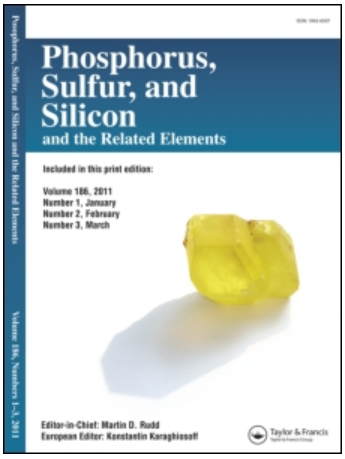

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Efficient One-Pot Synthesis of Naphthoquinone-Fused Phosphorous Heterocycles via Mannich-Type Reaction

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Efficient One-Pot Synthesis of Naphthoquinone-Fused Phosphorous Heterocycles via Mannich-Type Reaction

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A series of naphthoquinone-fused phosphorus heterocycles was synthesized via a three-component Mannich-type reaction. 2-Chloroethoxylphosphorodichloridite was used as a phosphorus substrate to explore the structure–activity relationships of the 2-position of the heterocycles. One spiral heterocycle compound was structurally characterized by a single-crystal X-ray diffraction analysis.

Keywords α-Aminophosphonates; heterocycles; Mannich reaction; quinine

INTRODUCTION

1,4-Naphthoquinones are essential constituents of a variety of biologically active natural products that include vitamin K,[1] plumbagin,[2–4] and shikonin.[5] Many 1,4-naphthoquinone-fused heterocycles containing nitrogen, sulfur, or selenium atoms have displayed antitumor, antibacterial, antimalarial, and antifungal activities.[6–11] As a consequence, considerable efforts have been directed toward developing methods that will construct heterocycles to various 1,4-naphthoquinone derivatives.[12–20] The three-component Mannich-type reaction of phosphorous acid, amines, and formaldehyde was developed by Moedritzer and Irani in 1966.[21] This method has proved facile for the preparation of α-aminomethylphosphonic acids (Scheme 1).[21]

Considerable progress has been made in recent years, particularly use of different trivalent phosphorus species, amines, and carbonyl materials as reactants. The phosphorus species can be PCl_3, $RPCl_2$,

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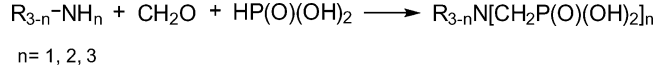
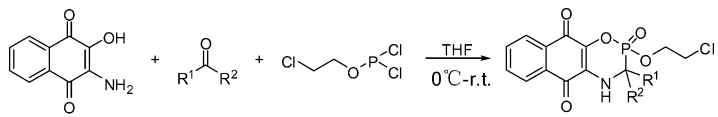


SCHEME 1 The three-component Mannich-type reaction developed by Moedritzer and Irani.[21]

$ROPCl_2$, $(RO)_3P$, and R_3P,[22–25] and carbonyl components can be different alkyl aldehydes, aromatic aldehydes, and ketones.[26,27] Amines can be replaced by phosphoamide, urea, and carbamate.[28–30] These building blocks can be easily utilized for the construction of various α-aminophosphonates or α-aminophosphonic acids by using this synthetic approach. Recently, we developed the first benzene-fused cyclic α-aminophosphonates formation via the three-component Mannich-type reaction in organophosphorus chemistry.[23,31] We became interested in the reaction of 1,4-naphthoquinone substrates and the formation of the naphthoquinone-fused phosphorous heterocycles. The synthesis and antitumor activities of 2-alkoxy-substituted cyclic α-aminophosphonates have been explored by our group.[22,25,32] To examine the structure–activity relationship (SAR) for the 2-position, we report here the synthesis of chloroethoxy-substituted naphthoquinone-fused cyclic α-aminophosphonates.

RESULTS AND DISCUSSION

As shown in Scheme 2, the starting material 2-amino-3-hydroxy-1,4-naphthoquinone **1**, was prepared by the use of a previously reported method.[25] When it was allowed to react with 2-chloroethoxyl phosphorodichloridite **10**[33] and ketones or aromatic aldehydes **2–9**, the target compounds **11–18** were obtained in 67–81% yields.



