Acta Cryst E
CRYSTALLOGRAPHIC COMMUNICATIONS
ISSN 2056-9890

Crystal structures of two bis-carbamoylmethylphosphine oxide (CMPO) compounds

Andrew I. VanderWeide,[a] Richard J. Staples[b] and Shannon M. Biros[a]*

[a]Department of Chemistry, Grand Valley State University, 1 Campus Dr., Allendale, MI 49401, USA, and [b]Center for Crystallographic Research, Department of Chemistry, Michigan State University, East Lansing, MI 48824, USA.
*Correspondence e-mail: biross@gvsu.edu

Received 21 May 2019
Accepted 6 June 2019

Edited by S. Parkin, University of Kentucky, USA

Keywords: crystal structure; carbamoylmethylphosphine oxide; multidentate ligand; hydrogen bonds; C—H···π interactions; π–π stacking interactions.

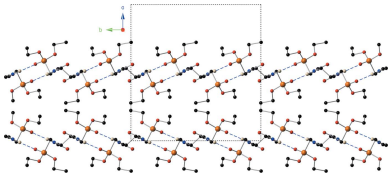
CCDC references: 1921486; 1921485

Supporting information: this article has supporting information at journals.iucr.org/e

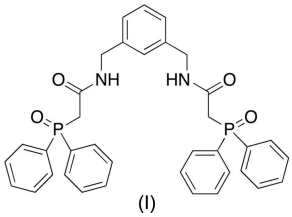
Two bis-carbamoylmethylphosphine oxide compounds, namely {[(3-{[2-(diphenylphosphinoyl)ethanamido]methyl}benzyl)carbamoyl]methyl}diphenylphosphine oxide, $C_{36}H_{34}N_2O_4P_2$, (I), and diethyl [({2-[2-(diethoxyphosphinoyl)-ethanamido]ethyl}carbamoyl)methyl]phosphonate, $C_{14}H_{30}N_2O_8P_2$, (II), were synthesized *via* nucleophilic acyl substitution reactions between an ester and a primary amine. Hydrogen-bonding interactions are present in both crystals, but these interactions are intramolecular in the case of compound (I) and intermolecular in compound (II). Intramolecular π–π stacking interactions are also present in the crystal of compound (I) with a centroid–centroid distance of 3.9479 (12) Å and a dihedral angle of 9.56 (12)°. Intermolecular C—H···π interactions [C···centroid distance of 3.622 (2) Å, C—H···centroid angle of 146°] give rise to supramolecular sheets that lie in the *ab* plane. Key geometric features for compound (I) involve a nearly planar, *trans*-amide group with a C—N—C—C torsion angle of 169.12 (17)°, and a torsion angle of −108.39 (15)° between the phosphine oxide phosphorus atom and the amide nitrogen atom. For compound (II), the electron density corresponding to the phosphoryl group was disordered, and was modeled as two parts with a 0.7387 (19):0.2613 (19) occupancy ratio. Compound (II) also boasts a *trans*-amide group that approaches planarity with a C—N—C—C torsion angle of −176.50 (16)°. The hydrogen bonds in this structure are intermolecular, with a *D*···*A* distance of 2.883 (2) Å and a *D*—H···*A* angle of 175.0 (18)° between the amide hydrogen atom and the P═O oxygen atom. These non-covalent interactions create ribbons that run along the *b*-axis direction.

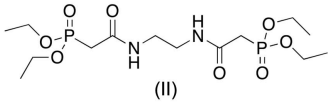
1. Chemical context

The carbamoylmethylphosphine oxide (CMPO) moiety has found use as the chelating portion of a ligand in the TRUEX process for the remediation of nuclear waste (Horwitz *et al.*, 1985). It has been shown that the CMPO group binds lanthanide (*Ln*) and actinide (*An*) metals in a 1:2 or 1:3 metal-ligand ratio in solution, depending on the size of the metal ion. Many researchers have attempted to mimic this solution stoichiometry by tethering two, three or four CMPO groups together *via* an organic scaffold (Dam *et al.*, 2007; Leoncini *et al.*, 2017; Miyazaki *et al.*, 2015; Sharova *et al.*, 2014; Werner & Biros, 2019). In some cases, these multidentate ligands have demonstrated an increased binding affinity for certain *Ln* and *An* ions, as well as an increased ability to extract these metals out of aqueous solutions. To this end, we report here the synthesis of compounds (I) and (II) and their characterization by ^{1}H, ^{13}C, and ^{31}P NMR spectroscopy, and by X-ray crystallography.



OPEN ACCESS





2. Structural commentary

The structure of compound (I) was solved in the monoclinic space group *C*2/*c*. Since the entire molecule straddles a twofold symmetry axis, the asymmetric unit is composed of one half of the compound. The complete molecular structure of compound (I) is shown in Fig. 1 along with the atom-labeling scheme. The P═O bond length is 1.4915 (13) Å, with P—C bond lengths that range from 1.7988 (18) to 1.8169 (19) Å. The τ_4 descriptor for fourfold coordination around the phosphorus atom P1 is 0.95, indicating a nearly perfect tetrahedral geometry of the phosphine oxide group (where 0.00 = square-planar, 0.85 = trigonal–pyramidal, and 1.00 = tetrahedral; Yang *et al.*, 2007). The geometry between the amide nitrogen atom N1 and the β-phosphine oxide phosphorus atom P1 is defined by a P1—C2—C1—N1 torsion angle of −108.39 (15)°. The amide group adopts a nearly perfect *trans* geometry with a C3—N1—C1—C2 torsion angle of 169.12 (17)°, and is staggered with respect to the plane of the C4–C7 aromatic ring with a H1—N1—C3—C4 torsion angle of 59.1 (17)°.

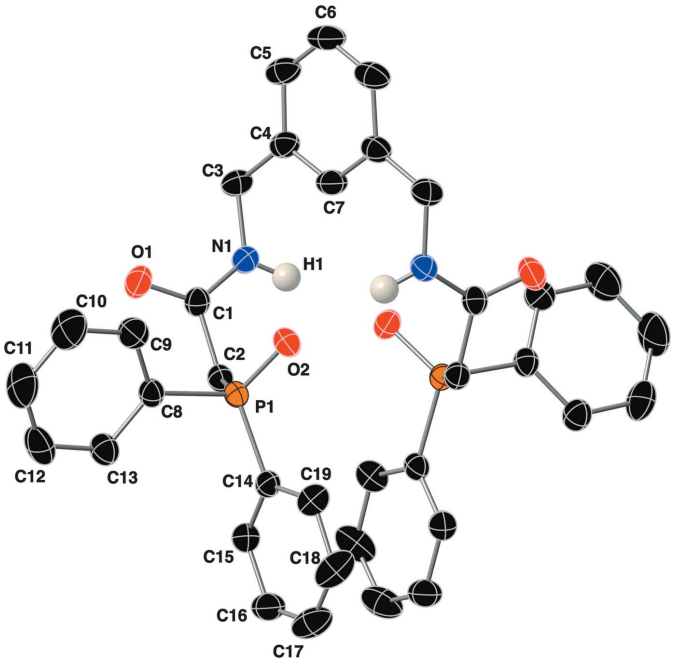


Figure 1
The complete molecular structure of compound (I), with the atom-labeling scheme. Unlabeled atoms are related to labeled atoms by the crystallographic twofold axis. Displacement ellipsoids are drawn at the 50% probability level, and hydrogen atoms bonded to carbon atoms have been omitted for clarity.

Table 1
Hydrogen-bond geometry (Å, °) for (I).

