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# Polymorphism in a secondary phosphine

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

Two crystal structures of bis(2,3,5,6-tetramethylphenyl)phosphine, $C_{20}H_{27}P$, are reported constituting the first recorded case of polymorphism in a secondary phosphine ($R_2PH$). The two structures differ in their conformation and, as a result, the steric hindrance experienced at the phosphorus centre is observed to be dependent on the packing environment. Each polymorph exhibits a distinct supramolecular structure; in polymorph **I** the molecules are arranged in columns in two directions, whereas polymorph **II** forms layers. There is a distinct lack of significant intermolecular interactions in either form, with the exception of some weak Me$\cdots\pi$ interactions observed in polymorph **II**. These interactions are likely the cause of the variation in the C—P—C angles observed between the two structures.

## 1. Introduction

Phosphines have become ubiquitous ligands for transition-metal centres due to the ease with which their electronic and steric properties may be tailored and to the many and varied applications of transition-metal phosphine complexes in catalysis. Secondary phosphines $R_2PH$ have the added advantage that the P—H proton may readily be removed to furnish anionic phosphanide $R_2P^-$ ligands (Izod, 2000). We have a long-standing interest in the application of such phosphanide ligands for the support of novel low-oxidation-state main group species; for example, the recently isolated fully phosphanyl-substituted ditetrelenes $\{(Mes)_2P\}_2E{=}{=}E\{P(Mes)_2\}_2$ ($E$ = Si or Ge; Mes = 2,4,6-$Me_3C_6H_2$) (Izod *et al.*, 2017*a*, 2022).

In the course of this work, we have striven to explore the impact of the steric profile and substitution pattern of the aromatic rings in diarylphosphanide ligands on the structures and stabilities of both low-oxidation-state main group compounds and their alkali metal precursors $R_2PM$ ($M$ = Li, Na or K) (Izod *et al.*, 2017*b*). While 2,6-disubstituted and 2,4,6-trisubstituted aromatic rings are common phosphine substituents, alternative substitution patterns, such as in the 2,3,5,6-tetramethylphenyl substituent described here, are rare.

Due to their reactivity, the structure determination of secondary phosphines using single-crystal X-ray crystallography can be challenging, with the first such structure being reported in 1987 (Bartlett *et al.*, 1987). As testament to this, at time of writing there are only 95 organic acyclic secondary phosphine structures in the Cambridge Structural Database (CSD, Version 5.45, update 2, June 2024; Groom *et al.*, 2016) and only one polymorph is reported for any one secondary phosphine compound, including cyclic phosphines and organometallic complexes.

In this work, we present the first known instance of polymorphism in a secondary phosphine. Bis(2,3,5,6-tetramethylphenyl)phosphine (Fig. 1) crystallizes in two distinct forms:

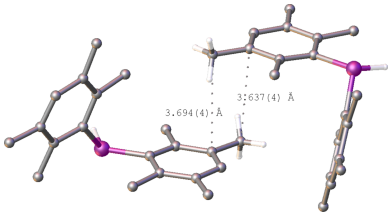

**Table 1**
Experimental details.

For both structures: $C_{20}H_{27}P$, $M_r$ = 298.38. Experiments were carried out at 150 K with Cu $K\alpha$ radiation using a Rigaku Xcalibur Gemini ultra diffractometer with an Atlas detector. The absorption correction was analytical [*CrysAlis PRO* (Rigaku OD, 2015), based on expressions derived by Clark & Reid (1995)]. H atoms were treated by a mixture of independent and constrained refinement.

| | Polymorph **I** | Polymorph **II** |
|---|---|---|
| Crystal data | | |
| Crystal system, space group | Monoclinic, $P2/n$ | Monoclinic, $P2_1/c$ |
| $a$, $b$, $c$ (Å) | 6.5476 (2), 5.9910 (2), 21.5676 (6) | 12.8874 (6), 8.8455 (3), 15.4635 (7) |
| $\beta$ (°) | 96.020 (2) | 104.108 (5) |
| $V$ (Å$^3$) | 841.36 (4) | 1709.61 (13) |
| $Z$ | 2 | 4 |
| $\mu$ (mm$^{-1}$) | 1.35 | 1.33 |
| Crystal size (mm) | 0.24 × 0.08 × 0.06 | 0.36 × 0.14 × 0.05 |
| Data collection | | |
| $T_{min}$, $T_{max}$ | 0.696, 0.866 | 0.746, 0.939 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 11409, 1500, 1299 | 12709, 3021, 2424 |
| $R_{int}$ | 0.040 | 0.039 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.596 | 0.596 |
| Refinement | | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.039, 0.118, 1.06 | 0.047, 0.135, 1.06 |
| No. of reflections | 1500 | 3021 |
| No. of parameters | 103 | 214 |
| No. of restraints | 0 | 182 |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.27, −0.24 | 0.43, −0.35 |

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

polymorph **I**, grown from tetrahydrofuran, which crystallizes in the monoclinic space group $P2/n$, and polymorph **II**, grown from fluorobenzene, which crystallizes in the monoclinic space group $P2_1/c$. As the first case of its kind, the structural analysis here should provide unique insights into the supramolecular chemistry of secondary phosphines.

## 2. Experimental

### 2.1. Preparation of bis(2,3,5,6-tetramethylphenyl)phosphine

All manipulations were performed under an inert atmosphere (argon gas) using standard Schlenk techniques unless otherwise stated. To a cold (−78 °C) solution of $PCl_3$ (2.9 ml, 23 mmol) in diethyl ether (50 ml) was added (2,3,5,6-$Me_4$-$C_6H$)MgBr (42 mmol) dissolved in tetrahydrofuran (THF, 200 ml). This mixture was allowed to warm to room temperature and was stirred for 12 h. To this solution was carefully added an excess of solid $LiAlH_4$ (1.0 g, 26.3 mmol) and the resulting mixture was stirred at room temperature for 2 h. Degassed water (50 ml) was carefully added and the organic phase was extracted into THF (3 × 30 ml). The combined organic extracts were dried over activated 4 Å molecular sieves, the solution was filtered and solvent was removed *in vacuo* from the filtrate to give bis(2,3,5,6-tetramethylphenyl)-phosphine as a colourless solid in 65% yield. Crystals suitable for single-crystal X-ray diffraction were grown from cold (3 °C) THF (polymorph **I**) or from cold (−30 °C) fluorobenzene (polymorph **II**).

