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Synthesis and structures of substituted triphenyl(phenylimino)phosphoranes

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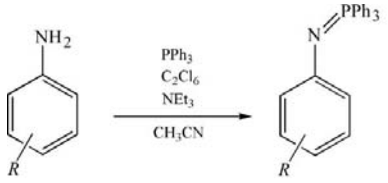
Three substituted triphenyl(phenylimino)phosphoranes, namely (4-cyanophenylimino)triphenylphosphorane, $C_{25}H_{19}N_2P$, (I), (4-nitrophenylimino)triphenylphosphorane, $C_{24}H_{19}N_2O_2P$, (II), and (3-nitrophenylimino)triphenylphosphorane, $C_{24}H_{19}N_2O_2P$, (III), were synthesized as precursors for the preparation of substituted diphenylcarbodiimides. All three compounds display a supramolecular arrangement in which the substituted benzene rings are organized in an antiparallel fashion. The nitro group on the ring participates in C—H$\cdots$O and O$\cdots\pi$ interactions, forming intermolecular dimers. Compound (III) shows disorder which involves the rotation of one of the phenyl rings of the triphenylphosphine group.

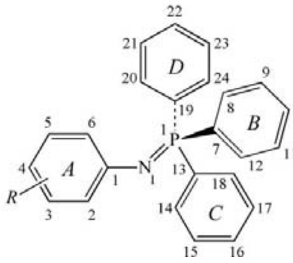
Comment

There is considerable current interest in organic semiconductors mainly because of the convenient and easy way in which their molecular structures, and hence their properties, can be tuned to a specific application. This is in stark contrast to the present-day CMOS (complementary metal oxide semiconductor) technology where such tuning is almost impossible. Switching from polymeric to oligomeric organic materials is a worthwhile undertaking because of the numerous opto-electronic applications for which these new compounds are used, as has been discussed in depth by Müllen & Wegner (1998) and Segura & Martin (2000). The most important reason for making this transition is the fact that it is far easier to produce, purify and structurally characterize oligomers than polymers, as they are monodisperse materials. For these reasons, oligomers have been extensively used as model compounds for polymers but it is far more rewarding to study the properties of these oligomeric organic semiconductor materials for their own sake.

The solid-state structures and electronic properties of one particular class of these oligomeric organic semiconductors, *viz.* distyrylbenzenes, oligomeric derivatives of PPV [poly(*p*-phenylene vinylene)], have already been extensively studied and the influence of substituents on the properties of the carbon backbone have been investigated (Baeke *et al.*, 2007; Vande Velde *et al.*, 2004; Vande Velde, Baeke *et al.* 2005; Vande Velde, De Borger *et al.*, 2005; Vande Velde, Geise *et al.*, 2005; Vande Velde *et al.*, 2006; Irngartinger *et al.*, 1994; Hakansson *et al.*, 1992; Bartholomew *et al.*, 2000*a*,*b*; Coates *et al.*, 1998; Nohra *et al.*, 2006; Renak *et al.*, 1999; Sancho-Garcia *et al.*, 2005; Zeller *et al.*, 2005). However, keeping in mind the tunability of these materials, it is equally interesting to determine the effects of the replacement of the ethenylic —CH═CH— link by other spacers such as —CH═N— (benzylideneanilines), —N═S═N— (sulfodiimides) or —N═C═N— (carbodiimides) fragments.