Cg is the centroid of the C14–C19 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.85 (2)	2.10 (2)	2.940 (2)	168 (2)
C3—H3*A*···*Cg*^{ii}	0.99	2.76	3.622 (2)	146

Symmetry codes: (i) $-x+1, y, -z+\frac{3}{2}$; (ii) $x-\frac{1}{2}, y+\frac{1}{2}, z$.

Intramolecular non-covalent interactions are also present in the crystal of compound (I). Hydrogen bonds between the amide hydrogen H1 and the phosphine oxide oxygen atom O2($-x+1, y, -z+\frac{3}{2}$) have a *D*···*A* distance of 2.940 (2) Å and a *D*—H···*A* angle of 168 (2)° (Fig. 3 and Table 1). The C14–C19 aromatic ring of this compound is engaged in an intramolecular π–π stacking interaction with its symmetry-derived counterpart with an intercentroid distance of 3.9479 (12) Å, slippage of 1.521 (1) Å and a dihedral angle of 9.56 (12)°.

Compound (II) crystallizes in the orthorhombic space group *Pbca*. Since the molecule lies on an inversion center (at $2-x, 1-y, 1-z$), the asymmetric unit comprises one half of the molecule. The electron density corresponding to the atoms of the phosphoryl group was disordered and was modeled over two positions with a 0.7387 (19):0.2613 (19) occupancy ratio (see the *Refinement* section for more details). The complete molecular structure of the major component of compound (II) is shown in Fig. 2 along with the labeling scheme. For the major component, the P═O bond length is 1.474 (2) Å, with P—O bond lengths of 1.5791 (16) and 1.5619 (15) Å, and a P—C bond length of 1.801 (2) Å. The τ_4 descriptor for fourfold coordination around the phosphorus atom of the major component, P1, is 0.93, indicating that the geometry of the phosphoryl group is slightly distorted from an ideal tetrahedron. The geometry between the amide nitrogen atom N1 and the β-phosphoryl group phosphorus atom P1 is defined by a N1—C1—C2—P1 torsion angle of −111.8 (2)°. The amide group of this compound also adopts a nearly perfect *trans* geometry with a C3—N1—C1—C2 torsion angle of −176.50 (16)°.

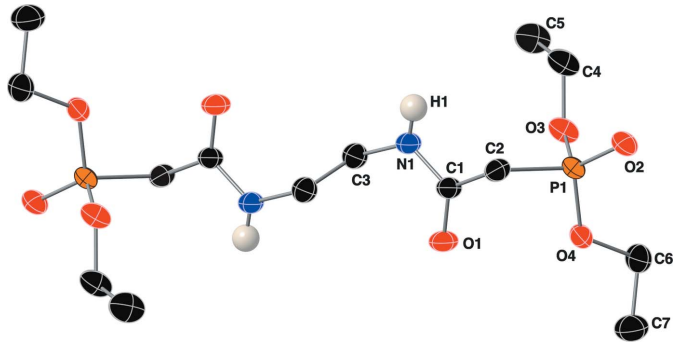


Figure 2
The molecular structure of compound (II), with the atom-labeling scheme. Unlabeled atoms are related to labeled atoms by a crystallographic inversion center. Displacement ellipsoids are drawn at the 50% probability level, only the major component and hydrogen atoms bonded to nitrogen atoms have been included for clarity.

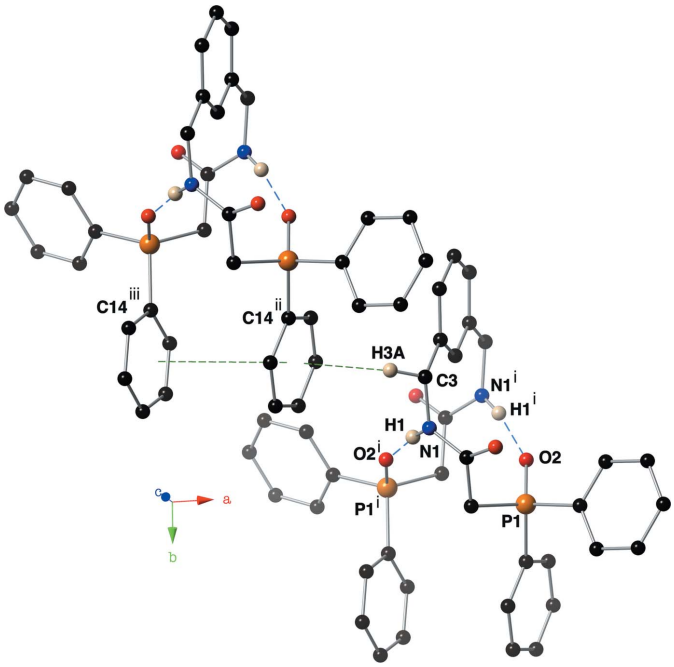


Figure 3
Depiction of non-covalent interactions present in the crystal of compound (I) using a ball-and-stick model with standard CPK colors. Intramolecular hydrogen bonds are shown as blue dashed lines; intramolecular π–π and intermolecular C—H···π interactions are shown with green dashed lines. Symmetry codes: (i) $1-x, y, \frac{3}{2}-z$; (ii) $-\frac{1}{2}+x, \frac{1}{2}+y, z$; (iii) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{3}{2}-z$.

Table 2
Hydrogen-bond geometry (Å, °) for (II).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.832 (19)	2.05 (2)	2.883 (2)	175.0 (18)
N1—H1···O2A^{i}	0.832 (19)	1.92 (2)	2.741 (8)	170.2 (18)

Symmetry code: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$.

3. Supramolecular features

The C14–C19 aromatic ring of compound (I) hosts a C—H···π interaction with H3A (symmetry code: $-\frac{1}{2}+x, \frac{1}{2}+y, z$) with a C···centroid distance of 3.622 (2) Å and a C—H···centroid angle of 146°. These non-covalent interactions create supramolecular sheets of compound (I) that lie in the *ab* plane (Fig. 4).

The crystal structure of compound (II) displays intermolecular hydrogen bonds between the amide hydrogen H1 and the oxygen atom O2 of the phosphoryl group of a neighboring molecule (symmetry code: $x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$; Fig. 5 and Table 2). This hydrogen bond is present for both parts of the disordered phosphoryl group. For the major component, this hydrogen bond has a D···A distance of 2.883 (2) Å with a D—H···A angle of 175.0 (18)°. This

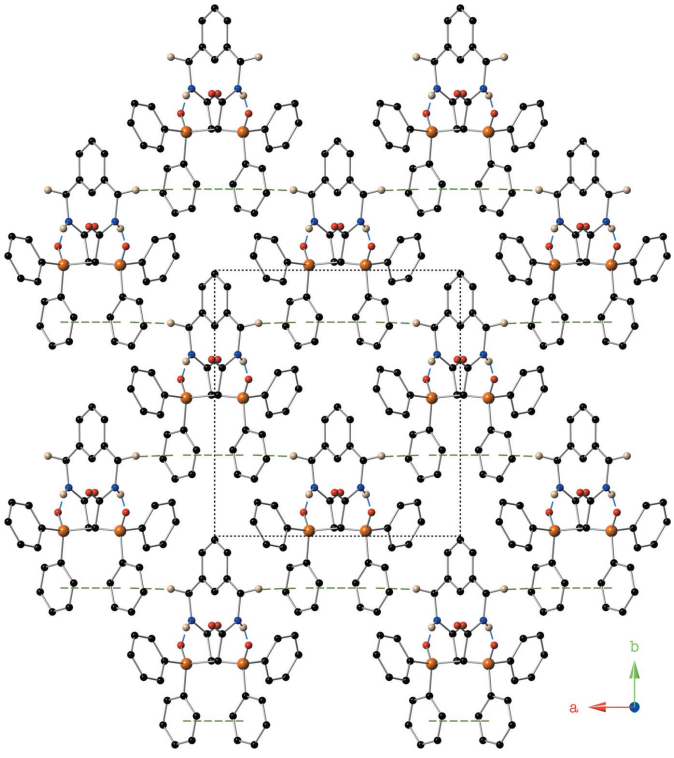


Figure 4
A view down the *c*-axis of compound (I) showing the supramolecular sheets that are held together with intramolecular C—H···π interactions using a ball-and-stick model with standard CPK colors. Hydrogen bonds are depicted with blue dashed lines, while π–π and C—H···π interactions are shown with green dashed lines. Only (N)H1 and (C)H3A are shown for clarity.