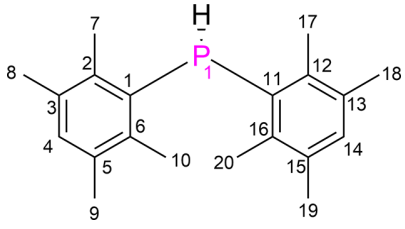


**Figure 1**
Bis(2,3,5,6-tetramethylphenyl)phosphine with the numbering scheme used in this article.

### 2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. H atoms bound to C atoms were positioned with idealized geometry. The displacement parameters of these H atoms were constrained using a riding model, with $U_{iso}$(H) values set to be an appropriate multiple of the $U_{eq}$ value of the parent atom.

The H atoms bound to phosphorus were located using peaks in the Fourier difference map. In both structures, the most prominent residual peaks about phosphorus after all other atoms were modelled were selected. In the case of polymorph **I**, the occupancy of this H atom was constrained to be 0.5 as it is disordered across a special position. For polymorph **II**, peaks corresponding to two proton positions with similar geometry were observed and hence the phosphine H atom was split across two positions with the occupancies refined to be approximately 63 and 37%. The displacement parameters of the phosphine H atoms in both structures were constrained using a riding model, with $U_{iso}$(H) values set to be 1.2$U_{eq}$ relative to the parent atom.

It is likely that the unrestrained P—H distances are shorter than the true bond lengths, but the direction of the bond

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

| | Polymorph **I** | Polymorph **II** |
|---|---|---|
| P1—C1 | 1.8471 (17) | 1.852 (2) |
| P1—C11 | | 1.856 (2) |
| C1—P1—C1/11 | 108.78 (11) | 105.74 (9) |
| C1—P1—H1 | 100 (2) | 109.1 (2) |
| C1—P1—H1$^{i}$/C11—P1—H1 | 99 (2) | 118.4 (2) |
| Aryl–aryl twist angle | 84.39 (10) | 94.12 (9) |

Symmetry code: (i) $-x+\frac{3}{2}, y, -z+\frac{3}{2}$.

vectors are likely to be accurate. Though some residual density remains, most prominently in the structure of polymorph **II**, there are no peaks greater than 0.5 e Å$^{-3}$ and they do not appear to be in positions that could correspond to atoms; the largest peak is altogether too close to the P atom (<1 Å) and/or in a position that would make little sense in terms of molecular geometry. It is possible that these residual peaks are the result of series termination errors (Fourier ripples).

## 3. Results and discussion

The two structures of bis(2,3,5,6-tetramethylphenyl)phosphine crystallize in different monoclinic space groups. Polymorph **I** crystallizes in the space group $P2/n$, with an asymmetric unit comprising half of the molecule ($Z'$ = 0.5). Here the P atom is located on the twofold rotation axis in the structure and the full molecule is generated through this symmetry operation. Polymorph **II** crystallizes in the space group $P2_1/c$, with one whole molecule in the asymmetric unit. In both structures, the proton on the P atom is disordered over two positions, as has been observed previously in similar bis(aryl) secondary phosphine structures (Izod *et al.*, 2017*b*; Clegg, 2017). Details of the refinements for both structures are presented in Table 1.

Though the bond distances do not differ significantly, the conformations of the molecules in the two polymorphs are somewhat different, as demonstrated by overlaying them (Fig. 2). The conformational variation can be attributed to differences in the geometry about the P atom and the angles between the planes of the aryl rings (Table 2). As polymorph **I** exhibits a wider C—P—C angle than polymorph **II**, this would

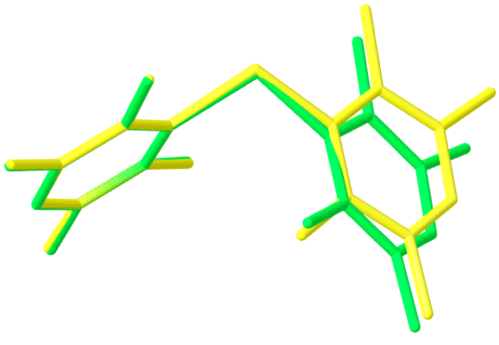

**Figure 2**
Overlay diagram of polymorphs **I** (yellow) and **II** (green).

**Table 3**
Intermolecular Me$\cdots\pi$ interactions (Å, °) in the structure of polymorph.

| $D$—H$\cdots A$ | $D\cdots A$ | H$\cdots A$ | $D$—H$\cdots A$ |
|---|---|---|---|
| C18—H18$A\cdots$C15$^{i}$ | 3.694 (4) | 2.83 (2) | 150 (1) |
| C19$^{i}$—H19$B^{i}\cdots$C15 | 3.637 (4) | 2.88 (2) | 136.6 (9) |

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

suggest that it experiences a greater degree of steric hindrance at the phosphorus centre (Rivard *et al.*, 2007).

The degree of steric pressure on the P atom in bis(aryl)phosphines can also be assessed by the sum of the angles about phosphorus, Σ°P (Boeré & Zhang, 2005). The values measured exceed 300°, with polymorph **I** exhibiting a Σ°P of 318 (2)° and the same sum being 333.2 (2)° for polymorph **II** (measured for the H atom of highest occupancy). This would seem to contradict the interpretation of the C—P—C bond angles as, according to the Σ°P, the P atom in polymorph **II** is under greater steric pressure in spite of its narrower C—P—C angle. Regardless of the trend in these measurements, that there should be such variation within the same molecule demonstrates the effect that polymorphism can potentially have on these compounds. These conformational perturbations are likely the result of the different packing environments and intermolecular interactions in the two solid-state structures.

As is common in the structures of secondary bis(aryl)phosphines, there are no significant contacts involving the H atom on the phosphorus in either structure (Izod *et al.*, 2017*b*; Bartlett *et al.*, 1987; Clegg, 2017; Rivard *et al.*, 2007; Fleming *et al.*, 2013; Ritch *et al.*, 2014). The lack of structure-directing interactions involving this atom may well be the root of the disorder of the P—H proton manifest in both polymorphs.

