III** in 50 ml of anhydrous 1,2-dichloroethane cooled to –3°C. The mixture was stirred for 30 min at 0°C, then for 8–10 h at 20°C, and, finally, for 2 h at 70°C. After cooling, the reaction mixture was poured into 50 ml of ice water. The layers were separated, and the aqueous layer was extracted with 25 ml of 1,2-dichloroethane. The combined organic layers were washed with 10% Na_2CO_3 and water, and dried with $MgSO_4$. The solvent was removed, and the solid residue was treated with diethyl ether and recrystallized from acetone–hexane. Yield 3.5 g (33%).

1-Benzyl-3,5-bis(2-bromomethyl)-1,3,5-triazine-2(1*H*),4(3*H*),6(5*H*)-trione (IIb) was prepared in a similar way from compound **IV** and purified by column chromatography on silica gel, eluent benzene. The eluate was evaporated to leave a transparent oily substance which quickly crystallized. Yield 36%.

1-Benzyl-3,5-bis(2-chloroethyl)-1,3,5-triazine-2(1*H*),4(3*H*),6(5*H*)-trione (IIb'). Thionyl chloride, 42.5 g (5-fold excess), was added dropwise at 20°C to a solution of 11 g of compound **IV** in 80 ml of absolute 1,2-dichloroethane. The reaction mixture was slowly heated to boiling and then boiled for 2 h. After cooling, the mixture was treated with 80 ml of water, the organic layer was separated, washed with two portions of water, and dried with $MgSO_4$. The residue was subjected to column chromatography on silica gel, eluent benzene–methanol, 20 : 1. Yield 10.3 g (84%), transparent oily substance crystallizing on handling.

1,3-Bis(hydroxymethyl)-5-methyl-1,3,5-triazine-2(1*H*),4(3*H*),6(5*H*)-trione (V). Methyl isocyanurate (**VII**), 2.86 g, was added to a solution of 5.5 ml of 34% formaldehyde in 30 ml of water. The mixture was stirred at 55–60°C until the precipitate had dissolved completely (30–40 min), evaporated in a vacuum at 50–60°C, the residue was dissolved in cold methanol, and the methyl isocyanurate precipitate was filtered off. The filtrate was evaporated in a vacuum at 40–50°C. Yield 3.1 g (76%), transparent colorless resin. R_f 0.45 (benzene–methanol, 4 : 1). IR spectrum (thin film), ν, cm^{-1}: 3500–3400 s (OH), 1690 s, 1670 v.s (C=O), 1440 s, 766 m (triazine ring), 1060 s (C–O). ^{1}H NMR spectrum (acetone-d_6), δ, ppm: 3.20 s (3H, CH_3), 5.30 d (2H, NCH_2O). Found, %: C 35.56; H 4.32; N 20.41. $C_6H_9N_3O_5$. Calculated, %: C 35.47; H 4.47; N 20.68.

1-Benzyl-3,5-bis(hydroxymethyl)-1,3,5-triazine-2(1*H*),4(3*H*),6(5*H*)-trione (VI). Benzyl isocyanurate, 5.48 g, and 2 g of pyridine were added to a solution of 5.5 ml of 34% of formaldehyde in 20 ml of water. The mixture was stirred at 40°C for 1 h, after which it was evaporated in a vacuum at 40–50°C, the residue was dissolved in cold methanol, and the residue was filtered off. The filtrate was evaporated in a vacuum at 40–50°C. Yield 4.0 g (57%), white amorphous substance. R_f 0.30 (benzene–methanol, 4 : 1). IR spectrum (thin film), ν, cm^{-1}: 3500–3300 s (OH), 1720 s, 1690 v.s (C=O), 1440 s, 766 m (triazine ring), 1080 s (C–O). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 4.93 s (2H, C_6H_5–CH_2), 5.23 d (2H, NCH_2O), 7.20 s (5H, C_6H_5). Found, %: C 51.88; H 4.98; N 14.84. $C_{12}H_{13}N_3O_5$. Calculated, %: C 51.61; H 4.69; N 15.05.