(I) R = 4-CN
(II) R = 4-NO$_2$
(III) R = 3-NO$_2$



The work we present here deals with the precursors to carbodiimide derivatives of distyrylbenzenes, *i.e.* triphenyl(phenylimino)phosphoranes. Because of the recent interest in carbodiimides as precursors for nitrogen-containing heterocycles, the synthesis of these compounds has received considerable attention (Ding *et al.*, 2000, 2004; Liu *et al.*, 2006; Okawa *et al.*, 1996; Yuan *et al.*, 2006; Zhao *et al.*, 2006; Wamhoff *et al.*, 1993; Eguchi, 2005). The most important pathway to obtain carbodiimides is through the aza-Wittig reaction of iminophosphoranes with isocyanates (Ding *et al.*, 2004, 2005; Liu *et al.*, 2006; Zhao *et al.*, 2006), since this reaction proceeds under mild conditions, requires only readily available precursors and does not involve the use of difficult-to-handle compounds such as phosgene (Kurzer & Douraghi, 1967; Mikolajczyk & Kielbasinski, 1981; Ulrich & Sayigh, 1966). The required iminophosphoranes can be obtained by a Staudinger reaction from azide derivatives (Ding *et al.*, 2000; Okawa *et al.*, 1996; Kurzer & Douraghi, 1967) or, more conveniently, by the direct reaction of an aromatic amine with triphenylphosphine (Ding *et al.*, 2004, 2005; Liu *et al.*, 2006; Okawa *et al.*, 1996; Yuan *et al.*, 2006; Zhao *et al.*, 2006), as depicted in the scheme above. In this work, we report the molecular and crystal structures of the three precursor iminophosphoranes (I), (II), and (III), which were obtained using the latter reaction.

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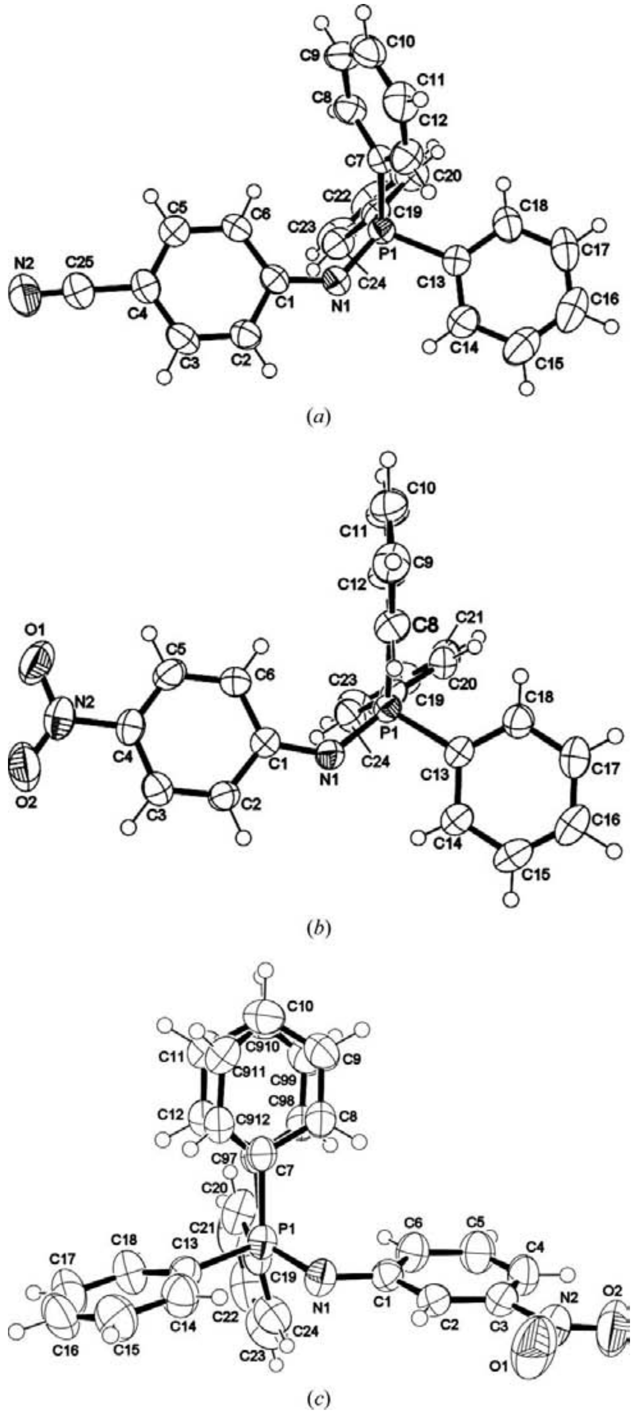

Figure 1
The molecular structures of (I), (II) and (III), the last showing the disorder in ring *B*, C7—C8—C9—C10—C11—C12 being the major conformer. Displacement ellipsoids are drawn at the 50% probability level.