The packing in both structures seems to prioritize the minimization of steric interactions as opposed to forming strong structure-directing intermolecular bonds. As such, the packing is best described in terms of the alignment of the aryl rings. The molecules in polymorph **I** stack forming continuous columns along both the crystallographic [100] direction, with P$\cdots$P distances of *ca* 6.55 Å (Fig. 3), and along the [010] direction, with an equivalent distance of *ca* 5.99 Å (the lengths

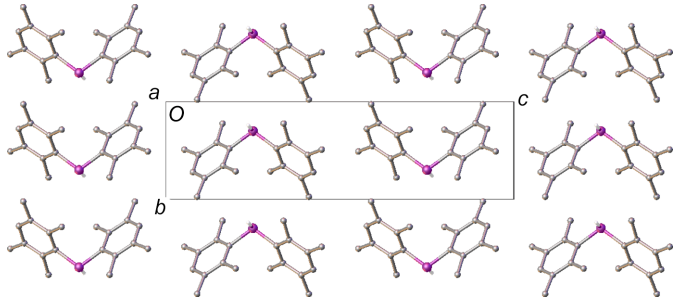


**Figure 3**
The structure of polymorph **I**, viewed along the [100] direction. Only one orientation of the H atoms bound to phosphorus is shown and the rest have been omitted for clarity.

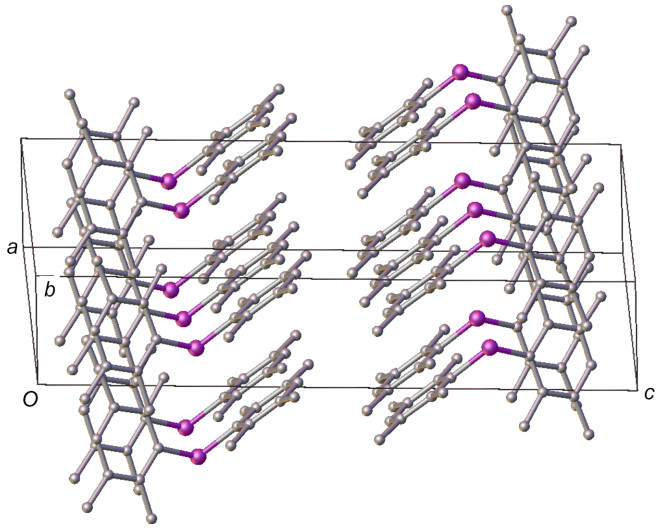


**Figure 4**
The structure of polymorph **I**, viewed approximately along the [$\bar{1}$10] direction, showing the direct alignment of the methyl groups hindering close approach of the molecules in this direction. H atoms have been omitted for clarity.

of the respective axes). The rings exhibit similar angles to their respective directions, *ca* 57° in [100] and *ca* 52° in [010].

The distance between the molecules along the columns appears to preclude direct $\pi$–$\pi$ interactions (Avashti *et al.*, 2014; Brunner *et al.*, 2014). In fact, there do not appear to be any salient intermolecular interactions observed in polymorph **I**. This suggests that the molecules are arranged in such a way as to minimize repulsive contacts rather than form attractive interactions. As a result, all the duryl rings in this structure are orientated coplanar to either the [110] or [$\bar{1}$10] directions. The orientation of the rings in these directions, with methyl groups directed towards each other in the same plane, further hinders the close approach of the molecules in the structure (Fig. 4).

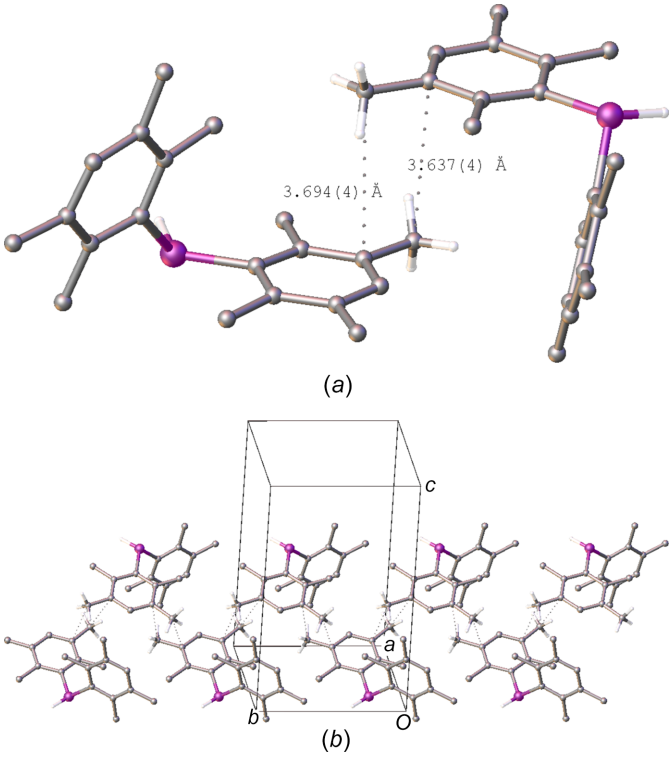


**Figure 5**
(*a*) A view of the ring motif formed in polymorph **II** of Me$\cdots\pi$ interactions between two molecules and (*b*) the continuous chain motif formed of these interactions in the [010] direction. Close contacts are depicted as dashed lines and H atoms, with the exception of those bound to phosphorus and the methyl groups involved in intermolecular interactions, have been omitted for clarity.

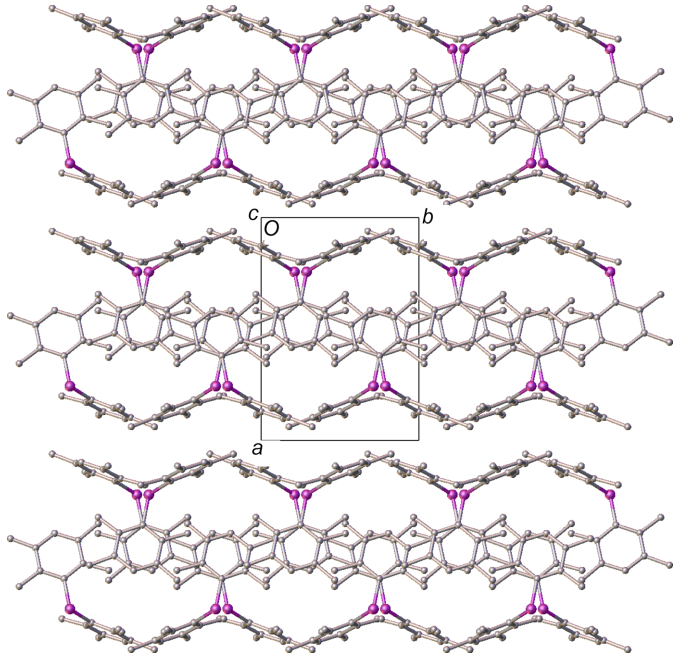


**Figure 6**
A view of the packing in polymorph **II**, showing the layers coplanar with the crystallographic (100) plane. H atoms have been omitted for clarity.

