Studies on Phosphoroheterocycle Chemistry I: A Facile Synthesis of New Tricyclic Phosphoroheterocycles 1,3,2-Oxazaphospholidino(or Oxazaphosphorino)[3,2-*a*][1,3,2]benzodiazaphosphorines

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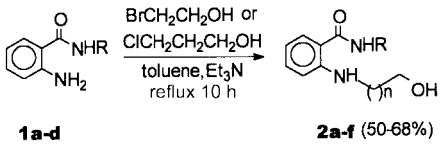
Received 19 February 2001; revised 7 May 2001

Abstract: Reaction procedures have been developed for the preparation of structurally different phosphoroheterocyclic compounds. Firstly, the treatment of PCl_3 with functionalized compound *N*-ethyl-2-(β-hydroxyethyl)aminobenzamide gave chlorinated phosphoroheterocycle, 1-(2-chloroethyl)-3-ethyl-2,3-dihydro-1,3,2-benzodiazaphosphorin-4(1*H*)-one 2-oxide. Secondly, novel tricyclic phosphoroheterocycles, 1,3,2-oxazaphospholidino(or oxazaphosphorino)[3,2-*a*][1,3,2]benzodiazaphosphorines, have been conveniently synthesized by cyclization of tris(diethylamino)phosphine with 2-(*N*-(β or γ-hydroxyalkyl)aminobenzamides. Finally, the reaction of 2-(*N*-ethoxycarbonylmethyl)amino-*N*-(2'-hydroxyethyl)-benzamide with $P(NEt_2)_3$ afforded five-membered phosphoroheterocycle, 3-(2'-*N*-ethoxycarbonylmethylamino)benzoyl-2-diethylamino-1,3,2-oxazaphospholidine-2-sulfide. Additionally, the preliminary bioassays showed that several title compounds possessed herbicidal activity to some extent.

Key words: multifunctional compounds, cyclization, facile synthesis, tricyclic phosphoroheterocycles, herbicidal activity

In past decades, there has been considerably growing interest in the use of tris(diethylamino)phosphine [$P(NEt_2)_3$] for the synthesis of pharmaceutically or agriculturally important phosphoroheterocyclic compounds.[1–4] As an useful reagent, $P(NEt_2)_3$ exhibited altered reactivity for a variety of cyclizing reactions in the preparation of cyclic products.[5,6] However, the use of $P(NEt_2)_3$ as a cyclocondensation reagent for the multifunctional compounds 2-(*N*-(β or γ-hydroxyalkyl)aminobenzamides has not been reported. Usually, the fused phosphoroheterocycles were prepared step by step in the ring closure,[7,8] while in this paper, we are interested in developing cyclocondensation methods for one-pot synthesis of fused-ring phosphoroheterocyclic compounds by utilizing $P(NEt_2)_3$ for the conversion of multifunctional compounds **2a**–**f** into the corresponding tricyclic heterocycles **3a**–**h** and **4a**–**c**.

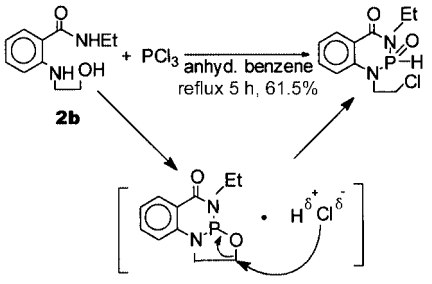
Starting materials of amides **2a**–**f** were routinely obtained by the reaction of 2-aminobenzamides **1a**–**d** with 2-bromoethanol or 3-chloropropanol in refluxing toluene (Scheme 1). It was found that triethylamine and 2-bromoethanol or 3-chloropropanol could be used in excess for



Scheme 1

improving the reaction yields. These materials **2a**–**f** could be readily prepared in large amounts and gave satisfactory analytical data (Table 1).

Cyclization reactions for the preparation of the desired tricyclic compounds were initially attempted by using PCl_3. Treatment of compound **2b** with PCl_3 in anhydrous benzene under reflux conditions gave the chlorinated bicyclic heterocycle, 1-(2-chloroethyl)-3-ethyl-2,3-dihydro-1,3,2-benzodiazaphosphorin-4(1*H*)-one 2-oxide. It is possible that the desired tricyclic phosphoroheterocycle was initially formed and subsequently underwent Arbuzov rearrangement under hydrochloride atmosphere to give chlorinated product (Scheme 2). These results suggested the use of alternative reagents, which should not release nucleophilic chloride ions.



