Heteroatom Chemistry
Volume 17, Number 2, 2006

Synthesis of Oxime Bearing Cyclophosphazenes and Their Reactions with Alkyl and Acyl Halides

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Received 26 April 2005; revised 29 June 2005

ABSTRACT: *Two novel cyclophosphazenes containing oxime groups were prepared from the hexakis(4-formylphenoxy)cyclotriphosphazene (***2***) and hexakis-(4-acetylphenoxy)cyclotriphosphazene (***7***). The reactions of these oximes with acetyl chloride, chloroacetyl chloride, methyl iodide, propyl chloride, monochloroacetone, and 1,4-dichlorobutane were studied. Hexasubstituted compounds were obtained from the reactions of hexakis(4-[(hydroxyimino)methyl]phenoxy)cyclotriphosphazene (***3***) with acetyl chloride (***4***) and chloroacetyl chloride (***5***); however, tetrasubstituted product was obtained from methyl iodide (***6***). Tetra- and trisubstituted products were obtained from the reactions of hexakis(4-[(1)-N-hydroxyethaneimidoyl]phenoxy)cyclotriphosphazene (***8***) with acetyl chloride (***9***) and chloroacetyl chloride (***10***), respectively. All products were obtained in high yields. Pure and defined product could not be obtained from the reaction of* **8** *with methyl iodide, and could not be also obtained from the reactions of* **3** *and* **8** *with propyl chloride, monochloroacetone, and 1,4-dichlorobuthane. The structures of the compounds were defined by elemental analysis, IR,* ^{1}H, ^{13}C, *and* ^{31}P *NMR spectroscopy.* © 2006 Wiley Periodicals, Inc. Heteroatom Chem 17:112–117, 2006; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20176

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Contract grant sponsor: Firat University Research Found.
Contract grant number: FUBAP 923.
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INTRODUCTION

The chemistry of linear (short-chain), cyclic, and polymeric phosphazenes has been a subject of extensive investigation [1–5]. These compounds are reported to possess interesting biomedical properties and promising applications [6–8]. Phosphazene polymers bearing flouralkoxy or aryloxy groups stimulated considerable interest in the past as biologically inert, water insoluble, polymers for blood vessel or heart valve construction [9]. Cyclophosphazene derivatives substituted with aziridine groups were investigated as biomedical products due to their strongly antitumor activity [10]. Antimicrobial and biological effects of some phosphazenes on bacterial and yeast cells have been studied [11–13]. On the other hand, it is known that phosphorus and nitrogen compounds are effective flame retardants for fiber materials [14]. The literature contains reports on the synthesis of different linear, cyclic, or polyphosphazenes [15–25]. There are also a large number of literature reports on reactions of the functional groups on phosphazene substituents [9,26–30].

In this paper, we have prepared new cyclophosphazenes bearing oxime groups from the hexakis(4-formylphenoxy)cyclotriphosphazene and hexakis(4-acetylphenoxy)cyclotriphosphazene. We were also studied their reactions with alkyl and acyl halides.

RESULTS AND DISCUSSION

The reaction of **1** with 6 equiv. of 4-hydroxybenzaldehyde and 4-hydroxyacetophenone in the

presence of K_2CO_3 in THF gave hexakis(4-formylphenoxy)cyclotriphosphazene (**2**) and hexzakis(4-acetylphenoxy)cyclotriphosphazene (**7**). Oxime compounds hexakis(4-[(hydroxyimino)methyl]-phenoxy)cyclotriphosphazene (**3**) and hexakis(4-[(1)-*N*-hydroxyethaneimidoyl]phenoxy)cyclotriphosphazene (**8**) were synthesized from the reactions of **2** and **7** with hydroxlaminehydrochloride in pyridine, respectively.

Hexasubstituted oxime derivatives were obtained from the reactions of oxime compound **3** with acetyl chloride and chloroacetyl chloride (in acetone in the presence of K_2CO_3) via all of the oxime protons replacement with acetyl and chloroacetyl groups. However, tetrasubstituted product was obtained from the reaction of **3** with methyl iodide at the same conditions as for acetyl chloride and chloroacetyl chloride. Tetra- and trisubstituted products were obtained from the reactions of **8** with acetyl chloride and chloroacetyl chloride (in acetone in the presence of triethylamine), respectively. Pure or defined products could not be obtained from the reactions of both oximes (**3** and **8**) with propyl chloride, monochloroacetone and 1,4-dichlorobutane, and **8** with methyl iodide.