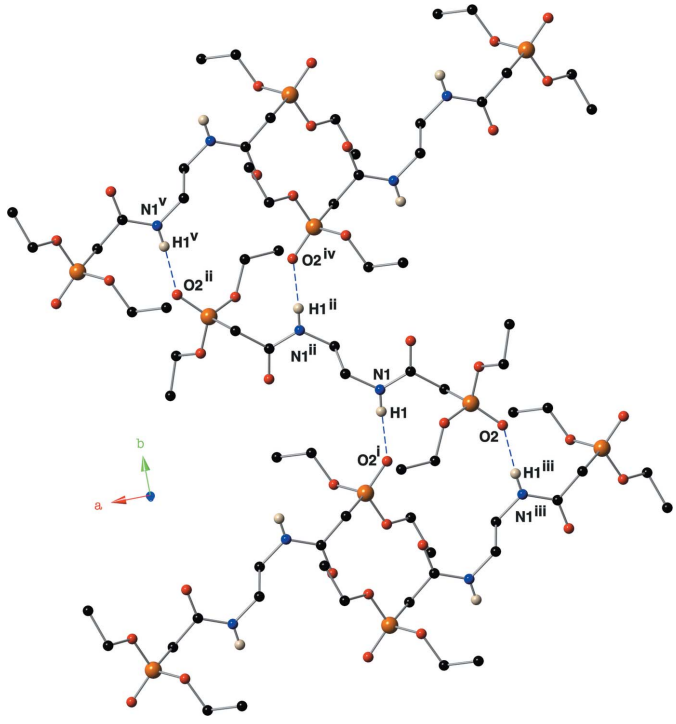


Figure 5
Depiction of the hydrogen-bonding network present in the crystal of compound (II) using a ball-and-stick model with standard CPK colors. The minor component of the disordered phosphoryl group is omitted for clarity. Intermolecular hydrogen bonds are shown with blue dashed lines. Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (ii) $-x+2, -y, -z+1$; (iii) $x-\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (iv) $-x+\frac{3}{2}, y-\frac{1}{2}, z$; (v) $-x+\frac{5}{2}, y-\frac{1}{2}, z$.

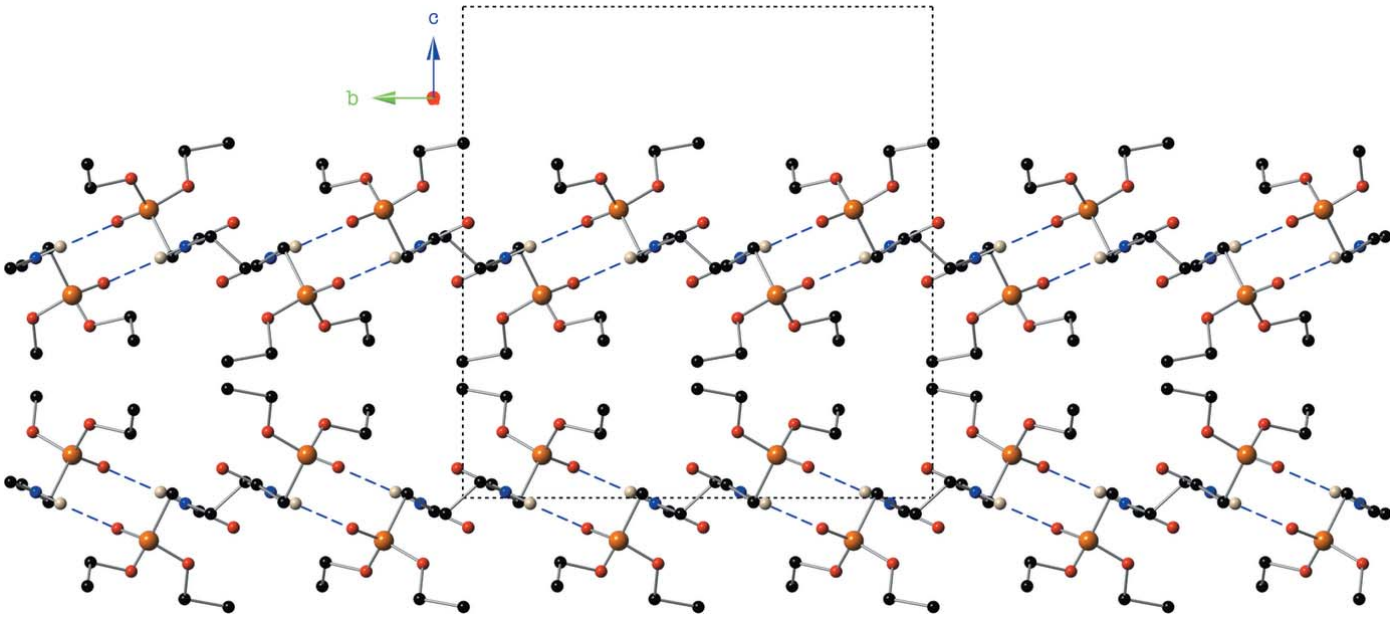


Figure 6
A view down the *a*-axis of the crystal of compound (II) showing the supramolecular ribbons that are formed *via* intermolecular hydrogen-bonding interactions. For clarity, only the major component of the disorder is shown.

hydrogen bond forms ribbons of compound (I) that run along the *b*-axis direction (Fig. 6).

4. Database survey

The Cambridge Structural Database (CSD, Version 5.40, November 2018; Groom *et al.*, 2016) contains 19 structures which have a CMPO group as part of an organic compound. (This count excludes metal complexes.) Of these, seven structures have two or more CMPO groups tethered to one another *via* an organic scaffold. The most similar structures to compound (I) are CIWFAR (Ouizem *et al.*, 2014) and SISLIQ (Artyushin *et al.*, 2006). Both structures use an aromatic ring as the scaffold to present two phenyl-substituted CMPO groups. In SISLIQ, a 1,2-disubstituted benzene ring is utilized to present the CMPO groups. In CIWFAR, the scaffold is a pyridine ring where the 2- and 6-positions bear CMPO groups, which makes it directly analogous to compound (I). The amide hydrogens of CIWFAR are engaged in intermolecular hydrogen bonds with the oxygen atoms of the phosphine oxide groups [rather than the intramolecular interaction observed for compound (I)], and the pyridine nitrogen is hydrogen bonded to the –OH group of a solvent methanol molecule. The hydrogen atoms of the pyridine scaffold interact with the phenyl rings of the phosphine oxide *via* intermolecular C—H$\cdots\pi$ interactions. A structure closely related to compound (II) was reported by the Rebek group as OGIVIJ (Amrhein, *et al.*, 2002). Here, a resorcin[4]arene scaffold presents two ethoxy-substituted CMPO units. We also note that the structure of compound (II) complexed with $Sm(NO_3)_3$ has been reported in this journal (Stoscup *et al.*, 2014).