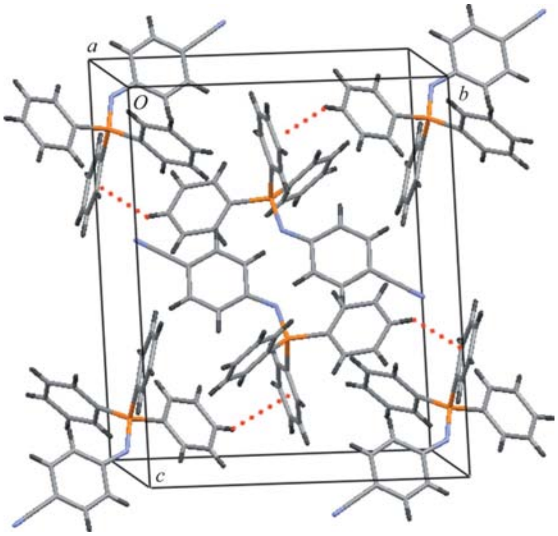

Figure 2
Packing and the C—H···π contacts of (I).

Table 1 presents selected geometric parameters of the three triphenyl(phenylimino)phosphoranes under investigation; the numbering of the atoms can be found in the scheme. The molecular geometries are similar for the three compounds. As can be seen from the values of the C6—C1—N1—P1 torsion angle, ring *A* is almost coplanar with the N=P bond, even though for the 3-nitro derivative (III) the deviation is somewhat larger. The values of the C1—N1—P1—C13 torsion angle indicate that ring *C* of the triphenylphosphine group is only slightly twisted compared to the plane formed by the P=N bond and ring *A*, whereas the torsion angles of rings *B* and *D* vary between approximately 40 and 85°. In comparison with the two other angles between the P=N bond and the rings of the triphenylphosphine group, the N1—P1—C13 angle is much smaller due to the intramolecular C14—H14···N1 interaction, of which the H···N contact distances and C—H···N angles are 2.51 Å/108°, 2.51 (2) Å/110° and 2.67 Å/105° for compounds (I), (II) and (III), respectively. This smaller N1—P1—C13 angle can also be found in the structures of related triphenyl(phenylimino)phosphoranes such as the parent triphenyl(phenylimino)phosphorane [refcode GEHRIU (Bohm *et al.*, 1988) in the Cambridge Structural Database (CSD; Allen, 2002)], (2-aminophenylimino)triphenylphosphorane (refcode JOZYIG; Llamas-Saiz *et al.*, 1992), [2-(*N*,*N*-dimethylamino)phenylimino]triphenylphosphorane (refcode HATXOP; Llamas-Saiz & Foces-Foces, 1994) and (4-bromophenylimino)triphenylphosphorane (refcode BPITPP; Hewlins, 1971).

The crystal structure of (4-cyanophenylimino)triphenylphosphorane, (I) (Figs. 1 and 2), is composed of alternating layers of cyanophenyl rings and triphenylphosphine groups. Within the cyanophenyl layer, the fragments are oriented in an antiparallel fashion. Successive molecules are linked in the *b*-axis direction in the triphenylphosphine layer by C16—H16···CgB^{i} contacts [2.83 Å/129°; symmetry code: (i) $1 - x$, $\frac{1}{2} + y, \frac{1}{2} - z$] (represented by the dotted lines in Fig. 2), in which *CgB* is the centroid of ring *B*. These two layers combine into two-dimensional layers, which are stacked on top of each other to give the three-dimensional structure. Successive stacked layers are connected by C21—H21···CgA^{ii} contacts [2.72 Å/159°; symmetry code: (ii) $1 + x, y, z$] (not shown in Fig. 2).