Scheme 2

$P(NEt_2)_3$ may promote the desired sequential cyclizations for the synthesis of tricyclic products. The sizes of *N*-alkyl groups in the starting materials determined the reaction conditions and methods for the conversion of substrates **2a**–**f** into the corresponding tricyclic products (Scheme 3). For small substituents such as methyl or ethyl group, reactions could readily take place in anhydrous benzene in the presence of catalytic amount of iodine (Method A). The reaction progress could be monitored by thin layer chromatography and the yields of **3a**–**h** were

Synthesis 2001, No. 11, 28 08 2001. Article Identifier:
1437-210X,E;2001,0,11,1631,1634,ftx,en;F01701SS.pdf.

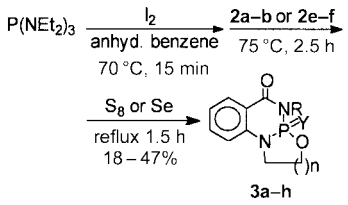
ISSN 0039-7881

Table 1 Compounds **2a**–**f** Prepared

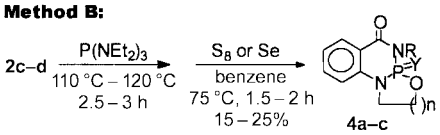
Product[a]	R	n	Yield (%)[b]	Mp (°C)	^{1}H NMR ($CDCl_3$/TMS) δ, *J* (Hz)
2a	Me	1	69	96–98	2.80 (d, 3 H, $^3J_{H-H}$ = 5.20, CH_3), 3.24 (t, 2 H, $^3J_{H-H}$ = 7.24, NHCH_2CH$_2$OH), 3.69 (t, 2 H, $^3J_{H-H}$ = 7.24, NHCH$_2$CH_2OH), 5.40 (br, 1 H, NH), 6.60–7.78 (m, 4 H_{arom})
2b	Et	1	53	70–72	1.21 (t, 3 H, $^3J_{H-H}$ = 7.24, NHCH$_2$CH_3), 3.30–3.50 (m, 4 H, NHCH_2CH$_3$ + NHCH_2CH$_2$OH), 3.79 (t, 2 H, $^3J_{H-H}$ = 7.36, NHCH$_2$CH_2OH), 6.20 (br, 1 H, NH), 6.55 –7.55 (m, 4 H_{arom})
2c	*i*-Pr	1	51	97–99	1.25 (d, 6 H, $^3J_{H-H}$=7.32, 2 × CH_3), 3.40 (t, 2 H, $^3J_{H-H}$ = 7.20, NHCH_2CH$_2$OH), 3.83 (t, 2 H, $^3J_{H-H}$ = 7.20, NHCH$_2$CH_2OH), 4.16–4.40 (d, 1 H, $^3J_{H-H}$ = 7.32, CH), 4.85 (br, 1 H, OH), 6.00 (br, 1 H, NH), 6.60–7.44 (m, 4 H_{arom})
2d	Bn	1	53	134–136	3.24 (t, 2 H, $^3J_{H-H}$ = 7.24, NCH_2CH$_2$OH), 3.70 (t, 2 H, $^3J_{H-H}$ = 7.24, NCH$_2$CH_2OH), 4.08 (br, 1 H, NH), 4.52 (d, 2 H, $^3J_{H-H}$ = 5.40, NHCH_2Ph), 6.60–8.01 (m, 9 H_{arom})
2e	Me	2	69	97–98	1.63–1.89 (m, 2 H, NCH$_2$CH_2CH$_2$OH), 2.81 (d, 3 H, $^3J_{H-H}$ = 4.80, CH_3), 3.21 (t, 2 H, $^3J_{H-H}$ = 7.26, NCH_2CH$_2$CH$_2$OH), 3.61 (t, 2 H, $^3J_{H-H}$ = 7.20, NCH$_2$CH$_2$CH_2OH), 4.56 (br, 1 H, NH), 6.48–7.62 (m, 4 H_{arom}), 8.15 (br, 1 H, CONH)
2f	Et	2	51	68–70	1.30 (t, 3 H, $^3J_{H-H}$ = 7.28, CH_3), 1.64–1.86 (m, 2 H, NCH$_2$CH_2CH$_2$O), 3.10–3.34 (m, 4 H_{arom} NCH_2CH$_2$CH$_2$O + NCH_2CH$_3$), 3.60 (t, 2 H, $^3J_{H-H}$ = 7.24, NCH$_2$CH$_2$CH_2OH), 4.24 (br, 1 H, NH), 6.50–7.65 (m, 4 H_{arom}), 8.08 (br, 1 H, CONH)

[a] Satisfactory microanalyses obtained: C, H, N ±0.4.
[b] Yields of isolated products.

