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New *rac*-*X*P(O)(OC_6H_5)(NHC_6H_4-*p*-CH_3) [*X* = N(CH_3)(*cyclo*-C_6H_{11}) and NH(C_3H_5)] and *rac*-($C_6H_5CH_2NH$)-P(O)(OC_6H_5)(NH-*cyclo*-C_6H_{11}) mixed-amide phosphinates

Mehrdad Pourayoubi,[a]* Fatemeh Karimi Ahmadabad,[a] Hossein Eshtiagh-Hosseini,[a] Monika Kučeráková,[b] Václav Eigner[b] and Michal Dušek[b]

[a]Department of Chemistry, Ferdowsi University of Mashhad, Mashhad, Iran, and [b]Institute of Physics, Academy of Sciences of the Czech Republic v.v.i., Na Slovance 2, 182 21 Praha 8, Czech Republic
Correspondence e-mail: pourayoubi@um.ac.ir

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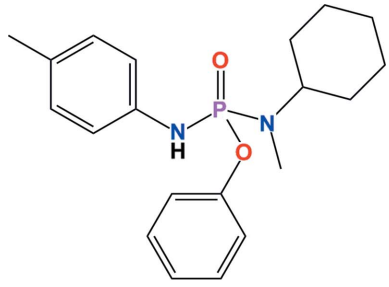
The mixed-amide phosphinates, *rac*-phenyl (*N*-methylcyclohexylamido)(*p*-tolylamido)phosphinate, $C_{20}H_{27}N_2O_2P$, (I), and *rac*-phenyl (allylamido)(*p*-tolylamido)phosphinate, $C_{16}H_{19}N_2O_2P$, (II), were synthesized from the racemic phosphorus–chlorine compound (*R*,*S*)-(Cl)P(O)(OC_6H_5)(NHC_6H_4-*p*-CH_3). Furthermore, the phosphorus–chlorine compound ClP(O)(OC_6H_5)(NH-*cyclo*-C_6H_{11}) was synthesized for the first time and used for the synthesis of *rac*-phenyl (benzylamido)(cyclohexylamido)phosphinate, $C_{19}H_{25}N_2O_2P$, (III). The strategies for the synthesis of racemic mixed-amide phosphinates are discussed. The P atom in each compound is in a distorted tetrahedral (N^1)P(=O)(O)(N^2) environment. In (I) and (II), the *p*-tolylamido substituent makes a longer P—N bond than those involving the *N*-methylcyclohexylamido and allylamido substituents. In (III), the differences between the P—N bond lengths involving the cyclohexylamido and benzylamido substituents are not significant. In all three structures, the phosphoryl O atom takes part with the N—H unit in hydrogen-bonding interactions, *viz*. an N—H···O=P hydrogen bond for (I) and (N—H)(N—H)···O=P hydrogen bonds for (II) and (III), building linear arrangements along [001] for (I) and along [010] for (III), and a ladder arrangement along [100] for (II).

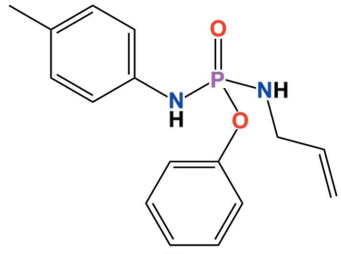
Keywords: crystal structure; racemic mixed-amide phosphinates; P—N bonds; N—H···O=P hydrogen bonds.

1. Introduction

Recently, the syntheses and diffraction experiments were described for some racemic mixed-amide phosphinates having an (N^1)P(=O)(O)(N^2) skeleton, where two different amide groups are bonded to the P atom (Pourayoubi, Karimi Ahmadabad *et al.*, 2011; Pourayoubi, Rheingold *et al.*, 2011; Sabbaghi *et al.*, 2011). [In compounds with a P(O)(NR^1R^2) segment, the NR^1R^2 group obtained from an NHR^1R^2 amine is named as an amido group.] The systematic study of such molecules may be useful for: (i) gaining insight into the reason why a particular amide group makes a stronger or weaker P—N bond; (ii) the preparation of racemic compounds (Pourayoubi *et al.*, 2007); (iii) finding new synthesis and purification procedures (Pourayoubi, Rheingold *et al.*, 2011); (iv) comparison of the hydrogen-bond patterns for finding empirical rules to predict hydrogen-bond patterns based on molecular structure (Toghraee *et al.*, 2011; Pourayoubi *et al.*, 2012).



(I)



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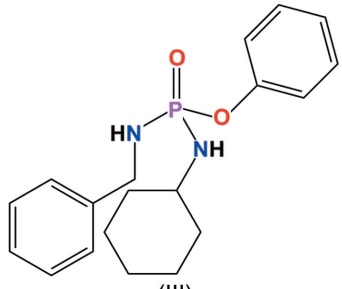

(III)

The strategy for the synthesis of racemic compounds with an (N^1)P(=O)(O)(N^2) molecular skeleton and containing a heterocyclic segment including an *X*—P—N (*X* = N*R*′ and O) fragment can be based on the reaction of a Cl_2P(O)(O*R*) initial compound with a diamine of the type NH*R*–*Y*–NH*R*′ (Gholivand *et al.*, 2007), or the reaction of [R^1R^2N]P(O)Cl_2 with an amino alcohol of the type NH*R*–*Z*–OH (Holmes *et al.*, 1984) [*Y* and *Z* are the hydrocarbon fragments between two functional groups in NH*R*–*Y*–NH*R*′ and NH*R*–*Z*–OH compounds, respectively].

In the case of acyclic compounds, Ghadimi *et al.* (2009) used a step-by-step nucleophilic substitution, using Cl_2P(O)(O*R*) as a starting compound and its reaction with an NHR^1R^2 amine in the presence of an HCl scavenger (such as C_5H_5N, or an

excess of an NHR^1R^2 amine as two moles of amine per mole of P—Cl bond; Pourayoubi, Karimi Ahmadabad *et al.*, 2011) in a suitable solvent to yield $ClP(O)(OR)(NR^1R^2)$ compounds. For such a reaction, a suitable solvent usually has low solubility of the C_5H_5NHCl (or $[R^1R^2NH_2]Cl$ alkyl/aryl ammonium chloride) by-product, while the solubility of $ClP(O)(OR)(NR^1R^2)$ is good in this solvent. In such a case, the by-product is simply removed by filtration.

The well-known purification method in the preparation of $(R^1R^2N)_2P(O)(OR)$ amidophosphinates is the dissolution of the above-mentioned by-product(s) in H_2O (Sabbaghi *et al.*, 2010). This strategy is not suitable for the purification of a $ClP(O)(OR)(NR^1R^2)$ compound, as the P—Cl bond is sensitive to moisture.

After purification, $ClP(O)(OR)(NR^1R^2)$ reacts with an NHR^3R^4 amine (1:2 molar ratio) to yield the racemic $(R^3R^4N)P(O)(OR)(NR^1R^2)$ mixed-amide phosphinate; the by-product of this step, $[R^3R^4NH_2]Cl$, is removed by dissolving in H_2O.

As examples where this or similar procedures have been used, we can mention $[(CH_3)_2N](p\text{-}CH_3\text{-}C_6H_4O)P(O)X$, where X = $NHC(CH_3)_3$, p-CH_3-C_6H_4NH (Ghadimi *et al.*, 2009) and $NHCH(CH_3)_2$ (Pourayoubi *et al.*, 2007). Another example is the compound $[(ClC_2H_4)_2N](C_6H_5O)P(O)[NHC_6H_5]$, which was studied by X-ray crystallography (Orji *et al.*, 1994). When using $[(CH_3)_2N](p\text{-}CH_3\text{-}C_6H_4\text{-}O)P(O)Cl$ as a starting material, some other compounds were also obtained, for example, with the (N)P(O)(O)(C) skeleton, such as $[(CH_3)_2N](p\text{-}CH_3\text{-}C_6H_4O)P(O)CN$ (Ghadimi *et al.*, 2007), which was also characterized by X-ray crystallography.

As has been noted in the literature (Pourayoubi, Karimi Ahmadabad *et al.*, 2011; Pourayoubi, Rheingold *et al.*, 2011; Sabbaghi *et al.*, 2011), the *p*-toluidine hydrochloride salt is relatively insoluble in CH_3CN and also in $CHCl_3$ (although the degree of solubility can be affected by the other starting materials used in the preparation method). The reaction of $C_6H_5OP(O)Cl_2$ and p-CH_3-$C_6H_4NH_2$ (1:2 molar ratio) was performed in CH_3CN (at ice-bath temperature), the $[p\text{-}CH_3\text{-}C_6H_4NH_3]Cl$ by-product was simply filtered off and the racemic product $ClP(O)(OC_6H_5)(NHC_6H_4\text{-}p\text{-}CH_3)$ was obtained from the solution.