The packing of (4-nitrophenylimino)triphenylphosphorane, (II), shown in Fig. 3(*a*), is based on dimers with the nitro-

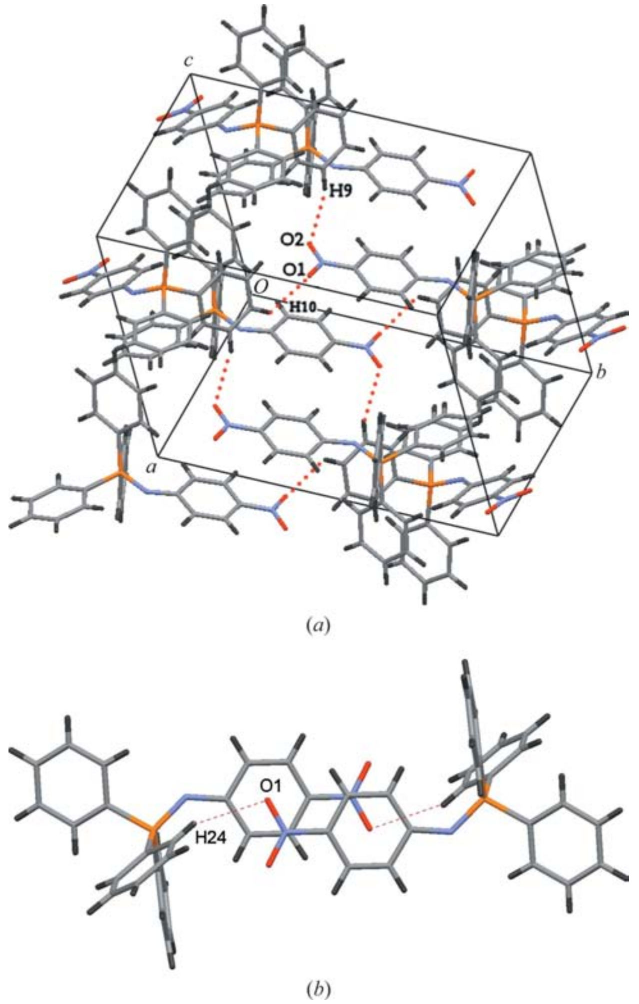

Figure 3
(*a*) Graphical representation of the packing and network of C—H···O contacts connecting the dimers of (II), and (*b*) a view of the C—H···O contact generating the dimers. The molecule on the left is related to that on the right by the symmetry code ($-x + 2, -y + 1, -z + 1$).

