

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

(Di-*tert*-butylphosphanyl)bis(diphenylphosphanyl)phosphaneAleksandra Wisniewska,^a Katarzyna Baranowska,^{a*}
Eberhard Matern^b and Jerzy Pikies^a^aDepartment of Inorganic Chemistry, Gdańsk University of Technology, 11/12 G. Narutowicz St. 80952-PL Gdańsk, Poland, and ^bInstitute for Inorganic Chemistry, University of Karlsruhe, 15 G. Engesserstrasse St., 76131 Karlsruhe, Germany
Correspondence e-mail: kasiab29@wp.pl

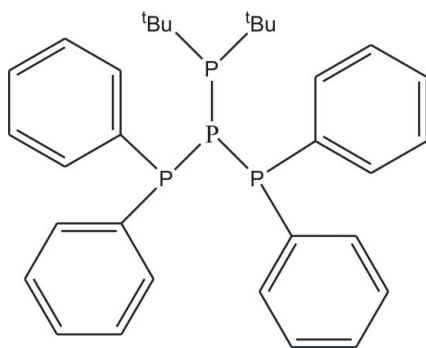
Received 20 June 2008; accepted 24 June 2008

Key indicators: single-crystal X-ray study; $T = 120$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.038; wR factor = 0.106; data-to-parameter ratio = 16.5.

The title phosphane, $\text{C}_{32}\text{H}_{38}\text{P}_4$ or $(\text{Ph}_2\text{P})_2\text{P}(\text{P}^t\text{Bu}_2)$, has a P atom that is linked to another three P atoms in a pyramidal configuration; the P—P distances in the range 2.2231 (7)–2.2446 (7) Å indicate that the P—P bonds are single bonds.

Related literature

For the synthesis of silylated triphosphanes, see: Kovacs *et al.* (1996). For other similar pyramidal isotetraphosphanes, see: Cowley *et al.* (1997); Fritz *et al.* (1987); Jones *et al.* (2002). For planar $(^t\text{Bu}_2\text{P})_3\text{P}$, see: Fritz *et al.* (1999). For evaluation of NMR data, see: Bruker (1999); Hägele *et al.* (1987).



Experimental

Crystal data

| | |
|--|-----------------------------------|
| $\text{C}_{32}\text{H}_{38}\text{P}_4$ | $\gamma = 90.900$ (5)° |
| $M_r = 546.5$ | $V = 1473.79$ (15) Å ³ |
| Triclinic, $P\bar{1}$ | $Z = 2$ |
| $a = 10.0161$ (6) Å | Mo $K\alpha$ radiation |
| $b = 11.9258$ (7) Å | $\mu = 0.28$ mm ⁻¹ |
| $c = 12.9951$ (7) Å | $T = 120$ (2) K |
| $\alpha = 104.831$ (5)° | $0.32 \times 0.15 \times 0.13$ mm |
| $\beta = 100.201$ (5)° | |

Data collection

| | |
|--|--|
| Oxford Diffraction KM-4-CCD diffractometer | 5474 independent reflections |
| Absorption correction: none | 4356 reflections with $I > 2\sigma(I)$ |
| 10351 measured reflections | $R_{\text{int}} = 0.023$ |

Refinement

| | |
|---------------------------------|---|
| $R[F^2 > 2\sigma(F^2)] = 0.038$ | 331 parameters |
| $wR(F^2) = 0.106$ | H-atom parameters constrained |
| $S = 1.07$ | $\Delta\rho_{\text{max}} = 0.45$ e Å ⁻³ |
| 5474 reflections | $\Delta\rho_{\text{min}} = -0.30$ e Å ⁻³ |

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); 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).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2467).

References

- Bruker (1999). *WINNMR* and *WINDAISY*. Bruker Daltonik, Bremen, Germany.
- Cowley, A. H., Dennis, S. M., Kamepalli, S., Carrano, C. J. & Bond, M. R. (1997). *J. Organomet. Chem.* **529**, 75–77.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Fritz, G., Matern, E., Krautscheid, H., Ahlrichs, R., Olkowska, J. W. & Pikies, J. (1999). *Z. Anorg. Allg. Chem.* **625**, 1604–1607.
- Fritz, G., Stoll, K., Hoenle, W. & von Schnering, H. G. (1987). *Z. Anorg. Allg. Chem.* **544**, 127–136.
- Hägele, G., Engelhardt, M. & Boenigk, W. (1987). *Simulation und automatisierte Analyse von NMR-Spektren*. Weinheim:VCH.
- Jones, C., Junk, P. C. & Williams, T. C. (2002). *J. Chem. Soc. Dalton Trans.* pp. 2417–2418.
- Kovacs, I., Matern, E. & Fritz, G. (1996). *Z. Anorg. Allg. Chem.* **622**, 935–941.
- Oxford Diffraction (2006). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Abingdon, Oxfordshire, England.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supporting information