Up to now, this racemic starting compound has been used for the preparation of *rac*-$LP(O)(OC_6H_5)(NHC_6H_4\text{-}p\text{-}CH_3)$ [L = NHC_6H_{11} (Sabbaghi *et al.*, 2011), $NHC(CH_3)_3$ (Pourayoubi, Rheingold *et al.*, 2011) and $NHCH_2C_6H_5$ (Pourayoubi, Karimi Ahmadabad *et al.*, 2011)]. Here, we used the above-mentioned starting racemic compound to study the syntheses and crystal structures of *rac*-phenyl (*N*-methylcyclohexylamido)(*p*-tolylamido)phosphinate, (I) (Fig. 1), and *rac*-phenyl (allylamido)(*p*-tolylamido)phosphinate, (II) (Fig. 2).

Moreover, as the cyclohexylamine (*cyclo*-$C_6H_{11}NH_2$) hydrochloride salt is also relatively insoluble in CH_3CN (and in $CHCl_3$), we used cyclohexylamine to obtain the starting phosphorus–chlorine compound $ClP(O)(OC_6H_5)(NH\text{-}cyclo\text{-}C_6H_{11})$ for the first time. This starting compound was used for the synthesis of *rac*-phenyl (benzylamido)(cyclohexylamido)-phosphinate, (III) (Fig. 3). All three compounds crystallize in centrosymmetric space groups and the racemic character is reflected by the symmetry.

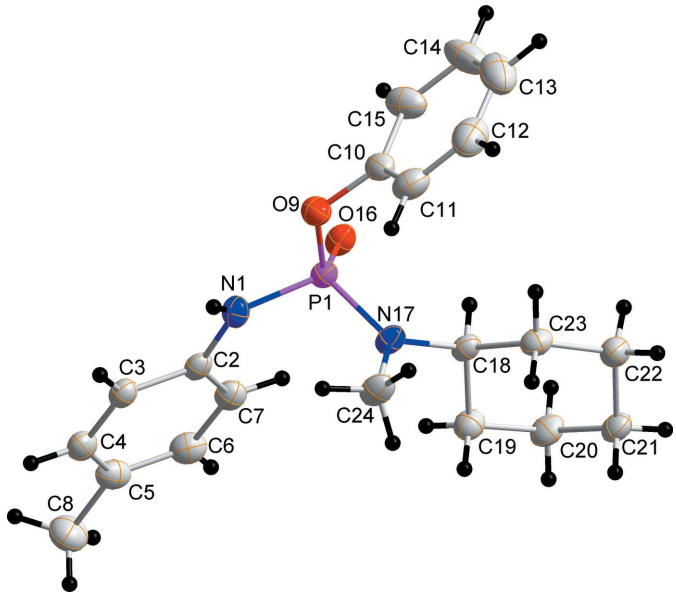

Figure 1
Displacement ellipsoid plot (50% probability level) and atom-numbering scheme for (I).

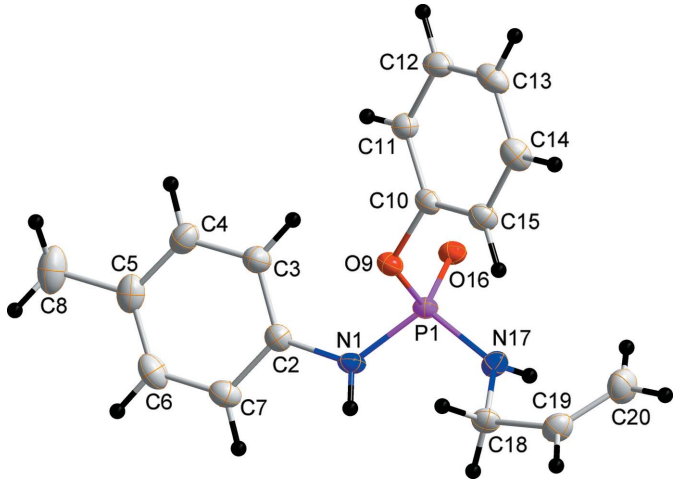

Figure 2
Displacement ellipsoid plot (50% probability level) and atom-numbering scheme for (II). There is disorder of the C8 methyl group and only the major part is shown, for clarity.

2. Experimental

2.1. Synthesis and crystallization

All of the syntheses described in this section begin with the reagents being combined at ice-bath temperature and the mixture then allowed to come to room temperature for the rest of the procedure. For the synthesis of $(Cl)P(O)(OC_6H_5)(NHC_6H_4\text{-}p\text{-}CH_3)$, a solution of 4-$CH_3$–$C_6H_4NH_2$ (20.0 mmol) in dry chloroform (20 ml) was added to a solution of $(C_6H_5O)P(O)Cl_2$ (10.0 mmol) in the same solvent (10 ml) at 273 K. After stirring for 5 h, the solid which formed was

Table 1
Experimental details.

	(I)	(II)	(III)
Crystal data			
Chemical formula	$C_{20}H_{27}N_2O_2P$	$C_{16}H_{19}N_2O_2P$	$C_{19}H_{25}N_2O_2P$
M_r	358.4	302.3	344.4
Crystal system, space group	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/n$
Temperature (K)	120	120	120
a, b, c (Å)	12.9137 (6), 14.6324 (4), 10.3114 (4)	5.4168 (2), 20.3357 (9), 14.2398 (6)	13.3675 (5), 8.0189 (2), 17.4416 (6)
β (°)	99.484 (4)	97.489 (3)	108.707 (3)
V (Å^3)	1921.79 (13)	1555.20 (11)	1770.84 (11)
Z	4	4	4
Radiation type	Cu $K\alpha$	Cu $K\alpha$	Cu $K\alpha$
μ (mm^{-1})	1.39	1.62	1.48
Crystal size (mm)	0.44 × 0.22 × 0.12	0.65 × 0.10 × 0.07	0.17 × 0.12 × 0.10
Data collection			
Diffractometer	Agilent Xcalibur Gemini Ultra diffractometer with Atlas detector	Agilent Xcalibur Gemini Ultra diffractometer with Atlas detector	Agilent Xcalibur Gemini Ultra diffractometer with Atlas detector
Absorption correction	Analytical [*CrysAlis PRO* (Agilent, 2013), using a multifaceted crystal model based on expressions derived by Clark & Reid (1995)]	Analytical [*CrysAlis PRO* (Agilent, 2013), using a multifaceted crystal model based on expressions derived by Clark & Reid (1995)]	Analytical [*CrysAlis PRO* (Agilent, 2013), analytical numerical absorption correction based on crystal shape]
T_{min}, T_{max}	0.706, 0.877	0.613, 0.904	0.88, 0.917
No. of measured, independent and observed [$I > 3\sigma(I)$] reflections	11561, 3420, 2928	10454, 2745, 2333	12735, 3146, 2774
R_{int}	0.031	0.026	0.024
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.597	0.598	0.597
Refinement			
$R[F^3 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.040, 0.111, 1.75	0.028, 0.070, 1.81	0.029, 0.087, 1.53
No. of reflections	3420	2745	3146
No. of parameters	229	198	223
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	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.55, −0.37	0.16, −0.26	0.16, −0.23

Computer programs: *CrysAlis PRO* (Agilent, 2013), *SUPERFLIP* (Palatinus & Chapuis, 2007), *JANA2006* (Petříček *et al.*, 2006), *DIAMOND* (Brandenburg & Putz, 2005) and *Mercury* (Macrae *et al.*, 2008).

filtered off. The product was obtained from the filtrate solution after removing the solvent *in vacuo*.

For the synthesis of (I), a solution of *N*-methylcyclohexylamine (4.6 mmol) in dry chloroform (10 ml) was added to a solution of (Cl)P(O)(OC$_6$H$_5$)(NHC$_6$H$_4$-*p*-CH$_3$) (2.3 mmol) in the same solvent (10 ml) at 273 K. After stirring for 5 h, the solvent was removed *in vacuo* and the solid obtained was washed with distilled water. Single crystals of (I) were obtained from a solution of the product in CHCl$_3$–CH$_3$OH (1:1 *v*/*v*) after slow evaporation at room temperature.

For the synthesis of (II), a solution of allylamine (6.0 mmol) in dry chloroform (10 ml) was added to a solution of (Cl)P(O)(OC$_6$H$_5$)(NHC$_6$H$_4$-*p*-CH$_3$) (3.0 mmol) in the same solvent (10 ml) at 273 K. After stirring for 5 h, the solvent was removed *in vacuo* and the solid obtained was washed with distilled water. Single crystals of (II) were obtained from a solution of the product in CH$_3$CN–CHCl$_3$ (5:1 *v*/*v*) after slow evaporation at room temperature.

For the synthesis of (Cl)P(O)(OC$_6$H$_5$)(NHC$_6$H$_{11}$), a solution of C$_6$H$_{11}$NH$_2$ (20.0 mmol) in dry chloroform (15 ml) was added to a solution of (C$_6$H$_5$O)P(O)Cl$_2$ (10.0 mmol) in the same solvent (15 ml) at 273 K. After stirring for 5 h, the solid was filtered off. The product was obtained from the filtrate solution after removing the solvent *in vacuo*.

For the synthesis of (III), a solution of benzylamine (4.6 mmol) in dry chloroform (10 ml) was added to a solution of (Cl)P(O)(OC$_6$H$_5$)(NHC$_6$H$_{11}$) (2.3 mmol) in the same solvent (10 ml) at 273 K. After stirring for 5 h, the solvent was removed *in vacuo* and the solid obtained was washed with distilled water. Single crystals of (III) were obtained from a solution of the product in CH$_3$CN–CHCl$_3$ (1:1 *v*/*v*) after slow evaporation at room temperature.

