

Two tris(3,5-disubstituted phenyl)phosphines and their isostructural P^V oxides

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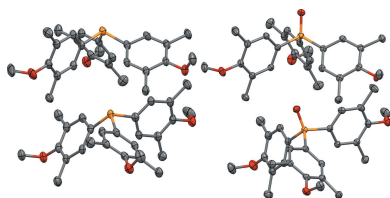
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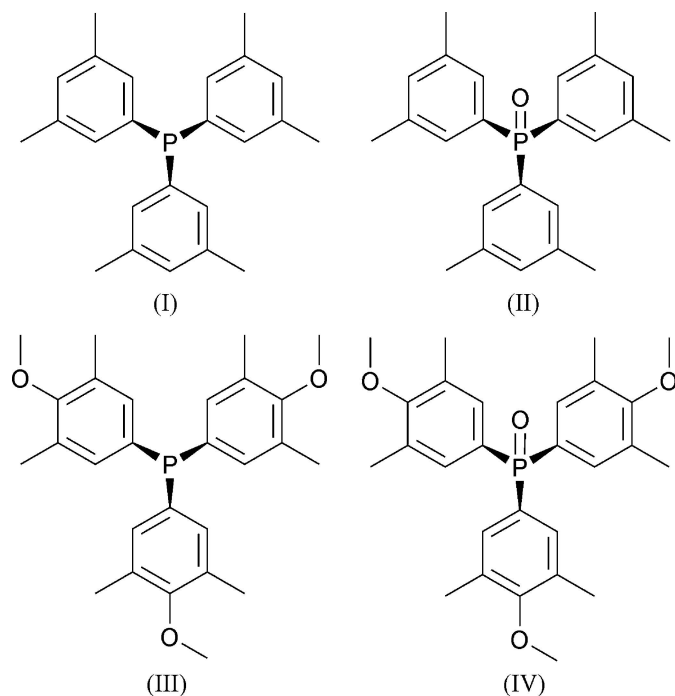
The crystal structures of tris(3,5-dimethylphenyl)phosphine (C₂₄H₂₇P), (I), tris(3,5-dimethylphenyl)phosphine oxide (C₂₄H₂₇OP), (II), tris(4-methoxy-3,5-dimethylphenyl)phosphine (C₂₇H₃₃O₃P), (III), and tris(4-methoxy-3,5-dimethylphenyl)phosphine oxide (C₂₇H₃₃O₄P), (IV), are reported. The structure of (III) has been described before [Romain *et al.* (2000). *Organometallics*, **19**, 2047–2050], but it is rereported here on the basis of modern area-detector data and to facilitate comparison with the other structures reported here. Compounds (I) and (II) crystallize isostructurally in *P2₁/c*. Similarly, (III) and (IV) crystallize isostructurally in *Pbca*. The conformations of (I) and (II) in the solid state deviate strongly from helical, whereas those of (III) and (IV) are found to be closer to an ideal threefold rotational symmetry. The pyramidal indices, $\sum(\text{C}-\text{P}-\text{C})$, are 305.35 (16), 317.23 (15), 307.2 (4) and 318.67 (18)° for (I), (II), (III) and (IV), respectively. Each is found to be more pyramidal than Ph₃P or Ph₃PO. Hybrid DFT calculations incorporating terms for dispersion provide evidence that the causes of the increased pyramidalities, despite the 3,5-dimethyl group substitution, include dispersion interactions. The calculated $\sum(\text{C}-\text{P}-\text{C})$ values are 304.8° for both (I) and (III) and 317.4° for both (II) and (IV), with no difference arising from the substitution at ring position 4.

1. Chemical context

The two bulky triarylphosphines (I) and (III) are of considerable interest in coordination chemistry and catalysis (Kakizoe *et al.*, 2017; Lian *et al.*, 2017; Ogiwara *et al.*, 2017; Nishikawa *et al.*, 2016; Naruto *et al.*, 2015; Jover *et al.*, 2010; Romain *et al.*, 2000) and have been investigated for frustrated Lewis-pair activity (Wang & Stephan, 2014; Ullrich *et al.*, 2010). The synthesis of (I) was first mentioned in the non-patent literature by Hengartner *et al.* (1979) and in more detail twelve years later (Culcasi *et al.*, 1991) and is now commercially available from several sources, but its crystal structure has not been reported. The preparation of (III) was reported by Romain *et al.* (2000) some 11 years after it appeared in the patent literature. These authors reported a crystal structure, Cambridge Structural Database (CSD, Version 5.39, with updates to November 2017; Groom *et al.*, 2016) refcode: FOQNOO. However, as this determination used molybdenum radiation and a serial diffractometer, we have repeated it here under the same conditions as the other three compounds to improve comparability. Phosphine oxide (II) was first mentioned for its use as an additive that enhances the enantiomeric excess in stoichiometric asymmetric epoxidation of *E*-methylstyrene (Kerrigan *et al.*, 2002) and a schematic synthesis was reported a year later (Henschke *et al.*, 2003) but



the characterization details are not found in the open literature. Similarly, phosphine oxide (IV) is mentioned only in the patent literature. Here we report the crystal structures of (I), (II) and (IV) and full details for synthesis and characterization of (II) and (IV), for the first time, and the redetermination of (III).



2. Structural commentary

Phosphine (I) crystallizes in $P2_1/c$ with one molecule in the asymmetric unit that is distinctly pyramidal (Fig. 1). It has a sum of angles around the central phosphorus atom, the **pyramidity index** (see Boéré & Zhang, 2013), $\sum(C-P-C) = 305.35$ (16°). This is a *smaller* value than that in PPh_3 , $\sum(C-P-C) = 308.3$ (2°) (Boéré & Zhang, 2005), indicating a more pyramidal structure, despite the potential steric interference of the three *endo*-oriented methyl substituents at C3, C13, and C23. Similarly, (III) crystallizes in $Pbca$ also with $Z' = 1$ and $\sum(C-P-C) = 307.2$ (4°). By contrast, phosphines with 2,6-disubstitution patterns have greatly reduced pyramidity. For example, $\sum(C-P-C) = 335.6$ (3°) in $Dipp_3P$, (Boéré *et al.*, 2008) 334.4 (3°) in $Tripp_3P$, (Sasaki *et al.*, 2002) and 329.1 (5°) in Mes_3P , (Blount *et al.*, 1994). Oxidation or protonation of Ar_3P always leads to some flattening at the phosphorus atom. Thus, although (II) is isostructural with (I), $\sum(C-P-C) = 317.23$ (15°) differs by some 12° , while (IV), which is isostructural with (II), has $\sum(C-P-C) = 318.67$ (18°) (Fig. 2). In sixteen independent structure determinations of Ph_3PO reported in the CSD, the average value with s.u. of $\sum(C-P-C)$ is 319.3 (3°). Thus, for both the title phosphines and their oxides, the pyramidity index for the title compounds is lower than in the corresponding Ph_3P or Ph_3PO .

That all these 3,5-dimethyl-substituted compounds should be *more* pyramidal than corresponding C_6H_5- derivatives is at first surprising. A plausible explanation for this is that the substitution induces greater intramolecular dispersion interactions, *i.e.* between the methyl groups and the π -clouds of adjacent rings. To find evidence for this, hybrid density functional theory (DFT) calculations [with Becke's non-local three parameter exchange and the Lee–Yang–Parr correlation functional (B3LYP) and also incorporating Grimme's D3 empirical dispersion corrections] with the 6-31G(2d,p) basis set, as implemented in the *Gaussian16* program package (Frisch *et al.*, 2016), were undertaken. The optimized geometries by DFT are characterized by common $\sum(C-P-C) = 304.8^\circ$ for both (I) and (III) and 317.4° for both (II) and (IV). This supports dispersion as an origin for the observed increased pyramidity caused by 3,5-dimethyl group substi-

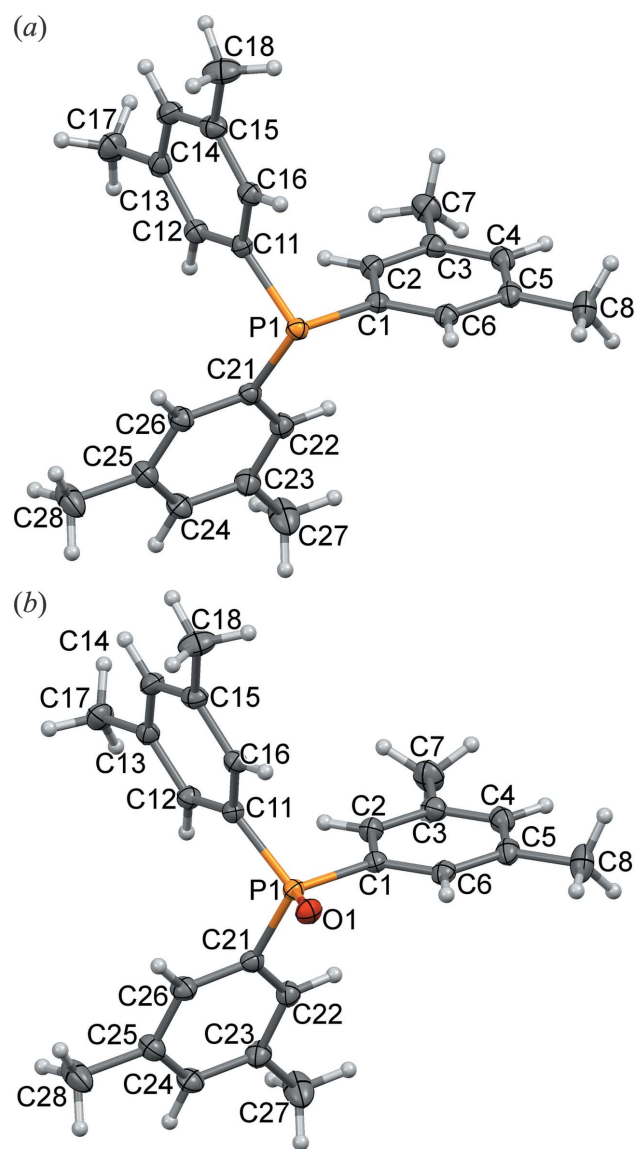


Figure 1
Displacement ellipsoid plots (50%) of (a) phosphine (I) and (b) phosphine oxide (II), including the atom-numbering schemes.

tution. Interestingly, whereas the crystal structures have flatter structures for the 4-CH₃O derivatives (III) and (IV), the DFT calculations have identical pyramidity indices whether the substituent at the 4-position is H or CH₃O. This indicates that intermolecular interactions in the extended structures involving the methoxy groups affect the observed structures compared to that predicted by computation.