The packing in the structure of polymorph **II** is markedly different to that in polymorph **I** and much of this can be attributed to the fact that the two aryl rings are crystallographically independent in polymorph **II**. The molecules align along the [010] direction, but the angles of the rings to this direction are shallower than in polymorph **I**; *ca* 28° for one and 0° for the other, where, once again, the methyl groups prevent the close approach of the $\pi$ systems. Though the arrangement of the rings in the ($\bar{1}$02) plane, propagating along [010], is reminiscent of a similar arrangement in polymorph **I**, also in the [010] direction, in polymorph **II** the spacing between the molecules in this direction is over 2 Å longer, with a P$\cdots$P distance of *ca* 8.85 Å along [010], likely the result of the shallower P—C—P angle.

In contrast to the structure of polymorph **I**, there do appear to be some weak intermolecular interactions in the structure of polymorph **II** in the form of C—H$\cdots$C contacts between methyl groups and the aromatic rings. Two such contacts, with C$\cdots$C distances < 3.7 Å, are observed (Table 3), which can be classified as weak Me$\cdots\pi$ interactions (Brunner *et al.*, 2014). The two contacts form a ring motif between two of the duryl rings and propagate along the [010] direction, forming a chain

of intermolecular interactions, with each molecule related to the next by the symmetry of the $2_1$ screw axis (Fig. 5).

By way of comparison, a similar relationship between the molecules is observed in both the [100] and [010] directions in polymorph **I**; however, in this case, the C···C distances are at least 0.1 Å too long to be considered Me···$\pi$ interactions. Given this, it is possible that the weak but nonetheless attractive interactions observed in polymorph **II** are the root of the shallower C—P—C angle observed in this structure compared to that of polymorph **I**.

Although there are no discernible close contacts between the chains of molecules in polymorph **II**, they appear to pack to form 2D layers coplanar to [100] (Fig. 6). Again, there do not appear to be any significant structure-directing interactions between these layers and the closest centroid–centroid distances between pairs of duryl rings across the layer boundary are *ca* 4.2 Å. As a result, it can be inferred that these rings are orientated simply to minimize steric interactions.

## 4. Conclusion

Bis(2,3,5,6-tetramethylphenyl)phosphine is the first secondary phosphine known to exhibit polymorphism and is observed to form two different crystalline forms depending on the solvent of crystallization. As structures of these reactive compounds are somewhat rare in the literature, this study expands the sum of structural knowledge of secondary phosphines, as well as revealing aspects of their supramolecular chemistry.

The molecules in each crystal structure vary in terms of their conformation, with the degree of steric pressure on the P atom observed to vary depending on the packing environment. Though polymorph **I** crystallizes with columnar motifs in the [100] and [010] directions, and no significant structure-directing intermolecular interactions, polymorph **II** forms a 2D layered structure with weak Me···$\pi$ interactions, forming a chain motif along the [010] direction.

The study of polymorphism in a secondary phosphine raises some interesting points in terms of the solid-state structures of these molecules. It should be noted that as the same molecule exhibits drastically different values for $\Sigma^{\circ}$P, a measure of the steric pressure on the P atom, that this is not an intrinsic molecular property and can be affected by the packing environment. This demonstrates that caution should be exercised when drawing conclusions based on these values, especially in the context of solution-phase calculations.

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## Conflict of interest

The author declares no competing financial interests.

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# supporting information

*Acta Cryst.* (2025). C**81**, 109-113 [https://doi.org/10.1107/S2053229625000555]

## Polymorphism in a secondary phosphine

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### Computing details

#### Bis(2,3,5,6-tetramethylphenyl)phosphine (kji190001_fa)

*Crystal data*

$C_{20}H_{27}P$
$M_r = 298.38$
Monoclinic, $P2/n$
$a = 6.5476$ (2) Å
$b = 5.9910$ (2) Å
$c = 21.5676$ (6) Å
$\beta = 96.020$ (2)°
$V = 841.36$ (4) Å$^3$
$Z = 2$
$F(000) = 324$
$D_x = 1.178$ Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 4245 reflections
$\theta = 4.1$–66.4°
$\mu = 1.35$ mm$^{-1}$
$T = 150$ K
Block, colourless
0.24 × 0.08 × 0.06 mm

*Data collection*

Rigaku Xcalibur Gemini ultra diffractometer with an Atlas detector
Radiation source: fine-focus sealed X-ray tube, Enhance Ultra (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.3968 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: analytical [CrysAlis PRO (Rigaku OD, 2015), based on expressions derived by Clark & Reid (1995)]
$T_{min} = 0.696$, $T_{max} = 0.866$
11409 measured reflections
1500 independent reflections
1299 reflections with $I > 2\sigma(I)$
$R_{int} = 0.040$
$\theta_{max} = 66.9°$, $\theta_{min} = 4.1°$
$h = -7\rightarrow7$
$k = -7\rightarrow7$
$l = -25\rightarrow24$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.118$
$S = 1.06$
1500 reflections
103 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0665P)^2 + 0.3189P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.27$ e Å$^{-3}$
$\Delta\rho_{min} = -0.24$ 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.

**Refinement**. Single-crystal diffraction was carried out on a Rigaku Xcalibur Altlas Gemini ultra diffractometer using Cu *Kα* radiation ($\lambda$ = 1.54184 Å). Data were collected at 150 K using an Oxford Cryosystems CryostreamPlus open-flow $N_2$ cooling device. Intensities were corrected for absorption using a multifaceted crystal model created by indexing the faces of the crystal for which data were collected (Clark & Reid, 1995). Cell refinement, data collection and data reduction were undertaken *via* the software *CrysAlis PRO* (Rigaku OD, 2024). Both structures were solved using *SHELXT* (Sheldrick, 2015) and refined by *SHELXL* (Sheldrick, 2008) using the *OLEX2* interface (Dolomanov *et al.*, 2009).