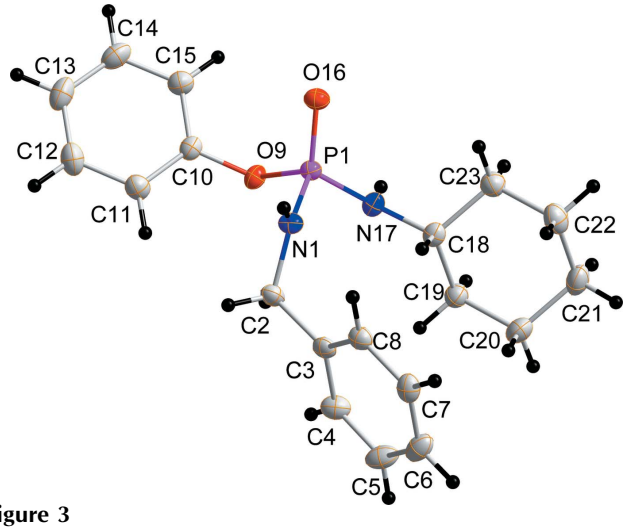

Figure 3
Displacement ellipsoid plot (50% probability level) and atom-numbering scheme for (III).

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. All H atoms were discernible in difference Fourier maps and could be refined to a reasonable geometry. H atoms bonded to C atoms were placed in ideal positions, with C—H = 0.96 Å, while the positions of H atoms bonded to N atoms were refined freely. In both cases, U_{iso}(H) = 1.2U_{eq}(C,N) and, for (I) and (II), 1.5U_{eq}(C) for methyl H atoms. All non-H atoms were refined using harmonic refinement. In (II), the H-atom positions of the disordered methyl group were found in a difference Fourier map, their geometry was fixed to tetrahedral and the occupancies were refined with a final ratio of 0.58 (2):0.42 (2).

3. Results and discussion

In compounds (I), (II) and (III), the P atom exhibits a distorted tetrahedral environment, as has been noted for other amidophosphoesters (Pourayoubi, Karimi Ahmadabad *et al.*, 2011; Pourayoubi, Rheingold *et al.*, 2011). In (I), the angles at the P atom are in the range 94.80 (7)–116.36 (7)°.

As can be seen in Table 2, the *p*-tolylamido group in (I) and (II) makes a longer P1—N1 bond relative to the P1—N17 bonds involving the *N*-methylcyclohexylamido and allylamido groups. This may be attributed to the electron-withdrawing effect of the phenyl group of the *p*-tolylamido substituent, which causes P—N bond weakening. Moreover, the hybridization of the C atom attached to the N atom in the *p*-tolylamido group is different from those of the *N*-methylcyclohexylamido and allylamido groups (sp^2 and sp^3). This is in accord with what was reported for *rac*-*L*P(O)(OC_6H_5)(NHC_6H_4-*p*-CH_3) [*L* = NHC_6H_{11} (Sabbaghi *et al.*, 2011), $NHC(CH_3)_3$ (Pourayoubi, Rheingold *et al.*, 2011) and $NHCH_2C_6H_5$ (Pourayoubi, Karimi Ahmadabad *et al.*, 2011)]. The P—N (*p*-tolylamido) bond is also longer than the related P—N (*L*) bond. In (III), though, the difference between the P—N bond lengths involving the cyclohexylamido and benzylamido fragments is not significant (see Table 2). Such a comparison may be useful for a study of the strengths of different P—N bonds in a given molecule in order to obtain an insight into the electronic effect produced by different amide fragments. For example, a comparison between the P—N bond lengths related to the dimethylamido fragment and the other amido fragments indicates that, in compounds of the general formula [$(CH_3)_2$N][*p*-CH_3-C_6H_4O]P(O)*X*, the P—N(dimethylamido) bond is longer than the P—N(*X*) bond [*X* = $NHC(CH_3)_3$ (Ghadimi *et al.*, 2009) and $NHCH(CH_3)_2$ (Pourayoubi *et al.*, 2007)] and shorter than the P—N(NHC_6H_4-*p*-CH_3) bond (Ghadimi *et al.*, 2009).

In all three structures, the phosphoryl O atom takes part with the N—H unit in hydrogen-bonding interactions, *viz.* an N—H···OP hydrogen bond for (I) (Table 3) and (N—H)(N—H)···O=P hydrogen bonds for (II) (Table 4) and (III)

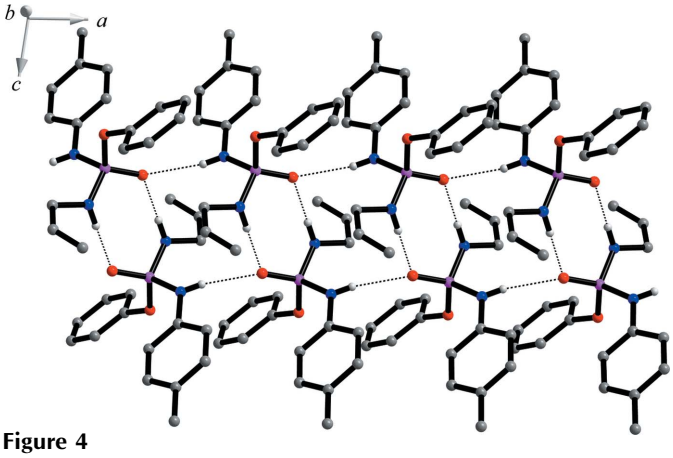

Figure 4
Part of the crystal packing of (II), viewed along the *b* axis, showing the ladder arrangement along [100] built *via* (N—H)(N—H)···O=P hydrogen bonds (dashed lines). Only H atoms involved in hydrogen bonds are shown.

Table 2
Selected geometric parameters (Å, °) for (I), (II) and (III).

Parameter	(I)	(II)	(III)
P1—N1	1.6398 (16)	1.6375 (12)	1.6243 (11)
P1—O9	1.6106 (13)	1.5991 (10)	1.6108 (9)
P1—O16	1.4683 (12)	1.4762 (10)	1.4804 (8)
P1—N17	1.6297 (14)	1.6184 (13)	1.6267 (12)
N1—C2	1.391 (2)	1.4208 (18)	1.4667 (14)
O9—C10	1.388 (2)	1.4027 (16)	1.3937 (17)
N17—C18	1.479 (2)	1.4672 (18)	1.4702 (19)
N1—P1—O9	94.80 (7)	101.30 (5)	108.53 (5)
N1—P1—O16	116.36 (7)	114.69 (6)	111.38 (5)
N1—P1—N17	109.59 (8)	108.13 (6)	107.19 (5)
O9—P1—O16	114.31 (7)	112.59 (5)	112.00 (5)
O9—P1—N17	108.78 (7)	106.79 (6)	96.58 (5)
O16—P1—N17	111.71 (7)	112.50 (6)	119.90 (6)
P1—N1—C2	126.67 (12)	126.96 (10)	123.30 (9)
O9—C10—C11	118.11 (17)	118.57 (12)	119.16 (11)
P1—N17—C18	122.39 (11)	124.40 (9)	123.61 (9)

Table 3
Hydrogen-bond geometry (Å, °) for (I).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N1···O16^{i}	0.87 (3)	1.99 (3)	2.8487 (19)	169 (2)

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

Table 4
Hydrogen-bond geometry (Å, °) for (II).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N1···O16^{i}	0.798 (19)	2.219 (19)	3.0063 (16)	169.0 (16)
N17—H1N17···O16^{ii}	0.873 (17)	2.057 (17)	2.9216 (15)	170.3 (16)

Symmetry codes: (i) $x+1, y, z$; (ii) $-x, -y, -z$.

Table 5
Hydrogen-bond geometry (Å, °) for (III).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N1···O16^{i}	0.839 (17)	2.351 (17)	3.1673 (14)	164.2 (16)
N17—H1N17···O16^{ii}	0.871 (17)	2.140 (17)	2.9977 (14)	168.2 (13)

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

(Table 5), building linear arrangements along [001] for (I) and along [010] for (III), and a ladder arrangement (Fig. 4) along [100] for (II). The phenoxy O atom and the N atoms bonded to the P atom do not take part in hydrogen bonding as acceptors in (I), (II) and (III).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: LG3117). Services for accessing these data are described at the back of the journal.

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supplementary materials

Acta Cryst. (2013). C**69**, 1181-1185 [doi:10.1107/S010827011302341X]

New *rac*-*X*P(O)(OC_6H_5)(NHC_6H_4-*p*-CH_3) [*X* = N(CH_3)(*cyclo*-C_6H_{11}) and NH(C_3H_5)] and *rac*-($C_6H_5CH_2NH$)P(O)(OC_6H_5)(NH-*cyclo*-C_6H_{11}) mixed-amide phosphinates

Mehrdad Pourayoubi, Fatemeh Karimi Ahmadabad, Hossein Eshtiagh-Hosseini, Monika Kučeráková, Václav Eigner and Michal Dušek

Computing details

For all compounds, data collection: *CrysAlis PRO* (Agilent, 2013); cell refinement: *CrysAlis PRO* (Agilent, 2013); data reduction: *CrysAlis PRO* (Agilent, 2013). Program(s) used to solve structure: Superflip (Palatinus & Chapuis, 2007) for (I), (II); SUPERFLIP (Palatinus & Chapuis, 2007) for (III). For all compounds, program(s) used to refine structure: JANA2006 (Petříček *et al.*, 2006). Molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005) for (I), (III); *DIAMOND* (Brandenburg & Putz, 2005) and *Mercury* (Macrae *et al.*, 2008) for (II). For all compounds, software used to prepare material for publication: JANA2006 (Petříček *et al.*, 2006).