In the isostructural pairs, the volumes of the unit cells are larger due to oxygen incorporation. For (I) and (II), the increase is a mere 14 Å³ (0.7%) for the whole unit cell, or 3.5 Å³ per oxygen atom, whereas for (III) and (IV) the increase in volume is larger at 106 Å³ (2.2%) or 13.3 Å³ per oxygen atom. The van der Waals volume of an oxygen atom is 14.7 Å³. In the extended structure, the oxygen atoms in (II) are oriented into a void space (Fig. 3), whereas in (IV) they are directed towards the backside of the next P=O pyramid

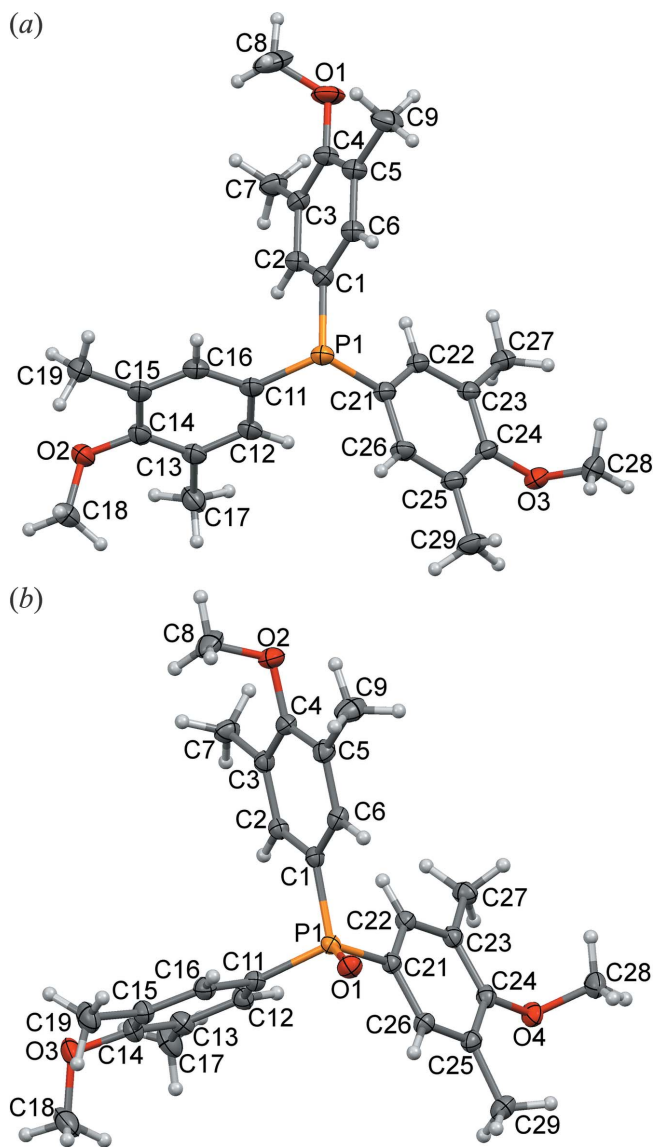


Figure 2
Displacement ellipsoid plots (50%) of (a) phosphine (III) and (b) phosphine oxide (IV), including the atom-numbering schemes.

(Fig. 4). Thus, the nearest P···Pⁱⁱ separations in the crystal increase from 5.148 (2) Å along the *b*-axis direction in (III) to 6.039 (2) Å in (IV) [Symmetry code: (ii) $\frac{3}{2} - x, -\frac{1}{2} + y, z$]. As a consequence, the *a*:*b* lattice parameter ratio changes from 12.30:10.27 in (III) to 11.29:11.90 in (IV).

3. Supramolecular features

As mentioned, the supramolecular organization in (III) and (IV) approximately stacks the Ar₃P structures along the *b*-axis direction [the P–O vectors in (IV) alternate 21.7° off the P···P directions] and the rings are arranged so that alternating molecules are approximately staggered (Fig. 4). This geometry facilitates helical structures, and thus the ring-tilt dihedral angles (defined from the molecular threefold axis through C1,11,21 to C6,16,26) are 26.2 (1), 44.3 (1) and 49.0 (1)° in (III) and 17.0 (1), 38.8 (1) and 39.3 (1)° in (IV).

By contrast, the molecules of (I) and (II) are not aligned in their crystals and are pronouncedly *less* helical in the crystals,

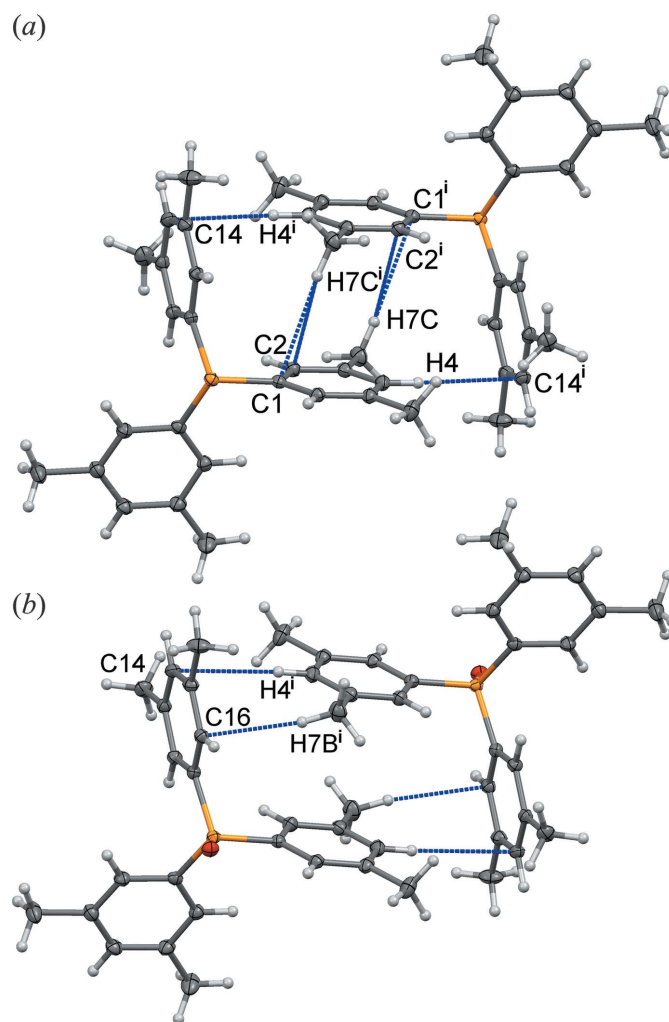


Figure 3
Stacking interactions (π - π and 'T' type) linking centrosymmetric pairs of (a) phosphine (I) and (b) phosphine oxide (II), which is a likely cause of the conformations adopted by the C1 rings. [Symmetry code: (i) $-x, 1 - y, -z$].

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C8-H8A\cdots O1^i$	0.98	2.54	3.3868 (19)	144

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C29-H29A\cdots O3^i$	0.98	2.58	3.524 (5)	161

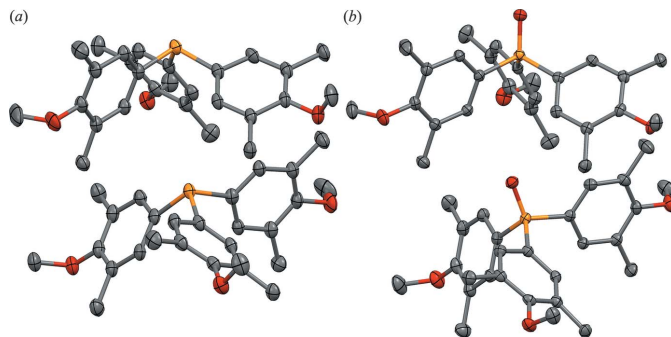
 Symmetry code: (i) $-x + 2, y - \frac{1}{2}, -z + \frac{3}{2}$.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C2-H2\cdots O1^i$	0.95	2.44	3.1533 (16)	132
$C7-H7A\cdots O1^i$	0.98	2.55	3.4033 (18)	145

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

as seen by ring-tilt dihedral angles of 35.6 (1), 8.3 (1) and 58.1 (1)° in (I) and 29.4 (1), 9.1 (1) and 61.2 (1)° in (II). In each of these structures, the C1 aryl rings are almost parallel to the molecular threefold axes, a geometry that was defined as the transition state for Mislow's 'one-ring flip' mechanism for racemization of propeller-shaped molecules (Gust & Mislow, 1973). As shown in Fig. 3*a*, the molecules in (I) are centrosymmetrically related to one another and there are short intermolecular contacts between the C1 rings on adjacent molecules (C2 and C1 to methyl hydrogen H7Cⁱ of 2.84 and 2.90 Å and H4 to C14ⁱ of 2.87 Å. It is likely that this packing preference is responsible for the non-helical arrangement of the rings in this structure. Similarly, in (II) short contacts link C14 with H4ⁱ at 2.88 Å and C16 with methyl hydrogen H7Bⁱ at 2.68 Å (Fig. 3*b*) [Symmetry code: (i) $-x, 1 - y, -z$]. There are some short intermolecular C—H⋯O interactions in structures (II)–(IV), as listed in Tables 1–3.