1,3-Bis(chloromethyl)-5-methyl- and 1-benzyl-3,5-bis(chloromethyl)-1,3,5-triazine-2(1*H*),4(3*H*), 6(5*H*)-triones (Ia, IIa). A solution of 22 g (5-fold excess) of thionyl chloride in 10 ml of 1,2-dichloroethane was added dropwise at 10–12°C to a suspension of 0.018 mol of compound **V** or **VI** in 40 ml of anhydrous 1,2-dichloroethane. The mixture was stirred at 20°C for 1 h, after which it was heated to boiling and boiled for 30 min. After cooling, it was treated with caution with 40 ml of water, the organic layer was separated, washed with water, and dried with $MgSO_4$. The filtrate was evaporated, and the residue was subjected to column chromatography on silica gel, eluent benzene. Compound **Ia**: yield 20%, white crystals. Compound **IIa**: yield 43%, white crystals.

***N*-Methylbiuret (IX).** Nitrobiuret, 32.2 g, was added with stirring to 120 ml of 25% methylamine cooled to 17°C. The reaction mixture warmed up to 23°C. It was stirred for 2 h at 22–23°C, for 30–40 min at 26–27°C, and for 30–40 min at 50°C. The water and excess methylamine were distilled off in a vacuum on a water bath (50–60°C), and the solid residue was recrystallized from isopropanol. Yield 16.5 g (67%), mp 168–168.5°C (published data [16]: mp 166.5–167°C).

REFERENCES

1. Balitskaya, L.G., Khomenkova, K.K., and Kornev, A.A., *Zh. Org. Khim.*, 1966, vol. 2, no. 8, pp. 1421–1423.
2. Fedorenko, V.P., Chernyavskaya, G.A., Zhilina, V.A., and Kornev, K.A., in *Sintez i fiziko-khimiya polimerov* (Synthesis and Physical Chemistry of Polymers), Kiev: Naukova Dumka, 1972, no. 10, pp. 15–18.
3. Novikova, O.A., Samoilenko, M.I., and Shevlyakov, A.S., in *Sintez i fiziko-khimiya polimerov* (Synthesis and Physical Chemistry of Polymers), Kiev: Naukova Dumka, 1973, no. 11, pp. 123–126.
4. Esayan, G.T., Airapetyan, A.N., Isayan, G.A., Gasparyan, Zh.M., and Bagdasaryan, R.O., *Arm. Khim. Zh.*, 1974, vol. 27, no. 2, pp. 163–164.
5. Airapetyan, A.N., Esayan, G.T., and Babayan, A.A., *Arm. Khim. Zh.*, 1982, vol. 35, no. 4, pp. 262–265.
6. Hofmann, A.W., *Ber. B*, 1886, vol. 19, pp. 2061–2098.
7. Pazenko, Z.N. and Chovnik, L.I., *Ukr. Khim. Zh.*, 1964, vol. 30, no. 2, pp. 195–198.
8. US Patent 4 266 055, *Ref. Zh. Khim.*, 1982, 12N233P.
9. Vodop'yanov, V.G., Golov, V.G., Dvoeglazova, N.K., Ivanov, M.G., Mysin, N.I., and Gerega, V.F., *Zh. Obshch. Khim.*, 1981, vol. 51, no. 8, pp. 1885–1890.
10. US Patent 4 250 309, *Ref. Zh. Khim.*, 1981, 18N149P.
11. Reznik, V.S., Salikhov, I.Sh., Shvetsov, Yu.S., Shirshov, A.N., Bakulin, V.S., and Ivanov, B.E., *Izv. Akad. Nauk SSSR, Ser. Khim.*, 1977, no. 4, pp. 880–884.
12. Reznik, V.S., Salikhov, I.Sh., Shvetsov, Yu.S. and Ivanov, B.E., *Izv. Akad. Nauk SSSR, Ser. Khim.*, 1980, no. 11, pp. 2568–2574.
13. Vorotyntseva, V.D., Kuz'mina, E.A., Roginskaya, I.N., Finkel'shtein, A.I., and Dergunov, Yu.I., *Zh. Prikl. Spektrosk.*, 1972, vol. 17, no. 2, pp. 286–292.
14. Bellami, L.J., *Infrared Spectra of Complex Molecules*, New York: Wiley, 1958.
15. Close, W.J., *J. Am. Chem. Soc.*, 1953, vol. 75, no. 15, pp. 3617–3618.
16. Tenney, L.D. and Blanchard, K.C., *J. Am. Chem. Soc.*, 1929, vol. 51, no. 6, pp. 1801.