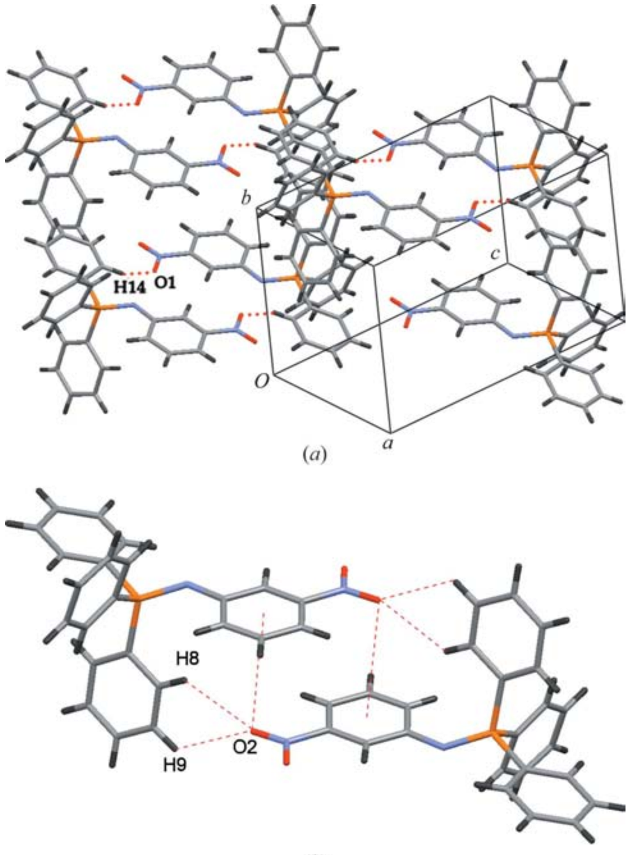

Figure 4
(*a*) Graphical representation of the packing and network of C—H···O contacts connecting the dimers of (III) (only the major conformer is shown), and (*b*) a view of the C—H···O and O···*Cg* contacts generating the dimers. The molecule on the right is related to that on the left by the symmetry code ($-x + 1, -y + 1, -z + 1$).

phenyl rings in an antiparallel cofacial arrangement, one of which is shown in Fig. 3(*b*). These dimers are due to intermolecular N2—O1···*CgA*$^{\rm iii}$ [3.473 (2) Å/84.81 (11)°; symmetry code: (iii) $2 - x, 1 - y, 1 - z$] (not shown in Fig. 3*b*) and C24—H24···O1$^{\rm iii}$ [2.68 (3) Å/145 (2)°] contacts (represented by the dotted lines in Fig. 3*b*). The former are possible most likely because of the fact that ring *A* is electron-deficient due to the presence of the electron-withdrawing nitro group. The dimers are then linked into a three-dimensional structure through C22—H22···N1$^{\rm iv}$ contacts [2.67 (2) Å/152.3 (6)°; symmetry code: (iv) $1 + x, \frac{3}{2} - y, \frac{1}{2} + z$] (not shown in Fig. 3*b*) and a network of C—H···O contacts consisting of C9—H9···O2$^{\rm i}$ [2.59 (3) Å/139.0 (16)°] and C10—H10···O1$^{\rm v}$ contacts [2.701 (19) Å/132.7 (15)°; symmetry code: (v) $2 - x, \frac{1}{2} + y, \frac{1}{2} - z$] (represented by the dotted lines in Fig. 3*a*). The latter generate extended chains in the *a*-axis direction, in which nitro groups alternate with rings *B* of the triphenylphosphine fragment, which contain H9 and H10.

The packing of (3-nitrophenylimino)triphenylphosphorane, (III), which is presented in Fig. 4(*a*), is similar to that of compound (II). Two molecules are involved in a dimer *via* three separate intermolecular interactions, which are represented by the dotted lines in Fig. 4(*b*). The first is the N2—O2···*CgA*$^{\rm vi}$ contact [3.798 (4) Å/92.77 (17)°; symmetry code: (vi) $1 - x, 1 - y, 1 - z$] which is possible again because of the lower electron density in ring *A* due to the presence of the nitro group. The other two are C—H···O contacts, *i.e.* C8—H8···O2$^{\rm vi}$ (2.63 Å/122°) and C9—H9···O2$^{\rm vi}$ (2.62 Å/123°). These dimers are then connected to adjacent molecules by a C—H···O interaction involving nitro atom O1, which was unused in the dimer contacts, *i.e.* C14—H14···O1$^{\rm vii}$ [2.70 Å/128°; symmetry code: (vii) $1 - x, 2 - y, 1 - z$] (represented by the dotted lines in Fig. 3*a*). The result is a supramolecular structure which displays alternating layers of nitrophenyl and triphenylphosphine groups.