Figure 4
 Views with the *b* axes vertical in the page, showing the staggered pyramids of (a) phosphine (III) and (b) phosphine oxide (IV) molecules in their respective crystal structures. [Symmetry code for upper molecules: (ii) $\frac{3}{2} - x, -\frac{1}{2} + y, z$].

4. Database survey

The structure of phosphine (I) can be profitably compared to six recently reported diffraction studies reported for its metal complexes or adducts. The cationic silver complex (undecamethyl-1*H*-1-carba-closo-dodecaborate)(tris(3,5-dimethylphenyl)phosphine)silver(I), [LAg][closo-1-*H*-CB₁₁Me₁₁] (refcode ASIZIL; Clarke *et al.*, 2004) employs the large distal steric bulk from the methyl groups in (I) to hinder aggregation in the crystal. The ruthenium(II) complex (μ^2 -aqua)bis(μ^2 -chloro)-dichlorotetrakis[tris(3,5-dimethylphenyl)phosphine]-diruthenium (COQDET01; Naruto & Saito, 2015) is part of a rational design strategy of catalysts for hydrogenation of carboxylic acids. In this complex, one ring in each unique coordinated phosphine re-oriens so as to be almost orthogonal to the coordination axis, with a Ru—P—C—C torsion angles of 83.9 (3) and 87.3 (3)°. The borane complex tris(3,5-dimethylphenyl)[tris(2,3,5,6-tetrafluorophenyl)- λ^5 -boranyl]phosphorane (OLAJIV; Ullrich *et al.*, 2010) is a classical rather than frustrated Lewis-pair adduct. The Tolman cone angle of (I) is estimated to be 151°. In the molybdenum complex *trans*-acetyl-dicarbonyl(cyclopentadienyl)[tris(3,5-dimethylphenyl)phosphine]molybdenum(II) (RAHHUG; Whited *et al.*, 2017), the methyl groups on the aromatic phosphine substituents impact supramolecular organization. The ruthenium complex dichloro-[(*R,R*)-1,2-diphenylethylenediamine]bis[tris(3,5-dimethylphenyl)phosphine]ruthenium(II) (XARCOJ; Jing *et al.*, 2005) is competitive with chiral bidentate ligands for the enantioselective hydrogenation of ketones. The cationic copper complex (1,10-phenanthroline)bis[tris(3,5-dimethylphenyl)phosphine]copper(I) tetrafluoroborate (BEKZOJ; Kakizoe *et al.*, 2017) is part of a study on the effects of bulky phosphines on photophysical properties of copper(I) phenanthroline complexes. Here one of the coordinated phosphines re-oriens so as to have one almost orthogonal ring, with a Cu—P—C—C torsion angle of 86.6 (2)°. The structure of phosphine (III) can be compared to a single crystal structure where it is coordinated to an iridium atom that is part of an Ir₂Mo₂ cyclopentadienyl-carbonyl complex in tris(μ^2 -carbonyl)[tris(4-methoxy-3,5-dimethylphenyl)phosphine]hexacarbonyl-bis(η^5 -cyclopentadienyl)diiridiumdimolybdenum (TUTJAV; Fu *et al.*, 2016). In this complex, one of the rings is also found almost orthogonal to the coordination axis, with an Ir—P—C—C torsion angle of 73 (2)°. Thus, having one of the three aryl rings orthogonal seems to be a common configuration in crowded environments around a metal.

No crystal structures of (II) or (IV), nor any of their derivatives, are reported in the CSD.

5. Synthesis and crystallization

Crystals of tris(3,5-dimethylphenyl)phosphine [69227-47-0], (I), and tris(4-methoxy-3,5-dimethylphenyl)phosphine [121898-64-4], (III), were selected for data collection as received from Sigma-Aldrich Inc. Solvents (BDH) were chromatographic grade and used as received. NMR spectra

Table 4
Experimental details.

	(I)	(II)	(III)	(IV)
Crystal data				
Chemical formula	C ₂₄ H ₂₇ P	C ₂₄ H ₂₇ OP	C ₂₇ H ₃₃ O ₃ P	C ₂₇ H ₃₃ O ₄ P
<i>M_r</i>	346.42	362.42	436.50	452.50
Crystal system, space group	Monoclinic, <i>P2₁/c</i>	Monoclinic, <i>P2₁/c</i>	Orthorhombic, <i>Pbca</i>	Orthorhombic, <i>Pbca</i>
Temperature (K)	108	108	109	108
<i>a</i> , <i>b</i> , <i>c</i> (Å)	14.38617 (9), 9.00514 (5), 17.22745 (12)	14.65624 (11), 8.97960 (5), 17.27940 (13)	12.3031 (6), 10.2629 (5), 37.856 (2)	11.28601 (11), 11.90008 (11), 36.3801 (3)
α , β , γ (°)	90, 112.6169 (7), 90	90, 114.2052 (9), 90	90, 90, 90	90, 90, 90
<i>V</i> (Å ³)	2060.17 (2)	2074.16 (3)	4780.0 (4)	4886.01 (8)
<i>Z</i>	4	4	8	8
Radiation type	Cu <i>K</i> α	Cu <i>K</i> α	Cu <i>K</i> α	Cu <i>K</i> α
μ (mm ⁻¹)	1.18	1.23	1.21	1.24
Crystal size (mm)	0.24 × 0.2 × 0.2	0.3 × 0.2 × 0.16	0.31 × 0.07 × 0.05	0.2 × 0.2 × 0.04
Data collection				
Diffractometer	Rigaku Oxford Diffraction SuperNova, Dual, Cu at zero, Pilatus 200/300K	Rigaku Oxford Diffraction SuperNova, Dual, Cu at zero, Pilatus 200/300K	Rigaku Oxford Diffraction SuperNova, Dual, Cu at zero, Pilatus 200K	Rigaku Oxford Diffraction SuperNova, Dual, Cu at zero, Pilatus 200/300K
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku OD, 2015)	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku OD, 2015)	Gaussian (<i>CrysAlis PRO</i> ; Rigaku OD, 2015)	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku OD, 2015)
<i>T_{min}</i> , <i>T_{max}</i>	0.907, 1.000	0.796, 1.000	0.792, 0.950	0.755, 1.000
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>) reflections]	42680, 4296, 4220	48104, 4542, 4390	19900, 5029, 4084	29719, 5325, 4821
<i>R_{int}</i> (<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.025 0.630	0.027 0.641	0.066 0.640	0.033 0.639
Refinement				
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.036, 0.099, 1.05	0.037, 0.102, 1.09	0.073, 0.198, 1.05	0.039, 0.100, 1.05
No. of reflections	4296	4542	5029	5325
No. of parameters	233	242	289	299
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.28, -0.30	0.31, -0.30	0.54, -0.67	0.35, -0.41

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *olex2.solve* (Bourhis *et al.*, 2015), *SHELXT* (Sheldrick, 2015a), *SHELXL2016* (Sheldrick, 2015b) and *OLEX2* (Dolomanov *et al.*, 2009).

were recorded on a 300 MHz Bruker Avance II spectrometer and are referenced to TMS at 0 (¹H), CDCl₃ at 77.23 (¹³C) and 85% H₃PO₄ at 0 ppm (capillary, ³¹P).

5.1. Preparation of (II)

Tris(3,5-dimethylphenyl)phosphine oxide [381212-20-0], (II), was prepared by dissolving 0.10 g (I), 0.29 mmol, in 15 ml of acetone (thin-layer chromatography, TLC, monitoring: *R_f* = 0.32 in 1:9 ethyl acetate/hexanes), heating to the boil, and adding 3.0 mL of 4% aqueous H₂O₂ dropwise. After gentle reflux for 1.5 h, the mixture was checked again by TLC (*R_f* = 0) indicating reaction completion. Removal of all volatiles, dissolving in 10 ml CH₂Cl₂ and drying overnight with Na₂SO₄, filtering and evaporating, left a dry solid. Recrystallization from mixed solvents of 5 ml heptane and 2 ml CH₂Cl₂ at the boil produced colourless blocks on cooling, recovered by slow evaporation to afford 0.06 g (II), 0.17 mmol, 57% yield. Identity was established by X-ray crystallography and very high purity by nuclear magnetic resonance (NMR) spectroscopy (atom numbers are those from the C1 ring in Fig. 1*b*). ¹H NMR (CDCl₃): δ 2.312 (CH₃, *s*, 18H); 7.144 (C4H, *s*, 3H); 7.282 (C2,6H, *d* ³*J*_{PH} = 12.3 Hz, 6H). ¹³C NMR (CDCl₃): δ 21.47 (CH₃, *s*); 129.74 (C2&6, *d* ²*J*_{PC} = 9.8 Hz); 132.67 (C1, *d*

¹*J*_{PC} = 102.6 Hz); 133.67 (C4, *d* ⁴*J*_{PC} = 3.0 Hz); 138.16 (C3&5, *d* ³*J*_{PC} = 12.8 Hz). ³¹P NMR (CDCl₃): δ +29.73, *s* (satellites: ¹*J*_{PC} = 102.6 Hz).