5. Synthesis and crystallization

Compound (I): 1,3-Bis(aminomethyl)benzene (128 mg, 0.124 mL, 0.785 mmol) and the *p*-nitrophenyl ester of diphenylphosphonoacetate (Arnaud-Neu *et al.*, 1996) (1.0 g, 3.14 mmol) were dissolved in anhydrous, ethanol-free chloroform (30 mL). The solution was heated to 313 K and stirred for three days. The reaction mixture was then allowed to cool to room temperature, a small amount of 40% KOH was added (*ca.* 3 mL) and the solution was stirred for 3.5 h. The organic layer was separated, washed with brine (3 × 10 mL), dried over solid magnesium sulfate and concentrated under reduced pressure. The crude product was triturated multiple times with ethyl acetate to give a white solid in 91% yield. X-ray quality crystals of compound (I) were grown by slow evaporation of a chloroform solution. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91 (*t*, *J* = 5.3 Hz, 2H, –N*H*), 7.7–7.3 (*m*, 20H), 7.1–6.8 (*m*, 4H), 4.24 (*d*, *J* = 7.2 Hz, 4H), 3.36 (*d*, J_{P-H} = 13.2 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 164.7 (*d*, J_{P-C} = 4.5 Hz), 138.3, 132.5, 131.9, 131.2–130.5 (*broad*), 129.5–128.3 (*broad*), 126.9–126.1 (*broad*), 43.5, 38.6; ^{31}P NMR (161 MHz, $CDCl_3$): δ 30.6.

Compound (II): Ethylene diamine (1.0 mL, 14.9 mmol) was dissolved in 8.3 mL of methanol. The solution was cooled to 195 K, and triethyl phosphonoacetate (8.8 mL, 44.8 mmol) was added dropwise. The reaction mixture was allowed to warm to room temperature and stirred overnight. The product precipitated from the solution, was isolated by vacuum filtration and rinsed with ethyl acetate. Some of this solid was crystalline and suitable for analysis by X-ray diffraction. The remainder of the isolated product was purified by silica gel chromatography (10:1 dichloromethane–methanol) to give

Table 3
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$C_{36}H_{34}N_2O_4P_2$	$C_{14}H_{30}N_2O_8P_2$
M_r	620.59	416.34
Crystal system, space group	Monoclinic, *C*2/*c*	Orthorhombic, *Pbca*
Temperature (K)	173	173
a, *b*, *c* (Å)	13.0352 (2), 14.1348 (4), 17.0471 (4)	8.9401 (1), 15.0535 (2), 15.7314 (3)
α, β, γ (°)	90, 90.217 (2), 90	90, 90, 90
V (Å^3)	3140.90 (13)	2117.13 (5)
Z	4	4
Radiation type	Cu *Kα*	Cu *Kα*
μ (mm^{-1})	1.60	2.23
Crystal size (mm)	0.38 × 0.11 × 0.08	0.34 × 0.23 × 0.06
Data collection		
Diffractometer	Bruker APEXII CCD	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2013)	Multi-scan (*SADABS*; Bruker, 2013)
T_{min}, T_{max}	0.617, 0.754	0.612, 0.754
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	16825, 3022, 2543	10282, 2057, 1839
R_{int}	0.050	0.028
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.617	0.617
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.041, 0.117, 1.03	0.035, 0.093, 1.05
No. of reflections	3022	2057
No. of parameters	204	154
No. of restraints	0	20
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.33, −0.27	0.21, −0.27

Computer programs: *APEX2* and *SAINT* (Bruker, 2013), *SHELXS* (Sheldrick, 2008), *olex2.solve* (Bourhis *et al.*, 2015), *SHELXL* (Sheldrick, 2015), *OLEX2* (Dolomanov *et al.*, 2009) and *CrystalMaker* (Palmer, 2007).

compound (II) as a white solid (37% yield). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.75 (*broad*, 2H, –N*H*), 4.15 (*q*, *J* = 7.0 Hz, 8H), 3.34 (*d*, *J* = 5.9 Hz, 8H), 2.85 (*q*, J_{P-H} = 15.8 Hz, 8H), 1.33 (*t*, *J* = 7.0 Hz, 12H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 165.4, 62.9, 35.8 (*d*, J_{P-C} = 128 Hz), 16.5; ^{31}P NMR (121 MHz, $CDCl_3$): δ 24.5.

6. Refinement

Crystal data, data collection and structure refinement details for both compounds are summarized in Table 3. For compounds (I) and (II), all hydrogen atoms bonded to carbon atoms were placed in calculated positions and refined as riding: C—H = 0.95–1.00 Å with U_{iso}(H) = 1.2U_{eq}(C) for methylene groups and aromatic hydrogen atoms, and U_{iso}(H) = 1.5U_{eq}(C) for methyl groups. For both compounds (I) and (II), the hydrogen atoms bonded to nitrogen atoms were located using electron-density difference maps. The disordered electron density corresponding to the phosphoryl group of compound (II) was modeled over two positions with a relative occupancy ratio of 0.7387 (19):0.2613 (19). The C5—C4 and C6—C7 bond lengths were restrained using DFIX instructions in *SHELXL* (Sheldrick, 2015) at 1.5 Å to agree with known values. Atoms of each part (P1, P1*A*, O2–O4, O2*A*–O4*A*, C2, C2*A*, C5–C7, C5*A*–C7*A*) were treated with SAME and EADP commands to produce bond lengths and angles that agree with known values, and to ensure physically reasonable displacement parameters.

Acknowledgements

The authors thank Pfizer, Inc. for the donation of a Varian INOVA 400 F T NMR. The CCD-based X-ray diffractometers at Michigan State University were upgraded and/or replaced by departmental funds.

Funding information

Funding for this research was provided by: National Science Foundation (grant No. MRI CHE-1725699; grant No. REU CHE-1092944 to A. VanderWeide); Grand Valley State University (OURS, CSCE, Chemistry Department's Weldon Fund).

References

Amrhein, P., Shivanyuk, A., Johnson, D. W. & Rebek, J. Jr (2002). *J. Am. Chem. Soc.* **124**, 10349–10358.
Arnaud-Neu, F., Böhmer, V., Dozol, J.-F., Grüttner, C., Jakobi, R., Kraft, D., Mauprivez, O., Rouquette, H., Schwing-Weill, M., Simon, N. & Vogt, W. (1996). *J. Chem. Soc. Perkin Trans. 2*, pp. 1175–1182.
Artyushin, O. I., Sharova, E. V., Odinets, I. L., Lyssenko, K. A., Golovanov, D. G., Mastryukova, T. A., Pribylova, G. A., Tananaev, I. G. & Myasoedova, G. V. (2006). *Russ. Chem. Bull.* **55**, 1440–1447.
Bourhis, L. J., Dolomanov, O. V., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2015). *Acta Cryst.* A**71**, 59–75.
Bruker (2013). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc. Madison, Wisconsin, USA.
Dam, H. H., Reinhoudt, D. N. & Verboom, W. (2007). *Chem. Soc. Rev.* **36**, 367–377.

Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.
Horwitz, E. P., Kalina, D. C., Diamond, H., Vandegrift, G. F. & Schulz, W. W. (1985). *Solvent Extr. Ion Exch.* **3**, 75–109.
Leoncini, A., Huskens, J. & Verboom, W. (2017). *Chem. Soc. Rev.* **46**, 7229–7273.
Miyazaki, Y., Suzuki, S., Kobayashi, T., Yaita, T., Inaba, Y., Takeshita, K. & Mori, A. (2015). *Chem. Lett.* **44**, 1626–1636.
Ouizem, S., Rosario-Amorin, D., Dickie, D. A., Paine, R. T., de Bettencourt-Dias, A., Hay, B. P., Podair, J. & Delmau, L. H. (2014). *Dalton Trans.* **43**, 8368–8386.
Palmer, D. (2007). *CrystalMaker*. CrystalMaker Software, Bicester, England.
Sharova, E. V., Artyushin, O. I. & Odinets, I. L. (2014). *Russ. Chem. Rev.* **83**, 95–119.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Stoscup, J. A., Staples, R. J. & Biros, S. M. (2014). *Acta Cryst.* E**70**, 188–191.
Werner, E. J. & Biros, S. M. (2019). *Org. Chem. Front.* **6**, 2067–2094.
Yang, L., Powell, D. R. & Houser, R. P. (2007). *Dalton Trans.* pp. 955–964.

supporting information

Acta Cryst. (2019). E**75**, 991-996 [https://doi.org/10.1107/S205698901900820X]

Crystal structures of two bis-carbamoylmethylphosphine oxide (CMPO) compounds

Andrew I. VanderWeide, Richard J. Staples and Shannon M. Biros

Computing details

For both structures, data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT* (Bruker, 2013). Program(s) used to solve structure: *SHELXS* (Sheldrick, 2008) for (I); *olex2.solve* (Bourhis *et al.*, 2015) for (II). For both structures, program(s) used to refine structure: *SHELXL* (Sheldrick, 2015). Molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009; Bourhis *et al.*, 2015) for (I); *OLEX2* (Dolomanov *et al.*, 2009) for (II). Software used to prepare material for publication: *CrystalMaker* (Palmer, 2007) for (I); *OLEX2* (Dolomanov *et al.*, 2009) for (II).

{[(3-{[2-(Diphenylphosphinoyl)ethanamido]methyl}benzyl)carbamoyl]methyl}diphenylphosphine oxide (I)

Crystal data

$C_{36}H_{34}N_2O_4P_2$
M_r = 620.59
Monoclinic, $C2/c$
a = 13.0352 (2) Å
b = 14.1348 (4) Å
c = 17.0471 (4) Å
β = 90.217 (2)°
V = 3140.90 (13) Å^3
Z = 4
$F(000)$ = 1304
D_x = 1.312 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 7639 reflections
θ = 4.6–71.9°
μ = 1.60 mm^{-1}
T = 173 K
Needle, colourless
0.38 × 0.11 × 0.08 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2013)
T_{min} = 0.617, T_{max} = 0.754
16825 measured reflections
3022 independent reflections
2543 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 72.1°, θ_{min} = 4.6°
$h = -16 \rightarrow 16$
$k = -17 \rightarrow 16$
$l = -21 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.117
S = 1.03
3022 reflections
204 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.071P)^2 + 1.6884P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
P1	0.61558 (3)	0.01942 (3)	0.65310 (2)	0.02456 (15)
O1	0.51178 (11)	0.16409 (10)	0.52941 (8)	0.0387 (4)
O2	0.63820 (10)	0.08953 (9)	0.71622 (8)	0.0319 (3)
N1	0.40562 (13)	0.18290 (11)	0.63349 (10)	0.0304 (4)
H1	0.3839 (17)	0.1569 (16)	0.6753 (14)	0.031 (6)*
C1	0.46922 (13)	0.13271 (13)	0.58793 (10)	0.0279 (4)
C2	0.48641 (14)	0.03112 (13)	0.61386 (10)	0.0279 (4)
H2A	0.477236	−0.012087	0.568671	0.033*
H2B	0.435750	0.013809	0.654532	0.033*
C3	0.39481 (17)	0.28492 (14)	0.62513 (12)	0.0367 (5)
H3A	0.321015	0.301452	0.625674	0.044*
H3B	0.422971	0.304441	0.573764	0.044*
C4	0.44947 (14)	0.33936 (13)	0.68985 (12)	0.0323 (4)
C5	0.44880 (15)	0.43806 (14)	0.69115 (14)	0.0388 (5)
H5	0.413150	0.472302	0.651594	0.047*
C6	0.500000	0.4858 (2)	0.750000	0.0418 (7)
H6	0.500001	0.553010	0.750000	0.050*
C7	0.500000	0.29186 (19)	0.750000	0.0337 (6)
H7	0.500000	0.224650	0.750000	0.040*
C8	0.70406 (14)	0.03028 (13)	0.57209 (10)	0.0282 (4)
C9	0.75675 (18)	0.11496 (16)	0.56285 (13)	0.0430 (5)
H9	0.745261	0.165338	0.598650	0.052*
C10	0.8259 (2)	0.12686 (19)	0.50205 (16)	0.0560 (7)
H10	0.862193	0.184740	0.496512	0.067*
C11	0.84148 (19)	0.0541 (2)	0.44976 (15)	0.0575 (7)
H11	0.888183	0.062054	0.407633	0.069*
C12	0.78956 (18)	−0.03057 (19)	0.45821 (14)	0.0495 (6)
H12	0.800717	−0.080434	0.421862	0.059*
C13	0.72129 (15)	−0.04301 (15)	0.51949 (12)	0.0355 (4)
H13	0.686365	−0.101540	0.525499	0.043*
C14	0.62569 (14)	−0.10082 (13)	0.68680 (11)	0.0278 (4)
C15	0.57441 (16)	−0.17625 (14)	0.65110 (12)	0.0342 (4)
H15	0.527354	−0.164934	0.609453	0.041*
C16	0.59268 (17)	−0.26772 (15)	0.67688 (13)	0.0407 (5)
H16	0.557730	−0.319191	0.652892	0.049*
C17	0.66130 (17)	−0.28459 (16)	0.73716 (16)	0.0472 (6)
H17	0.674521	−0.347566	0.753841	0.057*
C18	0.71074 (17)	−0.20969 (18)	0.77322 (15)	0.0481 (6)
H18	0.757181	−0.221448	0.815181	0.058*

C19	0.69321 (15)	−0.11785 (15)	0.74878 (12)	0.0359 (4)
H19	0.726984	−0.066640	0.774090	0.043*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0270 (2)	0.0245 (2)	0.0222 (2)	0.00211 (17)	0.00302 (17)	−0.00095 (16)
O1	0.0434 (8)	0.0417 (8)	0.0310 (7)	0.0046 (6)	0.0073 (6)	0.0093 (6)
O2	0.0351 (7)	0.0325 (7)	0.0281 (6)	0.0011 (6)	0.0016 (5)	−0.0055 (5)
N1	0.0345 (8)	0.0285 (8)	0.0283 (8)	0.0040 (7)	0.0030 (7)	0.0035 (6)
C1	0.0282 (9)	0.0317 (10)	0.0238 (9)	0.0006 (7)	−0.0023 (7)	0.0012 (7)
C2	0.0294 (9)	0.0291 (9)	0.0251 (8)	0.0009 (7)	0.0022 (7)	−0.0011 (7)
C3	0.0426 (11)	0.0291 (10)	0.0385 (11)	0.0092 (8)	0.0008 (9)	0.0063 (8)
C4	0.0296 (9)	0.0283 (9)	0.0392 (10)	0.0032 (7)	0.0094 (8)	0.0039 (8)
C5	0.0317 (10)	0.0297 (10)	0.0549 (13)	0.0048 (8)	0.0128 (9)	0.0078 (9)
C6	0.0384 (15)	0.0224 (13)	0.065 (2)	0.000	0.0157 (14)	0.000
C7	0.0376 (14)	0.0230 (12)	0.0406 (15)	0.000	0.0051 (12)	0.000
C8	0.0262 (8)	0.0327 (9)	0.0256 (8)	0.0033 (7)	0.0025 (7)	0.0008 (7)
C9	0.0521 (13)	0.0356 (11)	0.0415 (12)	−0.0065 (10)	0.0124 (10)	−0.0018 (9)
C10	0.0556 (14)	0.0547 (15)	0.0577 (15)	−0.0170 (12)	0.0174 (12)	0.0078 (12)
C11	0.0456 (13)	0.0782 (18)	0.0488 (14)	−0.0083 (13)	0.0236 (11)	0.0011 (13)
C12	0.0458 (13)	0.0615 (15)	0.0414 (12)	0.0029 (11)	0.0146 (10)	−0.0137 (11)
C13	0.0328 (10)	0.0391 (11)	0.0346 (10)	−0.0002 (8)	0.0049 (8)	−0.0050 (8)
C14	0.0293 (9)	0.0271 (9)	0.0269 (8)	0.0046 (7)	0.0069 (7)	0.0024 (7)
C15	0.0392 (10)	0.0327 (10)	0.0308 (9)	0.0015 (8)	0.0041 (8)	0.0007 (8)
C16	0.0451 (11)	0.0284 (10)	0.0487 (12)	−0.0008 (9)	0.0110 (10)	0.0017 (9)
C17	0.0375 (11)	0.0345 (11)	0.0698 (16)	0.0057 (9)	0.0081 (11)	0.0192 (11)
C18	0.0340 (11)	0.0506 (13)	0.0595 (14)	0.0032 (10)	−0.0052 (10)	0.0233 (12)
C19	0.0299 (9)	0.0381 (11)	0.0398 (11)	−0.0002 (8)	−0.0003 (8)	0.0062 (9)