Acta Cryst. (2008). E64, o1364 [doi:10.1107/S1600536808019077]

(Di-*tert*-butylphosphanyl)bis(diphenylphosphanyl)phosphane

Aleksandra Wisniewska, Katarzyna Baranowska, Eberhard Matern and Jerzy Pikies

S1. Comment

The reaction of Ph_2PCl with $t\text{Bu}_2\text{P}(\text{SiMe}_3)\text{Li}$ 1.5 THF in toluene at $-30\text{ }^\circ\text{C}$ (Kovacs *et al.* 1996) yielded unexpectedly $(\text{Ph}_2\text{P})_2\text{P}(t\text{Bu}_2)$ (1) in the place of expected $\text{Ph}_2\text{P}(\text{SiMe}_3)\text{P}t\text{Bu}_2$.

The molecular structure of (1) is shown in Fig.1. The geometry around P3 atom in (1) is pyramidal, the sum of angles around P3 is 310.26 degrees. The geometry around P1 atom indicates more pyramidal character (the sum of angles is 297.49 degrees) than around P3. The geometry around P4 atom is more planar (the sum of angles is 320.58 degrees) than around P3. The P–P distances (2.2327 Å - mean value) clearly indicate a single bond character of these bonds. The tendency of phosphane to planarity is more visible for compounds with big groups attached to the central P atom. This assumption is strongly supported by the planar geometry around the central P atom in $(t\text{Bu}_2\text{P})_3\text{P}$. This planarity is accompanied by a significant shortening of the P–P distances (2.198 Å) (Fritz *et al.* 1987).

S2. Experimental

A solution of $t\text{Bu}_2\text{P}(\text{SiMe}_3)\text{Li}$ 1.5 THF (855 mg 3.42 mmol) in 20 ml toluene at $-30\text{ }^\circ\text{C}$ was dropped to a solution of Ph_2PCl (792 mg, 3.59 mmol) in 20 ml toluene. The resulting solution was stirred for 3 h at $-30\text{ }^\circ\text{C}$ and for 12 h at ambient temp. Then the solvent was removed under vacuum at 1 mTorr for 1 h, the residue dissolved in pentane (40 ml), filtered and concentrated to about 8 ml. After 6 days at $-35\text{ }^\circ\text{C}$ the solution yielded 688 mg of colourless crystals of $(\text{Ph}_2\text{P})_2\text{P}(t\text{Bu}_2)$ (1).

$^3\text{P}\{^1\text{H}\}$ NMR of $(\text{Ph}_2\text{P}1,2)_2\text{P}3(\text{P}4t\text{Bu}_2)$ (1) (Bruker Av400, C_6D_6 , 298 K, external standard 85% H_3PO_4) (δ p.p.m.) 37.9 dt, P4; -19.5 dd*, P1, P2; -62.3 dt*, P3. $^1\text{J}(\text{P}3\text{—P}4) = -410.7\text{ Hz}$, $^1\text{J}(\text{P}1\text{—P}3) = -253.3\text{ Hz}$, $^2\text{J}(\text{P}1\text{—P}4) = +33.9\text{ Hz}$ (*= multiplet of higher order).

Chemical shifts and coupling constants of (1) were optimized using Bruke software (Bruker 1999, Hägele *et al.* 1987).

S3. Refinement

All H atoms were refined as riding on C atoms with aromatic C—H = 0.95 Å, methyl C—H = 0.98 Å, and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for CH groups, $1.5U_{\text{eq}}(\text{C})$ for CH_3 groups.