5.2. Preparation of (IV)

Tris(4-methoxy-3,5-dimethylphenyl)phosphine oxide [540743-36-0], (IV), was similarly prepared from 0.10 g (III), 0.23 mmol, (TLC: *R_f* = 0.38 in 1:9 ethyl acetate/hexanes) and 3.0 ml of 4% aqueous H₂O₂. 1.5 h gentle reflux also sufficed for reaction completion (TLC: *R_f* = 0). A similar workup and recrystallization procedure afforded colourless plates by slow evaporation, 0.08 g (II), 0.18 mmol, 77% yield. Identity was established by X-ray crystallography and very high purity by nuclear magnetic resonance (NMR) spectroscopy (atom numbers are those from the C1 ring in Fig. 2*b*). ¹H NMR (CDCl₃): δ 2.282 (CH₃, *s*, 18H); 3.747 (CH₃O, *s*, 9H); 7.311 (C2,6H, *d* ³*J*_{PH} = 12.0 Hz, 6H). ¹³C NMR (CDCl₃): δ 16.37 (CH₃, *s*); 59.75 (CH₃O, *s*); 127.84 (C1, *d* ¹*J*_{PC} = 105.7 Hz); 131.41 (C3&5, *d* ³*J*_{PC} = 13.6 Hz); 132.81 (C2&6, *d* ²*J*_{PC} = 10.6 Hz); 160.09 (C4, *d* ⁴*J*_{PC} = 3.0 Hz). ³¹P NMR (CDCl₃): δ +28.49, *s* (satellites: ¹*J*_{PC} = 105.8 Hz).

6. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 4. H atoms attached to C atoms were treated as riding, with C–H = 0.98 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl and C–H = 0.95 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aromatic H atoms.

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References

- Blount, J. F., Camp, D., Hart, R. D., Healy, P. C., Skelton, B. W. & White, A. H. (1994). *Aust. J. Chem.* **47**, 1631–1639.
- Boéré, R. T., Bond, A. M., Cronin, S., Duffy, N. W., Hazendonk, P., Masuda, J. D., Pollard, K., Roemmele, T. L., Tran, P. & Zhang, Y. (2008). *New J. Chem.* **32**, 214–231.
- Boéré, R. T. & Zhang, Y. (2005). *J. Organomet. Chem.* **690**, 2651–2657.
- Boéré, R. T. & Zhang, Y. (2013). *Acta Cryst.* **C69**, 1051–1054.
- Bourhis, L. J., Dolomanov, O. V., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2015). *Acta Cryst.* **A71**, 59–75.
- Clarke, A. J., Ingleson, M. J., Kociok-Köhn, G., Mahon, M. F., Patmore, N. J., Rourke, J. P., Ruggiero, G. D. & Weller, A. S. (2004). *J. Am. Chem. Soc.* **126**, 1503–1517.
- Culcasi, M., Berchadsky, Y., Gronchi, G. & Tordo, P. (1991). *J. Org. Chem.* **56**, 3537–3542.
- Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
- Frisch, M. J., *et al.* (2016). Gaussian 16. Gaussian, Inc., Wallingford CT, USA.
- Fu, J., Moxey, G. J., Morshedi, M., Barlow, A., Randles, M. D., Simpson, P. V., Schwich, T., Cifuentes, M. P. & Humphrey, M. G. (2016). *J. Organomet. Chem.* **812**, 135–144.
- Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.
- Gust, D. & Mislow, K. (1973). *J. Am. Chem. Soc.* **95**, 1535–1547.
- Hengartner, U., Valentine, D. Jr, Johnson, K. K., Larscheid, M. E., Pigott, F., Scheidl, F., Scott, J. W., Sun, R. C. & Townsend, J. M. (1979). *J. Org. Chem.* **44**, 3741–3747.
- Henschke, J. P., Zanotti-Gerosa, A., Moran, P., Harrison, P., Mullen, B., Casey, G. & Lennon, I. C. (2003). *Tetrahedron Lett.* **44**, 4379–4383.
- Jing, Q., Zhang, X., Sun, J. & Ding, K. (2005). *Adv. Synth. Catal.* **347**, 1193–1197.
- Jover, J., Fey, N., Harvey, J. N., Lloyd-Jones, G. C., Orpen, A. G., Owen-Smith, G. J. J., Murray, P., Hose, D. R. J., Osborne, R. & Purdie, M. (2010). *Organometallics*, **29**, 6245–6258.
- Kakizoe, D., Nishikawa, M., Fujii, Y. & Tsubomura, T. (2017). *Dalton Trans.* **46**, 14804–14811.
- Kerrigan, N. J., Langan, I. J., Dalton, C. T., Daly, A. M., Bousquet, C. & Gilheany, D. G. (2002). *Tetrahedron Lett.* **43**, 2107–2110.
- Lian, Z., Bhawal, B. N., Yu, P. & Morandi, B. (2017). *Science*, **356**, 1059–1063.
- Naruto, M. & Saito, S. (2015). *Nat. Commun.* **6**, 8140p.
- Nishikawa, D., Hirano, K. & Miura, M. (2016). *Org. Lett.* **18**, 4856–4859.
- Ogiwara, Y., Miyake, M., Kochi, T. & Kakiuchi, F. (2017). *Organometallics*, **36**, 159–164.
- Rigaku OD (2015). *CrysAlis PRO*. Rigaku Oxford Diffraction, Yarnton, England.
- Romain, J. K., Ribblett, J. W., Byrn, R. W., Snyder, R. D., Storhoff, B. N. & Huffman, J. C. (2000). *Organometallics*, **19**, 2047–2050.
- Sasaki, S., Sutoh, K., Murakami, M. & Yoshifuji, M. (2002). *J. Am. Chem. Soc.* **124**, 14830–14831.
- Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.
- Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.
- Ullrich, M., Lough, A. J. & Stephan, D. W. (2010). *Organometallics*, **29**, 3647–3654.
- Wang, T. & Stephan, D. W. (2014). *Chem. Commun.* **50**, 7007–7010.
- Whited, M. T., Ruffer, E. J., Zhang, J., Rabaey, D. J. & Janzen, D. E. (2017). *IUCrData*, **2**, x170042.

supporting information

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Two tris(3,5-disubstituted phenyl)phosphines and their isostructural P^V oxides

Nathan D. D. Hill and René T. Boéré

Computing details

For all structures, data collection: *CrysAlis PRO* (Rigaku OD, 2015); cell refinement: *CrysAlis PRO* (Rigaku OD, 2015); data reduction: *CrysAlis PRO* (Rigaku OD, 2015). Program(s) used to solve structure: *olex2.solve* (Bourhis *et al.*, 2015) for (I), (II), (IV); SHELXT (Sheldrick, 2015a) for (III). For all structures, program(s) used to refine structure: *SHELXL2016* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

Tris(3,5-dimethylphenyl)phosphane (I)

Crystal data

C₂₄H₂₇P

$M_r = 346.42$

Monoclinic, $P2_1/c$

$a = 14.38617$ (9) Å

$b = 9.00514$ (5) Å

$c = 17.22745$ (12) Å

$\beta = 112.6169$ (7)°

$V = 2060.17$ (2) Å³

$Z = 4$

$F(000) = 744$

$D_x = 1.117$ Mg m⁻³

Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å

Cell parameters from 33406 reflections

$\theta = 4.9\text{--}76.1^\circ$

$\mu = 1.18$ mm⁻¹

$T = 108$ K

Prism, clear colourless

0.24 × 0.2 × 0.2 mm

Data collection

Rigaku Oxford Diffraction SuperNova, Dual,
Cu at zero, Pilatus 200/300K
diffractometer

Radiation source: micro-focus sealed X-ray
tube, SuperNova (Cu) X-ray Source

Mirror monochromator

ω scans

Absorption correction: multi-scan
(*CrysAlis PRO*; Rigaku OD, 2015)

$T_{\min} = 0.907$, $T_{\max} = 1.000$

42680 measured reflections

4296 independent reflections

4220 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\max} = 76.3^\circ$, $\theta_{\min} = 3.3^\circ$

$h = -18 \rightarrow 18$

$k = -11 \rightarrow 11$

$l = -21 \rightarrow 21$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.036$

$wR(F^2) = 0.099$

$S = 1.05$

4296 reflections

233 parameters

0 restraints

Primary atom site location: iterative

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0521P)^2 + 1.0098P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.28$ e Å⁻³

$\Delta\rho_{\min} = -0.29$ e Å⁻³

Extinction correction: SHELXL2016
 (Sheldrick, 2015b),
 $F_c^* = kFc[1 + 0.001x\lambda^3/\sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.0008 (2)