Geometric parameters (Å, º)

P1—O2	1.4915 (13)	C8—C13	1.389 (3)
P1—C2	1.8169 (19)	C9—H9	0.9500
P1—C8	1.8091 (18)	C9—C10	1.386 (3)
P1—C14	1.7988 (18)	C10—H10	0.9500
O1—C1	1.226 (2)	C10—C11	1.377 (4)
N1—H1	0.85 (2)	C11—H11	0.9500
N1—C1	1.341 (2)	C11—C12	1.382 (4)
N1—C3	1.456 (2)	C12—H12	0.9500
C1—C2	1.519 (3)	C12—C13	1.386 (3)
C2—H2A	0.9900	C13—H13	0.9500
C2—H2B	0.9900	C14—C15	1.397 (3)
C3—H3A	0.9900	C14—C19	1.393 (3)
C3—H3B	0.9900	C15—H15	0.9500
C3—C4	1.520 (3)	C15—C16	1.386 (3)
C4—C5	1.395 (3)	C16—H16	0.9500
C4—C7	1.390 (2)	C16—C17	1.381 (3)

C5—H5	0.9500	C17—H17	0.9500
C5—C6	1.379 (3)	C17—C18	1.382 (4)
C6—H6	0.9500	C18—H18	0.9500
C7—H7	0.9500	C18—C19	1.382 (3)
C8—C9	1.389 (3)	C19—H19	0.9500

O2—P1—C2	112.68 (8)	C9—C8—P1	118.45 (15)
O2—P1—C8	111.68 (8)	C13—C8—P1	122.32 (15)
O2—P1—C14	112.54 (8)	C13—C8—C9	119.22 (18)
C8—P1—C2	107.69 (8)	C8—C9—H9	119.6
C14—P1—C2	105.68 (9)	C10—C9—C8	120.8 (2)
C14—P1—C8	106.13 (8)	C10—C9—H9	119.6
C1—N1—H1	117.7 (15)	C9—C10—H10	120.3
C1—N1—C3	121.81 (17)	C11—C10—C9	119.4 (2)
C3—N1—H1	118.5 (15)	C11—C10—H10	120.3
O1—C1—N1	124.21 (18)	C10—C11—H11	119.8
O1—C1—C2	120.80 (17)	C10—C11—C12	120.4 (2)
N1—C1—C2	114.98 (15)	C12—C11—H11	119.8
P1—C2—H2A	109.8	C11—C12—H12	119.9
P1—C2—H2B	109.8	C11—C12—C13	120.3 (2)
C1—C2—P1	109.19 (12)	C13—C12—H12	119.9
C1—C2—H2A	109.8	C8—C13—H13	120.1
C1—C2—H2B	109.8	C12—C13—C8	119.9 (2)
H2A—C2—H2B	108.3	C12—C13—H13	120.1
N1—C3—H3A	109.1	C15—C14—P1	123.20 (15)
N1—C3—H3B	109.1	C19—C14—P1	116.79 (15)
N1—C3—C4	112.67 (16)	C19—C14—C15	119.90 (18)
H3A—C3—H3B	107.8	C14—C15—H15	120.2
C4—C3—H3A	109.1	C16—C15—C14	119.5 (2)
C4—C3—H3B	109.1	C16—C15—H15	120.2
C5—C4—C3	120.98 (19)	C15—C16—H16	119.8
C7—C4—C3	120.69 (18)	C17—C16—C15	120.5 (2)
C7—C4—C5	118.3 (2)	C17—C16—H16	119.8
C4—C5—H5	120.1	C16—C17—H17	120.1
C6—C5—C4	119.8 (2)	C16—C17—C18	119.9 (2)
C6—C5—H5	120.1	C18—C17—H17	120.1
C5—C6—C5^{i}	121.4 (3)	C17—C18—H18	119.7
C5^{i}—C6—H6	119.3	C19—C18—C17	120.6 (2)
C5—C6—H6	119.3	C19—C18—H18	119.7
C4—C7—C4^{i}	122.2 (3)	C14—C19—H19	120.2
C4—C7—H7	118.9	C18—C19—C14	119.6 (2)
C4^{i}—C7—H7	118.9	C18—C19—H19	120.2

Symmetry code: (i) $-x+1, y, -z+3/2$.

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C14–C19 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.85 (2)	2.10 (2)	2.940 (2)	168 (2)
C3—H3*A*···*Cg*^{ii}	0.99	2.76	3.622 (2)	146

Symmetry codes: (i) −*x*+1, *y*, −*z*+3/2; (ii) *x*−1/2, *y*+1/2, *z*.

Diethyl [({2-[2-(diethoxyphosphinoyl)ethanamido]ethyl}carbamoyl)methyl]phosphonate (II)

Crystal data

$C_{14}H_{30}N_2O_8P_2$
M_r = 416.34
Orthorhombic, *Pbca*
a = 8.9401 (1) Å
b = 15.0535 (2) Å
c = 15.7314 (3) Å
V = 2117.13 (5) Å^3
Z = 4
F(000) = 888
D_x = 1.306 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54178 Å
Cell parameters from 6034 reflections
θ = 4.1–72.0°
μ = 2.23 mm^{-1}
T = 173 K
Plate, colourless
0.34 × 0.23 × 0.06 mm

Data collection

Bruker APEXII CCD diffractometer
φ and *ω* scans
Absorption correction: multi-scan (SADABS; Bruker, 2013)
T_{min} = 0.612, T_{max} = 0.754
10282 measured reflections
2057 independent reflections
1839 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 72.2°, θ_{min} = 5.6°
h = −11→10
k = −18→12
l = −19→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.093
S = 1.05
2057 reflections
154 parameters
20 restraints
Primary atom site location: iterative
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.045P)^2 + 0.8277P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
P1	0.49865 (8)	0.16848 (6)	0.58724 (5)	0.0291 (2)	0.7387 (19)
O2	0.3858 (2)	0.23508 (12)	0.56270 (16)	0.0402 (5)	0.7387 (19)
O3	0.6214 (2)	0.20596 (15)	0.64985 (15)	0.0364 (4)	0.7387 (19)