Method A:



Method B:



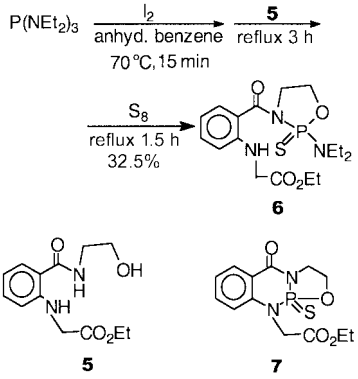
Compds.	n	R	Y	Compds.	n	R	Y
3a	1	Me	S	3g	2	Et	S
3b	1	Me	Se	3h	2	Et	Se
3c	1	Et	S	4a	1	Pr^i	S
3d	1	Et	Se	4b	1	Pr^i	Se
3e	2	Me	S	4c	1	Bn	S
3f	2	Me	Se				

Scheme 3

generally in the range of 18–47%. For several other groups such as isopropyl and benzyl in the substrates, Method A appeared to be unsatisfactory for ring-closure due to the steric effect of *N*-alkyl groups. Alternatively, Method B, which was to heat the compounds **2c**–**d** directly with $P(NEt_2)_3$, became effective in affording the target products **4a**–**c**. Reaction temperatures should be controlled in the range of 110–120 °C. Whereas, higher temperature (>150 °C) usually led to the formation of polymers, lower temperature (<90 °C) gave poor yields of **4a**–**c**. In addition, a slight excess of $P(NEt_2)_3$ was found to improve the yield in both the methods. The title compounds were characterized by spectroscopy and elemental analyses (Table 2), and the molecular structure of compound **3c** was further confirmed by X-ray diffraction determination.[9]

For the purpose of synthesizing another type of tricyclic phosphoroheterocycle **7** by similar methods, reacting $P(NEt_2)_3$ with multifunctional compound **5** was carried out. Although the target product **7** was not produced (Scheme 4), a five-membered phosphoroheterocycle **6** was generated with moderate yields (33% by Method A, 48% by Method B). The different results from compound **5** will be explained after further studies by the use of a variety of other substrates.

The preliminary bioassays showed that several compounds had herbicidal activity against *Brassica campestris*. Under the concentration of 100mg/L, the compounds **3a**, **3c** and **6** showed inhibition against *Brassica campestris* of 32.4%, 23.7% and 47.6%, respectively.