(I) *rac*-Phenyl (*N*-methylcyclohexylamido)(*p*-tolylamido)phosphinate

Crystal data

$C_{20}H_{27}N_2O_2P$
M_r = 358.4
Monoclinic, $P2_1/c$
Hall symbol: -P 2ycb
a = 12.9137 (6) Å
b = 14.6324 (4) Å
c = 10.3114 (4) Å
β = 99.484 (4)°
V = 1921.79 (13) Å^3
Z = 4
$F(000)$ = 768
D_x = 1.238 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.5418 Å
Cell parameters from 6300 reflections
θ = 4.6–66.8°
μ = 1.39 mm^{-1}
T = 120 K
Prism, colourless
0.44 × 0.22 × 0.12 mm

Data collection

Agilent Xcalibur Gemini Ultra diffractometer with Atlas detector
Radiation source: Enhance Ultra (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.3784 pixels mm^{-1}
ω scans
Absorption correction: analytical [*CrysAlis PRO* (Agilent, 2013); using a multifaceted crystal model based on expressions derived by Clark & Reid (1995)]
T_{min} = 0.706, T_{max} = 0.877
11561 measured reflections
3420 independent reflections
2928 reflections with $I > 3\sigma(I)$
R_{int} = 0.031
θ_{max} = 67°, θ_{min} = 3.5°
h = −14→15
k = −17→16
l = −12→11

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.111$
$S = 1.75$
3420 reflections
229 parameters
0 restraints
105 constraints

H atoms treated by a mixture of independent and constrained refinement
Weighting scheme based on measured s.u.'s $w = 1/(\sigma^2(I) + 0.0016I^2)$
$(\Delta/\sigma)_{max} = 0.013$
$\Delta\rho_{max} = 0.55$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ e Å^{-3}

Special details

Experimental. CrysAlis PRO (Agilent Technologies, 2013) Version 1.171.36.28 (release 01-02-2013 CrysAlis171 .NET) (compiled Feb 1 2013,16:14:44) Analytical numeric absorption correction using a multifaceted crystal model based on expressions derived by Clark & Reid (1995).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
P1	0.92505 (3)	0.22177 (3)	0.12180 (4)	0.02155 (14)
N1	1.01936 (11)	0.22570 (10)	0.03315 (15)	0.0251 (4)
C2	1.12027 (13)	0.19066 (11)	0.06727 (16)	0.0233 (5)
C3	1.18909 (13)	0.19806 (11)	−0.02420 (17)	0.0258 (5)
C4	1.29106 (13)	0.16683 (12)	0.00592 (18)	0.0294 (5)
C5	1.32966 (14)	0.12681 (13)	0.1265 (2)	0.0323 (5)
C6	1.26075 (14)	0.11880 (13)	0.21662 (18)	0.0319 (6)
C7	1.15761 (13)	0.14976 (12)	0.18874 (17)	0.0281 (5)
C8	1.44047 (16)	0.09078 (16)	0.1563 (2)	0.0454 (7)
O9	0.84656 (9)	0.28686 (8)	0.02516 (12)	0.0288 (4)
C10	0.74259 (13)	0.29973 (12)	0.03959 (17)	0.0279 (5)
C11	0.66557 (16)	0.26084 (13)	−0.0524 (2)	0.0364 (6)
C12	0.56139 (17)	0.27827 (15)	−0.0460 (3)	0.0486 (8)
C13	0.53456 (18)	0.33402 (18)	0.0503 (3)	0.0544 (8)
C14	0.6125 (2)	0.37254 (18)	0.1414 (2)	0.0540 (8)
C15	0.71737 (17)	0.35658 (15)	0.1363 (2)	0.0402 (6)
O16	0.95110 (10)	0.25560 (8)	0.25732 (12)	0.0283 (4)
N17	0.87575 (11)	0.11905 (9)	0.11646 (14)	0.0253 (4)
C18	0.83979 (12)	0.07626 (11)	0.23119 (16)	0.0234 (5)
C19	0.91248 (13)	−0.00213 (12)	0.28778 (18)	0.0290 (5)
C20	0.87503 (14)	−0.04334 (13)	0.40807 (19)	0.0321 (5)
C21	0.76305 (14)	−0.07870 (12)	0.37201 (18)	0.0301 (5)
C22	0.68872 (13)	−0.00268 (12)	0.31399 (19)	0.0302 (5)
C23	0.72622 (13)	0.04343 (12)	0.19689 (18)	0.0275 (5)
C24	0.86069 (16)	0.06812 (13)	−0.00773 (18)	0.0331 (6)
H1C3	1.165023	0.225126	−0.108593	0.031*
H1C4	1.336682	0.172925	−0.058271	0.0353*
H1C6	1.285182	0.0911	0.300471	0.0383*
H1C7	1.112005	0.143072	0.252838	0.0337*
H1C8	1.458208	0.078111	0.248684	0.0681*
H2C8	1.445749	0.035652	0.10738	0.0681*
H3C8	1.487992	0.135657	0.13198	0.0681*
H1C11	0.68413	0.222092	−0.120092	0.0437*

H1C12	0.507325	0.251167	−0.109353	0.0583*
H1C13	0.462061	0.346108	0.05414	0.0653*
H1C14	0.593701	0.410961	0.209344	0.0648*
H1C15	0.771418	0.384522	0.198816	0.0483*
H1C18	0.842586	0.122356	0.297929	0.028*
H1C19	0.912802	−0.04852	0.222015	0.0348*
H2C19	0.982692	0.020613	0.312445	0.0348*
H1C20	0.920598	−0.092723	0.441723	0.0385*
H2C20	0.877759	0.002341	0.475379	0.0385*
H1C21	0.740111	−0.102893	0.449046	0.0361*
H2C21	0.761251	−0.127409	0.309316	0.0361*
H1C22	0.683221	0.042115	0.380535	0.0363*
H2C22	0.619749	−0.027462	0.286591	0.0363*
H1C23	0.720209	0.001297	0.124666	0.033*
H2C23	0.68146	0.094393	0.16815	0.033*
H1C24	0.901843	0.095599	−0.066782	0.0496*
H2C24	0.882516	0.005905	0.008876	0.0496*
H3C24	0.78784	0.069513	−0.046572	0.0496*
H1N1	1.0030 (17)	0.2382 (15)	−0.050 (3)	0.0301*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0277 (2)	0.0199 (2)	0.0169 (2)	−0.00163 (14)	0.00304 (16)	−0.00052 (15)
N1	0.0290 (7)	0.0292 (8)	0.0172 (7)	−0.0022 (5)	0.0043 (6)	0.0014 (6)
C2	0.0295 (8)	0.0187 (8)	0.0213 (8)	−0.0053 (6)	0.0034 (6)	−0.0037 (7)
C3	0.0301 (8)	0.0217 (8)	0.0257 (8)	−0.0036 (6)	0.0043 (6)	−0.0009 (7)
C4	0.0300 (8)	0.0249 (8)	0.0332 (9)	−0.0046 (6)	0.0052 (7)	−0.0038 (7)
C5	0.0278 (8)	0.0279 (9)	0.0390 (10)	0.0001 (7)	−0.0011 (7)	−0.0063 (8)
C6	0.0357 (9)	0.0301 (10)	0.0273 (9)	0.0017 (7)	−0.0024 (7)	−0.0014 (8)
C7	0.0327 (9)	0.0271 (9)	0.0243 (8)	−0.0028 (7)	0.0040 (7)	−0.0040 (7)
C8	0.0333 (10)	0.0488 (12)	0.0529 (13)	0.0081 (9)	0.0035 (9)	0.0033 (11)
O9	0.0332 (6)	0.0290 (6)	0.0250 (6)	0.0053 (5)	0.0075 (5)	0.0062 (5)
C10	0.0331 (9)	0.0254 (8)	0.0262 (9)	0.0064 (7)	0.0083 (7)	0.0082 (7)
C11	0.0443 (10)	0.0258 (9)	0.0378 (11)	−0.0005 (7)	0.0032 (8)	0.0012 (8)
C12	0.0363 (10)	0.0438 (12)	0.0633 (15)	−0.0010 (8)	0.0011 (10)	0.0136 (11)
C13	0.0422 (12)	0.0622 (15)	0.0616 (15)	0.0167 (10)	0.0167 (11)	0.0245 (13)
C14	0.0635 (15)	0.0612 (15)	0.0428 (12)	0.0317 (12)	0.0253 (11)	0.0101 (11)
C15	0.0543 (12)	0.0389 (11)	0.0276 (10)	0.0125 (9)	0.0071 (9)	0.0007 (8)
O16	0.0400 (7)	0.0257 (6)	0.0194 (6)	−0.0066 (5)	0.0052 (5)	−0.0023 (5)
N17	0.0354 (7)	0.0216 (7)	0.0188 (7)	−0.0048 (5)	0.0040 (5)	−0.0025 (6)
C18	0.0304 (8)	0.0189 (8)	0.0205 (8)	−0.0018 (6)	0.0034 (6)	−0.0011 (7)
C19	0.0278 (8)	0.0279 (9)	0.0300 (9)	0.0003 (6)	0.0011 (7)	0.0045 (7)
C20	0.0364 (9)	0.0280 (9)	0.0298 (9)	0.0004 (7)	−0.0004 (7)	0.0067 (8)
C21	0.0387 (9)	0.0223 (8)	0.0298 (9)	−0.0019 (7)	0.0071 (7)	0.0043 (7)
C22	0.0305 (9)	0.0259 (9)	0.0348 (10)	−0.0011 (7)	0.0071 (7)	0.0006 (8)
C23	0.0289 (8)	0.0235 (8)	0.0287 (9)	0.0023 (6)	0.0004 (7)	0.0024 (7)
C24	0.0501 (11)	0.0268 (9)	0.0226 (9)	−0.0066 (7)	0.0070 (7)	−0.0068 (7)