1 **2-9** **10** **11-18**

2, 11: $R^1 = R^2 = Me$ **6, 15:** $R^1, R^2 = (CH_2)_5$
3, 12: $R^1 = R^2 = Et$ **7, 16:** $R^1 = Ph, R^2 = H$
4, 13: $R^1 = Me, R^2 = Et$ **8, 17:** $R^1 = 4\text{-}MeOPh, R^2 = H$
5, 14: $R^1, R^2 = (CH_2)_4$ **9, 18:** $R^1 = 2\text{-}NO_2Ph, R^2 = H$

SCHEME 2 Synthesis of naphthoquinone-fused phosphorus heterocycles **11–18**.

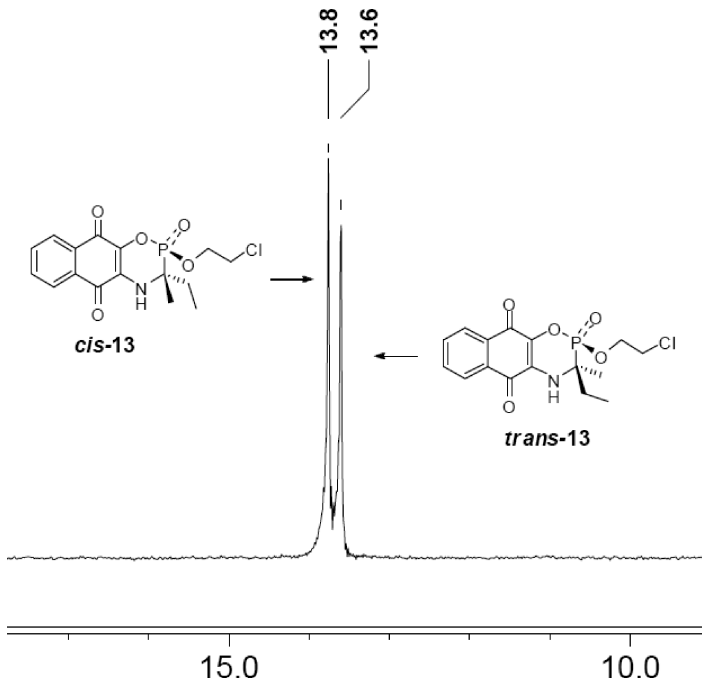


FIGURE 1 The structures and ^{31}P NMR spectra of isomers of compound **13**.

In the case of compound **13**, the mixture of two isomers was isolated based on the ^{31}P NMR (13.8 ppm and 13.6 ppm) and ^{1}H NMR spectra. The determination of *cis* and *trans* isomer was according to previous reports.[23,25,32] Unfortunately, they could not be separated by column chromatography. The structures and ^{31}P NMR spectra of two isomers of compound **13** are shown in Figure 1. On the basis of ^{31}P NMR analyses, we consider that the *cis*-isomers are generated preferably because of steric hindrance effects. Consequently, the *cis*-isomer is the major product, and its ^{31}P NMR chemical shift should be 13.8 ppm. The other *trans*-isomer is the minor product (^{31}P NMR, 13.6 ppm).

As for compounds **16**–**18**, the ^{31}P NMR of the crude product shows only one peak. We consider that the *cis*-isomers were generated preferably because of steric hindrance effects, and the other *trans*-isomers were not formed. The results are similar to our previous reports.[23,32]

A red platelet crystal of compound **14** ($C_{18}H_{20}ClNO_4P$) having approximate dimensions of 0.24 × 0.20 × 0.12 mm was mounted on a glass fiber for X-ray crystallographic analysis. The ORTEP diagram of compound **14**[34] is shown in Figure 2, and it is a spiral