The structures of the compounds were defined by elemental analysis, IR, ^{1}H, ^{13}C, and ^{31}P NMR spectroscopy (structures of **2–10** are shown in Scheme 1). Physical properties and analytical data of **2–10** are given in Table 1. Compounds **2–9** were synthesized in high yields. The solvents used for the purification of the compounds **9** and **10** could not be removed completely as observed by ^{1}H, ^{13}C, and NMR spectra. Thus, the presence of these trace amounts of solvent affects the elemental analysis, in particular the carbon value.

TABLE 1 Physical Properties, Molecular Weight, and Analytical Data of Compounds **2–10**

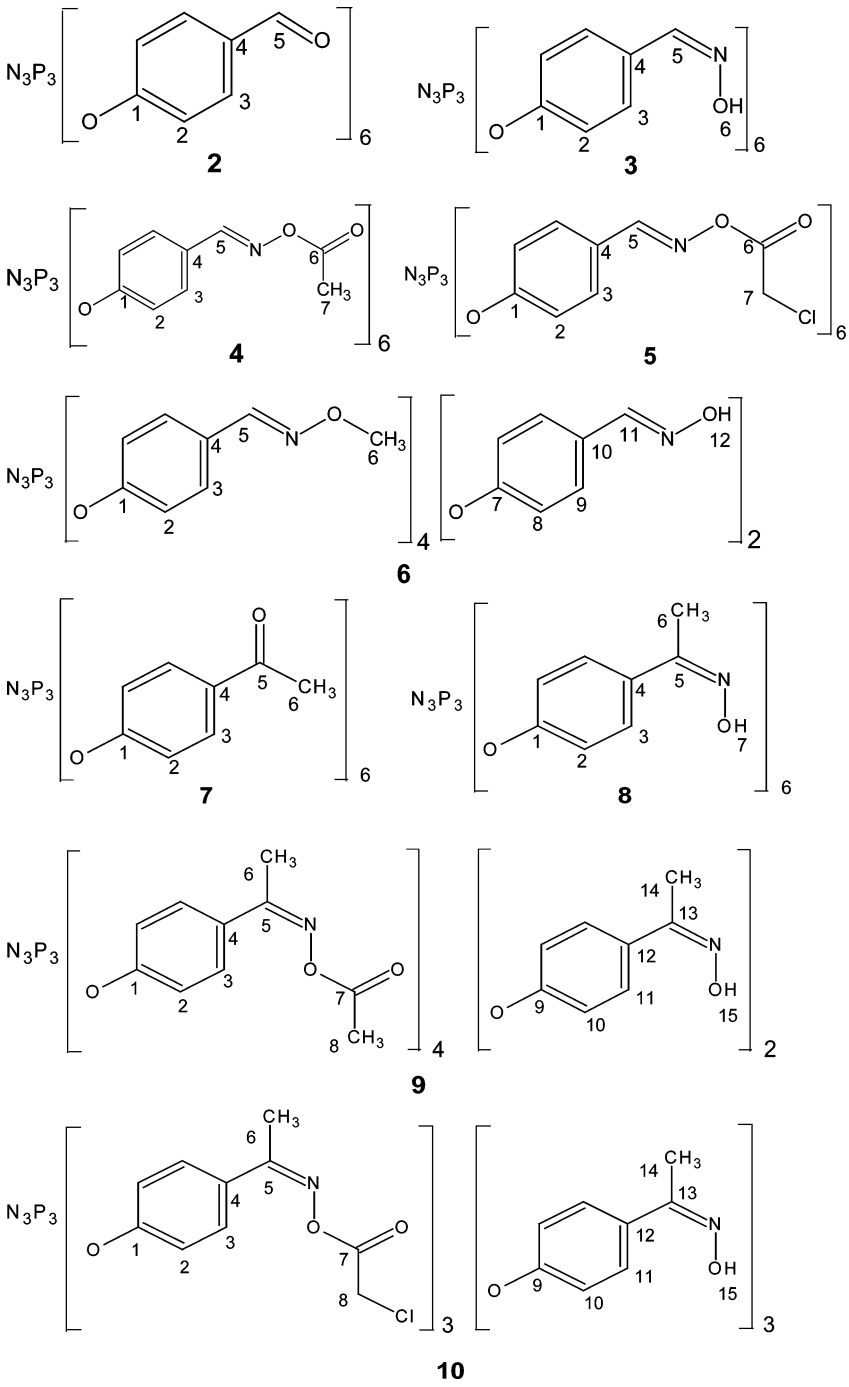
	Yield (%)		*Found*	*Calcd*		*Yield (%)*		*Found*	*Calcd*
2	92	C	58.52	58.55	**7**	87	C	60.93	60.96
		H	3.39	3.51			H	4.50	4.48
		N	4.51	4.88			N	4.40	4.44
3	83	C	53.43	53.00	**8**	99	C	55.67	55.66
		H	3.75	3.81			H	4.69	4.67
		N	12.98	13.25			N	11.98	12.17
4	75	C	54.88	53.87	**9**	79	C	54.12	55.86
		H	4.05	4.02			H	4.96	4.69
		N	10.47	10.47			N	8.34	10.47
5	72	C	45.98	47.92	**10**	57	C	49.37	51.26
		H	3.00	3.25			H	3.69	4.06
		N	8.94	9.09			N	7.40	9.96
6	80	C	54.37	54.82					
		H	4.64	4.40					
		N	11.12	12.51					