O4	0.43649 (17)	0.08482 (10)	0.63416 (10)	0.0361 (4)	0.7387 (19)
C2	0.5970 (5)	0.1213 (3)	0.49806 (18)	0.0255 (6)	0.7387 (19)
H2A	0.526044	0.087298	0.462353	0.031*	0.7387 (19)
H2B	0.639568	0.169666	0.462907	0.031*	0.7387 (19)
C4	0.6892 (3)	0.2924 (2)	0.6313 (3)	0.0433 (7)	0.7387 (19)
H4A	0.620650	0.340728	0.648672	0.052*	0.7387 (19)
H4B	0.708400	0.297943	0.569581	0.052*	0.7387 (19)
C5	0.8336 (4)	0.2991 (3)	0.6797 (3)	0.0521 (8)	0.7387 (19)
H5A	0.901244	0.251520	0.661636	0.078*	0.7387 (19)
H5B	0.813603	0.293490	0.740726	0.078*	0.7387 (19)
H5C	0.880239	0.356862	0.668357	0.078*	0.7387 (19)
C6	0.3339 (3)	0.09235 (16)	0.70583 (18)	0.0470 (6)	0.7387 (19)
H6A	0.250321	0.132632	0.691026	0.056*	0.7387 (19)
H6B	0.386886	0.117380	0.755628	0.056*	0.7387 (19)
C7	0.2754 (9)	0.0033 (4)	0.7266 (5)	0.0514 (10)	0.7387 (19)
H7A	0.209468	−0.016933	0.680694	0.077*	0.7387 (19)
H7B	0.218830	0.006108	0.779863	0.077*	0.7387 (19)
H7C	0.358860	−0.038321	0.732867	0.077*	0.7387 (19)
O1	0.69589 (11)	−0.01159 (7)	0.56001 (7)	0.0369 (3)	
N1	0.85841 (14)	0.09068 (9)	0.51170 (9)	0.0333 (3)	
C1	0.72039 (15)	0.06112 (9)	0.52745 (9)	0.0287 (3)	
C3	0.98811 (16)	0.03699 (10)	0.53232 (10)	0.0348 (3)	
H3A	0.974766	0.010625	0.589473	0.042*	
H3B	1.078035	0.075326	0.533945	0.042*	
P1A	0.5246 (3)	0.19107 (16)	0.60724 (17)	0.0291 (2)	0.2613 (19)
O2A	0.4150 (8)	0.2586 (4)	0.5806 (5)	0.0402 (5)	0.2613 (19)
O3A	0.6639 (8)	0.2236 (5)	0.6594 (5)	0.0364 (4)	0.2613 (19)
O4A	0.4656 (5)	0.1195 (3)	0.6726 (3)	0.0361 (4)	0.2613 (19)
C2A	0.5883 (17)	0.1303 (10)	0.5188 (7)	0.0255 (6)	0.2613 (19)
H2AA	0.501156	0.097716	0.495619	0.031*	0.2613 (19)
H2AB	0.618606	0.173979	0.474973	0.031*	0.2613 (19)
C4A	0.7429 (11)	0.3020 (7)	0.6334 (9)	0.0433 (7)	0.2613 (19)
H4AA	0.678295	0.354818	0.640873	0.052*	0.2613 (19)
H4AB	0.770059	0.297421	0.572537	0.052*	0.2613 (19)
C5A	0.8806 (14)	0.3114 (11)	0.6859 (11)	0.0521 (8)	0.2613 (19)
H5AA	0.953206	0.265798	0.669311	0.078*	0.2613 (19)
H5AB	0.855187	0.304251	0.746069	0.078*	0.2613 (19)
H5AC	0.924122	0.370407	0.676741	0.078*	0.2613 (19)
C6A	0.3201 (9)	0.0845 (5)	0.6622 (5)	0.0470 (6)	0.2613 (19)
H6AA	0.303480	0.070051	0.601507	0.056*	0.2613 (19)
H6AB	0.245523	0.129747	0.679118	0.056*	0.2613 (19)
C7A	0.299 (3)	0.0026 (12)	0.7147 (16)	0.0514 (10)	0.2613 (19)
H7AA	0.272633	−0.047288	0.677574	0.077*	0.2613 (19)
H7AB	0.218138	0.012506	0.755778	0.077*	0.2613 (19)
H7AC	0.391725	−0.010925	0.745083	0.077*	0.2613 (19)
H1	0.871 (2)	0.1397 (13)	0.4883 (11)	0.038 (5)*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0313 (3)	0.0177 (4)	0.0384 (4)	0.0034 (3)	0.0024 (3)	0.0043 (3)
O2	0.0366 (11)	0.0231 (11)	0.0610 (14)	0.0061 (8)	0.0015 (8)	0.0068 (9)
O3	0.0456 (14)	0.0265 (12)	0.0372 (9)	0.0008 (8)	−0.0094 (9)	0.0011 (7)
O4	0.0430 (8)	0.0243 (8)	0.0409 (9)	0.0049 (6)	0.0152 (7)	0.0033 (6)
C2	0.0263 (9)	0.0256 (12)	0.0248 (18)	0.0006 (8)	0.0027 (13)	0.0054 (13)
C4	0.052 (2)	0.0284 (12)	0.0494 (10)	0.0007 (15)	−0.0097 (18)	0.0029 (9)
C5	0.053 (3)	0.0453 (18)	0.0576 (14)	−0.0050 (17)	−0.012 (2)	0.0038 (12)
C6	0.0605 (13)	0.0392 (11)	0.0413 (14)	−0.0010 (10)	0.0244 (14)	−0.0052 (12)
C7	0.054 (3)	0.0554 (12)	0.045 (2)	−0.0040 (14)	0.0188 (16)	0.0094 (12)
O1	0.0330 (5)	0.0251 (5)	0.0526 (6)	−0.0028 (4)	−0.0023 (5)	0.0110 (5)
N1	0.0263 (6)	0.0241 (6)	0.0496 (8)	−0.0009 (5)	0.0012 (5)	0.0071 (6)
C1	0.0285 (7)	0.0231 (7)	0.0343 (7)	−0.0023 (5)	−0.0019 (5)	0.0030 (5)
C3	0.0257 (7)	0.0333 (8)	0.0453 (8)	0.0002 (6)	−0.0029 (6)	0.0010 (7)
P1A	0.0313 (3)	0.0177 (4)	0.0384 (4)	0.0034 (3)	0.0024 (3)	0.0043 (3)
O2A	0.0366 (11)	0.0231 (11)	0.0610 (14)	0.0061 (8)	0.0015 (8)	0.0068 (9)
O3A	0.0456 (14)	0.0265 (12)	0.0372 (9)	0.0008 (8)	−0.0094 (9)	0.0011 (7)
O4A	0.0430 (8)	0.0243 (8)	0.0409 (9)	0.0049 (6)	0.0152 (7)	0.0033 (6)
C2A	0.0263 (9)	0.0256 (12)	0.0248 (18)	0.0006 (8)	0.0027 (13)	0.0054 (13)
C4A	0.052 (2)	0.0284 (12)	0.0494 (10)	0.0007 (15)	−0.0097 (18)	0.0029 (9)
C5A	0.053 (3)	0.0453 (18)	0.0576 (14)	−0.0050 (17)	−0.012 (2)	0.0038 (12)
C6A	0.0605 (13)	0.0392 (11)	0.0413 (14)	−0.0010 (10)	0.0244 (14)	−0.0052 (12)
C7A	0.054 (3)	0.0554 (12)	0.045 (2)	−0.0040 (14)	0.0188 (16)	0.0094 (12)