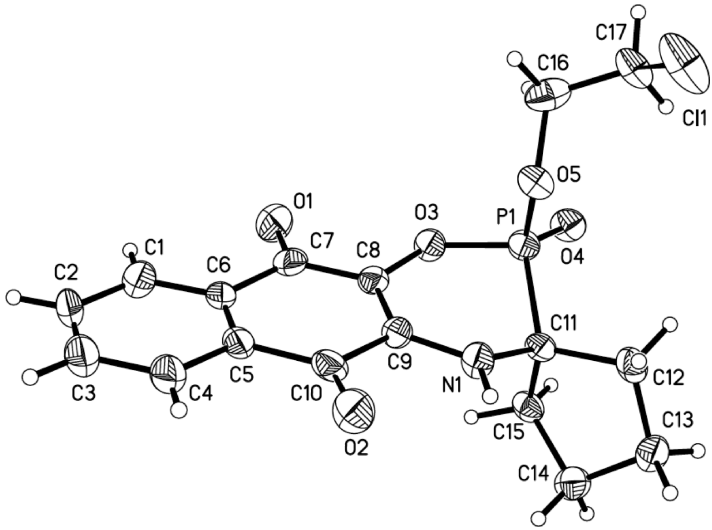


FIGURE 2 ORTEP drawing of compound **14**.

phosphorus-heterocycle compound. The data refinement parameters of compound **14** are listed in Table I. All measurements were made on a Rigaku Saturn CCD area detector with graphite-monochromated MoKα radiation. The structure was solved by direct methods and expanded using Fourier techniques. Some non-hydrogen atoms were refined anisotropically, while the rest were refined isotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement on F^2 was based on 2939 observed reflections and 238 variable parameters. The final agreement factors are based on the reflections with $I > 2\sigma(I)$. All calculations were performed using the CrystalStructure crystallographic software package,[35] except for refinement, which was performed using SHELXL-97.[35]

The selected bond lengths and angles for compound **14** are listed in Table II. The bond length of P(1)–O(4) (1.478 Å) is shorter than that of P(1)–O(3) (1.607 Å) and P(1)–O(5) (1.584 Å). The bond angles of O(4)-P(1)-O(5) (115.09), O(4)-P(1)-O(3) (110.70), O(5)-P(1)-O(3) (104.83), O(4)-P(1)-C(11) (117.16), O(5)-P(1)-C(11) (106.57○), and O(3)-P(1)-C(11) (102.57) indicate that the phosphorus atom adopts a distorted tetrahedral configuration. The bond angles between the alpha carbon atom (C11) and adjacent atoms (N1, C12, P1, C15) also display the distorted tetrahedral configuration. Because of anchoring of naphthyl ring, the six-member phosphorus heterocycle is almost coplanar, and to some extent, the confirmation of cyclopentyl ring cannot invert

TABLE I Crystal Structure and Data Refinement Parameters of Compound 14

Empirical Formula	$C_{17}H_{17}ClNO_5P$	μ (mm^{-1})	0.352
Formula Weight	381.74	Crystal size (mm)	0.24 × 0.20 × 0.12
Crystal System/Space Group	Monoclinic, $P2_1/c$	Color/Shape	Red/platelet
a/Å	8.3947(16) A	Temp (K)	113(2)
b/Å	16.079(3)	Theta range for collection	3.02 to 25.03°
c/Å	12.547(2)	Reflections collected	12235
α/°	90	Independent reflections	2947
β/°	98.156(10)	Data/restraints/parameters	2947/5/243
γ/°	90	Goodness of fit on F^2	0.902
V/$Å^3$	1676.4(5)	Final R indices [$I > 2\sigma(I)$]	R1 = 0.0728, wR2 = 0.1596
Z	4	R indices (all data)	R1 = 0.1054, wR2 = 0.1756
D_{calc} (g/cm^3)	1.512	Largest difference peak/hole	0.618 and –0.559 $e.A^{-3}$