The characteristic stretching peaks in the IR spectra of the phosphazenes have been assigned as in Table 2. The P=N stretching vibrations, which are observed between 1172 and 1208 cm^{-1}, are characteristic of cyclophosphazenes. Compared to **1**, which appear at 1218 cm^{-1}, these peaks are shifted to longer wavelengths for **2–10**. The OH stretching vibrations in the IR spectra of **3, 6, 8, 9**, and **10** indicate the oxime compounds. Although **3** and **8** are initial oximes, all hydrogen atoms of OH groups of **6, 9**, and **10** could not be replaced by the alkyl and acyl substituents.

The NMR data of **2–10** are presented in Tables 3–5. The ^{31}P NMR shifts of **2–10** change between 8.05 and 17.21 ppm. Although there is only one peak in the ^{31}P NMR spectra of **1, 2, 7, 9**, and **10** at 20.12, 8.33, 8.05, 8.77, and 8.75 ppm, two peaks, where the second signals are very weak, are observed at $\delta = 17.21$, 17.26, $\delta = 9.06$, 8.93, $\delta = 9.86$, 10.30, $\delta = 9.28$, 9.18, and $\delta = 9.04$, 9.01 ppm for **3, 4, 5, 6**, and **8**, respectively. It is assumed that the weak peaks due to the *cyn* and *anti* isomerism of −C=N− groups. The effects of the *cyn* and *anti* isomerism are also observed at the ^{13}C NMR spectra of **3, 5**, and **6** (Table 5). From this data, it is explained that compounds **3–6** and **8** consist of a mixture of two isomers, but others have one isomer. Although there are different phosphorus environments in the molecules of **6, 9**, and **10**, the main peak is observed as a singlet. It is understood that the phosphorus peaks are not affected from these changes because of the substituted groups are far off the phosphorus atoms.

The ^{1}H and ^{13}C NMR data also confirm the structures of **2–10** (Scheme 1). In the ^{1}H NMR spectra (Table 4), the OH protons are observed at 10.17, 11.31, 11.25, 11.25, and 11.25 ppm for **3, 6, 8, 9**, and **10**, respectively. It is understood from the integral intensities that there are six OH protons in **3** and **8**, which are original oxime-phosphazenes, two OH protons in **6** and **9**, and three OH protons in **10**. This observation indicates that alkyl or acyl groups have not replaced all OH protons in **3** and **8**. Aldehyde proton for **2** appears at 9.90 ppm. The azomethine protons for **3, 4, 5**, and **6** are observed at 7.96 (H^5), 8.00 (H^5), 8.32 (H^5), and 8.17 (H^5)–8.18 (H^{11}), respectively. The aromatic protons for all the compounds appear between 6.85 and 7.85 ppm.