Scheme 4

Table 2 Compounds **3a**–**h** and **4a**–**c** Prepared

Product[a]	Yield (%)[b]	Mp(°C)	^{31}P NMR δ	^{1}H NMR ($CDCl_3$/TMS) δ, *J* (Hz)
3a	24	108–110	72.38	3.22 (d, 3 H, $^{3}J_{P\text{-}H}$ = 9.35, CH_3), 3.70–3.85 (m, 1 H, $NCH_aH_bCH_2$), 4.31–4.68 (m, 3 H, $NCH_aH_bCH_2O$), 7.05–8.15 (m, 4 H_{arom})
3b	19	114–116	–	3.22 (d, 3 H, $^{3}J_{P\text{-}H}$ = 9.43, CH_3), 3.70–3.85 (m, 1 H, NCH_aH_b), 4.29–4.66 (m, 3 H, $NCH_aH_bCH_2O$), 7.10–8.14 (m, 4 H_{arom})
3c	47	130–132	71.94	1.33 (t, 3 H, $^{3}J_{H\text{-}H}$ = 7.07, CH_3), 3.74–3.95 (m, 3 H, $NCH_aH_bCH_2O + CH_2CH_3$), 4.28–4.75 (m, 3 H, $NCH_aH_bCH_2O$), 7.04–8.09 (m, 4 H_{arom})
3d	25	132–134	–	1.33 (t, 3 H, $^{3}J_{H\text{-}H}$ = 7.02, CH_3), 3.72–3.85 (m, 3 H, $NCH_aH_bCH_2O + CH_2CH_3$), 4.30–4.73 (m, 3 H, $NCH_aH_bCH_2O$), 7.06–8.10 (m, 4 H_{arom})
3e	31	178–180	65.42	2.14–2.40 (m, 2 H, $NCH_2CH_2CH_2O$), 3.21 (d, 3 H, $^{3}J_{P\text{-}H}$ = 8.04, NCH_3), 3.83–3.90 (m, 2 H, $NCH_2CH_2CH_2O$), 4.24–4.80 (m, 2 H, $NCH_2CH_2CH_2O$), 6.89– 8.14 (m, 4 H_{arom})
3f	22	182–184	–	2.03–2.42 (m, 2 H, $NCH_2CH_2CH_2O$), 3.19 (d, 3 H, $^{3}J_{P\text{-}H}$ = 10.11, NCH_3), 3.75–3.85 (m, 2 H, $NCH_2CH_2CH_2O$), 4.28–4.83 (m, 2 H, $NCH_2CH_2CH_2O$), 6.87–8.14 (m, 4 H_{arom})
3g	41	186–188	65.44	1.35 (t, 3 H, $^{3}J_{H\text{-}H}$ = 7.12, CH_2CH_3), 2.02–2.40 (m, 2 H, $NCH_2CH_2CH_2O$), 3.72–4.84 (m, 6 H, $NCH_2CH_2CH_2O + CH_2CH_3$), 6.88–8.13 (m, 4 H_{arom})
3h	25	193–195	66.95	1.33 (t, 3 H, $^{3}J_{H\text{-}H}$ = 7.24, CH_3), 2.05–2.40 (m, 2 H, $NCH_2CH_2CH_2O$), 3.70–4.83 (m, 6 H, $NCH_2CH_2CH_2O + CH_2CH_3$), 6.65–8.13 (m, 4 H_{arom})
4a	25	157–159	70.49	1.54–1.58 (q, 6 H, $^{4}J_{P\text{-}H}$ = 3.27, $^{3}J_{H\text{-}H}$ = 4.74, 2 × CH_3), 3.65–3.73 (m, 1 H, $NCH_aH_bCH_2O$), 4.21–4.63 (m, 4 H, $NCH_aH_bCH_2O$ + NCH), 6.99–8.08 (m, 4 H_{arom})
4b	15	162–164	67.77	1.56–1.63 (t, 6 H, $^{4}J_{P\text{-}H}$ = 6.87, $^{3}J_{H\text{-}H}$ = 7.06, 2 × CH_3), 3.66–3.70 (m, 1 H, $NCH_aH_bCH_2O$), 4.25–4.66 (m, 4 H, $NCH_aH_bCH_2O$ + NCH), 7.03–8.11 (m, 4 H_{arom})
4c	16	148–150	–	3.62–4.01 (m, 2 H, NCH_2CH_2O), 4.22–4.52 (m, 2 H, NCH_2CH_2O), 4.60– 5.36 (qq, 2 H, $^{2}J_{H\text{-}H}$ = 14.56, $^{3}J_{P\text{-}H}$ = 10.25, NCH_2Ph), 7.04–8.17 (m, 9 H_{arom})

[a] Satisfactory microanalyses obtained: C, H, N ±0.4.
[b] Yields of isolated pure products.

In summary, we have studied the reaction of $P(NEt_2)_3$ with multifunctional compounds and found convenient methods for generating interesting tricyclic phosphoroheterocyclic compounds such as 1,3,2-oxazaphospholidino(or oxazaphosphorino)[3,2-*a*][1,3,2] benzodiazaphosphorines, which, to some extent, exhibited herbicidal activity.

^{1}H NMR and ^{31}P NMR spectra were recorded with a Bruker AC-P 200 Spectrometer ($CDCl_3$ as solvent, TMS as internal, 85% H_3PO_4 as external standard) with exception of the compounds **2a**–**f** (recorded on a Jeol-FX-90Q Spectrometer). Melting Points were determined using Thomashoover melting point apparatus and are uncorrected. Elemental analyses were carried out with Yanaco CHN CORDER MT-3 autoanalysis apparatus. EI-MS spectra were recorded on a VG-7070E spectrometer. X-ray analysis was performed on a Bruker Smart 1000 diffractometer with graphite monochromated MoK_α radiation (λ = 0.71073 Å) and ω–2θ scan mode. 2-Bromoethanol and $P(NEt_2)_3$ were purchased from Aldrich (Germany). $P(NEt_2)_3$ and PCl_3 were distilled before use. Benzene was dried over sodium. Petroleum ether used had the boiling range 30–60 °C.