Geometric parameters (Å, º)

P1—N1	1.6398 (16)	C13—C14	1.379 (3)
P1—O9	1.6106 (13)	C13—H1C13	0.96
P1—O16	1.4683 (12)	C14—C15	1.384 (3)
P1—N17	1.6297 (14)	C14—H1C14	0.96
N1—C2	1.391 (2)	C15—H1C15	0.96
N1—H1N1	0.87 (3)	N17—C18	1.479 (2)
C2—C3	1.403 (3)	N17—C24	1.467 (2)
C2—C7	1.400 (2)	C18—C19	1.535 (2)
C3—C4	1.380 (2)	C18—C23	1.528 (2)
C3—H1C3	0.96	C18—H1C18	0.96
C4—C5	1.390 (3)	C19—C20	1.528 (3)
C4—H1C4	0.96	C19—H1C19	0.96
C5—C6	1.394 (3)	C19—H2C19	0.96
C5—C8	1.508 (3)	C20—C21	1.523 (2)
C6—C7	1.391 (2)	C20—H1C20	0.96
C6—H1C6	0.96	C20—H2C20	0.96
C7—H1C7	0.96	C21—C22	1.526 (2)
C8—H1C8	0.96	C21—H1C21	0.96
C8—H2C8	0.96	C21—H2C21	0.96
C8—H3C8	0.96	C22—C23	1.530 (3)
O9—C10	1.388 (2)	C22—H1C22	0.96
C10—C11	1.379 (3)	C22—H2C22	0.96
C10—C15	1.378 (3)	C23—H1C23	0.96
C11—C12	1.381 (3)	C23—H2C23	0.96
C11—H1C11	0.96	C24—H1C24	0.96
C12—C13	1.373 (4)	C24—H2C24	0.96
C12—H1C12	0.96	C24—H3C24	0.96
N1—P1—O9	94.80 (7)	C10—C15—C14	118.61 (19)
N1—P1—O16	116.36 (7)	C10—C15—H1C15	120.7
N1—P1—N17	109.59 (8)	C14—C15—H1C15	120.7
O9—P1—O16	114.31 (7)	P1—N17—C18	122.39 (11)
O9—P1—N17	108.78 (7)	P1—N17—C24	119.54 (12)
O16—P1—N17	111.71 (7)	C18—N17—C24	117.99 (13)
P1—N1—C2	126.67 (12)	N17—C18—C19	111.55 (14)
P1—N1—H1N1	118.5 (15)	N17—C18—C23	111.32 (13)
C2—N1—H1N1	113.1 (15)	N17—C18—H1C18	107.23
N1—C2—C3	118.21 (14)	C19—C18—C23	110.88 (13)
N1—C2—C7	123.65 (16)	C19—C18—H1C18	107.7
C3—C2—C7	118.13 (15)	C23—C18—H1C18	107.95
C2—C3—C4	120.66 (16)	C18—C19—C20	110.36 (15)
C2—C3—H1C3	119.67	C18—C19—H1C19	109.47
C4—C3—H1C3	119.67	C18—C19—H2C19	109.47
C3—C4—C5	121.94 (18)	C20—C19—H1C19	109.47
C3—C4—H1C4	119.03	C20—C19—H2C19	109.47
C5—C4—H1C4	119.03	H1C19—C19—H2C19	108.57
C4—C5—C6	117.23 (16)	C19—C20—C21	110.53 (14)
C4—C5—C8	121.21 (19)	C19—C20—H1C20	109.47

C6—C5—C8	121.53 (18)	C19—C20—H2C20	109.47
C5—C6—C7	121.97 (17)	C21—C20—H1C20	109.47
C5—C6—H1C6	119.01	C21—C20—H2C20	109.47
C7—C6—H1C6	119.01	H1C20—C20—H2C20	108.39
C2—C7—C6	120.06 (17)	C20—C21—C22	111.03 (15)
C2—C7—H1C7	119.97	C20—C21—H1C21	109.47
C6—C7—H1C7	119.97	C20—C21—H2C21	109.47
C5—C8—H1C8	109.47	C22—C21—H1C21	109.47
C5—C8—H2C8	109.47	C22—C21—H2C21	109.47
C5—C8—H3C8	109.47	H1C21—C21—H2C21	107.87
H1C8—C8—H2C8	109.47	C21—C22—C23	111.75 (15)
H1C8—C8—H3C8	109.47	C21—C22—H1C22	109.47
H2C8—C8—H3C8	109.47	C21—C22—H2C22	109.47
O9—C10—C11	118.11 (17)	C23—C22—H1C22	109.47
O9—C10—C15	120.51 (16)	C23—C22—H2C22	109.47
C11—C10—C15	121.13 (18)	H1C22—C22—H2C22	107.1
C10—C11—C12	119.2 (2)	C18—C23—C22	111.87 (14)
C10—C11—H1C11	120.38	C18—C23—H1C23	109.47
C12—C11—H1C11	120.38	C18—C23—H2C23	109.47
C11—C12—C13	120.5 (2)	C22—C23—H1C23	109.47
C11—C12—H1C12	119.73	C22—C23—H2C23	109.47
C13—C12—H1C12	119.73	H1C23—C23—H2C23	106.96
C12—C13—C14	119.5 (2)	N17—C24—H1C24	109.47
C12—C13—H1C13	120.24	N17—C24—H2C24	109.47
C14—C13—H1C13	120.24	N17—C24—H3C24	109.47
C13—C14—C15	120.9 (2)	H1C24—C24—H2C24	109.47
C13—C14—H1C14	119.53	H1C24—C24—H3C24	109.47
C15—C14—H1C14	119.54	H2C24—C24—H3C24	109.47

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*1···O16^{i}	0.87 (3)	1.99 (3)	2.8487 (19)	169 (2)

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

(II) *rac*-Phenyl (allylamido)(*p*-tolylamido)phosphinate

Crystal data

$C_{16}H_{19}N_2O_2P$
M_r = 302.3
Monoclinic, $P2_1/c$
Hall symbol: -P 2ycb
a = 5.4168 (2) Å
b = 20.3357 (9) Å
c = 14.2398 (6) Å
β = 97.489 (3)°
V = 1555.20 (11) Å^3
Z = 4
$F(000)$ = 640
D_x = 1.291 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.5418 Å
Cell parameters from 5797 reflections
θ = 3.1–67.0°
μ = 1.62 mm^{-1}
T = 120 K
Prism, colourless
0.65 × 0.10 × 0.07 mm

Data collection

Agilent Xcalibur Gemini Ultra diffractometer with Atlas detector
Radiation source: Enhance Ultra (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.3784 pixels mm^{-1}
ω scans

Absorption correction: analytical [*CrysAlis PRO* (Agilent, 2013); using a multifaceted crystal model based on expressions derived by Clark & Reid (1995)]
T_{min} = 0.613, T_{max} = 0.904
10454 measured reflections
2745 independent reflections
2333 reflections with $I > 3\sigma(I)$
R_{int} = 0.026
θ_{max} = 67.2°, θ_{min} = 3.8°
h = −6→6
k = −24→24
l = −16→16

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.070
S = 1.81
2745 reflections
198 parameters
0 restraints
87 constraints

H atoms treated by a mixture of independent and constrained refinement
Weighting scheme based on measured s.u.'s $w = 1/(\sigma^2(I) + 0.0004I^2)$
$(\Delta/\sigma)_{max}$ = 0.019
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Extinction correction: B-C type 1 Gaussian isotropic (Becker & Coppens, 1974)
Extinction coefficient: 1070 (170)

Special details

Experimental. CrysAlis PRO (Agilent Technologies, 2013) Version 1.171.35.21 (release 20-01-2012 CrysAlis171 .NET) (compiled Jan 23 2012,18:06:46) Analytical numeric absorption correction using a multifaceted crystal model based on expressions derived by Clark & Reid (1995).