The detailed ^{13}C NMR spectral data are given in Table 5. Aldehyde carbon atom for **2** and ketone carbon atom for **7** are observed at 190.83 and 197.00 ppm at the lowest downfield of the carbon atoms. Compared to **3** and **8**, the azomethine carbon atoms in the substituted moiety of the molecules are shifted to the lower downfield for compound **4, 5, 9**, and **10** except **6**, in which methoxy group



SCHEME 1 The structures of compounds **2–10**.

releases electron to the molecule. But the azomethine resonances do not change in the nonsubstituted moiety of **6, 9**, and **10**. In addition, the ^{13}C NMR data clearly show that compounds **6** and **9** are referred to as geminal isomer, but **10** is nongeminal isomer. There are essentially two different sets of carbon atoms within the geminal structures, but three sets within the nongeminal structures for the tetrasubstituted cyclotriphosphazenes. The resonances of two sets of carbon atoms were observed in the ^{13}C NMR spectra of **6** and **9**. These results indicate the geminal structure. There are essentially two different sets of carbon atoms in the nongeminal structure, but four sets in the geminal isomer for the trisubstituted cyclotriphosphazene. The carbon resonances for **10** are in accordance with the nongeminal

TABLE 2 Characteristic IR Vibrations (in cm^{-1}) of Compounds **2–10**

Compound	ν_{OH}	$\nu_{C-H\,arom}$	$\nu_{C-H\,aliph}$	$\nu_{C=O}$	$\nu_{P=N}$	ν_{N-O-C}	ν_{P-O-C}
2	–	3040, 3100	2732, 2820	1706	1184	–	962
3	3340	3044, 3086	2903, 2980	–	1187	–	949
4	–	3012, 3073	2941, 3000	1768	1182	1051	965
5	–	3069, 3098	2957, 3005	1780	1183	1020	954
6	3343	3068, 3098	2895, 2935	–	1176	1057	954
7	–	3067, 3105	2927, 3002	1685	1188	–	949
8	3460	3058, 3109	2913, 2981	–	1208	–	960
9	3341	3069, 3103	2917, 2981	1768	1188	1042	953
10	3440	3017, 3073	2854, 2956	1774	1172	1082	958

TABLE 3 The ^{31}P NMR Data of Compounds **1–10**

	Main Compound	*Minor Isomer*		*Main Compound*	*Minor Isomer*
1	20.12	–	**6**	9.28	9.18
2	8.33	–	**7**	8.05	–
3	17.21	17.26	**8**	9.04	9.01
4	9.06	8.93	**9**	8.77	–
5	9.86	10.30	**10**	8.75	–

isomer. A triplet and a doublet are expected in the ^{31}P NMR spectra of geminal structures for tetrasubstituted cyclotriphosphazenes (**6** and **9**), but the main peak was observed as a singlet. These results show that the phosphorus signals are not affected from the binding groups because of the substituents at the end of the molecule are far from the phosphorus atoms.

EXPERIMENTAL

General Remarks

Solvents and other liquids used in the experimental works were dried by conventional methods. Hexachlorocyclotriphosphazene $[N_3P_3Cl_6]$ (**1**) was recrystallized from hexane. Other chemicals were used as purchased. Hexakis(4-formylphenoxy)cyclotriphosphazene (**2**) and hexakis(4-acetylphenoxy)-cyclotriphosphazene (**7**) were prepared as described by Carriedo et al. [23]. The reactions of $[N_3P_3Cl_6]$ with the phenols were carried out under dry nitrogen.

The IR spectra were recorded on an ATI Unicam Mattson 1000 FTIR spectrometer. ^1H, ^{13}C, and ^{31}P NMR spectra were recorded using a Bruker DPX-300 spectrometer operating at 300.13, 75.46, and 121.49 MHz, respectively. The ^1H and ^{13}C chemical shifts were measured using $SiMe_4$ as an internal standard; the ^{31}P chemical shifts were measured using 85% H_3PO_4 as an external standard. Chemical shifts downfield from the standard are assigned positive δ values. Microanalysis was carried out by LECO 932 CHNS-O apparatus.

TABLE 4 ^1H NMR Data of Compounds **2–10**

2 7.10 (d): H^2 ($^4J_{POCCH}$: 8.50), 7.60 (d): H^3 ($^5J_{POCCCH}$: 8.56), 9.90 (s): H^5, H^2:H3:H^5 = 2:2:1

3 6.86 (d): H^2 ($^4J_{POCCH}$: 8.45), 7.28 (d): H^3 ($^5J_{POCCCH}$: 8.50), 7.96 (s): H^5, 10.17 (s): H^6, 7.20 (w): H^2 (is), 7.77 (w): H^3 (is), H^2:H^3:H^5:H^6 = 2:2:1:1

4 6.95 (d): H^2 ($^4J_{POCCH}$: 8.50), 7.50 (d): H^3 ($^5J_{POCCCH}$: 8.51), 8.00 (s): H^5, 2.19 (s): H^7, H^2:H^3:H^5:H^7 = 2:2:1:3