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| P1 | 0.7500 | 0.70011 (11) | 0.7500 | 0.0370 (2) | |
| H1 | 0.930 (8) | 0.760 (8) | 0.736 (2) | 0.055* | 0.5 |
| C1 | 0.6681 (2) | 0.5206 (3) | 0.68230 (8) | 0.0305 (4) | |
| C2 | 0.4837 (3) | 0.5804 (3) | 0.64606 (8) | 0.0334 (4) | |
| C3 | 0.4182 (3) | 0.4548 (3) | 0.59272 (8) | 0.0355 (4) | |
| C4 | 0.5370 (3) | 0.2772 (3) | 0.57701 (8) | 0.0382 (4) | |
| H4 | 0.4904 | 0.1910 | 0.5413 | 0.046* | |
| C5 | 0.7214 (3) | 0.2184 (3) | 0.61090 (8) | 0.0345 (4) | |
| C6 | 0.7905 (2) | 0.3439 (3) | 0.66413 (8) | 0.0311 (4) | |
| C7 | 0.3561 (3) | 0.7772 (4) | 0.66223 (10) | 0.0461 (5) | |
| H7A | 0.2539 | 0.7281 | 0.6894 | 0.069* | |
| H7B | 0.2860 | 0.8413 | 0.6239 | 0.069* | |
| H7C | 0.4453 | 0.8903 | 0.6838 | 0.069* | |
| C8 | 0.2213 (3) | 0.5095 (4) | 0.55258 (9) | 0.0465 (5) | |
| H8A | 0.1051 | 0.4965 | 0.5775 | 0.070* | |
| H8B | 0.2028 | 0.4051 | 0.5175 | 0.070* | |
| H8C | 0.2284 | 0.6623 | 0.5367 | 0.070* | |
| C9 | 0.8429 (3) | 0.0249 (4) | 0.58956 (9) | 0.0461 (5) | |
| H9A | 0.7649 | −0.0477 | 0.5539 | 0.069* | |
| H9B | 0.8689 | −0.0828 | 0.6237 | 0.069* | |
| H9C | 0.9740 | 0.0791 | 0.5773 | 0.069* | |
| C10 | 0.9941 (3) | 0.2855 (3) | 0.70003 (8) | 0.0353 (4) | |
| H10A | 1.0520 | 0.4184 | 0.7218 | 0.053* | |
| H10B | 1.0888 | 0.2315 | 0.6711 | 0.053* | |
| H10C | 0.9734 | 0.1686 | 0.7305 | 0.053* | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| P1 | 0.0399 (4) | 0.0324 (4) | 0.0362 (4) | 0.000 | −0.0071 (3) | 0.000 |
| C1 | 0.0282 (8) | 0.0338 (9) | 0.0288 (8) | −0.0022 (7) | −0.0002 (6) | 0.0041 (7) |
| C2 | 0.0288 (8) | 0.0381 (9) | 0.0324 (9) | 0.0010 (7) | −0.0003 (7) | 0.0066 (7) |
| C3 | 0.0276 (8) | 0.0485 (11) | 0.0294 (9) | −0.0006 (8) | −0.0014 (7) | 0.0091 (8) |
| C4 | 0.0366 (9) | 0.0509 (11) | 0.0259 (8) | −0.0027 (8) | −0.0024 (7) | −0.0018 (8) |
| C5 | 0.0335 (9) | 0.0401 (10) | 0.0295 (8) | 0.0007 (7) | 0.0011 (7) | 0.0017 (7) |
| C6 | 0.0282 (8) | 0.0355 (9) | 0.0290 (8) | −0.0011 (7) | 0.0004 (7) | 0.0056 (7) |
| C7 | 0.0369 (10) | 0.0486 (11) | 0.0506 (11) | 0.0103 (9) | −0.0063 (8) | 0.0003 (9) |
| C8 | 0.0337 (9) | 0.0683 (14) | 0.0351 (10) | 0.0034 (9) | −0.0070 (8) | 0.0075 (9) |
| C9 | 0.0490 (11) | 0.0484 (11) | 0.0397 (10) | 0.0083 (9) | −0.0010 (8) | −0.0068 (9) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C10 | 0.0298 (9) | 0.0410 (10) | 0.0340 (9) | 0.0030 (7) | −0.0024 (7) | 0.0025 (8) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| P1—H1 | 1.29 (5) | C6—C10 | 1.510 (2) |
| P1—C1$^{i}$ | 1.8472 (17) | C7—H7A | 0.9800 |
| P1—C1 | 1.8471 (17) | C7—H7B | 0.9800 |
| C1—C2 | 1.414 (2) | C7—H7C | 0.9800 |
| C1—C6 | 1.408 (2) | C8—H8A | 0.9800 |
| C2—C3 | 1.404 (3) | C8—H8B | 0.9800 |
| C2—C7 | 1.507 (3) | C8—H8C | 0.9800 |
| C3—C4 | 1.381 (3) | C9—H9A | 0.9800 |
| C3—C8 | 1.512 (2) | C9—H9B | 0.9800 |
| C4—H4 | 0.9500 | C9—H9C | 0.9800 |
| C4—C5 | 1.390 (3) | C10—H10A | 0.9800 |
| C5—C6 | 1.407 (3) | C10—H10B | 0.9800 |
| C5—C9 | 1.505 (3) | C10—H10C | 0.9800 |
| | | | |
| C1$^{i}$—P1—H1 | 98 (2) | C2—C7—H7C | 109.5 |
| C1—P1—H1 | 100 (2) | H7A—C7—H7B | 109.5 |
| C1—P1—C1$^{i}$ | 108.78 (11) | H7A—C7—H7C | 109.5 |
| C2—C1—P1 | 116.72 (13) | H7B—C7—H7C | 109.5 |
| C6—C1—P1 | 122.08 (12) | C3—C8—H8A | 109.5 |
| C6—C1—C2 | 120.98 (16) | C3—C8—H8B | 109.5 |
| C1—C2—C7 | 122.17 (16) | C3—C8—H8C | 109.5 |
| C3—C2—C1 | 119.14 (16) | H8A—C8—H8B | 109.5 |
| C3—C2—C7 | 118.68 (15) | H8A—C8—H8C | 109.5 |
| C2—C3—C8 | 121.47 (17) | H8B—C8—H8C | 109.5 |
| C4—C3—C2 | 118.86 (15) | C5—C9—H9A | 109.5 |
| C4—C3—C8 | 119.67 (17) | C5—C9—H9B | 109.5 |
| C3—C4—H4 | 118.4 | C5—C9—H9C | 109.5 |
| C3—C4—C5 | 123.14 (17) | H9A—C9—H9B | 109.5 |
| C5—C4—H4 | 118.4 | H9A—C9—H9C | 109.5 |
| C4—C5—C6 | 118.78 (17) | H9B—C9—H9C | 109.5 |
| C4—C5—C9 | 119.49 (17) | C6—C10—H10A | 109.5 |
| C6—C5—C9 | 121.73 (16) | C6—C10—H10B | 109.5 |
| C1—C6—C10 | 121.94 (15) | C6—C10—H10C | 109.5 |
| C5—C6—C1 | 119.01 (15) | H10A—C10—H10B | 109.5 |
| C5—C6—C10 | 119.05 (15) | H10A—C10—H10C | 109.5 |
| C2—C7—H7A | 109.5 | H10B—C10—H10C | 109.5 |
| C2—C7—H7B | 109.5 | | |

Symmetry code: (i) −*x*+3/2, *y*, −*z*+3/2.