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
P1	0.18488 (6)	0.050592 (16)	0.12760 (2)	0.01772 (11)	
N1	0.4263 (2)	0.00759 (6)	0.17371 (9)	0.0211 (3)	
C2	0.4578 (2)	−0.02454 (6)	0.26308 (10)	0.0209 (4)	
C3	0.2879 (3)	−0.01881 (7)	0.32763 (11)	0.0267 (4)	
C4	0.3286 (3)	−0.05143 (8)	0.41368 (11)	0.0302 (5)	
C5	0.5360 (3)	−0.09073 (7)	0.43834 (11)	0.0301 (5)	
C6	0.7029 (3)	−0.09599 (7)	0.37260 (12)	0.0324 (5)	
C7	0.6668 (3)	−0.06367 (7)	0.28651 (11)	0.0276 (4)	
C8	0.5764 (4)	−0.12675 (9)	0.53139 (13)	0.0445 (6)	
O9	0.19758 (16)	0.11180 (4)	0.19856 (7)	0.0205 (3)	
C10	0.0084 (2)	0.15911 (6)	0.19621 (10)	0.0203 (4)	
C11	−0.1630 (2)	0.15344 (7)	0.25954 (10)	0.0234 (4)	
C12	−0.3391 (3)	0.20295 (7)	0.26244 (11)	0.0271 (4)	
C13	−0.3425 (3)	0.25665 (7)	0.20307 (11)	0.0293 (5)	
C14	−0.1713 (3)	0.26086 (7)	0.13908 (12)	0.0287 (5)	
C15	0.0069 (3)	0.21214 (7)	0.13540 (11)	0.0248 (4)	
O16	−0.05512 (17)	0.01539 (4)	0.12077 (7)	0.0212 (3)	
N17	0.2421 (2)	0.07885 (6)	0.02639 (9)	0.0213 (3)	

C18	0.4585 (3)	0.11949 (7)	0.01298 (10)	0.0231 (4)	
C19	0.4016 (2)	0.17120 (7)	−0.06164 (10)	0.0276 (4)	
C20	0.1826 (2)	0.18363 (8)	−0.10972 (12)	0.0349 (5)	
H1C3	0.141812	0.007832	0.312621	0.0321*	
H1C4	0.209455	−0.046676	0.457565	0.0362*	
H1C6	0.848234	−0.122956	0.387411	0.0389*	
H1C7	0.786537	−0.068285	0.242815	0.0331*	
H1C8	0.48946	−0.104452	0.576643	0.0667*	0.58 (2)
H2C8	0.514335	−0.170849	0.52273	0.0667*	0.58 (2)
H3C8	0.751119	−0.127927	0.554159	0.0667*	0.58 (2)
H1C8′	0.420119	−0.143386	0.546166	0.0667*	0.42 (2)
H2C8′	0.689223	−0.162681	0.526954	0.0667*	0.42 (2)
H3C8′	0.645572	−0.097161	0.580413	0.0667*	0.42 (2)
H1C11	−0.160725	0.116033	0.300773	0.0281*	
H1C12	−0.459333	0.199726	0.306159	0.0325*	
H1C13	−0.463015	0.29094	0.206089	0.0352*	
H1C14	−0.175758	0.297786	0.096892	0.0345*	
H1C15	0.126701	0.215237	0.091492	0.0297*	
H1N17	0.181 (3)	0.0547 (8)	−0.0218 (13)	0.0255*	
H1C18	0.590475	0.091843	−0.003039	0.0277*	
H2C18	0.522375	0.139952	0.071923	0.0277*	
H1C19	0.538375	0.197845	−0.075719	0.0332*	
H1C20	0.165555	0.217769	−0.156689	0.0419*	
H2C20	0.03963	0.158574	−0.097868	0.0419*	
H1N1	0.555 (3)	0.0116 (8)	0.1526 (12)	0.0253*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.01564 (18)	0.01839 (18)	0.01964 (19)	−0.00093 (13)	0.00424 (13)	−0.00026 (13)
N1	0.0157 (6)	0.0238 (6)	0.0252 (6)	0.0002 (5)	0.0077 (5)	0.0025 (5)
C2	0.0205 (7)	0.0179 (6)	0.0239 (7)	−0.0025 (5)	0.0014 (5)	−0.0002 (5)
C3	0.0236 (7)	0.0291 (7)	0.0279 (8)	0.0026 (6)	0.0052 (6)	0.0023 (6)
C4	0.0332 (8)	0.0332 (8)	0.0246 (8)	−0.0038 (7)	0.0060 (6)	0.0011 (6)
C5	0.0380 (8)	0.0225 (7)	0.0277 (8)	−0.0068 (7)	−0.0042 (6)	0.0025 (6)
C6	0.0297 (8)	0.0244 (7)	0.0409 (9)	0.0043 (6)	−0.0040 (7)	0.0040 (7)
C7	0.0240 (7)	0.0243 (7)	0.0349 (8)	0.0020 (6)	0.0057 (6)	0.0013 (6)
C8	0.0609 (12)	0.0354 (9)	0.0334 (10)	−0.0058 (8)	−0.0076 (8)	0.0087 (8)
O9	0.0168 (5)	0.0212 (5)	0.0235 (5)	0.0017 (4)	0.0024 (4)	−0.0024 (4)
C10	0.0169 (6)	0.0199 (6)	0.0233 (7)	0.0001 (5)	−0.0003 (5)	−0.0046 (5)
C11	0.0226 (7)	0.0248 (7)	0.0231 (7)	0.0004 (6)	0.0035 (6)	−0.0006 (6)
C12	0.0223 (7)	0.0310 (7)	0.0284 (8)	0.0020 (6)	0.0055 (6)	−0.0057 (6)
C13	0.0216 (7)	0.0250 (7)	0.0402 (9)	0.0028 (6)	−0.0002 (6)	−0.0070 (6)
C14	0.0247 (7)	0.0219 (7)	0.0386 (9)	−0.0022 (6)	0.0005 (6)	0.0040 (6)
C15	0.0204 (7)	0.0245 (7)	0.0295 (8)	−0.0034 (6)	0.0035 (6)	0.0011 (6)
O16	0.0182 (5)	0.0227 (5)	0.0232 (5)	−0.0025 (4)	0.0055 (4)	−0.0006 (4)
N17	0.0213 (6)	0.0231 (6)	0.0196 (6)	−0.0065 (5)	0.0039 (5)	−0.0012 (5)
C18	0.0186 (7)	0.0251 (7)	0.0261 (8)	−0.0043 (6)	0.0054 (6)	0.0011 (6)
C19	0.0294 (8)	0.0269 (7)	0.0278 (8)	−0.0056 (6)	0.0081 (6)	0.0034 (6)
C20	0.0366 (9)	0.0359 (8)	0.0320 (9)	−0.0007 (7)	0.0037 (7)	0.0105 (7)

Geometric parameters (Å, º)

P1—N1	1.6375 (12)	C10—C15	1.382 (2)
P1—O9	1.5991 (10)	C11—C12	1.391 (2)
P1—O16	1.4762 (10)	C11—H1C11	0.96
P1—N17	1.6184 (13)	C12—C13	1.380 (2)
N1—C2	1.4208 (18)	C12—H1C12	0.96
N1—H1N1	0.798 (19)	C13—C14	1.385 (2)
C2—C3	1.388 (2)	C13—H1C13	0.96
C2—C7	1.3886 (19)	C14—C15	1.389 (2)
C3—C4	1.386 (2)	C14—H1C14	0.96
C3—H1C3	0.96	C15—H1C15	0.96
C4—C5	1.386 (2)	N17—C18	1.4672 (18)
C4—H1C4	0.96	N17—H1N17	0.873 (17)
C5—C6	1.388 (2)	C18—C19	1.498 (2)
C5—C8	1.505 (2)	C18—H1C18	0.96
C6—C7	1.382 (2)	C18—H2C18	0.96
C6—H1C6	0.96	C19—C20	1.3148 (18)
C7—H1C7	0.96	C19—H1C19	0.96
C8—H1C8	0.96	C20—H1C20	0.96
C8—H2C8	0.96	C20—H2C20	0.96
C8—H3C8	0.96	H1C8—H1C8′	0.9559
C8—H1C8′	0.96	H1C8—H3C8′	0.8533
C8—H2C8′	0.96	H2C8—H1C8′	0.8533
C8—H3C8′	0.96	H2C8—H2C8′	0.9559
O9—C10	1.4027 (16)	H3C8—H2C8′	0.8533
C10—C11	1.381 (2)	H3C8—H3C8′	0.9559
N1—P1—O9	101.30 (5)	H2C8—C8—H3C8	109.47
N1—P1—O16	114.69 (6)	H1C8′—C8—H2C8′	109.47
N1—P1—N17	108.13 (6)	H1C8′—C8—H3C8′	109.47
O9—P1—O16	112.59 (5)	H2C8′—C8—H3C8′	109.47
O9—P1—N17	106.79 (6)	P1—O9—C10	123.21 (8)
O16—P1—N17	112.50 (6)	O9—C10—C11	118.57 (12)
P1—N1—C2	126.96 (10)	O9—C10—C15	119.65 (12)
P1—N1—H1N1	119.1 (12)	C11—C10—C15	121.65 (13)
C2—N1—H1N1	112.0 (12)	C10—C11—C12	118.74 (13)
N1—C2—C3	122.82 (12)	C10—C11—H1C11	120.63
N1—C2—C7	118.54 (13)	C12—C11—H1C11	120.63
C3—C2—C7	118.64 (13)	C11—C12—C13	120.60 (14)
C2—C3—C4	120.19 (13)	C11—C12—H1C12	119.7
C2—C3—H1C3	119.9	C13—C12—H1C12	119.7
C4—C3—H1C3	119.9	C12—C13—C14	119.70 (14)
C3—C4—C5	122.03 (15)	C12—C13—H1C13	120.15
C3—C4—H1C4	118.99	C14—C13—H1C13	120.15
C5—C4—H1C4	118.99	C13—C14—C15	120.60 (14)
C4—C5—C6	116.83 (14)	C13—C14—H1C14	119.7
C4—C5—C8	121.59 (15)	C15—C14—H1C14	119.7
C6—C5—C8	121.57 (14)	C10—C15—C14	118.70 (14)
C5—C6—C7	122.16 (14)	C10—C15—H1C15	120.65