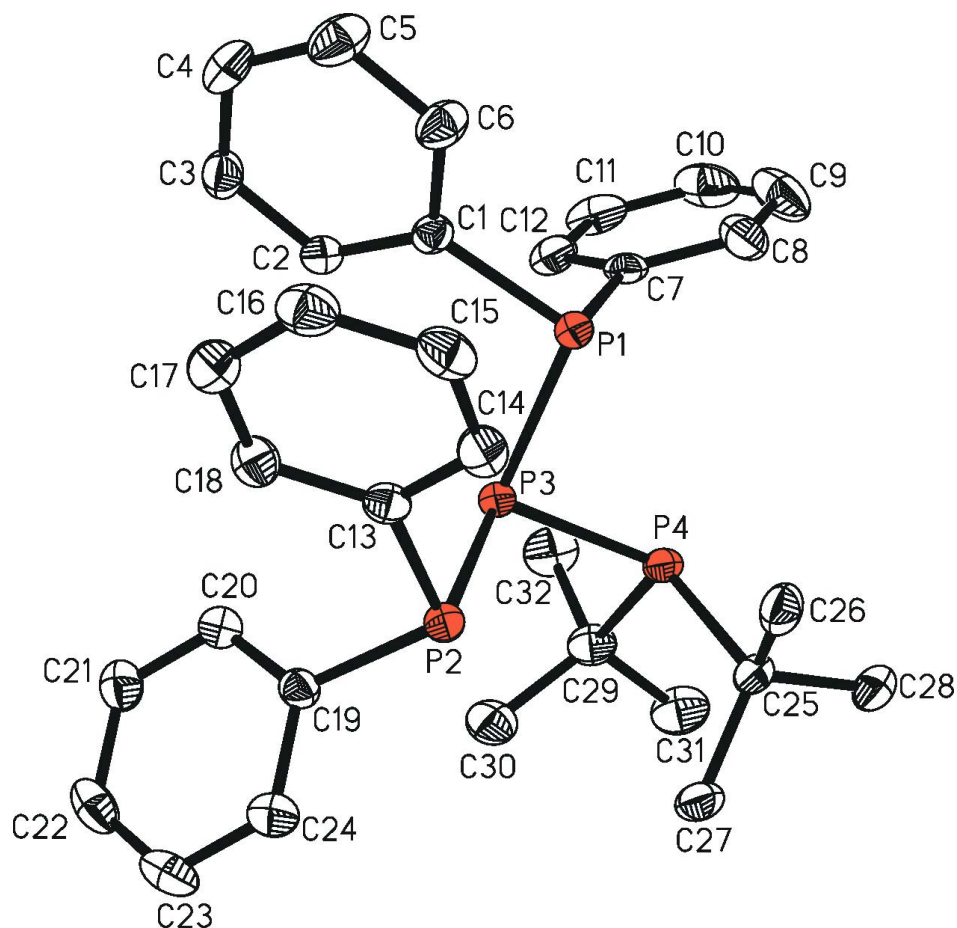


Figure 1

A view of the title molecule, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity.

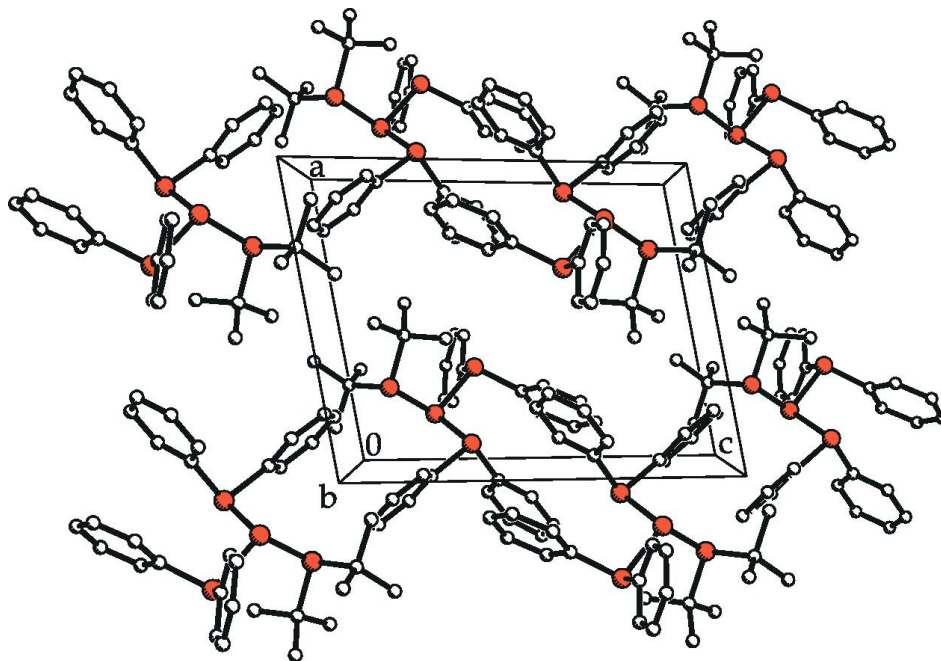


Figure 2

Crystal packing of the title compound, viewed approximately along the b axis.