**Bis(2,3,5,6-tetramethylphenyl)phosphine (kji190003_fa)**

*Crystal data*

$C_{20}H_{27}P$
$M_r = 298.38$
Monoclinic, $P2_1/c$
$a = 12.8874$ (6) Å
$b = 8.8455$ (3) Å
$c = 15.4635$ (7) Å
$\beta = 104.108$ (5)°
$V = 1709.61$ (13) Å$^3$
$Z = 4$
$F(000) = 648$
$D_x = 1.159$ Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 3351 reflections
$\theta = 5.8$–$66.7°$
$\mu = 1.33$ mm$^{-1}$
$T = 150$ K
Block, colourless
0.36 × 0.14 × 0.05 mm

*Data collection*

Rigaku Xcalibur Gemini ultra diffractometer with an Atlas detector
Radiation source: fine-focus sealed X-ray tube, Enhance Ultra (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.3968 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: analytical [CrysAlis PRO (Rigaku OD, 2015), based on expressions derived by Clark & Reid (1995)]
$T_{min} = 0.746$, $T_{max} = 0.939$
12709 measured reflections
3021 independent reflections
2424 reflections with $I > 2\sigma(I)$
$R_{int} = 0.039$
$\theta_{max} = 66.8°$, $\theta_{min} = 3.5°$
$h = -15 \rightarrow 15$
$k = -10 \rightarrow 9$
$l = -18 \rightarrow 18$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.135$
$S = 1.06$
3021 reflections
214 parameters
182 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0698P)^2 + 0.7357P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.43$ e Å$^{-3}$
$\Delta\rho_{min} = -0.35$ 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.

**Refinement**. The hydrogen atom bound to phosphorous in this structure has been modelled over two positions. The P-H bond lengths were constrained to be similar using the SADI card. To prevent the close contact of one of these disordered hydrogens with an adjacent methyl group, this methyl group was also modelled as disordered over two positions. Single-crystal diffraction was carried out on a Rigaku Xcalibur Altlas Gemini ultra diffractometer using Cu $K\alpha$ radiation ($\lambda = 1.54184$ Å). Data were collected at 150 K using an Oxford Cryosystems CryostreamPlus open-flow $N_2$ cooling device. Intensities were corrected for absorption using a multifaceted crystal model created by indexing the faces of the crystal for which data were collected (Clark & Reid, 1995). Cell refinement, data collection and data reduction were undertaken *via* the software *CrysAlis PRO* (Rigaku OD, 2024). Both structures were solved using *SHELXT* (Sheldrick, 2015) and refined by *SHELXL* (Sheldrick, 2008) using the *OLEX2* interface (Dolomanov *et al.*, 2009).

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| P1 | 0.75650 (5) | 0.71204 (7) | 0.50367 (4) | 0.0411 (2) | |

| | | | | | |
|---|---|---|---|---|---|
| H1A | 0.822 (3) | 0.827 (4) | 0.527 (3) | 0.049* | 0.6323 |
| H1B | 0.745 (6) | 0.639 (7) | 0.574 (3) | 0.049* | 0.3677 |
| C1 | 0.81783 (16) | 0.5738 (2) | 0.44111 (15) | 0.0357 (5) | |
| C2 | 0.82942 (16) | 0.6093 (2) | 0.35486 (15) | 0.0353 (5) | |
| C3 | 0.87100 (17) | 0.4999 (3) | 0.30694 (16) | 0.0401 (5) | |
| C4 | 0.90209 (18) | 0.3610 (3) | 0.34647 (17) | 0.0431 (5) | |
| H4 | 0.9272 | 0.2876 | 0.3136 | 0.052* | |
| C5 | 0.89728 (17) | 0.3273 (2) | 0.43253 (17) | 0.0407 (5) | |
| C6 | 0.85536 (16) | 0.4348 (2) | 0.48175 (15) | 0.0381 (5) | |
| C7 | 0.80033 (19) | 0.7628 (3) | 0.31418 (16) | 0.0422 (5) | |
| H7A | 0.7252 | 0.7802 | 0.3072 | 0.063* | |
| H7B | 0.8168 | 0.7672 | 0.2570 | 0.063* | |
| H7C | 0.8404 | 0.8389 | 0.3525 | 0.063* | |
| C8 | 0.8851 (2) | 0.5296 (3) | 0.21425 (18) | 0.0549 (6) | |
| H8A | 0.9130 | 0.4407 | 0.1924 | 0.082* | |
| H8B | 0.9340 | 0.6121 | 0.2161 | 0.082* | |
| H8C | 0.8172 | 0.5549 | 0.1753 | 0.082* | |
| C9 | 0.93877 (19) | 0.1764 (3) | 0.47214 (19) | 0.0504 (6) | |
| H9A | 0.8850 | 0.1278 | 0.4958 | 0.076* | |
| H9B | 1.0020 | 0.1917 | 0.5191 | 0.076* | |
| H9C | 0.9557 | 0.1139 | 0.4267 | 0.076* | |
| C11 | 0.62051 (16) | 0.7494 (2) | 0.43286 (14) | 0.0327 (4) | |
| C12 | 0.55112 (17) | 0.6284 (2) | 0.39960 (14) | 0.0339 (5) | |
| C13 | 0.44829 (17) | 0.6597 (2) | 0.34621 (14) | 0.0359 (5) | |
| C14 | 0.41651 (17) | 0.8083 (3) | 0.32953 (14) | 0.0373 (5) | |
| H14 | 0.3488 | 0.8279 | 0.2935 | 0.045* | |
| C15 | 0.48205 (17) | 0.9295 (2) | 0.36467 (14) | 0.0346 (5) | |
| C16 | 0.58531 (16) | 0.9002 (2) | 0.41712 (14) | 0.0326 (5) | |
| C17 | 0.58273 (19) | 0.4660 (2) | 0.42098 (17) | 0.0423 (5) | |
| H17A | 0.5214 | 0.4094 | 0.4271 | 0.063* | |
| H17B | 0.6372 | 0.4615 | 0.4758 | 0.063* | |
| H17C | 0.6098 | 0.4237 | 0.3737 | 0.063* | |
| C18 | 0.37332 (19) | 0.5335 (3) | 0.30559 (17) | 0.0464 (6) | |
| H18A | 0.4081 | 0.4686 | 0.2716 | 0.070* | |
| H18B | 0.3099 | 0.5754 | 0.2672 | 0.070* | |
| H18C | 0.3543 | 0.4761 | 0.3521 | 0.070* | |
| C19 | 0.44052 (19) | 1.0886 (3) | 0.34468 (17) | 0.0435 (5) | |
| H19A | 0.3728 | 1.0857 | 0.3017 | 0.065* | |
| H19B | 0.4906 | 1.1461 | 0.3211 | 0.065* | |
| H19C | 0.4318 | 1.1352 | 0.3985 | 0.065* | |
| C20 | 0.65494 (19) | 1.0335 (2) | 0.45548 (16) | 0.0416 (5) | |
| H20A | 0.6931 | 1.0680 | 0.4131 | 0.062* | |
| H20B | 0.7051 | 1.0034 | 0.5094 | 0.062* | |
| H20C | 0.6108 | 1.1138 | 0.4683 | 0.062* | |
| C10A | 0.848 (2) | 0.404 (3) | 0.5765 (8) | 0.047 (3) | 0.6323 |
| H10A | 0.8322 | 0.4960 | 0.6033 | 0.071* | 0.6323 |
| H10B | 0.9150 | 0.3641 | 0.6103 | 0.071* | 0.6323 |
| H10C | 0.7923 | 0.3314 | 0.5758 | 0.071* | 0.6323 |