C5—C6—H1C6	118.92	C14—C15—H1C15	120.65
C7—C6—H1C6	118.92	P1—N17—C18	124.40 (9)
C2—C7—C6	120.14 (15)	P1—N17—H1N17	113.8 (12)
C2—C7—H1C7	119.93	C18—N17—H1N17	116.2 (12)
C6—C7—H1C7	119.93	N17—C18—C19	113.18 (11)
C5—C8—H1C8	109.47	N17—C18—H1C18	109.47
C5—C8—H2C8	109.47	N17—C18—H2C18	109.47
C5—C8—H3C8	109.47	C19—C18—H1C18	109.47
C5—C8—H1C8′	109.47	C19—C18—H2C18	109.47
C5—C8—H2C8′	109.47	H1C18—C18—H2C18	105.48
C5—C8—H3C8′	109.47	C18—C19—C20	126.35 (13)
H1C8—C8—H2C8	109.47	C18—C19—H1C19	116.82
H1C8—C8—H3C8	109.47	C20—C19—H1C19	116.83

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*1···O16^{i}	0.798 (19)	2.219 (19)	3.0063 (16)	169.0 (16)
N17—H1*N*17···O16^{ii}	0.873 (17)	2.057 (17)	2.9216 (15)	170.3 (16)

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

(III) *rac*-Phenyl (benzylamido)(cyclohexylamido)phosphinate

Crystal data

$C_{19}H_{25}N_2O_2P$
M_r = 344.4
Monoclinic, $P2_1/n$
Hall symbol: -P 2yabc
a = 13.3675 (5) Å
b = 8.0189 (2) Å
c = 17.4416 (6) Å
β = 108.707 (3)°
V = 1770.84 (11) Å^3
Z = 4
$F(000)$ = 736
D_x = 1.291 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.5418 Å
Cell parameters from 8367 reflections
θ = 3.5–67.1°
μ = 1.48 mm^{-1}
T = 120 K
Prism, colourless
0.17 × 0.12 × 0.10 mm

Data collection

Agilent Xcalibur Gemini Ultra diffractometer with Atlas detector
Radiation source: Enhance Ultra (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.3784 pixels mm^{-1}
ω scans
Absorption correction: analytical [*CrysAlis PRO* (Agilent, 2013); analytical numerical absorption correction based on crystal shape]
T_{min} = 0.88, T_{max} = 0.917
12735 measured reflections
3146 independent reflections
2774 reflections with $I > 3\sigma(I)$
R_{int} = 0.024
θ_{max} = 67.1°, θ_{min} = 3.7°
h = −15→15
k = −9→9
l = −20→20

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.087$
$S = 1.53$
3146 reflections
223 parameters
0 restraints
94 constraints

H atoms treated by a mixture of independent and constrained refinement
Weighting scheme based on measured s.u.'s $w = 1/(\sigma^2(I) + 0.0016I^2)$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
P1	0.71355 (2)	0.11093 (4)	0.189090 (18)	0.01674 (12)
N1	0.66090 (8)	0.28539 (13)	0.14825 (6)	0.0194 (3)
C2	0.58563 (10)	0.29715 (16)	0.06605 (7)	0.0218 (4)
C3	0.47414 (10)	0.34243 (16)	0.06186 (7)	0.0202 (4)
C4	0.38903 (11)	0.27421 (16)	0.00134 (8)	0.0255 (4)
C5	0.28614 (11)	0.31666 (17)	−0.00480 (9)	0.0309 (5)
C6	0.26684 (11)	0.42688 (17)	0.05023 (9)	0.0291 (5)
C7	0.35120 (11)	0.49612 (16)	0.11023 (8)	0.0250 (4)
C8	0.45453 (10)	0.45565 (16)	0.11581 (8)	0.0220 (4)
O9	0.75651 (7)	0.01290 (10)	0.12532 (5)	0.0209 (3)
C10	0.84955 (10)	0.05993 (16)	0.11136 (8)	0.0197 (4)
C11	0.84434 (11)	0.16886 (17)	0.04868 (8)	0.0255 (4)
C12	0.93691 (12)	0.20763 (18)	0.03224 (9)	0.0313 (5)
C13	1.03197 (12)	0.13873 (18)	0.07779 (9)	0.0321 (5)
C14	1.03541 (11)	0.03000 (18)	0.14028 (9)	0.0312 (5)
C15	0.94397 (11)	−0.01049 (17)	0.15729 (8)	0.0252 (4)
O16	0.79798 (7)	0.13961 (11)	0.26706 (5)	0.0220 (3)
N17	0.61720 (8)	−0.01643 (13)	0.18485 (7)	0.0204 (4)
C18	0.52451 (10)	0.03001 (15)	0.20812 (8)	0.0201 (4)
C19	0.42742 (10)	−0.05824 (18)	0.15236 (8)	0.0252 (4)
C20	0.32791 (11)	−0.01393 (19)	0.17285 (9)	0.0319 (5)
C21	0.34172 (11)	−0.0506 (2)	0.26121 (9)	0.0341 (5)
C22	0.43866 (12)	0.03763 (19)	0.31688 (9)	0.0320 (5)
C23	0.53794 (11)	−0.00843 (17)	0.29628 (9)	0.0266 (5)
H1C2	0.584902	0.193837	0.038069	0.0261*
H2C2	0.611017	0.376324	0.035348	0.0261*
H1C4	0.401669	0.196911	−0.036564	0.0306*
H1C5	0.228154	0.269723	−0.047199	0.0371*
H1C6	0.195679	0.454834	0.04672	0.0349*
H1C7	0.338278	0.572705	0.148303	0.0301*
H1C8	0.51245	0.505976	0.156993	0.0263*
H1C11	0.778131	0.216807	0.017123	0.0306*
H1C12	0.93459	0.282867	−0.01111	0.0376*
H1C13	1.095558	0.166089	0.066206	0.0386*
H1C14	1.101613	−0.017658	0.172033	0.0375*
H1C15	0.946258	−0.086406	0.200379	0.0303*
H1C18	0.516071	0.148726	0.202415	0.0241*