(Di-*tert*--butylphosphanyl)bis(diphenylphosphanyl)phosphane

Crystal data

$C_{32}H_{38}P_4$

$M_r = 546.5$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 10.0161(6) \text{ \AA}$

$b = 11.9258(7) \text{ \AA}$

$c = 12.9951(7) \text{ \AA}$

$\alpha = 104.831(5)^\circ$

$\beta = 100.201(5)^\circ$

$\gamma = 90.900(5)^\circ$

$V = 1473.79(15) \text{ \AA}^3$

$Z = 2$

$F(000) = 580$

$D_x = 1.232 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 6775 reflections

$\theta = 2.4\text{--}32.4^\circ$

$\mu = 0.28 \text{ mm}^{-1}$

$T = 120 \text{ K}$

Prism, colourless

$0.32 \times 0.15 \times 0.13 \text{ mm}$

Data collection

Oxford Diffraction KM-4-CCD
diffractometer

Graphite monochromator

Detector resolution: $8.1883 \text{ pixels mm}^{-1}$

0.75° wide ω scans

10351 measured reflections

5474 independent reflections

4356 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.023$

$\theta_{\text{max}} = 25.5^\circ$, $\theta_{\text{min}} = 2.4^\circ$

$h = -11 \rightarrow 12$

$k = -14 \rightarrow 13$

$l = -15 \rightarrow 14$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.038$

$wR(F^2) = 0.106$

$S = 1.07$

5474 reflections

331 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier map
 Hydrogen site location: inferred from neighbouring sites
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0689P)^2]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.45 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.30 \text{ e } \text{\AA}^{-3}$

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|--------------|--------------|----------------------------------|
| C1 | -0.05525 (19) | 0.27686 (16) | 0.36738 (15) | 0.0197 (4) |
| C2 | -0.10603 (19) | 0.16151 (17) | 0.33015 (16) | 0.0223 (4) |
| H2 | -0.0706 | 0.11 | 0.2741 | 0.027* |
| C3 | -0.2075 (2) | 0.12062 (18) | 0.37356 (17) | 0.0267 (5) |
| H3 | -0.2412 | 0.0416 | 0.347 | 0.032* |
| C4 | -0.2601 (2) | 0.19420 (19) | 0.45549 (17) | 0.0299 (5) |
| H4 | -0.3292 | 0.1659 | 0.4857 | 0.036* |
| C5 | -0.2118 (2) | 0.3082 (2) | 0.49266 (18) | 0.0354 (5) |
| H5 | -0.2479 | 0.3592 | 0.5486 | 0.042* |
| C6 | -0.1106 (2) | 0.34949 (18) | 0.44923 (17) | 0.0288 (5) |
| H6 | -0.0783 | 0.4288 | 0.4757 | 0.035* |
| C7 | -0.03069 (19) | 0.37273 (17) | 0.19424 (16) | 0.0222 (4) |
| C8 | -0.0180 (2) | 0.48242 (19) | 0.17833 (18) | 0.0325 (5) |
| H8 | 0.0439 | 0.5402 | 0.2289 | 0.039* |
| C9 | -0.0951 (3) | 0.5082 (2) | 0.0890 (2) | 0.0446 (7) |
| H9 | -0.0871 | 0.5841 | 0.0792 | 0.054* |
| C10 | -0.1834 (3) | 0.4244 (2) | 0.0143 (2) | 0.0431 (6) |
| H10 | -0.2353 | 0.4423 | -0.0474 | 0.052* |
| C11 | -0.1966 (2) | 0.3146 (2) | 0.02890 (18) | 0.0368 (6) |
| H11 | -0.2575 | 0.2569 | -0.0227 | 0.044* |
| C12 | -0.1212 (2) | 0.28863 (19) | 0.11857 (17) | 0.0278 (5) |
| H12 | -0.1309 | 0.2131 | 0.1288 | 0.033* |
| C13 | 0.24915 (19) | 0.19694 (17) | 0.47598 (16) | 0.0214 (4) |
| C14 | 0.2700 (2) | 0.31269 (17) | 0.53800 (16) | 0.0258 (5) |
| H14 | 0.3222 | 0.3667 | 0.5163 | 0.031* |
| C15 | 0.2149 (2) | 0.34903 (18) | 0.63082 (18) | 0.0297 (5) |
| H15 | 0.2286 | 0.428 | 0.6719 | 0.036* |
| C16 | 0.1403 (2) | 0.27079 (19) | 0.66379 (17) | 0.0313 (5) |
| H16 | 0.1035 | 0.2958 | 0.7278 | 0.038* |
| C17 | 0.1193 (2) | 0.15651 (19) | 0.60370 (18) | 0.0313 (5) |