TABLE II Selected Bond Lengths [Å] and Angles [°] for Compound 14

Bond	Dist.	Bond	Dist.
P(1)-O(4)	1.478(3)	N(1)-H(1A)	0.856(10)
P(1)-O(5)	1.584(3)	C(8)-C(9)	1.365(6)
P(1)-O(3)	1.607(3)	C(11)-C(12)	1.547(6)
P(1)-C(11)	1.825(4)	C(11)-C(15)	1.552(6)
O(1)-C(7)	1.249(5)	C(12)-C(13)	1.536(6)
O(2)-C(10)	1.219(5)	C(13)-C(14)	1.557(6)
O(3)-C(8)	1.422(5)	C(14)-C(15)	1.519(6)
O(5)-C(16)	1.462(5)	C(16)-C(17)	1.429(8)
N(1)-C(9)	1.356(5)	C(16)-C(18)	1.541(12)
N(1)-C(11)	1.483(5)	C(17)-Cl(1)	1.753(11)
Angle	[°]	Angle	[°]
O(4)-P(1)-O(5)	115.09(18)	N(1)-C(11)-P(1)	106.3(3)
O(4)-P(1)-O(3)	110.70(17)	C(12)-C(11)-P(1)	114.3(3)
O(5)-P(1)-O(3)	104.83(17)	C(15)-C(11)-P(1)	109.2(3)
O(4)-P(1)-C(11)	117.16(19)	C(13)-C(12)-C(11)	106.5(3)
O(5)-P(1)-C(11)	105.09(18)	O(3)-P(1)-C(11)	102.57(18)

freely, which is similar to the previously reported spiral heterocyclic compounds.[22,31,32]

CONCLUSIONS

In conclusion, we report that the three-component Mannich reaction, which is applied to prepare chloroethoxy-substituted naphthoquinone-fused phosphorus heterocycles. The synthetic processes described here provide valuable routes to various phosphorus heterocycles. The structures of isomers of compound **13** have been confirmed based on the ^{31}P NMR spectra. X-ray analysis reveals the structure of a spiral ring for compound **14**, and its structural characteristics have been discussed. Exploration of the biological properties of these compounds is in progress.

EXPERIMENTAL

All melting points were measured on a Yanaco apparatus and uncorrected. NMR spectra were determined on a Bruker Avance 300 NMR instrument in $CDCl_3$, and chemical shifts are expressed as δ. Coupling constants J are given in Hz. Tetramethylsilane was used as an internal standard for ^1H NMR, and 85% H_3PO_4 as an external standard for

^{31}P NMR spectroscopy. MS were recorded on a Polaris-Q instrument of Thermofinnigan. Elemental analysis was carried out on a Yanaco CHN-CORDER MT-3 Analyzer. X-Ray analysis was performed on a Rigaku Saturn detector diffractometer with MoKa radiation ($\lambda = 0.71070$ Å). Column chromatograghy was performed using silica gel H (10–40 mm, Haiyang chemical Factory of Qingdao). THF was dried with sodium and redistilled prior to use.

The General Procedure for the Synthesis of Compounds 11–18

2-Amino-3-hydroxy-1,4-naphthoquinone (0.5 g, 2.6 mmol) and 2-chloroethoxyl phosphorodichloridite (2.6 mmol) were dissolved in anhydrous THF (15 mL) with stirring at 0°C. After 15 min, the ketone or aromatic aldehyde (2.6 mmol) was added. The reaction mixture was allowed to warm to room temperature and continuously stirred for 18 h. The resulting mixture was filtered, and the filtrate was concentrated in vacuo. The residue was purified by column chromatography on silica gel and eluted with EtOAc:hexane (1:1, v/v) to afford the analytically pure products.

2-(2-Chloroethoxy)-3,3-dimethyl-3,4-dihydro-2H-naphtho[2,3-e][1,4,2]oxazaphosphinane-5,10-dione 2-oxide (11)

Orange power, yield 75%; mp 180–182°C. ^{1}H NMR (300 MHz, CD_3Cl): δ_H 1.44 (d, $^3J_{PCH} = 16.2$ Hz, 3 H, CH_3), 1.57 (d, $^3J_{PCH} = 16.2$ Hz, 3 H, CH_3), 3.81 ($J = 5.06$ Hz, 2 H, CH_2Cl), 4.32–4.41 (m, 2 H, OCH_2), 5.34–5.42 (br, 1 H, NH), 7.65–7.98 (m, 4 H, C_6H_4). ^{31}P NMR (121 MHz, CD_3Cl): δ_P 14.8. Calcd. (%) for $C_{15}H_{15}ClNO_5P$: C, 50.65; H, 4.25; N, 3.94. Found (%): C, 50.67; H, 4.30; N, 4.01. MS: m/z 355 (M^+).