| | | | | | |
|---|---|---|---|---|---|
| C10B | 0.859 (4) | 0.384 (5) | 0.5759 (14) | 0.046 (5) | 0.3677 |
| H10D | 0.8657 | 0.4701 | 0.6143 | 0.069* | 0.3677 |
| H10E | 0.9192 | 0.3178 | 0.5965 | 0.069* | 0.3677 |
| H10F | 0.7943 | 0.3302 | 0.5765 | 0.069* | 0.3677 |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| P1 | 0.0397 (3) | 0.0397 (3) | 0.0411 (3) | 0.0051 (2) | 0.0046 (2) | −0.0048 (2) |
| C1 | 0.0302 (10) | 0.0325 (10) | 0.0424 (11) | 0.0001 (8) | 0.0050 (8) | 0.0008 (9) |
| C2 | 0.0294 (10) | 0.0320 (10) | 0.0429 (11) | −0.0008 (8) | 0.0059 (9) | 0.0022 (8) |
| C3 | 0.0346 (11) | 0.0386 (11) | 0.0479 (12) | 0.0009 (9) | 0.0116 (9) | 0.0002 (9) |
| C4 | 0.0384 (12) | 0.0362 (12) | 0.0566 (14) | 0.0044 (10) | 0.0152 (10) | −0.0037 (10) |
| C5 | 0.0288 (10) | 0.0310 (11) | 0.0616 (14) | 0.0008 (9) | 0.0094 (10) | 0.0045 (10) |
| C6 | 0.0282 (10) | 0.0361 (11) | 0.0482 (12) | 0.0017 (9) | 0.0058 (9) | 0.0075 (9) |
| C7 | 0.0409 (12) | 0.0355 (11) | 0.0496 (13) | 0.0039 (9) | 0.0097 (10) | 0.0071 (9) |
| C8 | 0.0587 (15) | 0.0579 (16) | 0.0528 (15) | 0.0046 (13) | 0.0225 (12) | 0.0012 (12) |
| C9 | 0.0368 (12) | 0.0352 (12) | 0.0777 (18) | 0.0062 (10) | 0.0114 (12) | 0.0112 (11) |
| C11 | 0.0342 (10) | 0.0293 (10) | 0.0372 (11) | −0.0006 (8) | 0.0135 (8) | −0.0039 (8) |
| C12 | 0.0363 (11) | 0.0301 (10) | 0.0394 (11) | −0.0027 (8) | 0.0168 (9) | −0.0030 (8) |
| C13 | 0.0376 (11) | 0.0352 (11) | 0.0386 (11) | −0.0067 (9) | 0.0162 (9) | −0.0068 (9) |
| C14 | 0.0314 (10) | 0.0428 (12) | 0.0391 (11) | −0.0008 (9) | 0.0115 (9) | −0.0032 (9) |
| C15 | 0.0360 (11) | 0.0326 (10) | 0.0384 (11) | 0.0015 (9) | 0.0151 (9) | −0.0006 (8) |
| C16 | 0.0342 (10) | 0.0296 (10) | 0.0365 (11) | −0.0017 (8) | 0.0132 (8) | −0.0024 (8) |
| C17 | 0.0445 (12) | 0.0283 (11) | 0.0571 (14) | −0.0024 (9) | 0.0180 (11) | −0.0011 (10) |
| C18 | 0.0428 (13) | 0.0443 (13) | 0.0549 (14) | −0.0128 (10) | 0.0172 (11) | −0.0139 (11) |
| C19 | 0.0429 (12) | 0.0362 (12) | 0.0532 (14) | 0.0063 (10) | 0.0152 (11) | 0.0031 (10) |
| C20 | 0.0411 (12) | 0.0293 (11) | 0.0536 (14) | −0.0030 (9) | 0.0102 (10) | −0.0043 (9) |
| C10A | 0.043 (6) | 0.042 (6) | 0.053 (3) | 0.013 (5) | 0.006 (3) | 0.017 (3) |
| C10B | 0.045 (9) | 0.045 (10) | 0.050 (5) | 0.019 (6) | 0.016 (5) | 0.009 (4) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| P1—H1A | 1.32 (4) | C12—C13 | 1.408 (3) |
| P1—H1B | 1.31 (4) | C12—C17 | 1.508 (3) |
| P1—C1 | 1.852 (2) | C13—C14 | 1.382 (3) |
| P1—C11 | 1.856 (2) | C13—C18 | 1.510 (3) |
| C1—C2 | 1.413 (3) | C14—H14 | 0.9300 |
| C1—C6 | 1.411 (3) | C14—C15 | 1.391 (3) |
| C2—C3 | 1.402 (3) | C15—C16 | 1.404 (3) |
| C2—C7 | 1.505 (3) | C15—C19 | 1.511 (3) |
| C3—C4 | 1.387 (3) | C16—C20 | 1.513 (3) |
| C3—C8 | 1.511 (3) | C17—H17A | 0.9600 |
| C4—H4 | 0.9300 | C17—H17B | 0.9600 |
| C4—C5 | 1.380 (3) | C17—H17C | 0.9600 |
| C5—C6 | 1.406 (3) | C18—H18A | 0.9600 |
| C5—C9 | 1.511 (3) | C18—H18B | 0.9600 |
| C6—C10A | 1.516 (8) | C18—H18C | 0.9600 |