Compounds **1a**–**d** were prepared according to Lit.[10] **1a**: R = Me; Yield: ~100%; mp 128–130 °C; **1b**: R = Et; Yield: 91%; mp 100–101 °C (Lit.[10] mp 102–103 °C); **1c**: R = *i*-Pr; Yield: 93%; mp 148 °C; **1d**: R = Bn; Yield: 85%; mp 120–121 °C (Lit.[10] mp123–123.5 °C). β-Chloropropanol was prepared according to Lit.[11].

2-(β- or γ-Hydroxyalkyl)aminobenzamides 2a–f; General Procedure

To a 100 mL three-necked flask containing a mixture of compound **1a**–**d** (0.037 mol), anhyd toluene (35 mL) and Et_3N (5.65g, 0.056 mol) was added 2-bromoethanol (or β-chloropropanol) (0.074 mol) at r.t. The mixture was heated to reflux for 10 h, and then cooled to r.t. After the removal of precipitate, the filtrate was concentrated under reduced pressure to give a sticky oil, which was purified on a silica gel column (100 × 4.5 cm, 250–300 mesh) using petroleum ether–EtOAc (1:1 v/v) as eluent. Products **2a**–**f** were obtained and recrystallized from EtOAc–EtOH (4:1 v/v).

1-(2-Chloroethyl)-3-ethyl-2,3-dihydro-1,3,2-benzodiazaphosphorin-4(1*H*)-one 2-Oxide

A suspension of **2b** (2.08g, 0.01mol) in anhydrous benzene (100 mL) in a 500 mL three-necked flask was heated with stirring until the mixture became transparent. PCl_3 (1 equiv) was added dropwise within 30 min under N_2 and the resulting solution was refluxed for another 5 h. The mixture was then concentrated and the crude product was purified by flash chromatography using EtOAc–petroleum ether (1:3 v/v) as eluent. Recrystallization from Et_2O–petroleum ether (1:5 v/v) gave a solid (1.67 g, 62%; mp 99–101 °C).

Anal. Calcd for $C_{11}H_{14}ClN_2O_2P$ (272.5): C, 48.44; H, 5.14; N, 10.28. Found: C, 48.52; H, 5.08; N, 10.54.

^{1}H NMR: δ =1.32 (t, 3 H, $^3J_{H-H}$ = 7.07 Hz, CH_3), 3.60–4.10 (m, 6 H, 3 × CH_2), 7.87 (d, 1 H $^1J_{P-H}$ = 642.03 Hz, PH), 6.9–8.2 (m, 4 H_{arom}).

EIMS: *m*/*z* (%) = 272 (M^+, 22.7) 274 (M^+ + 2, 7.6).

1,3,2-Oxazaphospholidino-(or Oxazaphosphorino)-[3,2-*a*][1,3,2]benzodiazaphosphorines 3a–h; General Procedure

Method A: To a solution of $P(NEt_2)_3$ (0.55g, 2.2 mmol) and anhyd benzene (30 mL) in a 100 mL three-necked flask was added I_2 (0.051 g, 0.2 mmol) under N_2 at r.t. The reaction mixture was heated at 70 °C for 15 min, and then cooled to r.t. After the addition of corresponding **2a**,**b**,**e**,**f** (2 mmol), the mixture was subsequently heated at 75 °C for another 2.5 h. After cooling to r.t., S_8 or Se (2 mmol, 1 equiv) was added and the mixture was then heated to reflux for another 1.5 h. After the solvent was removed under reduced pressure, the desired product was isolated by flash chromatography with EtOAc–petroleum ether (1:1 v/v) as eluent and further purified by recrystallization from a mixture of $CHCl_3$ and petroleum ether (1:5 v/v).