H1C19	0.418091	−0.028201	0.097216	0.0302*
H2C19	0.438263	−0.176671	0.156645	0.0302*
H1C20	0.269303	−0.076851	0.138885	0.0382*
H2C20	0.312149	0.102214	0.162088	0.0382*
H1C21	0.280087	−0.014415	0.273467	0.0409*
H2C21	0.348851	−0.168686	0.270407	0.0409*
H1C22	0.447909	0.007838	0.372041	0.0384*
H2C22	0.428115	0.156088	0.312102	0.0384*
H1C23	0.55251	−0.125067	0.306332	0.0319*
H2C23	0.597085	0.052864	0.330611	0.0319*
H1N1	0.6846 (13)	0.376 (2)	0.1713 (10)	0.0233*
H1N17	0.6347 (13)	−0.121 (2)	0.1925 (9)	0.0244*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.01671 (18)	0.01421 (18)	0.01837 (19)	0.00020 (11)	0.00431 (13)	0.00039 (11)
N1	0.0202 (5)	0.0145 (5)	0.0205 (5)	−0.0008 (4)	0.0024 (4)	−0.0003 (4)
C2	0.0231 (6)	0.0223 (6)	0.0187 (6)	0.0038 (5)	0.0051 (5)	0.0026 (5)
C3	0.0224 (6)	0.0166 (6)	0.0201 (6)	0.0019 (5)	0.0049 (5)	0.0046 (5)
C4	0.0279 (7)	0.0195 (6)	0.0252 (7)	0.0017 (5)	0.0031 (5)	−0.0027 (5)
C5	0.0239 (7)	0.0240 (7)	0.0375 (8)	−0.0019 (6)	−0.0002 (6)	−0.0020 (6)
C6	0.0215 (7)	0.0220 (7)	0.0440 (9)	0.0011 (5)	0.0106 (6)	0.0048 (6)
C7	0.0295 (7)	0.0187 (6)	0.0304 (7)	0.0010 (5)	0.0145 (6)	0.0019 (5)
C8	0.0246 (7)	0.0183 (6)	0.0218 (6)	−0.0009 (5)	0.0059 (5)	0.0016 (5)
O9	0.0195 (5)	0.0186 (4)	0.0260 (5)	−0.0017 (3)	0.0092 (4)	−0.0033 (3)
C10	0.0200 (6)	0.0175 (6)	0.0228 (6)	−0.0015 (5)	0.0085 (5)	−0.0049 (5)
C11	0.0285 (7)	0.0237 (7)	0.0255 (7)	0.0031 (6)	0.0102 (5)	0.0014 (5)
C12	0.0398 (8)	0.0275 (7)	0.0330 (8)	−0.0022 (6)	0.0206 (6)	0.0009 (6)
C13	0.0295 (7)	0.0297 (7)	0.0438 (9)	−0.0057 (6)	0.0210 (6)	−0.0085 (6)
C14	0.0212 (7)	0.0314 (8)	0.0402 (8)	0.0021 (6)	0.0086 (6)	−0.0056 (6)
C15	0.0252 (7)	0.0233 (7)	0.0266 (7)	0.0010 (5)	0.0075 (5)	−0.0005 (5)
O16	0.0223 (5)	0.0193 (4)	0.0219 (5)	0.0007 (3)	0.0036 (4)	0.0004 (3)
N17	0.0204 (5)	0.0141 (5)	0.0280 (6)	0.0011 (4)	0.0098 (4)	0.0010 (4)
C18	0.0205 (6)	0.0154 (6)	0.0256 (7)	0.0007 (5)	0.0092 (5)	0.0000 (5)
C19	0.0224 (7)	0.0272 (7)	0.0264 (7)	−0.0019 (5)	0.0083 (5)	−0.0006 (5)
C20	0.0202 (7)	0.0391 (8)	0.0360 (8)	0.0011 (6)	0.0086 (6)	0.0019 (6)
C21	0.0273 (7)	0.0384 (8)	0.0429 (9)	0.0026 (6)	0.0199 (6)	0.0049 (7)
C22	0.0387 (8)	0.0324 (8)	0.0297 (8)	0.0037 (6)	0.0177 (6)	0.0005 (6)
C23	0.0270 (7)	0.0272 (7)	0.0257 (7)	−0.0010 (5)	0.0087 (5)	−0.0002 (5)

Geometric parameters (Å, º)

P1—N1	1.6243 (11)	C12—H1C12	0.96
P1—O9	1.6108 (9)	C13—C14	1.385 (2)
P1—O16	1.4804 (8)	C13—H1C13	0.96
P1—N17	1.6267 (12)	C14—C15	1.386 (2)
N1—C2	1.4667 (14)	C14—H1C14	0.96
N1—H1N1	0.839 (17)	C15—H1C15	0.96
C2—C3	1.5128 (19)	N17—C18	1.4702 (19)

C2—H1C2	0.96	N17—H1N17	0.871 (17)
C2—H2C2	0.96	C18—C19	1.5236 (16)
C3—C4	1.3919 (16)	C18—C23	1.521 (2)
C3—C8	1.3917 (19)	C18—H1C18	0.96
C4—C5	1.387 (2)	C19—C20	1.526 (2)
C4—H1C4	0.96	C19—H1C19	0.96
C5—C6	1.388 (2)	C19—H2C19	0.96
C5—H1C5	0.96	C20—C21	1.521 (2)
C6—C7	1.3849 (18)	C20—H1C20	0.96
C6—H1C6	0.96	C20—H2C20	0.96
C7—C8	1.392 (2)	C21—C22	1.5218 (19)
C7—H1C7	0.96	C21—H1C21	0.96
C8—H1C8	0.96	C21—H2C21	0.96
O9—C10	1.3937 (17)	C22—C23	1.527 (2)
C10—C11	1.3835 (19)	C22—H1C22	0.96
C10—C15	1.3803 (17)	C22—H2C22	0.96
C11—C12	1.392 (2)	C23—H1C23	0.96
C11—H1C11	0.96	C23—H2C23	0.96
C12—C13	1.380 (2)		

N1—P1—O9	108.53 (5)	C13—C14—C15	120.47 (13)
N1—P1—O16	111.38 (5)	C13—C14—H1C14	119.76
N1—P1—N17	107.19 (5)	C15—C14—H1C14	119.76
O9—P1—O16	112.00 (5)	C10—C15—C14	119.02 (13)
O9—P1—N17	96.58 (5)	C10—C15—H1C15	120.49
O16—P1—N17	119.90 (6)	C14—C15—H1C15	120.49
P1—N1—C2	123.30 (9)	P1—N17—C18	123.61 (9)
P1—N1—H1N1	119.3 (10)	P1—N17—H1N17	115.4 (11)
C2—N1—H1N1	116.6 (11)	C18—N17—H1N17	114.3 (13)
N1—C2—C3	114.78 (11)	N17—C18—C19	109.08 (11)
N1—C2—H1C2	109.47	N17—C18—C23	113.02 (10)
N1—C2—H2C2	109.47	N17—C18—H1C18	107.7
C3—C2—H1C2	109.47	C19—C18—C23	110.57 (12)
C3—C2—H2C2	109.47	C19—C18—H1C18	110.29
H1C2—C2—H2C2	103.58	C23—C18—H1C18	106.12
C2—C3—C4	119.69 (12)	C18—C19—C20	111.74 (12)
C2—C3—C8	121.34 (10)	C18—C19—H1C19	109.47
C4—C3—C8	118.93 (12)	C18—C19—H2C19	109.47
C3—C4—C5	120.74 (13)	C20—C19—H1C19	109.47
C3—C4—H1C4	119.63	C20—C19—H2C19	109.47
C5—C4—H1C4	119.63	H1C19—C19—H2C19	107.1
C4—C5—C6	120.14 (12)	C19—C20—C21	111.15 (11)
C4—C5—H1C5	119.93	C19—C20—H1C20	109.47
C6—C5—H1C5	119.93	C19—C20—H2C20	109.47
C5—C6—C7	119.39 (14)	C21—C20—H1C20	109.47
C5—C6—H1C6	120.3	C21—C20—H2C20	109.47
C7—C6—H1C6	120.3	H1C20—C20—H2C20	107.74
C6—C7—C8	120.58 (14)	C20—C21—C22	111.00 (14)
C6—C7—H1C7	119.71	C20—C21—H1C21	109.47

C8—C7—H1C7	119.71	C20—C21—H2C21	109.47
C3—C8—C7	120.19 (11)	C22—C21—H1C21	109.47
C3—C8—H1C8	119.9	C22—C21—H2C21	109.47
C7—C8—H1C8	119.91	H1C21—C21—H2C21	107.9
O9—C10—C11	119.16 (11)	C21—C22—C23	111.33 (13)
O9—C10—C15	119.28 (12)	C21—C22—H1C22	109.47
C11—C10—C15	121.45 (14)	C21—C22—H2C22	109.47
C10—C11—C12	118.75 (12)	C23—C22—H1C22	109.47
C10—C11—H1C11	120.62	C23—C22—H2C22	109.47
C12—C11—H1C11	120.63	H1C22—C22—H2C22	107.54
C11—C12—C13	120.49 (14)	C18—C23—C22	111.04 (11)
C11—C12—H1C12	119.76	C18—C23—H1C23	109.47
C13—C12—H1C12	119.76	C18—C23—H2C23	109.47
C12—C13—C14	119.82 (16)	C22—C23—H1C23	109.47
C12—C13—H1C13	120.09	C22—C23—H2C23	109.47
C14—C13—H1C13	120.09	H1C23—C23—H2C23	107.85

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*1···O16^{i}	0.839 (17)	2.351 (17)	3.1673 (14)	164.2 (16)
N17—H1*N*17···O16^{ii}	0.871 (17)	2.140 (17)	2.9977 (14)	168.2 (13)

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

Selected geometric parameters (Å, °) for (I), (II) and (III)

Parameter	(I)	(II)	(III)
P1—N1	1.6398 (16)	1.6375 (12)	1.6243 (11)
P1—O9	1.6106 (13)	1.5991 (10)	1.6108 (9)
P1—O16	1.4683 (12)	1.4762 (10)	1.4804 (8)
P1—N17	1.6297 (14)	1.6184 (13)	1.6267 (12)
N1—C2	1.391 (2)	1.4208 (18)	1.4667 (14)
O9—C10	1.388 (2)	1.4027 (16)	1.3937 (17)
N17—C18	1.479 (2)	1.4672 (18)	1.4702 (19)
N1—P1—O9	94.80 (7)	101.30 (5)	108.53 (5)
N1—P1—O16	116.36 (7)	114.69 (6)	111.38 (5)
N1—P1—N17	109.59 (8)	108.13 (6)	107.19 (5)
O9—P1—O16	114.31 (7)	112.59 (5)	112.00 (5)
O9—P1—N17	108.78 (7)	106.79 (6)	96.58 (5)
O16—P1—N17	111.71 (7)	112.50 (6)	119.90 (6)
P1—N1—C2	126.67 (12)	126.96 (10)	123.30 (9)
O9—C10—C11	118.11 (17)	118.57 (12)	119.16 (11)
P1—N17—C18	122.39 (11)	124.40 (9)	123.61 (9)