5 6.94 (d): H^2 ($^4J_{POCCH}$: 8.38), 7.50 (d): H^3 ($^5J_{POCCCH}$: 8.40), 8.32 (s): H^5, 4.22 (s): H^7, H^2:H^3:H^5:H^7 = 2:2:1:2

6 11.31 (s): H^{12}, 8.18 (s): H^{11}, 8.17 (s): H^5, 6.95: $H^2 + H^8$, 7.50: $H^3 + H^9$, 3.96 (s): H^6, 3.79 (s): H^6 (is), H^{12}:$(H^5 + H^{11})$:$(H^3 + H^9)$:$(H^2 + H^8)$:H^6 = 1:3:6:6:6

7 7.05 (d): H^2, 7.85 (d): H^3, 2.45 (s): H^6, H^2:H^3 = 1:1

8 6.85 (d): H^2, 7.50 (d): H^3, 2.10 (s): H^6, 11.25 (s): H^7, H^2:H^3:H^6:H^7 = 2:2:3:1

9 11.25 (s): H^{15}, 7.00: $H^2 + H^{10}$, 7.60: $H^3 + H^{11}$, 2.3 (s): H^8, 2.10 (s): H^6, 1.96 (s): H^{14}, H^{15}:$(H^2 + H^{10})$:$(H^3 + H^{11})$:H^8:H^6:H^{14} = 1:6:6:6:6:3

10 11.25 (s): H^{15}, 6.90: $H^2 + H^{10}$, 7.50–7.70: $H^3 + H^{11}$, 4.2 (s): H^8, 2.3 (s): H^6, 2.10 (s): H^{14}, H^{15}:$(H^2 + H^{10})$:$(H^3 + H^{11})$:H^8:H^6:H^{14} = 1:4:4:2:3:3

Notes: For numbering see Scheme 1; coupling constant *J* (Hz); acetone-d (for **3**), $CDCl_3$-d (for **2, 4**, and **5**), and DMSO-d (for **6–10**) were used as solvents in NMR analyses; is: isomer, s: singlet, w: weak.

Synthesis of Compound **2**. A mixture of $[N_3P_3Cl_6]$ (10.34 g, 29.74 mmol), 4-hydroxybenzaldehyde (22.05 g, 180.56 mmol), and K_2CO_3 (50.00 g, 361.76 mmol) was stirred in THF (250 mL) at 0°C and then was reacted at ambient temperature for 48 h. The solvent was removed under vacuum. The residue was extracted with CH_2Cl_2 (4 × 75 mL). After the solvent was removed, a white solid (**2**) formed in 92% (23.57 g) yield.

TABLE 5 ^{13}C NMR Data of Compounds **2–10**

2	121.60: C^2, 131.40: C^3, 134.14: C^4, 154.80: C^1, 190.83: C^5
3	121.50: C^2, 121.33: C^2 (is), 129.00: C^3, 129.37: C^3 (is), 131.00: C^4, 131.00: C^4 (is), 148.19: C_5, 151.70: C^1
4	19.95: C^7, 121.62: C^2, 127.84: C^4, 130.17: C^3, 152.91: C^1, 155.17: C^5, 168.90: C^6
5	40.23: C^7, 40.94: C^7 (is), 121.81: C^2, 127.33: C^4, 128.75: C_4 (is), 130.45: C^3, 153.16: C^1, 156.56: C^5, 165.51: C^6, 170.96: C^6 (is)
6	151.44: C^7, 151.02: C^1, 148.38: C^{11}, 147.93: C^5, 131.38: C^{10}, 130.12: C^4, 130.29: C^4 (is), 129.16: C^9, 128.69: C^3, 121.72: C^8, 121.16: C^2, 62.51: C^6, 61.15: C^6 (is)
7	27.04: C^6, 120.96: C^2, 131.20: C^3, 134.54: C^4, 153.27: C^1, 197.00: C^5
8	11.90: C^6, 120.93: C^2, 127.36: C^3, 134.64: C^4, 150.39: C^1, 152.51: C^5
9	168.80: C^7, 161.74: C^5, 152.53: C^{13}, 151.73: C^1, 150.27: C^9, 134.77: C^4, 132.15: C^{12}, 128.90: C^3, 127.40: C^{11}, 121.12: C^2, 120.93: C^{10}, 19.98: C^8, 14.24: C^6 , 11.84: C^{14}
10	165.95: C^7, 163.46: C^5, 152.52: C^{13}, 152.00: C^1, 150.29: C^9, 134.66: C^4, 131.56: C^{12}, 129.09: C^3, 127.38: C^{11}, 121.16: C^2, 120.93: C^{10}, 41.94: C^8, 14.46: C^6, 11.89: C^{14}

Notes: For numbering see Scheme 1; acetone-d (for **3**), $CDCl_3$-d (for **2, 4**, and **5**), and DMSO-d (for **6–10**) were used as solvents in NMR analyses; is: isomer.