| | | | | |
|------|-------------|---------------|---------------|--------------|
| H17 | 0.0675 | 0.103 | 0.6263 | 0.038* |
| C18 | 0.1735 (2) | 0.11927 (18) | 0.51040 (17) | 0.0260 (5) |
| H18 | 0.1588 | 0.0402 | 0.4697 | 0.031* |
| C19 | 0.3207 (2) | -0.00176 (16) | 0.32067 (15) | 0.0206 (4) |
| C20 | 0.2027 (2) | -0.07357 (17) | 0.27282 (16) | 0.0253 (4) |
| H20 | 0.1186 | -0.0399 | 0.2577 | 0.03* |
| C21 | 0.2071 (2) | -0.19302 (18) | 0.24733 (17) | 0.0291 (5) |
| H21 | 0.126 | -0.2409 | 0.2153 | 0.035* |
| C22 | 0.3292 (2) | -0.24324 (19) | 0.26830 (17) | 0.0317 (5) |
| H22 | 0.3317 | -0.3255 | 0.251 | 0.038* |
| C23 | 0.4468 (2) | -0.17404 (19) | 0.31416 (18) | 0.0334 (5) |
| H23 | 0.5308 | -0.2084 | 0.3279 | 0.04* |
| C24 | 0.4427 (2) | -0.05407 (18) | 0.34036 (16) | 0.0269 (5) |
| H24 | 0.5242 | -0.0068 | 0.3722 | 0.032* |
| C25 | 0.4464 (2) | 0.34098 (17) | 0.19073 (17) | 0.0241 (4) |
| C26 | 0.4359 (2) | 0.42053 (18) | 0.30219 (18) | 0.0300 (5) |
| H26A | 0.5207 | 0.469 | 0.3328 | 0.045* |
| H26B | 0.4204 | 0.3729 | 0.3508 | 0.045* |
| H26C | 0.36 | 0.4703 | 0.2941 | 0.045* |
| C27 | 0.5567 (2) | 0.25572 (19) | 0.20422 (18) | 0.0280 (5) |
| H27A | 0.5694 | 0.2103 | 0.1327 | 0.042* |
| H27B | 0.529 | 0.2032 | 0.2447 | 0.042* |
| H27C | 0.6423 | 0.299 | 0.2439 | 0.042* |
| C28 | 0.4878 (2) | 0.41895 (19) | 0.1227 (2) | 0.0334 (5) |
| H28A | 0.4212 | 0.4777 | 0.1184 | 0.05* |
| H28B | 0.4908 | 0.3714 | 0.0496 | 0.05* |
| H28C | 0.5778 | 0.4575 | 0.1567 | 0.05* |
| C29 | 0.2723 (2) | 0.14372 (18) | 0.00756 (16) | 0.0277 (5) |
| C30 | 0.3239 (2) | 0.03535 (18) | 0.04003 (18) | 0.0333 (5) |
| H30A | 0.267 | 0.014 | 0.087 | 0.05* |
| H30B | 0.4181 | 0.0515 | 0.0791 | 0.05* |
| H30C | 0.3196 | -0.0289 | -0.0251 | 0.05* |
| C31 | 0.3551 (2) | 0.1739 (2) | -0.07156 (18) | 0.0383 (6) |
| H31A | 0.3423 | 0.1103 | -0.138 | 0.058* |
| H31B | 0.4516 | 0.1854 | -0.0379 | 0.058* |
| H31C | 0.3242 | 0.2455 | -0.0893 | 0.058* |
| C32 | 0.1237 (2) | 0.1177 (2) | -0.05120 (19) | 0.0367 (5) |
| H32A | 0.1174 | 0.0502 | -0.1139 | 0.055* |
| H32B | 0.091 | 0.1852 | -0.0757 | 0.055* |
| H32C | 0.0678 | 0.1009 | -0.0015 | 0.055* |
| P1 | 0.07579 (5) | 0.34576 (4) | 0.31567 (4) | 0.01952 (13) |
| P2 | 0.33268 (5) | 0.15796 (4) | 0.35823 (4) | 0.02017 (14) |
| P3 | 0.17163 (5) | 0.19161 (4) | 0.22775 (4) | 0.01832 (13) |
| P4 | 0.26732 (5) | 0.27752 (4) | 0.12351 (4) | 0.02045 (14) |



Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C1 | 0.0156 (9) | 0.0236 (10) | 0.0209 (10) | 0.0015 (8) | 0.0024 (8) | 0.0079 (8) |
| C2 | 0.0207 (10) | 0.0228 (10) | 0.0249 (10) | 0.0040 (8) | 0.0074 (8) | 0.0069 (8) |
| C3 | 0.0237 (11) | 0.0252 (11) | 0.0326 (12) | -0.0018 (9) | 0.0030 (9) | 0.0117 (9) |
| C4 | 0.0218 (11) | 0.0421 (13) | 0.0278 (11) | -0.0058 (10) | 0.0057 (9) | 0.0126 (10) |
| C5 | 0.0308 (12) | 0.0417 (13) | 0.0296 (12) | -0.0043 (10) | 0.0124 (10) | -0.0021 (10) |
| C6 | 0.0249 (11) | 0.0296 (12) | 0.0279 (11) | -0.0054 (9) | 0.0081 (9) | -0.0011 (9) |
| C7 | 0.0197 (10) | 0.0263 (11) | 0.0252 (11) | 0.0094 (8) | 0.0118 (8) | 0.0093 (8) |
| C8 | 0.0415 (13) | 0.0272 (12) | 0.0341 (12) | 0.0122 (10) | 0.0153 (10) | 0.0115 (10) |
| C9 | 0.0699 (19) | 0.0379 (14) | 0.0377 (14) | 0.0269 (13) | 0.0232 (14) | 0.0204 (11) |
| C10 | 0.0430 (14) | 0.0630 (17) | 0.0331 (13) | 0.0277 (13) | 0.0118 (11) | 0.0257 (13) |
| C11 | 0.0233 (11) | 0.0606 (16) | 0.0294 (12) | 0.0093 (11) | 0.0081 (10) | 0.0145 (11) |
| C12 | 0.0197 (10) | 0.0381 (12) | 0.0294 (11) | 0.0035 (9) | 0.0071 (9) | 0.0140 (9) |
| C13 | 0.0172 (9) | 0.0241 (10) | 0.0224 (10) | 0.0036 (8) | 0.0008 (8) | 0.0068 (8) |
| C14 | 0.0235 (10) | 0.0244 (11) | 0.0268 (11) | -0.0003 (9) | -0.0014 (9) | 0.0062 (9) |
| C15 | 0.0304 (11) | 0.0241 (11) | 0.0299 (11) | 0.0056 (9) | 0.0012 (9) | 0.0013 (9) |
| C16 | 0.0343 (12) | 0.0359 (13) | 0.0251 (11) | 0.0107 (10) | 0.0097 (10) | 0.0072 (9) |
| C17 | 0.0331 (12) | 0.0313 (12) | 0.0337 (12) | 0.0045 (10) | 0.0128 (10) | 0.0115 (10) |
| C18 | 0.0285 (11) | 0.0234 (10) | 0.0255 (11) | 0.0018 (9) | 0.0046 (9) | 0.0057 (8) |
| C19 | 0.0219 (10) | 0.0215 (10) | 0.0206 (10) | 0.0048 (8) | 0.0062 (8) | 0.0080 (8) |
| C20 | 0.0234 (10) | 0.0248 (11) | 0.0294 (11) | 0.0037 (8) | 0.0063 (9) | 0.0087 (9) |
| C21 | 0.0339 (12) | 0.0244 (11) | 0.0293 (11) | -0.0002 (9) | 0.0068 (10) | 0.0072 (9) |
| C22 | 0.0490 (14) | 0.0218 (11) | 0.0259 (11) | 0.0116 (10) | 0.0095 (10) | 0.0070 (9) |
| C23 | 0.0366 (13) | 0.0332 (12) | 0.0315 (12) | 0.0180 (10) | 0.0062 (10) | 0.0100 (10) |
| C24 | 0.0245 (11) | 0.0298 (11) | 0.0260 (11) | 0.0054 (9) | 0.0039 (9) | 0.0070 (9) |
| C25 | 0.0176 (10) | 0.0260 (11) | 0.0313 (11) | 0.0001 (8) | 0.0060 (8) | 0.0112 (9) |
| C26 | 0.0274 (11) | 0.0248 (11) | 0.0360 (12) | -0.0062 (9) | 0.0071 (10) | 0.0046 (9) |
| C27 | 0.0171 (10) | 0.0351 (12) | 0.0346 (12) | 0.0035 (9) | 0.0054 (9) | 0.0138 (9) |
| C28 | 0.0248 (11) | 0.0353 (13) | 0.0473 (14) | 0.0004 (9) | 0.0126 (10) | 0.0196 (11) |
| C29 | 0.0271 (11) | 0.0330 (12) | 0.0227 (11) | 0.0047 (9) | 0.0073 (9) | 0.0048 (9) |
| C30 | 0.0362 (13) | 0.0275 (11) | 0.0338 (12) | 0.0067 (10) | 0.0133 (10) | -0.0010 (9) |
| C31 | 0.0382 (13) | 0.0515 (15) | 0.0263 (12) | 0.0066 (11) | 0.0125 (10) | 0.0074 (10) |
| C32 | 0.0332 (12) | 0.0413 (14) | 0.0287 (12) | -0.0010 (11) | 0.0000 (10) | 0.0009 (10) |
| P1 | 0.0169 (3) | 0.0182 (3) | 0.0239 (3) | 0.00124 (19) | 0.0052 (2) | 0.0054 (2) |
| P2 | 0.0165 (3) | 0.0201 (3) | 0.0244 (3) | 0.0010 (2) | 0.0029 (2) | 0.0074 (2) |
| P3 | 0.0153 (2) | 0.0183 (3) | 0.0219 (3) | 0.00182 (19) | 0.0037 (2) | 0.0060 (2) |
| P4 | 0.0165 (3) | 0.0232 (3) | 0.0239 (3) | 0.0039 (2) | 0.0057 (2) | 0.0088 (2) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|-------|-----------|---------|-----------|
| C1—C2 | 1.391 (3) | C20—C21 | 1.381 (3) |
| C1—C6 | 1.393 (3) | C20—H20 | 0.95 |
| C1—P1 | 1.847 (2) | C21—C22 | 1.385 (3) |
| C2—C3 | 1.385 (3) | C21—H21 | 0.95 |
| C2—H2 | 0.95 | C22—C23 | 1.376 (3) |
| C3—C4 | 1.384 (3) | C22—H22 | 0.95 |