Special details

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

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
P1	0.24237 (2)	0.22483 (3)	0.19991 (2)	0.01773 (10)
C1	0.13769 (9)	0.35790 (14)	0.17356 (7)	0.0191 (2)
C2	0.13216 (9)	0.48677 (14)	0.12664 (8)	0.0217 (3)
H2	0.185332	0.509281	0.108761	0.026*
C3	0.05030 (9)	0.58256 (14)	0.10568 (8)	0.0230 (3)
C4	-0.02748 (9)	0.54669 (15)	0.13197 (8)	0.0244 (3)
H4	-0.084271	0.610509	0.117124	0.029*
C5	-0.02410 (9)	0.41981 (14)	0.17940 (8)	0.0236 (3)
C6	0.05937 (9)	0.32556 (14)	0.19978 (7)	0.0207 (2)
H6	0.062758	0.238468	0.231871	0.025*
C7	0.04651 (11)	0.72101 (15)	0.05519 (9)	0.0301 (3)
H7A	0.113295	0.740191	0.054603	0.045*
H7B	0.026011	0.805557	0.080747	0.045*
H7C	-0.002220	0.707187	-0.002560	0.045*
C8	-0.10812 (10)	0.38474 (17)	0.20867 (10)	0.0335 (3)
H8A	-0.114279	0.465469	0.244548	0.050*
H8B	-0.092999	0.291750	0.240681	0.050*
H8C	-0.171574	0.374269	0.159769	0.050*
C11	0.25424 (9)	0.21396 (13)	0.09776 (7)	0.0188 (2)
C12	0.30867 (9)	0.31418 (14)	0.06972 (8)	0.0205 (2)
H12	0.346853	0.390234	0.106351	0.025*
C13	0.30765 (9)	0.30395 (14)	-0.01141 (8)	0.0228 (3)
C14	0.25218 (10)	0.18977 (15)	-0.06370 (8)	0.0250 (3)
H14	0.250737	0.182438	-0.119163	0.030*
C15	0.19901 (10)	0.08650 (14)	-0.03684 (8)	0.0246 (3)
C16	0.20062 (9)	0.10030 (14)	0.04441 (8)	0.0209 (2)
H16	0.164506	0.030941	0.063658	0.025*
C17	0.36423 (11)	0.41385 (17)	-0.04288 (9)	0.0323 (3)
H17A	0.395402	0.489222	0.000397	0.048*
H17B	0.317411	0.461883	-0.094009	0.048*
H17C	0.416634	0.361845	-0.055460	0.048*
C18	0.14109 (13)	-0.03762 (17)	-0.09353 (9)	0.0374 (3)
H18A	0.131846	-0.015172	-0.151700	0.056*
H18B	0.075139	-0.047668	-0.089723	0.056*
H18C	0.178614	-0.130746	-0.076018	0.056*

C21	0.35253 (9)	0.33617 (13)	0.26264 (7)	0.0192 (2)
C22	0.34586 (9)	0.47403 (14)	0.29659 (8)	0.0221 (3)
H22	0.282059	0.520911	0.280729	0.027*
C23	0.43125 (10)	0.54477 (15)	0.35355 (8)	0.0256 (3)
C24	0.52445 (9)	0.47645 (15)	0.37487 (8)	0.0254 (3)
H24	0.582867	0.523752	0.413673	0.031*
C25	0.53395 (9)	0.33965 (15)	0.34040 (8)	0.0255 (3)
C26	0.44748 (9)	0.26980 (14)	0.28504 (8)	0.0233 (3)
H26	0.452905	0.175786	0.262120	0.028*
C27	0.42188 (12)	0.69326 (18)	0.39067 (12)	0.0417 (4)
H27A	0.453772	0.770149	0.369034	0.063*
H27B	0.455274	0.688699	0.452071	0.063*
H27C	0.350522	0.717280	0.375004	0.063*
C28	0.63625 (11)	0.26868 (18)	0.36317 (11)	0.0396 (4)
H28A	0.678464	0.332243	0.343964	0.059*
H28B	0.628465	0.171292	0.336009	0.059*
H28C	0.668170	0.256499	0.424317	0.059*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.01727 (16)	0.01831 (17)	0.01719 (16)	-0.00102 (10)	0.00616 (12)	0.00027 (10)
C1	0.0182 (5)	0.0207 (6)	0.0169 (5)	-0.0014 (4)	0.0050 (4)	-0.0030 (4)
C2	0.0201 (5)	0.0239 (6)	0.0208 (6)	-0.0007 (5)	0.0074 (5)	-0.0003 (5)
C3	0.0233 (6)	0.0216 (6)	0.0205 (6)	0.0004 (5)	0.0045 (5)	-0.0021 (5)
C4	0.0198 (6)	0.0242 (6)	0.0265 (6)	0.0028 (5)	0.0058 (5)	-0.0052 (5)
C5	0.0208 (6)	0.0247 (6)	0.0260 (6)	-0.0023 (5)	0.0097 (5)	-0.0071 (5)
C6	0.0215 (6)	0.0209 (6)	0.0197 (6)	-0.0022 (5)	0.0081 (5)	-0.0031 (4)
C7	0.0308 (7)	0.0251 (7)	0.0318 (7)	0.0049 (5)	0.0090 (6)	0.0050 (5)
C8	0.0281 (7)	0.0311 (7)	0.0486 (9)	0.0004 (6)	0.0229 (6)	-0.0040 (6)
C11	0.0172 (5)	0.0195 (6)	0.0187 (6)	0.0030 (4)	0.0058 (4)	0.0007 (4)
C12	0.0191 (5)	0.0199 (6)	0.0218 (6)	0.0002 (5)	0.0071 (5)	-0.0005 (5)
C13	0.0215 (6)	0.0247 (6)	0.0234 (6)	0.0054 (5)	0.0098 (5)	0.0049 (5)
C14	0.0286 (6)	0.0283 (6)	0.0185 (6)	0.0074 (5)	0.0094 (5)	0.0010 (5)
C15	0.0263 (6)	0.0226 (6)	0.0213 (6)	0.0042 (5)	0.0052 (5)	-0.0021 (5)
C16	0.0200 (5)	0.0194 (6)	0.0218 (6)	0.0009 (4)	0.0064 (5)	0.0000 (4)
C17	0.0318 (7)	0.0382 (8)	0.0305 (7)	-0.0001 (6)	0.0160 (6)	0.0084 (6)
C18	0.0504 (9)	0.0310 (8)	0.0254 (7)	-0.0049 (7)	0.0086 (6)	-0.0081 (6)
C21	0.0196 (5)	0.0214 (6)	0.0165 (5)	-0.0021 (4)	0.0069 (4)	0.0007 (4)
C22	0.0201 (6)	0.0236 (6)	0.0235 (6)	-0.0008 (5)	0.0091 (5)	-0.0014 (5)
C23	0.0242 (6)	0.0246 (6)	0.0283 (6)	-0.0037 (5)	0.0106 (5)	-0.0056 (5)
C24	0.0210 (6)	0.0271 (7)	0.0257 (6)	-0.0059 (5)	0.0062 (5)	-0.0039 (5)
C25	0.0205 (6)	0.0271 (7)	0.0259 (6)	0.0001 (5)	0.0057 (5)	0.0000 (5)
C26	0.0218 (6)	0.0230 (6)	0.0230 (6)	0.0001 (5)	0.0064 (5)	-0.0023 (5)
C27	0.0296 (7)	0.0347 (8)	0.0567 (10)	-0.0047 (6)	0.0119 (7)	-0.0227 (7)
C28	0.0217 (7)	0.0366 (8)	0.0501 (9)	0.0032 (6)	0.0022 (6)	-0.0104 (7)

Geometric parameters (Å, °)

P1—C1	1.8396 (12)	C14—C15	1.3915 (19)
P1—C11	1.8350 (12)	C15—C16	1.3965 (17)
P1—C21	1.8350 (12)	C15—C18	1.5071 (18)
C1—C2	1.3988 (17)	C16—H16	0.9500
C1—C6	1.3963 (16)	C17—H17A	0.9800
C2—H2	0.9500	C17—H17B	0.9800
C2—C3	1.3908 (17)	C17—H17C	0.9800
C3—C4	1.3970 (18)	C18—H18A	0.9800
C3—C7	1.5091 (18)	C18—H18B	0.9800
C4—H4	0.9500	C18—H18C	0.9800
C4—C5	1.3946 (19)	C21—C22	1.3912 (17)
C5—C6	1.4005 (17)	C21—C26	1.4023 (17)
C5—C8	1.5113 (17)	C22—H22	0.9500
C6—H6	0.9500	C22—C23	1.3967 (18)
C7—H7A	0.9800	C23—C24	1.3900 (18)
C7—H7B	0.9800	C23—C27	1.5105 (19)
C7—H7C	0.9800	C24—H24	0.9500
C8—H8A	0.9800	C24—C25	1.3969 (19)
C8—H8B	0.9800	C25—C26	1.3929 (18)
C8—H8C	0.9800	C25—C28	1.5114 (18)
C11—C12	1.3971 (17)	C26—H26	0.9500
C11—C16	1.3936 (17)	C27—H27A	0.9800
C12—H12	0.9500	C27—H27B	0.9800
C12—C13	1.3950 (17)	C27—H27C	0.9800
C13—C14	1.3974 (19)	C28—H28A	0.9800
C13—C17	1.5083 (18)	C28—H28B	0.9800
C14—H14	0.9500	C28—H28C	0.9800
C11—P1—C1	99.63 (5)	C16—C15—C18	120.49 (12)
C11—P1—C21	102.48 (5)	C11—C16—C15	121.20 (12)
C21—P1—C1	103.24 (5)	C11—C16—H16	119.4
C2—C1—P1	122.84 (9)	C15—C16—H16	119.4
C6—C1—P1	118.04 (9)	C13—C17—H17A	109.5
C6—C1—C2	119.09 (11)	C13—C17—H17B	109.5
C1—C2—H2	119.4	C13—C17—H17C	109.5
C3—C2—C1	121.27 (11)	H17A—C17—H17B	109.5
C3—C2—H2	119.4	H17A—C17—H17C	109.5
C2—C3—C4	118.43 (12)	H17B—C17—H17C	109.5
C2—C3—C7	120.09 (12)	C15—C18—H18A	109.5
C4—C3—C7	121.47 (12)	C15—C18—H18B	109.5
C3—C4—H4	119.1	C15—C18—H18C	109.5
C5—C4—C3	121.85 (11)	H18A—C18—H18B	109.5
C5—C4—H4	119.1	H18A—C18—H18C	109.5
C4—C5—C6	118.47 (11)	H18B—C18—H18C	109.5
C4—C5—C8	120.97 (12)	C22—C21—P1	123.48 (9)
C6—C5—C8	120.56 (12)	C22—C21—C26	118.81 (11)