| | | | |
|---|---|---|---|
| C6—C10B | 1.514 (13) | C19—H19A | 0.9600 |
| C7—H7A | 0.9600 | C19—H19B | 0.9600 |
| C7—H7B | 0.9600 | C19—H19C | 0.9600 |
| C7—H7C | 0.9600 | C20—H20A | 0.9600 |
| C8—H8A | 0.9600 | C20—H20B | 0.9600 |
| C8—H8B | 0.9600 | C20—H20C | 0.9600 |
| C8—H8C | 0.9600 | C10A—H10A | 0.9600 |
| C9—H9A | 0.9600 | C10A—H10B | 0.9600 |
| C9—H9B | 0.9600 | C10A—H10C | 0.9600 |
| C9—H9C | 0.9600 | C10B—H10D | 0.9600 |
| C11—C12 | 1.409 (3) | C10B—H10E | 0.9600 |
| C11—C16 | 1.411 (3) | C10B—H10F | 0.9600 |

| | | | |
|---|---|---|---|
| C1—P1—H1A | 109.1 (18) | C12—C13—C18 | 120.9 (2) |
| C1—P1—H1B | 105 (3) | C14—C13—C12 | 119.39 (19) |
| C1—P1—C11 | 105.74 (9) | C14—C13—C18 | 119.7 (2) |
| C11—P1—H1A | 118.4 (18) | C13—C14—H14 | 118.8 |
| C11—P1—H1B | 108 (3) | C13—C14—C15 | 122.4 (2) |
| C2—C1—P1 | 120.22 (16) | C15—C14—H14 | 118.8 |
| C6—C1—P1 | 119.09 (17) | C14—C15—C16 | 118.89 (19) |
| C6—C1—C2 | 120.7 (2) | C14—C15—C19 | 119.2 (2) |
| C1—C2—C7 | 121.5 (2) | C16—C15—C19 | 121.96 (19) |
| C3—C2—C1 | 119.18 (19) | C11—C16—C20 | 122.31 (19) |
| C3—C2—C7 | 119.3 (2) | C15—C16—C11 | 119.61 (19) |
| C2—C3—C8 | 122.1 (2) | C15—C16—C20 | 118.08 (18) |
| C4—C3—C2 | 119.0 (2) | C12—C17—H17A | 109.5 |
| C4—C3—C8 | 118.9 (2) | C12—C17—H17B | 109.5 |
| C3—C4—H4 | 118.6 | C12—C17—H17C | 109.5 |
| C5—C4—C3 | 122.7 (2) | H17A—C17—H17B | 109.5 |
| C5—C4—H4 | 118.6 | H17A—C17—H17C | 109.5 |
| C4—C5—C6 | 119.3 (2) | H17B—C17—H17C | 109.5 |
| C4—C5—C9 | 119.2 (2) | C13—C18—H18A | 109.5 |
| C6—C5—C9 | 121.5 (2) | C13—C18—H18B | 109.5 |
| C1—C6—C10A | 119.5 (9) | C13—C18—H18C | 109.5 |
| C1—C6—C10B | 128.0 (15) | H18A—C18—H18B | 109.5 |
| C5—C6—C1 | 118.9 (2) | H18A—C18—H18C | 109.5 |
| C5—C6—C10A | 121.5 (9) | H18B—C18—H18C | 109.5 |
| C5—C6—C10B | 113.1 (15) | C15—C19—H19A | 109.5 |
| C2—C7—H7A | 109.5 | C15—C19—H19B | 109.5 |
| C2—C7—H7B | 109.5 | C15—C19—H19C | 109.5 |
| C2—C7—H7C | 109.5 | H19A—C19—H19B | 109.5 |
| H7A—C7—H7B | 109.5 | H19A—C19—H19C | 109.5 |
| H7A—C7—H7C | 109.5 | H19B—C19—H19C | 109.5 |
| H7B—C7—H7C | 109.5 | C16—C20—H20A | 109.5 |
| C3—C8—H8A | 109.5 | C16—C20—H20B | 109.5 |
| C3—C8—H8B | 109.5 | C16—C20—H20C | 109.5 |
| C3—C8—H8C | 109.5 | H20A—C20—H20B | 109.5 |
| H8A—C8—H8B | 109.5 | H20A—C20—H20C | 109.5 |

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| H8A—C8—H8C | 109.5 | H20B—C20—H20C | 109.5 |
| H8B—C8—H8C | 109.5 | C6—C10A—H10A | 109.5 |
| C5—C9—H9A | 109.5 | C6—C10A—H10B | 109.5 |
| C5—C9—H9B | 109.5 | C6—C10A—H10C | 109.5 |
| C5—C9—H9C | 109.5 | H10A—C10A—H10B | 109.5 |
| H9A—C9—H9B | 109.5 | H10A—C10A—H10C | 109.5 |
| H9A—C9—H9C | 109.5 | H10B—C10A—H10C | 109.5 |
| H9B—C9—H9C | 109.5 | C6—C10B—H10D | 109.5 |
| C12—C11—P1 | 120.29 (16) | C6—C10B—H10E | 109.5 |
| C12—C11—C16 | 120.43 (19) | C6—C10B—H10F | 109.5 |
| C16—C11—P1 | 119.18 (15) | H10D—C10B—H10E | 109.5 |
| C11—C12—C17 | 122.0 (2) | H10D—C10B—H10F | 109.5 |
| C13—C12—C11 | 119.16 (19) | H10E—C10B—H10F | 109.5 |
| C13—C12—C17 | 118.81 (19) | | |