| | | | |
|----------|-------------|--------------|-------------|
| C3—H3 | 0.95 | C23—C24 | 1.386 (3) |
| C4—C5 | 1.369 (3) | C23—H23 | 0.95 |
| C4—H4 | 0.95 | C24—H24 | 0.95 |
| C5—C6 | 1.385 (3) | C25—C27 | 1.530 (3) |
| C5—H5 | 0.95 | C25—C26 | 1.537 (3) |
| C6—H6 | 0.95 | C25—C28 | 1.535 (3) |
| C7—C8 | 1.383 (3) | C25—P4 | 1.902 (2) |
| C7—C12 | 1.395 (3) | C26—H26A | 0.98 |
| C7—P1 | 1.845 (2) | C26—H26B | 0.98 |
| C8—C9 | 1.382 (3) | C26—H26C | 0.98 |
| C8—H8 | 0.95 | C27—H27A | 0.98 |
| C9—C10 | 1.376 (4) | C27—H27B | 0.98 |
| C9—H9 | 0.95 | C27—H27C | 0.98 |
| C10—C11 | 1.378 (4) | C28—H28A | 0.98 |
| C10—H10 | 0.95 | C28—H28B | 0.98 |
| C11—C12 | 1.380 (3) | C28—H28C | 0.98 |
| C11—H11 | 0.95 | C29—C30 | 1.529 (3) |
| C12—H12 | 0.95 | C29—C31 | 1.535 (3) |
| C13—C18 | 1.394 (3) | C29—C32 | 1.535 (3) |
| C13—C14 | 1.400 (3) | C29—P4 | 1.902 (2) |
| C13—P2 | 1.833 (2) | C30—H30A | 0.98 |
| C14—C15 | 1.387 (3) | C30—H30B | 0.98 |
| C14—H14 | 0.95 | C30—H30C | 0.98 |
| C15—C16 | 1.382 (3) | C31—H31A | 0.98 |
| C15—H15 | 0.95 | C31—H31B | 0.98 |
| C16—C17 | 1.378 (3) | C31—H31C | 0.98 |
| C16—H16 | 0.95 | C32—H32A | 0.98 |
| C17—C18 | 1.388 (3) | C32—H32B | 0.98 |
| C17—H17 | 0.95 | C32—H32C | 0.98 |
| C18—H18 | 0.95 | P1—P3 | 2.2305 (7) |
| C19—C24 | 1.394 (3) | P2—P3 | 2.2446 (7) |
| C19—C20 | 1.397 (3) | P3—P4 | 2.2231 (7) |
| C19—P2 | 1.838 (2) | | |
| C2—C1—C6 | 117.64 (18) | C22—C23—C24 | 119.9 (2) |
| C2—C1—P1 | 126.30 (15) | C22—C23—H23 | 120 |
| C6—C1—P1 | 116.03 (15) | C24—C23—H23 | 120 |
| C3—C2—C1 | 120.96 (18) | C23—C24—C19 | 121.0 (2) |
| C3—C2—H2 | 119.5 | C23—C24—H24 | 119.5 |
| C1—C2—H2 | 119.5 | C19—C24—H24 | 119.5 |
| C2—C3—C4 | 120.36 (19) | C27—C25—C26 | 109.64 (17) |
| C2—C3—H3 | 119.8 | C27—C25—C28 | 108.98 (17) |
| C4—C3—H3 | 119.8 | C26—C25—C28 | 107.24 (17) |
| C5—C4—C3 | 119.4 (2) | C27—C25—P4 | 117.57 (14) |
| C5—C4—H4 | 120.3 | C26—C25—P4 | 106.54 (13) |
| C3—C4—H4 | 120.3 | C28—C25—P4 | 106.38 (14) |
| C4—C5—C6 | 120.4 (2) | C25—C26—H26A | 109.5 |
| C4—C5—H5 | 119.8 | C25—C26—H26B | 109.5 |