C1—C6—C5	120.89 (12)	C26—C21—P1	117.28 (9)
C1—C6—H6	119.6	C21—C22—H22	119.4
C5—C6—H6	119.6	C21—C22—C23	121.21 (11)
C3—C7—H7A	109.5	C23—C22—H22	119.4
C3—C7—H7B	109.5	C22—C23—C27	120.24 (12)
C3—C7—H7C	109.5	C24—C23—C22	118.83 (12)
H7A—C7—H7B	109.5	C24—C23—C27	120.93 (12)
H7A—C7—H7C	109.5	C23—C24—H24	119.3
H7B—C7—H7C	109.5	C23—C24—C25	121.34 (12)
C5—C8—H8A	109.5	C25—C24—H24	119.3
C5—C8—H8B	109.5	C24—C25—C28	120.48 (12)
C5—C8—H8C	109.5	C26—C25—C24	118.79 (12)
H8A—C8—H8B	109.5	C26—C25—C28	120.72 (12)
H8A—C8—H8C	109.5	C21—C26—H26	119.5
H8B—C8—H8C	109.5	C25—C26—C21	120.99 (12)
C12—C11—P1	124.64 (9)	C25—C26—H26	119.5
C16—C11—P1	116.08 (9)	C23—C27—H27A	109.5
C16—C11—C12	119.22 (11)	C23—C27—H27B	109.5
C11—C12—H12	119.6	C23—C27—H27C	109.5
C13—C12—C11	120.82 (11)	H27A—C27—H27B	109.5
C13—C12—H12	119.6	H27A—C27—H27C	109.5
C12—C13—C14	118.54 (12)	H27B—C27—H27C	109.5
C12—C13—C17	121.17 (12)	C25—C28—H28A	109.5
C14—C13—C17	120.29 (12)	C25—C28—H28B	109.5
C13—C14—H14	119.1	C25—C28—H28C	109.5
C15—C14—C13	121.88 (12)	H28A—C28—H28B	109.5
C15—C14—H14	119.1	H28A—C28—H28C	109.5
C14—C15—C16	118.32 (12)	H28B—C28—H28C	109.5
C14—C15—C18	121.19 (12)		

Tris(3,5-dimethylphenyl)(oxo)- λ^5 -phosphane (II)

Crystal data

C₂₄H₂₇OP
M_r = 362.42
 Monoclinic, *P*2₁/*c*
a = 14.65624 (11) Å
b = 8.97960 (5) Å
c = 17.27940 (13) Å
 β = 114.2052 (9)°
V = 2074.16 (3) Å³
Z = 4

F(000) = 776
D_x = 1.161 Mg m⁻³
 Cu *K* α radiation, λ = 1.54184 Å
 Cell parameters from 35213 reflections
 θ = 5.2–80.3°
 μ = 1.23 mm⁻¹
T = 108 K
 Prism, clear colourless
 0.3 × 0.2 × 0.16 mm

Data collection

Rigaku Oxford Diffraction SuperNova, Dual,
 Cu at zero, Pilatus 200/300K
 diffractometer
 Radiation source: micro-focus sealed X-ray
 tube, SuperNova (Cu) X-ray Source
 Mirror monochromator
 ω scans

Absorption correction: multi-scan
 (CrysAlis PRO; Rigaku OD, 2015)
T_{min} = 0.796, *T_{max}* = 1.000
 48104 measured reflections
 4542 independent reflections
 4390 reflections with *I* > 2 σ (*I*)
R_{int} = 0.027

$\theta_{\max} = 81.1^\circ$, $\theta_{\min} = 3.3^\circ$
 $h = -18 \rightarrow 18$

$k = -11 \rightarrow 11$
 $l = -22 \rightarrow 22$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.102$

$S = 1.09$

4542 reflections

242 parameters

0 restraints

Primary atom site location: iterative

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0539P)^2 + 0.8817P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.31 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.29 \text{ e } \text{\AA}^{-3}$

Extinction correction: SHELXL2016

(Sheldrick, 2015b),

$F_c^* = kFc[1 + 0.001x\text{Fc}^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0012 (2)

Special details

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

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
P1	0.24572 (2)	0.24916 (3)	0.18639 (2)	0.01699 (10)
O1	0.23144 (6)	0.10379 (10)	0.22115 (5)	0.02266 (19)
C1	0.14094 (8)	0.37373 (13)	0.16344 (7)	0.0196 (2)
C2	0.13398 (9)	0.50699 (14)	0.11963 (7)	0.0228 (2)
H2	0.184870	0.532338	0.101282	0.027*
C3	0.05362 (9)	0.60297 (14)	0.10254 (8)	0.0246 (3)
C4	-0.02072 (9)	0.56194 (15)	0.12945 (8)	0.0259 (3)
H4	-0.076327	0.626245	0.117578	0.031*
C5	-0.01577 (9)	0.42953 (14)	0.17318 (8)	0.0244 (3)
C6	0.06620 (9)	0.33548 (13)	0.19023 (7)	0.0214 (2)
H6	0.071067	0.245050	0.220211	0.026*
C7	0.04832 (12)	0.74754 (16)	0.05675 (10)	0.0339 (3)
H7A	0.084944	0.824684	0.097889	0.051*
H7B	-0.021764	0.777612	0.026657	0.051*
H7C	0.078336	0.734439	0.015891	0.051*
C8	-0.09711 (11)	0.38991 (16)	0.20202 (10)	0.0338 (3)
H8A	-0.106203	0.472170	0.235453	0.051*
H8B	-0.078025	0.299696	0.236953	0.051*
H8C	-0.159846	0.372154	0.152347	0.051*
C11	0.25717 (8)	0.22998 (13)	0.08669 (7)	0.0181 (2)
C12	0.30739 (8)	0.33312 (13)	0.05762 (7)	0.0198 (2)
H12	0.342407	0.413590	0.093035	0.024*
C13	0.30635 (9)	0.31846 (13)	-0.02321 (7)	0.0208 (2)
C14	0.25486 (9)	0.19849 (14)	-0.07324 (7)	0.0226 (2)
H14	0.253070	0.188474	-0.128577	0.027*

C15	0.20594 (9)	0.09278 (13)	-0.04505 (7)	0.0224 (2)
C16	0.20744 (8)	0.11037 (13)	0.03577 (7)	0.0195 (2)
H16	0.174160	0.039944	0.056146	0.023*
C17	0.35972 (10)	0.42842 (16)	-0.05602 (8)	0.0283 (3)
H17A	0.370845	0.521303	-0.023642	0.043*
H17B	0.318780	0.448660	-0.116113	0.043*
H17C	0.424234	0.386794	-0.049635	0.043*
C18	0.15251 (12)	-0.03725 (16)	-0.09981 (9)	0.0347 (3)
H18A	0.149360	-0.023358	-0.157102	0.052*
H18B	0.084510	-0.043764	-0.102530	0.052*
H18C	0.188859	-0.129304	-0.075450	0.052*
C21	0.35568 (9)	0.34982 (13)	0.25651 (7)	0.0193 (2)
C22	0.34906 (9)	0.48414 (14)	0.29400 (8)	0.0223 (2)
H22	0.285703	0.530544	0.278167	0.027*
C23	0.43410 (9)	0.55168 (15)	0.35450 (8)	0.0256 (3)
C24	0.52640 (9)	0.48300 (15)	0.37547 (8)	0.0255 (3)
H24	0.584722	0.527923	0.416774	0.031*
C25	0.53559 (9)	0.34976 (15)	0.33741 (8)	0.0258 (3)
C26	0.44935 (9)	0.28327 (14)	0.27831 (8)	0.0234 (2)
H26	0.454120	0.191864	0.252540	0.028*
C27	0.42575 (11)	0.69639 (18)	0.39538 (11)	0.0417 (4)
H27A	0.455221	0.776861	0.374843	0.063*
H27B	0.461488	0.687914	0.457132	0.063*
H27C	0.355104	0.718447	0.380735	0.063*
C28	0.63753 (11)	0.28224 (18)	0.35922 (11)	0.0401 (4)
H28A	0.676833	0.348165	0.339455	0.060*
H28B	0.629938	0.184983	0.331532	0.060*
H28C	0.671967	0.269752	0.420846	0.060*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.01757 (16)	0.01839 (16)	0.01570 (16)	-0.00143 (10)	0.00751 (12)	-0.00066 (9)
O1	0.0256 (4)	0.0219 (4)	0.0222 (4)	-0.0026 (3)	0.0116 (3)	0.0018 (3)
C1	0.0188 (5)	0.0227 (6)	0.0170 (5)	-0.0005 (4)	0.0070 (4)	-0.0031 (4)
C2	0.0209 (5)	0.0264 (6)	0.0214 (5)	0.0002 (5)	0.0090 (4)	0.0008 (5)
C3	0.0236 (6)	0.0258 (6)	0.0214 (6)	0.0016 (5)	0.0062 (5)	-0.0006 (5)
C4	0.0213 (6)	0.0262 (6)	0.0282 (6)	0.0028 (5)	0.0082 (5)	-0.0044 (5)
C5	0.0218 (6)	0.0257 (6)	0.0272 (6)	-0.0033 (5)	0.0115 (5)	-0.0085 (5)
C6	0.0226 (6)	0.0212 (6)	0.0215 (5)	-0.0023 (4)	0.0103 (4)	-0.0047 (4)
C7	0.0321 (7)	0.0320 (7)	0.0362 (8)	0.0079 (5)	0.0126 (6)	0.0096 (5)
C8	0.0313 (7)	0.0284 (7)	0.0517 (9)	-0.0016 (5)	0.0271 (6)	-0.0074 (6)
C11	0.0169 (5)	0.0198 (5)	0.0176 (5)	0.0014 (4)	0.0071 (4)	-0.0001 (4)
C12	0.0194 (5)	0.0201 (5)	0.0195 (5)	-0.0006 (4)	0.0074 (4)	-0.0005 (4)
C13	0.0194 (5)	0.0231 (6)	0.0200 (5)	0.0027 (4)	0.0084 (4)	0.0033 (4)
C14	0.0251 (6)	0.0265 (6)	0.0172 (5)	0.0038 (5)	0.0095 (4)	0.0001 (5)
C15	0.0243 (6)	0.0211 (6)	0.0194 (5)	0.0015 (4)	0.0066 (4)	-0.0017 (4)
C16	0.0193 (5)	0.0188 (5)	0.0198 (5)	0.0005 (4)	0.0072 (4)	0.0006 (4)