2-(2-Chloroethoxy)-3,3-diethyl-3,4-dihydro-2H-naphtho[2,3-e][1,4,2]oxazaphosphinane-5,10-dione 2-oxide (12)

Orange power, yield 79%; mp 212–214°C. ^{1}H NMR (300 MHz, CD_3Cl): 1.01 (t, 3H, $J = 7.35$ Hz, CH_2CH_3), 1.12 (t, 3H, $J = 7.35$ Hz, CH_2CH_3), 1.75–2.18 (m, 4H, 2×CH_2CH_3), 3.81 ($J = 5.06$ Hz, 2 H, CH_2Cl), 4.32–4.41 (m, 2 H, OCH_2), 5.34–5.42 (br, 1 H, NH), 7.65–7.98 (m, 4 H, C_6H_4). ^{31}P NMR (121 MHz, CD_3Cl): δ_P 14.8. Calcd. (%) for $C_{17}H_{19}ClNO_5P$: C, 53.21; H, 4.99; N, 3.65;. Found (%):C, 53.28; H, 5.02; N, 3.87. MS: m/z 383 (M^+).

2-(2-Chloroethoxy)-3-ethyl-3-methyl-3,4-dihydro-2H-naphtho[2,3-e][1,4,2]oxazaphosphinane-5,10-dione 2-oxide (13)

Orange power, yield 70%; mp 153–155°C. ^{1}H NMR (300 MHz, CD_3Cl): δ_H *cis*-**13** 1.23 (t, $J = 8.29$ Hz, 3 H, CH_2CH_3), 1.61 (d, $^3J_{PCCH} = 16.4$

Hz, 3 H, CH_3), 1.94–2.26 (m, 2 H, CH_2CH_3), 3.81 (J = 5.06 Hz, 2 H, CH_2Cl), 4.32–4.41 (m, 2 H, OCH_2), 5.43–5.51 (br, 1 H, NH), 7.65–8.14 (m, 4 H, C_6H_4); *trans*-**13** 1.07 (t, J = 6.78 Hz, 3 H, CH_2CH_3), 1.71 (d, $^3J_{PCCH}$ = 16.4 Hz, 3 H, CH_3), 1.94–2.26 (m, 2 H, CH_2CH_3), 3.81 (J = 5.06 Hz, 2 H, CH_2Cl), 4.32–4.41 (m, 2 H, OCH_2), 5.43–5.51 (br, 1 H, NH), 7.65–8.14 (m, 4 H, C_6H_4); ^{31}P NMR (121 MHz, CD_3Cl) δ *cis*-**13** 13.8; *trans*-**13** 13.6; Calcd. (%) for $C_{16}H_{17}ClNO_5P$: C, 51.98; H, 4.63; N, 3.79. Found (%):C, 52.04; H, 4.67; N, 3.59. MS: m/z 369 (M^+).

2-(2-Chloroethoxy)-4H-spiro{naphtho[2,3-e][1,4,2]oxazaphosphinane-5,10-dione,1′-cyclopentane} 2-oxide (14)

Orange power, yield 81%; mp 179–181°C. ^{1}H NMR (300 MHz, CD_3Cl): δ_H 1.44–2.27 (m, 8 H, $(CH_2)_4$), 3.71 (t, J = 6.1 Hz, 2 H, CH_2Cl), 4.37–4.52 (m, 2 H, OCH_2), 5.34–5.42 (br, 1 H, NH), 7.65–8.61 (m, 4 H, C_6H_4). ^{31}P NMR (121 MHz, CD_3Cl): δ_P 13.7. Calcd. (%) for $C_{17}H_{17}ClNO_5P$: C, 53.49; H, 4.49; N, 3.67. Found (%):C, 53.52; H, 4.52; N, 3.55. MS: m/z 381 (M^+).