The fact that the crystal structures of (II) and (III) are based on intermolecular dimers sets them apart from (I). These dimers owe their existence mainly to the presence of the nitro groups: they generate one [for (II)] or two [for (III)] C—H···O interactions and, because of the reduced electron density in the rings *A* of (II) and (III), as a consequence of the electron-withdrawing properties of the nitro group, one O···*Cg* interaction. Thus, the nitro group in the 3-position in (III) generates one more intermolecular contact within the dimer than the nitro group in the 4-position in (II) does. The supramolecular structure of (III), on the other hand, can count on just one intermolecular C—H···O contact, while for (II), one C—H···N and two additional C—H···O interactions become available from the nitro group in the 4-position. For (I), the nitrile functionalities are not involved in the supramolecular structure and the whole is based on weaker C—H···π interactions. Since the previously mentioned related triphenyl(phenylimino)phosphoranes all have electron-donating substituents, the typical dimers found in compounds (II) and (III) are absent due to the resulting higher electron density in ring *A*. Their packing is mainly based on C—H···π contacts as in compound (I), although π–π interactions can be found in HATXOP (Llamas-Saiz & Foces-Foces, 1994) and JOZYIG (Llamas-Saiz *et al.*, 1992), and the structure of HATXOP displays C—H···N interactions.

Experimental

All reagents and solvents were obtained from ACROS and used as received. The synthetic pathway for the preparation of compounds (I), (II) and (III) is shown in the scheme: the appropriately substituted aniline was stirred at room temperature with the triphenylphosphine/hexachloroethane/triethylamine system in acetonitrile and the resulting precipitate was recrystallized from ethanol to obtain large crystals of the desired products. The melting points are uncorrected. For (I), triethylamine (18.2 g, 0.18 mol) was added slowly to a solution of 4-cyanoaniline (7.1 g, 0.06 mol), triphenylphosphine (23.6 g, 0.09 mol) and hexachloroethane (21.3 g, 0.09 mol) in dry acetonitrile (200 ml). The mixture was stirred at room temperature overnight and then cooled in a refrigerator. The precipitated yellow solid was filtered off and recrystallized from ethanol. The yield was 15.4 g (68%); m.p. 461 K. For (II), triethylamine (18.2 g, 0.18 mol) was added slowly to a solution of 4-nitroaniline (8.3 g, 0.06 mol), triphenylphosphine (23.6 g, 0.09 mol) and hexachloroethane (21.3 g, 0.09 mol) in dry acetonitrile (200 ml). The mixture was stirred at room temperature overnight and then cooled in a refrigerator. The precipitated yellow solid was filtered off and recrystallized from ethanol. The yield was 16.4 g (69%); m.p. 428 K. For (III), triethylamine (18.2 g, 0.18 mol) was added slowly to a solution of 3-nitroaniline (8.3 g, 0.06 mol), triphenylphosphine (23.6 g, 0.09 mol) and hexachloroethane (21.3 g, 0.09 mol) in dry acetonitrile (200 ml). The mixture was stirred at room temperature overnight and then cooled in a refrigerator. The precipitate was filtered off but proved to be mostly triethylammonium chloride. The filtrate was partially evaporated, during which an orange precipitate formed. This new precipitate was filtered off and recrystallized from ethanol. The yield was 12.3 g (52%); m.p. 407 K. X-ray quality crystals of compounds (I), (II) and (III) were grown by slow cooling of hot ethanol solutions and were cut to the appropriate dimensions using a razor blade.

Compound (I)

Crystal data

$C_{25}H_{19}N_2P$
$M_r = 378.39$
Monoclinic, $P2_1/c$
$a = 8.581$ (3) Å
$b = 13.8620$ (10) Å
$c = 17.170$ (4) Å
$\beta = 104.53$ (2)°

$V = 1977.1$ (9) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.15$ mm^{-1}
$T = 293$ K
0.4 × 0.2 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
7279 measured reflections
3481 independent reflections
2198 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.041$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.095$
$S = 1.01$
3481 reflections

253 parameters
H-atom parameters constrained
$\Delta\rho_{\rm max} = 0.20$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.23$ e Å^{-3}

Compound (II)