C17	0.0286 (6)	0.0343 (7)	0.0244 (6)	-0.0028 (5)	0.0131 (5)	0.0059 (5)
C18	0.0493 (8)	0.0285 (7)	0.0238 (6)	-0.0088 (6)	0.0124 (6)	-0.0073 (5)
C21	0.0211 (5)	0.0208 (5)	0.0163 (5)	-0.0023 (4)	0.0080 (4)	0.0000 (4)
C22	0.0209 (5)	0.0230 (6)	0.0241 (6)	-0.0013 (4)	0.0104 (5)	-0.0023 (5)
C23	0.0253 (6)	0.0253 (6)	0.0276 (6)	-0.0044 (5)	0.0121 (5)	-0.0071 (5)
C24	0.0223 (6)	0.0268 (6)	0.0245 (6)	-0.0060 (5)	0.0068 (5)	-0.0048 (5)
C25	0.0214 (6)	0.0261 (6)	0.0258 (6)	-0.0003 (5)	0.0056 (5)	-0.0010 (5)
C26	0.0233 (6)	0.0221 (5)	0.0227 (6)	0.0000 (5)	0.0072 (5)	-0.0039 (5)
C27	0.0298 (7)	0.0370 (8)	0.0556 (10)	-0.0064 (6)	0.0148 (7)	-0.0250 (7)
C28	0.0233 (7)	0.0365 (8)	0.0482 (9)	0.0045 (6)	0.0023 (6)	-0.0119 (7)

Geometric parameters (Å, °)

P1—O1	1.4872 (9)	C14—C15	1.3925 (17)
P1—C1	1.8077 (12)	C15—C16	1.3966 (16)
P1—C11	1.8063 (12)	C15—C18	1.5047 (17)
P1—C21	1.8113 (12)	C16—H16	0.9500
C1—C2	1.3971 (17)	C17—H17A	0.9800
C1—C6	1.3957 (16)	C17—H17B	0.9800
C2—H2	0.9500	C17—H17C	0.9800
C2—C3	1.3900 (17)	C18—H18A	0.9800
C3—C4	1.3981 (18)	C18—H18B	0.9800
C3—C7	1.5057 (18)	C18—H18C	0.9800
C4—H4	0.9500	C21—C22	1.3912 (16)
C4—C5	1.3947 (19)	C21—C26	1.3999 (17)
C5—C6	1.3975 (17)	C22—H22	0.9500
C5—C8	1.5109 (17)	C22—C23	1.3949 (17)
C6—H6	0.9500	C23—C24	1.3927 (18)
C7—H7A	0.9800	C23—C27	1.5077 (18)
C7—H7B	0.9800	C24—H24	0.9500
C7—H7C	0.9800	C24—C25	1.3982 (18)
C8—H8A	0.9800	C25—C26	1.3922 (17)
C8—H8B	0.9800	C25—C28	1.5100 (18)
C8—H8C	0.9800	C26—H26	0.9500
C11—C12	1.3982 (16)	C27—H27A	0.9800
C11—C16	1.3904 (16)	C27—H27B	0.9800
C12—H12	0.9500	C27—H27C	0.9800
C12—C13	1.3967 (16)	C28—H28A	0.9800
C13—C14	1.3938 (17)	C28—H28B	0.9800
C13—C17	1.5072 (16)	C28—H28C	0.9800
C14—H14	0.9500		
O1—P1—C1	112.59 (5)	C14—C15—C16	118.26 (11)
O1—P1—C11	112.64 (5)	C14—C15—C18	121.25 (11)
O1—P1—C21	113.69 (5)	C16—C15—C18	120.49 (11)
C1—P1—C21	106.31 (5)	C11—C16—C15	120.62 (11)
C11—P1—C1	104.63 (5)	C11—C16—H16	119.7
C11—P1—C21	106.29 (5)	C15—C16—H16	119.7

C2—C1—P1	121.03 (9)	C13—C17—H17A	109.5
C6—C1—P1	119.12 (9)	C13—C17—H17B	109.5
C6—C1—C2	119.85 (11)	C13—C17—H17C	109.5
C1—C2—H2	119.6	H17A—C17—H17B	109.5
C3—C2—C1	120.89 (11)	H17A—C17—H17C	109.5
C3—C2—H2	119.6	H17B—C17—H17C	109.5
C2—C3—C4	118.29 (12)	C15—C18—H18A	109.5
C2—C3—C7	120.19 (12)	C15—C18—H18B	109.5
C4—C3—C7	121.51 (12)	C15—C18—H18C	109.5
C3—C4—H4	119.0	H18A—C18—H18B	109.5
C5—C4—C3	122.03 (11)	H18A—C18—H18C	109.5
C5—C4—H4	119.0	H18B—C18—H18C	109.5
C4—C5—C6	118.60 (11)	C22—C21—P1	122.10 (9)
C4—C5—C8	120.44 (12)	C22—C21—C26	119.52 (11)
C6—C5—C8	120.96 (12)	C26—C21—P1	118.19 (9)
C1—C6—C5	120.34 (11)	C21—C22—H22	119.5
C1—C6—H6	119.8	C21—C22—C23	120.97 (11)
C5—C6—H6	119.8	C23—C22—H22	119.5
C3—C7—H7A	109.5	C22—C23—C27	120.44 (12)
C3—C7—H7B	109.5	C24—C23—C22	118.46 (12)
C3—C7—H7C	109.5	C24—C23—C27	121.10 (12)
H7A—C7—H7B	109.5	C23—C24—H24	119.1
H7A—C7—H7C	109.5	C23—C24—C25	121.82 (11)
H7B—C7—H7C	109.5	C25—C24—H24	119.1
C5—C8—H8A	109.5	C24—C25—C28	120.16 (12)
C5—C8—H8B	109.5	C26—C25—C24	118.58 (11)
C5—C8—H8C	109.5	C26—C25—C28	121.25 (12)
H8A—C8—H8B	109.5	C21—C26—H26	119.7
H8A—C8—H8C	109.5	C25—C26—C21	120.64 (12)
H8B—C8—H8C	109.5	C25—C26—H26	119.7
C12—C11—P1	123.22 (9)	C23—C27—H27A	109.5
C16—C11—P1	116.59 (9)	C23—C27—H27B	109.5
C16—C11—C12	120.06 (11)	C23—C27—H27C	109.5
C11—C12—H12	119.8	H27A—C27—H27B	109.5
C13—C12—C11	120.35 (11)	H27A—C27—H27C	109.5
C13—C12—H12	119.8	H27B—C27—H27C	109.5
C12—C13—C17	121.32 (11)	C25—C28—H28A	109.5
C14—C13—C12	118.31 (11)	C25—C28—H28B	109.5
C14—C13—C17	120.37 (11)	C25—C28—H28C	109.5
C13—C14—H14	118.8	H28A—C28—H28B	109.5
C15—C14—C13	122.38 (11)	H28A—C28—H28C	109.5
C15—C14—H14	118.8	H28B—C28—H28C	109.5

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
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C8—H8A···O1ⁱ 0.98 2.54 3.3868 (19) 144

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

Tris(4-methoxy-3,5-dimethylphenyl)phosphane (III)

Crystal data

$C_{27}H_{33}O_3P$

$M_r = 436.50$

Orthorhombic, *Pbca*

$a = 12.3031$ (6) Å

$b = 10.2629$ (5) Å

$c = 37.856$ (2) Å

$V = 4780.0$ (4) Å³

$Z = 8$

$F(000) = 1872$

$D_x = 1.213$ Mg m⁻³

Cu *Kα* radiation, $\lambda = 1.54184$ Å

Cell parameters from 6866 reflections

$\theta = 4.3$ – 78.8°

$\mu = 1.21$ mm⁻¹

$T = 109$ K

Plate, clear colourless

$0.31 \times 0.07 \times 0.05$ mm

Data collection

Rigaku Oxford Diffraction SuperNova, Dual,
Cu at zero, Pilatus 200K
diffractometer

Radiation source: micro-focus sealed X-ray
tube, SuperNova (Cu) X-ray Source

Mirror monochromator

ω scans

Absorption correction: gaussian
(CrysAlis PRO; Rigaku OD, 2015)

$T_{\min} = 0.792$, $T_{\max} = 0.950$

19900 measured reflections

5029 independent reflections

4084 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.066$

$\theta_{\max} = 80.6^\circ$, $\theta_{\min} = 4.7^\circ$

$h = -15 \rightarrow 12$

$k = -13 \rightarrow 12$

$l = -48 \rightarrow 46$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.073$

$wR(F^2) = 0.198$

$S = 1.05$

5029 reflections

289 parameters

0 restraints

Primary atom site location: dual

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0756P)^2 + 11.1312P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.54$ e Å⁻³

$\Delta\rho_{\min} = -0.67$ e Å⁻³

Special details

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

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
P1	0.76672 (6)	0.54686 (8)	0.62877 (2)	0.0256 (2)
O1	0.29598 (18)	0.6985 (3)	0.63244 (7)	0.0422 (6)
O2	0.92563 (18)	0.7706 (3)	0.49141 (6)	0.0338 (5)
O3	0.96498 (17)	0.8338 (3)	0.75085 (6)	0.0335 (5)
C1	0.6240 (2)	0.5994 (3)	0.62927 (7)	0.0263 (6)
C2	0.5880 (2)	0.7150 (3)	0.61423 (8)	0.0266 (6)