2-(2-Chloroethoxy)-4H-spiro{naphtho[2,3-e][1,4,2]oxazaphosphinane-5,10-dione,1′-cyclohexane} 2-oxide (15)

Orange power, yield 80%; mp 188–190°C. ^{1}H NMR (300 MHz, CD_3Cl): δ_H 1.15–1.94 (m, 10 H, $(CH_2)_5$), 3.72 (t, J = 5.7 Hz, 2 H, CH_2Cl), 4.42–4.54 (m, 2 H, OCH_2), 5.29–5.38 (br, 1 H, NH), 7.67–8.63 (m, 4 H, C_6H_4). ^{31}P NMR (121 MHz, CD_3Cl): δ_P 13.6. Calcd. (%) for $C_{18}H_{19}ClNO_5P$: C, 54.63; H, 4.84; N, 3.54. Found (%): C, 54.65; H, 4.90; N, 3.49. MS: m/z 395 (M^+).

2-(2-Chloroethoxy)-3-phenyl-3,4-dihydro-2H-naphtho[2,3-e][1,4,2]oxazaphosphinane-5,10-dione 2-oxide (16)

Orange power, yield 67%; mp 164–166°C. ^{1}H NMR (300 MHz, CD_3Cl): δ_H 3.72 (t, J = 5.06 Hz, 2 H, CH_2Cl), 3.91(d, $^2J_{PH}$ = 11.3 Hz, 1 H, CH), 4.28–4.37 (m, 2 H, OCH_2), 5.28–5.45 (br, 1 H, NH), 7.42–8.17 (m, 9 H, C_6H_4, Ph). ^{31}P NMR (121 MHz, CD_3Cl): δ_P 7.82. Calcd. (%) for $C_{19}H_{15}ClNO_5P$: C, 56.52; H, 3.74; N, 3.47. Found (%): C, 56.62; H, 3.81; N, 3.48. MS: m/z 403 (M^+).

2-(2-Chloroethoxy)-3-(4-methoxyphenyl)-3,4-dihydro-2H-naphtho[2,3-e][1,4,2]oxazaphosphinane-5,10-dione 2-oxide (17)

Orange power, yield 58%; mp 178–180°C. ^{1}H NMR (300 MHz, CD_3Cl): δ_H 3.70 (t, $J = 5.08$ Hz, 2 H, CH_2Cl), 3.73 (s, 3 H, OCH_3), 3.78 (d, $^2J_{PH} = 11.3$ Hz, 1 H, CH), 4.29–4.36 (m, 2 H, OCH_2), 5.28–5.49 (br, 1 H, NH), 7.39–8.17 (m, 8 H, C_6H_4, C_6H_4). ^{31}P NMR (121 MHz, CD_3Cl): δ_P 10.1. Calcd. (%) for $C_{20}H_{17}ClNO_6P$: C, 55.38; H, 3.95; N, 3.23. Found (%): C, 55.40; H, 3.95; N, 3.27. MS: m/z 433 (M^+).

2-(2-Chloroethoxy)-3-(2-nitrophenyl)-3,4-dihydro-2H-naphtho[2,3-e][1,4,2]oxazaphosphinane-5,10-dione 2-oxide (18)

Orange power, yield 72%; mp 198–200°C. ^{1}H NMR (300 MHz, CD_3Cl): δ_H 3.72 (t, $J = 5.06$ Hz, 2 H, CH_2Cl), 3.90(d, $^2J_{PH} = 11.3$ Hz, 1 H, CH), 4.26–4.37 (m, 2 H, OCH_2), 5.28–5.45 (br, 1 H, NH), 7.42–8.17 (m, 8 H, C_6H_4, C_6H_4). ^{31}P NMR (121 MHz, CD_3Cl): δ_P 9.01. Calcd. (%) for $C_{19}H_{14}ClN_2O_7P$: C, 50.85; H, 3.14; N, 6.24. Found (%): C, 50.89; H, 3.24; N, 6.28. MS: m/z 448 (M^+).

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