Geometric parameters (Å, º)

P1—O2	1.474 (2)	C1—C2A	1.580 (15)
P1—O3	1.5791 (16)	C3—C3^{i}	1.523 (3)
P1—O4	1.5619 (15)	C3—H3A	0.9900
P1—C2	1.801 (2)	C3—H3B	0.9900
O3—C4	1.464 (3)	P1A—O2A	1.473 (7)
O4—C6	1.458 (3)	P1A—O3A	1.570 (6)
C2—H2A	0.9900	P1A—O4A	1.581 (5)
C2—H2B	0.9900	P1A—C2A	1.760 (9)
C2—C1	1.501 (5)	O3A—C4A	1.435 (10)
C4—H4A	0.9900	O4A—C6A	1.413 (9)
C4—H4B	0.9900	C2A—H2AA	0.9900
C4—C5	1.502 (4)	C2A—H2AB	0.9900
C5—H5A	0.9800	C4A—H4AA	0.9900
C5—H5B	0.9800	C4A—H4AB	0.9900
C5—H5C	0.9800	C4A—C5A	1.489 (10)
C6—H6A	0.9900	C5A—H5AA	0.9800
C6—H6B	0.9900	C5A—H5AB	0.9800
C6—C7	1.475 (6)	C5A—H5AC	0.9800
C7—H7A	0.9800	C6A—H6AA	0.9900
C7—H7B	0.9800	C6A—H6AB	0.9900

C7—H7C	0.9800	C6A—C7A	1.495 (12)
O1—C1	1.2282 (17)	C7A—H7AA	0.9800
N1—C1	1.3348 (18)	C7A—H7AB	0.9800
N1—C3	1.4502 (19)	C7A—H7AC	0.9800
N1—H1	0.832 (19)		

O2—P1—O3	113.34 (11)	N1—C1—C2A	117.1 (6)
O2—P1—O4	115.38 (11)	N1—C3—C3^{i}	111.71 (16)
O2—P1—C2	113.46 (18)	N1—C3—H3A	109.3
O3—P1—C2	106.70 (17)	N1—C3—H3B	109.3
O4—P1—O3	103.93 (11)	C3^{i}—C3—H3A	109.3
O4—P1—C2	102.95 (16)	C3^{i}—C3—H3B	109.3
C4—O3—P1	118.74 (19)	H3A—C3—H3B	107.9
C6—O4—P1	121.77 (14)	O2A—P1A—O3A	117.5 (4)
P1—C2—H2A	109.5	O2A—P1A—O4A	115.7 (4)
P1—C2—H2B	109.5	O2A—P1A—C2A	110.4 (6)
H2A—C2—H2B	108.0	O3A—P1A—O4A	97.9 (4)
C1—C2—P1	110.9 (2)	O3A—P1A—C2A	108.6 (6)
C1—C2—H2A	109.5	O4A—P1A—C2A	105.6 (5)
C1—C2—H2B	109.5	C4A—O3A—P1A	119.8 (7)
O3—C4—H4A	110.0	C6A—O4A—P1A	119.0 (5)
O3—C4—H4B	110.0	C1—C2A—P1A	121.1 (8)
O3—C4—C5	108.4 (3)	C1—C2A—H2AA	107.1
H4A—C4—H4B	108.4	C1—C2A—H2AB	107.1
C5—C4—H4A	110.0	P1A—C2A—H2AA	107.1
C5—C4—H4B	110.0	P1A—C2A—H2AB	107.1
C4—C5—H5A	109.5	H2AA—C2A—H2AB	106.8
C4—C5—H5B	109.5	O3A—C4A—H4AA	109.9
C4—C5—H5C	109.5	O3A—C4A—H4AB	109.9
H5A—C5—H5B	109.5	O3A—C4A—C5A	109.1 (9)
H5A—C5—H5C	109.5	H4AA—C4A—H4AB	108.3
H5B—C5—H5C	109.5	C5A—C4A—H4AA	109.9
O4—C6—H6A	109.9	C5A—C4A—H4AB	109.9
O4—C6—H6B	109.9	C4A—C5A—H5AA	109.5
O4—C6—C7	108.9 (3)	C4A—C5A—H5AB	109.5
H6A—C6—H6B	108.3	C4A—C5A—H5AC	109.5
C7—C6—H6A	109.9	H5AA—C5A—H5AB	109.5
C7—C6—H6B	109.9	H5AA—C5A—H5AC	109.5
C6—C7—H7A	109.5	H5AB—C5A—H5AC	109.5
C6—C7—H7B	109.5	O4A—C6A—H6AA	109.4
C6—C7—H7C	109.5	O4A—C6A—H6AB	109.4
H7A—C7—H7B	109.5	O4A—C6A—C7A	111.1 (13)
H7A—C7—H7C	109.5	H6AA—C6A—H6AB	108.0
H7B—C7—H7C	109.5	C7A—C6A—H6AA	109.4
C1—N1—C3	120.79 (12)	C7A—C6A—H6AB	109.4
C1—N1—H1	119.9 (13)	C6A—C7A—H7AA	109.5
C3—N1—H1	119.3 (13)	C6A—C7A—H7AB	109.5
O1—C1—C2	122.4 (2)	C6A—C7A—H7AC	109.5

O1—C1—N1	122.64 (13)	H7AA—C7A—H7AB	109.5
O1—C1—C2A	119.3 (6)	H7AA—C7A—H7AC	109.5
N1—C1—C2	114.9 (2)	H7AB—C7A—H7AC	109.5

P1—O3—C4—C5	−160.9 (3)	C1—N1—C3—C3^{i}	77.0 (2)
P1—O4—C6—C7	170.3 (4)	C3—N1—C1—C2	−176.50 (16)
P1—C2—C1—O1	71.4 (3)	C3—N1—C1—O1	0.4 (2)
P1—C2—C1—N1	−111.8 (2)	C3—N1—C1—C2A	169.1 (4)
O2—P1—O3—C4	−45.5 (3)	P1A—O3A—C4A—C5A	−172.5 (10)
O2—P1—O4—C6	−48.7 (2)	P1A—O4A—C6A—C7A	165.3 (11)
O2—P1—C2—C1	174.3 (2)	O2A—P1A—O3A—C4A	−44.3 (11)
O3—P1—O4—C6	76.0 (2)	O2A—P1A—O4A—C6A	40.5 (6)
O3—P1—C2—C1	48.8 (3)	O2A—P1A—C2A—C1	172.8 (8)
O4—P1—O3—C4	−171.6 (2)	O3A—P1A—O4A—C6A	166.2 (6)
O4—P1—C2—C1	−60.3 (3)	O3A—P1A—C2A—C1	42.7 (11)
C2—P1—O3—C4	80.1 (3)	O4A—P1A—O3A—C4A	−168.7 (8)
C2—P1—O4—C6	−172.9 (2)	O4A—P1A—C2A—C1	−61.4 (11)
O1—C1—C2A—P1A	76.3 (10)	C2A—P1A—O3A—C4A	81.9 (10)
N1—C1—C2A—P1A	−92.8 (9)	C2A—P1A—O4A—C6A	−81.9 (7)

Symmetry code: (i) −*x*+2, −*y*, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{ii}	0.832 (19)	2.05 (2)	2.883 (2)	175.0 (18)
N1—H1···O2*A*^{ii}	0.832 (19)	1.92 (2)	2.741 (8)	170.2 (18)

Symmetry code: (ii) *x*+1/2, −*y*+1/2, −*z*+1.