Crystal data

$C_{24}H_{19}N_2O_2P$
$M_r = 398.38$
Monoclinic, $P2_1/c$
$a = 9.118$ (2) Å
$b = 17.536$ (4) Å
$c = 14.245$ (4) Å
$\beta = 117.591$ (17)°

$V = 2018.7$ (9) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.16$ mm^{-1}
$T = 293$ K
0.4 × 0.2 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
7417 measured reflections
3544 independent reflections
2682 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.029$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.089$
$S = 1.02$
3544 reflections
338 parameters

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\rm max} = 0.21$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.23$ e Å^{-3}

Compound (III)

Crystal data

$C_{24}H_{19}N_2O_2P$
$M_r = 398.38$
Triclinic, $P\overline{1}$
$a = 9.001$ (2) Å
$b = 9.770$ (7) Å
$c = 12.098$ (5) Å
$\alpha = 84.340$ (4)°
$\beta = 78.190$ (4)°

$\gamma = 78.410$ (3)°
$V = 1018.3$ (9) Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.16$ mm^{-1}
$T = 293$ K
0.4 × 0.4 × 0.4 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
7180 measured reflections
3590 independent reflections
3018 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.016$
3 standard reflections
frequency: 60 min
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.110$
$S = 1.04$
3590 reflections
281 parameters

13 restraints
H-atom parameters constrained
$\Delta\rho_{\rm max} = 0.21$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.24$ e Å^{-3}

When possible, H atoms were located in a difference map and left free to refine, resulting in C—H distances between 0.90 (2) and

Table 1
Selected geometric parameters (Å, °) for (I), (II) and the major disorder component of (III).

	(I)	(II)	(III)
C1—N1	1.372 (3)	1.365 (2)	1.386 (2)
P1—N1	1.5729 (18)	1.5752 (15)	1.5622 (16)
P1—C7	1.808 (2)	1.8107 (17)	1.816 (3)
P1—C13	1.803 (2)	1.8015 (17)	1.799 (2)
P1—C19	1.803 (2)	1.8051 (17)	1.8071 (19)
C1—N1—P1	131.35 (15)	131.16 (12)	128.93 (13)
N1—P1—C7	113.44 (10)	117.17 (8)	112.56 (18)
N1—P1—C13	106.12 (10)	104.86 (9)	107.72 (8)
N1—P1—C19	116.93 (10)	113.77 (8)	116.08 (9)
C6—C1—N1—P1	1.1 (3)	0.2 (3)	14.7 (3)
C1—N1—P1—C7	78.4 (2)	43.30 (19)	−71.1 (2)
C1—N1—P1—C13	165.70 (19)	161.58 (16)	172.32 (15)
C1—N1—P1—C19	48.5 (2)	82.24 (17)	54.46 (19)

0.99 (2) Å; $U_{\rm iso}$(H) values were constrained to $1.5U_{\rm eq}$(C). When not, they were placed in calculated positions and refined as riding with C—H distances of 0.93 Å and with $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C). Ring *B* of the triphenylphosphine group in compound (III) was disordered. The displacement parameters of the atoms in the minor conformer were restrained to be the same as for the corresponding atoms in the major conformer. Furthermore, the P1—C7 and P1—C97 distances were restrained to be equal within a standard uncertainty of 0.02 Å. The disorder involves a rotation of the ring of approximately 40° around the axis through C7···C10, which atoms are not drastically displaced by the disorder, as shown in Fig. 1. Site-occupancy factors are 0.791 (3) and 0.209 (3) for the major and minor conformers, respectively. The major form is obviously energetically more favourable, since the C8—H8···O2$^{\rm vi}$ and C9—H9···O2$^{\rm vi}$ contacts [symmetry code: (vi) $x - 1, y - 1, z - 1$] seen in the major conformer are absent in the minor conformer.

For all compounds, data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1989); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 2008) for (I) and (II); *SHELXS97* (Sheldrick, 2008) for (III). For all compounds, program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: UK3009). Services for accessing these data are described at the back of the journal.

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