1,3,2-Oxazaphospholidino-(or Oxazaphosphorino)-[3,2-*a*][1,3,2]benzodiazaphosphorines 4a–c; General Procedure (Method B)

A mixture of **2c** or **2d** (2 mmol) and $P(NEt_2)_3$ (0.5 g, 2 mmol) was heated under N_2 in a 25 mL two-necked flask at 110–120 °C for 2.5–3 h. After the solution was cooled to r.t., a solution of a slight excess of S_8 or Se (2.2 mmol) in anhyd benzene (15 mL) was added. The mixture was heated at 75 °C for 1.5–2 h and the solvent was evaporated under reduced pressure. The product was purified by chromatography on a silica gel (100 × 3.5, 250–300 mesh) using a mixture of petroleum ether and EtOAc (3:1 v/v) as eluent to give solid materials, which were recrystallized from a mixture of $CHCl_3$ and petroleum ether (1:5 v/v).

2-(*N*-Ethoxycarbonylmethyl)amino-*N*-(2'-hydroxyethylene)-benzamide (5)

To a solution of 1-ethoxycarbonylmethyl-2*H*-3,1-benzoxazine-2,4(1*H*)-dione[12] (1.25 g, 5 mmol) and MeCN (40 mL) in a 100 mL three-necked flask was added aminoethanol (0.31 g, 5.1 mmol) at r.t. After the mixture had been stirred at r.t. for 10 h and 50 °C for another 3 h, the solution was concentrated under reduced pressure. The residue was washed with a small amount of Et_2O (4 mL) to give a pale solid material. Colorless crystals (1.26 g, 95%) were obtained after recrystallization of the crude product from a mixture of $CHCl_3$ and EtOAc (1:1 v/v); mp 95–97 °C.

Anal. Calcd for $C_{13}H_{18}N_2O_4$ (266.3): C, 58.65; H, 6.77; N, 10.53. Found: C, 58.82; H, 6.53; N, 10.77.

^{1}H NMR ($CDCl_3$/TMS): δ = 1.30 (t, 3 H, $^3J_{H-H}$ = 7.20 Hz, CH_3), 3.45–3.70 (m, 2 H, NCH_2CH_2O), 3.75–3.92 (m, 2 H, NCH_2CH_2O), 4.01 (s, 2 H, NCH_2C=O), 4.18–4.40 (q, 2 H, OCH_2CH_3), 5.32 (br, 1 H, NH), 6.60–7.59 (m, 4 H_{arom}).

3-(2'-*N*-Ethoxycarbonylmethylamino)benzoyl-2-diethylamino-1,3,2-oxazaphospholidine-2-sulfide (6)

To a solution of $P(NEt_2)_3$ (0.74 g, 3 mmol) and anhyd benzene (30 mL), kept in a 100 mL three-necked flask, was added I_2 (0.076 g, 0.3 mmol) under N_2. The reaction mixture was subsequently warmed up to 70 °C and stirred for 15 min. After cooling to r.t., compound **5** (0.79 g, 3 mmol) was added to the solution. The reaction mixture was kept at 75 °C for another 2.5 h. and then S_8 (0.1 g, 3.1 mmol) was added. After heating the solution at reflux for another 1.5 h, the solvent was evaporated under reduced pressure to give a yellow oil, which was purified on a silica gel column (100 × 3.5, 250–300 mesh) using petroleum ether–EtOAc (3:1 v/v) as eluent. The obtained crude product **6** was further purified by recrystallization from a mixture of $CHCl_3$ and petroleum ether (1:5 v/v) to afford 0.39 g (33%) of a colorless product; mp 92 °C.

Anal. Calcd for $C_{17}H_{26}N_3O_4PS$ (399.5): C, 51.13; H, 6.52; N, 10.53. Found: C, 50.81; H, 6.51; N, 10.21.

^{1}H NMR ($CDCl_3$/TMS) δ = 0.97 [t, 6 H, $^3J_{H-H}$ = 7.13 Hz, $N(CH_2CH_3)_2$], 1.27 (t, 3 H, $^3J_{H-H}$ = 7.14 Hz, OCH_2CH_3), 2.92–3.02 [m, 4 H, $N(CH_2CH_3)_2$], 3.95–4.15 (m, 4 H, NCH_2CH_2O + NCH_2C=O), 4.21–4.49 (m, 4 H, NCH_2CH_2O + OCH_2CH_3), 5.82 (br, 1 H, NH), 6.89–7.89 (m, 4 H_{arom}).

^{31}P NMR ($CDCl_3$/85% H_3PO_4): δ =76.32.

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