| | | | |
|-------------|-------------|---------------|-------------|
| C6—C5—H5 | 119.8 | H26A—C26—H26B | 109.5 |
| C5—C6—C1 | 121.2 (2) | C25—C26—H26C | 109.5 |
| C5—C6—H6 | 119.4 | H26A—C26—H26C | 109.5 |
| C1—C6—H6 | 119.4 | H26B—C26—H26C | 109.5 |
| C8—C7—C12 | 119.11 (19) | C25—C27—H27A | 109.5 |
| C8—C7—P1 | 117.40 (16) | C25—C27—H27B | 109.5 |
| C12—C7—P1 | 123.49 (15) | H27A—C27—H27B | 109.5 |
| C9—C8—C7 | 120.2 (2) | C25—C27—H27C | 109.5 |
| C9—C8—H8 | 119.9 | H27A—C27—H27C | 109.5 |
| C7—C8—H8 | 119.9 | H27B—C27—H27C | 109.5 |
| C10—C9—C8 | 120.3 (2) | C25—C28—H28A | 109.5 |
| C10—C9—H9 | 119.8 | C25—C28—H28B | 109.5 |
| C8—C9—H9 | 119.8 | H28A—C28—H28B | 109.5 |
| C9—C10—C11 | 120.1 (2) | C25—C28—H28C | 109.5 |
| C9—C10—H10 | 120 | H28A—C28—H28C | 109.5 |
| C11—C10—H10 | 120 | H28B—C28—H28C | 109.5 |
| C10—C11—C12 | 120.0 (2) | C30—C29—C31 | 110.04 (18) |
| C10—C11—H11 | 120 | C30—C29—C32 | 108.77 (18) |
| C12—C11—H11 | 120 | C31—C29—C32 | 107.76 (18) |
| C11—C12—C7 | 120.3 (2) | C30—C29—P4 | 115.98 (14) |
| C11—C12—H12 | 119.8 | C31—C29—P4 | 109.73 (15) |
| C7—C12—H12 | 119.8 | C32—C29—P4 | 104.12 (14) |
| C18—C13—C14 | 118.53 (19) | C29—C30—H30A | 109.5 |
| C18—C13—P2 | 124.94 (15) | C29—C30—H30B | 109.5 |
| C14—C13—P2 | 116.45 (15) | H30A—C30—H30B | 109.5 |
| C15—C14—C13 | 120.47 (19) | C29—C30—H30C | 109.5 |
| C15—C14—H14 | 119.8 | H30A—C30—H30C | 109.5 |
| C13—C14—H14 | 119.8 | H30B—C30—H30C | 109.5 |
| C16—C15—C14 | 120.2 (2) | C29—C31—H31A | 109.5 |
| C16—C15—H15 | 119.9 | C29—C31—H31B | 109.5 |
| C14—C15—H15 | 119.9 | H31A—C31—H31B | 109.5 |
| C17—C16—C15 | 120.0 (2) | C29—C31—H31C | 109.5 |
| C17—C16—H16 | 120 | H31A—C31—H31C | 109.5 |
| C15—C16—H16 | 120 | H31B—C31—H31C | 109.5 |
| C16—C17—C18 | 120.3 (2) | C29—C32—H32A | 109.5 |
| C16—C17—H17 | 119.8 | C29—C32—H32B | 109.5 |
| C18—C17—H17 | 119.8 | H32A—C32—H32B | 109.5 |
| C17—C18—C13 | 120.51 (19) | C29—C32—H32C | 109.5 |
| C17—C18—H18 | 119.7 | H32A—C32—H32C | 109.5 |
| C13—C18—H18 | 119.7 | H32B—C32—H32C | 109.5 |
| C24—C19—C20 | 118.16 (18) | C7—P1—C1 | 99.44 (9) |
| C24—C19—P2 | 115.67 (15) | C7—P1—P3 | 96.19 (6) |
| C20—C19—P2 | 126.16 (15) | C1—P1—P3 | 101.86 (6) |
| C21—C20—C19 | 120.70 (19) | C13—P2—C19 | 103.58 (9) |
| C21—C20—H20 | 119.7 | C13—P2—P3 | 101.37 (6) |
| C19—C20—H20 | 119.7 | C19—P2—P3 | 100.37 (6) |
| C20—C21—C22 | 120.2 (2) | P4—P3—P1 | 97.92 (3) |
| C20—C21—H21 | 119.9 | P4—P3—P2 | 109.51 (3) |

supporting information

| | | | |
|-------------|-------------|------------|------------|
| C22—C21—H21 | 119.9 | P1—P3—P2 | 102.93 (3) |
| C23—C22—C21 | 119.98 (19) | C25—P4—C29 | 110.55 (9) |
| C23—C22—H22 | 120 | C25—P4—P3 | 112.04 (7) |
| C21—C22—H22 | 120 | C29—P4—P3 | 97.98 (7) |