Synthesis of Compound **3**. A mixture of **2** (20.00 g, 23.21 mmol) and hydroxlaminehydrochloride (10.00 g, 143.90 mmol) was refluxed in pyridine (15 mL) for 3 h. After the reaction was complete, the mixture was allowed to cool and was slowly poured into water (100 mL) and reprecipitated twice from water. The white solid (**3**) was washed with alcohol and dried at 50°C in vacuum. Yield: 18.33 g, 83%.

Reaction of **3** *with Acetyl Chloride*. The solution of 0.40 mL (0.45 g, 5.80 mmol) acetyl chloride in acetone (10 mL) was slowly added dropwise to a stirred and cooled (0–5°C) mixture of **3** (0.80 g, 0.84 mmol) and K_2CO_3 (1.55 g, 11.25 mmol) in acetone (30 mL). The reaction was carried out at room temperature for 12 h. After the reaction was complete, the mixture was slowly poured into water (50 mL) and reprecipitated twice from water. The white solid (**4**) was recrystallized from alcohol. Yield: 0.76 g, 75%.

Reaction of **3** *with Chloroacetyl Chloride*. The solution of 0.46 mL (0.65 g, 5.77 mmol) chloroacetyl chloride in acetone (10 mL), **3** (0.80 g, 0.84 mmol), and K_2CO_3 (1.60 g, 11.57 mmol) in acetone (30 mL) were used for the preparation of **5** as for **4**. The white solid (**5**) was obtained in 72% (0.85 g) yield.

Reaction of **3** *with Methyl Iodide*. The solution of 0.40 mL (0.91 g, 6.42 mmol) methyl iodide in acetone (10 mL), **3** (0.80 g, 0.84 mmol), and K_2CO_3 (2.00 g, 14.47 mmol) in acetone (30 mL) were used for the preparation of **6** as for **4**. After the reaction was complete, the precipitated salt was filtered off and the solvent was removed under vacuum. The oily residue was solved in acetone and reprecipitated (as oily) from hexane. Compound **6** was obtained as solid in 80% (0.68 g) yield after the solvent was removed in vacuum for 24 h.

Synthesis of Compound **7**. A mixture of $[N_3P_3Cl_6]$ (7.00 g, 20.13 mmol), 4-hydroxyacetophenone (16.72 g, 122.80 mmol), and K_2CO_3 (34.00 g, 245.99 mmol) was refluxed in acetone (250 mL) for 3 h. The solvent was evaporated in vacuum, and the residue was extracted with CH_2Cl_2 (4×75 mL). After the evaporation of the solvent in vacuum, a white solid (**7**) formed in 87% (16.50 g) yield.

Synthesis of Compound **8**. Hydroxlaminehydrochloride (4.50 g, 64.75 mmol) and **7** (10.00 g, 10.57 mmol) were used for the preparation of **8** as for **3**. After the reaction was complete, the mixture was allowed to cool and the mixture was slowly poured into water (100 mL) and reprecipitated twice from water. The white solid (**8)** was obtained in 99% (10.89 g) yield.

Reaction of **8** *with Acetyl Chloride*. The solution of 0.40 mL (0.46 g, 5.80 mmol) acetyl chloride in acetone (10 mL), **8** (0.60 g, 0.58 mmol), and triethylamine (2 mL) in acetone (30 mL) were used for the preparation of **9** as for **4**. The oily product (**9**) was obtained in 79% (0.55 g) yield.

Reaction of **8** *with Chloroacetyl Chloride*. The solution of 0.40 mL (0.56 g, 5.02 mmol) acetyl chloride in acetone (10 mL), **8** (0.60 g, 0.58 mmol), and triethylamine (2 mL) in acetone (30 mL) were used for the preparation of **10** as for **4**. The solid (**10**) was obtained in 57% (0.42 g) yield.

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