H2	0.638858	0.771017	0.603024	0.032*
C3	0.4788 (2)	0.7505 (3)	0.61522 (8)	0.0277 (6)
C4	0.4056 (2)	0.6640 (3)	0.63115 (8)	0.0292 (7)
C5	0.4387 (2)	0.5500 (3)	0.64752 (8)	0.0294 (6)
C6	0.5494 (2)	0.5181 (3)	0.64638 (8)	0.0273 (6)
H6	0.574042	0.440235	0.657356	0.033*
C7	0.4408 (3)	0.8787 (4)	0.60070 (9)	0.0338 (7)
H7A	0.503947	0.933229	0.595125	0.051*
H7B	0.395514	0.922845	0.618304	0.051*
H7C	0.398309	0.863773	0.579192	0.051*
C8	0.2388 (3)	0.6623 (5)	0.60101 (12)	0.0579 (12)
H8A	0.269918	0.708578	0.580746	0.087*
H8B	0.161910	0.685379	0.603448	0.087*
H8C	0.245547	0.568119	0.597310	0.087*
C9	0.3589 (3)	0.4620 (4)	0.66631 (10)	0.0417 (8)
H9A	0.314595	0.415649	0.648830	0.062*
H9B	0.311580	0.514465	0.681517	0.062*
H9C	0.398694	0.398700	0.680751	0.062*
C11	0.8173 (2)	0.6251 (3)	0.58820 (8)	0.0270 (6)
C12	0.8840 (2)	0.7339 (3)	0.58696 (8)	0.0283 (6)
H12	0.903538	0.775954	0.608390	0.034*
C13	0.9232 (2)	0.7836 (3)	0.55488 (8)	0.0286 (6)
C14	0.8920 (2)	0.7209 (3)	0.52394 (8)	0.0297 (7)
C15	0.8245 (2)	0.6114 (4)	0.52399 (8)	0.0310 (7)
C16	0.7885 (2)	0.5642 (3)	0.55636 (8)	0.0291 (6)
H16	0.743434	0.489078	0.556934	0.035*
C17	0.9947 (3)	0.9028 (4)	0.55430 (9)	0.0379 (8)
H17A	1.071010	0.876368	0.556053	0.057*
H17B	0.976211	0.959229	0.574308	0.057*
H17C	0.983239	0.950390	0.532164	0.057*
C18	1.0338 (3)	0.7319 (4)	0.48189 (9)	0.0376 (8)
H18A	1.055911	0.777853	0.460361	0.056*
H18B	1.035495	0.637713	0.477671	0.056*
H18C	1.083902	0.753749	0.501123	0.056*
C19	0.7907 (3)	0.5467 (4)	0.48979 (8)	0.0385 (8)
H19A	0.854690	0.508952	0.478277	0.058*
H19B	0.757642	0.611649	0.474132	0.058*
H19C	0.737842	0.477731	0.494810	0.058*
C21	0.8248 (2)	0.6460 (3)	0.66423 (8)	0.0264 (6)
C22	0.7673 (2)	0.7393 (3)	0.68323 (7)	0.0261 (6)
H22	0.695207	0.760186	0.676218	0.031*
C23	0.8124 (2)	0.8032 (3)	0.71231 (8)	0.0266 (6)
C24	0.9190 (2)	0.7691 (3)	0.72226 (8)	0.0269 (6)
C25	0.9801 (2)	0.6791 (3)	0.70352 (8)	0.0296 (7)
C26	0.9313 (2)	0.6163 (3)	0.67470 (8)	0.0292 (7)
H26	0.971283	0.552442	0.661997	0.035*
C27	0.7505 (3)	0.9075 (4)	0.73180 (8)	0.0315 (7)
H27A	0.777763	0.993395	0.724809	0.047*

H27B	0.760265	0.895847	0.757295	0.047*
H27C	0.673034	0.901011	0.725968	0.047*
C28	0.9540 (3)	0.7629 (4)	0.78319 (9)	0.0434 (9)
H28A	0.877062	0.743845	0.787437	0.065*
H28B	0.982687	0.815264	0.802728	0.065*
H28C	0.994753	0.681096	0.781545	0.065*
C29	1.0948 (3)	0.6435 (4)	0.71393 (10)	0.0392 (8)
H29A	1.094164	0.561385	0.727137	0.059*
H29B	1.125434	0.712627	0.728759	0.059*
H29C	1.139325	0.633387	0.692632	0.059*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0188 (4)	0.0319 (4)	0.0261 (4)	0.0016 (3)	0.0008 (3)	0.0008 (3)
O1	0.0155 (10)	0.0541 (17)	0.0568 (15)	0.0058 (11)	0.0011 (9)	0.0071 (12)
O2	0.0254 (11)	0.0470 (14)	0.0290 (11)	0.0017 (10)	0.0051 (8)	0.0047 (10)
O3	0.0246 (10)	0.0459 (14)	0.0300 (11)	-0.0056 (10)	-0.0063 (8)	0.0003 (10)
C1	0.0219 (13)	0.0353 (16)	0.0216 (12)	-0.0012 (12)	-0.0018 (10)	-0.0025 (11)
C2	0.0192 (13)	0.0343 (17)	0.0264 (13)	-0.0011 (12)	0.0005 (10)	-0.0007 (12)
C3	0.0214 (13)	0.0370 (17)	0.0247 (13)	0.0002 (12)	-0.0036 (10)	-0.0010 (12)
C4	0.0177 (13)	0.0384 (18)	0.0314 (15)	0.0027 (12)	0.0005 (11)	-0.0023 (13)
C5	0.0213 (14)	0.0367 (17)	0.0302 (14)	-0.0009 (13)	0.0013 (11)	0.0004 (12)
C6	0.0216 (14)	0.0344 (17)	0.0259 (14)	0.0001 (12)	0.0000 (10)	0.0000 (12)
C7	0.0248 (14)	0.0368 (19)	0.0399 (17)	0.0028 (14)	-0.0044 (12)	0.0039 (14)
C8	0.0264 (17)	0.066 (3)	0.081 (3)	-0.0147 (19)	-0.0197 (18)	0.019 (2)
C9	0.0253 (16)	0.045 (2)	0.054 (2)	-0.0017 (16)	0.0080 (14)	0.0120 (17)
C11	0.0175 (12)	0.0344 (17)	0.0290 (14)	0.0053 (12)	0.0005 (10)	0.0010 (12)
C12	0.0201 (13)	0.0359 (18)	0.0289 (14)	0.0036 (12)	0.0032 (11)	-0.0004 (12)
C13	0.0209 (13)	0.0348 (17)	0.0300 (14)	0.0043 (12)	0.0036 (11)	-0.0001 (12)
C14	0.0220 (14)	0.0405 (18)	0.0266 (14)	0.0075 (13)	0.0028 (11)	0.0037 (12)
C15	0.0221 (13)	0.0428 (19)	0.0281 (14)	0.0047 (14)	0.0005 (11)	-0.0003 (13)
C16	0.0196 (13)	0.0340 (18)	0.0336 (15)	-0.0009 (12)	0.0006 (11)	0.0000 (12)
C17	0.0372 (17)	0.044 (2)	0.0320 (16)	-0.0053 (16)	0.0097 (13)	-0.0028 (14)
C18	0.0281 (16)	0.051 (2)	0.0337 (16)	-0.0012 (15)	0.0088 (13)	-0.0014 (15)
C19	0.0308 (16)	0.056 (2)	0.0284 (15)	-0.0071 (16)	-0.0001 (12)	-0.0009 (15)
C21	0.0191 (13)	0.0341 (17)	0.0260 (13)	-0.0004 (12)	-0.0005 (10)	0.0043 (11)
C22	0.0172 (13)	0.0357 (17)	0.0255 (13)	-0.0010 (12)	-0.0016 (10)	0.0037 (12)
C23	0.0177 (13)	0.0347 (17)	0.0275 (14)	-0.0025 (12)	-0.0002 (10)	0.0045 (12)
C24	0.0190 (13)	0.0348 (17)	0.0268 (14)	-0.0039 (12)	-0.0043 (10)	0.0039 (12)
C25	0.0178 (13)	0.0372 (18)	0.0337 (15)	-0.0013 (12)	-0.0028 (11)	0.0061 (13)
C26	0.0188 (13)	0.0357 (18)	0.0330 (15)	0.0055 (13)	0.0009 (11)	0.0028 (12)
C27	0.0211 (13)	0.0410 (19)	0.0324 (15)	-0.0009 (13)	-0.0031 (12)	-0.0031 (13)
C28	0.0354 (17)	0.068 (3)	0.0267 (15)	-0.0023 (18)	-0.0078 (13)	0.0063 (16)
C29	0.0213 (15)	0.048 (2)	0.048 (2)	0.0043 (15)	-0.0091 (13)	0.0041 (16)

Geometric parameters (Å, °)

P1—C1	1.836 (3)	C13—C17	1.507 (5)
P1—C11	1.841 (3)	C14—C15	1.398 (5)
P1—C21	1.829 (3)	C15—C16	1.390 (4)
O1—C4	1.396 (4)	C15—C19	1.513 (4)
O1—C8	1.431 (5)	C16—H16	0.9500
O2—C14	1.395 (4)	C17—H17A	0.9800
O2—C18	1.435 (4)	C17—H17B	0.9800
O3—C24	1.390 (4)	C17—H17C	0.9800
O3—C28	1.431 (4)	C18—H18A	0.9800
C1—C2	1.389 (4)	C18—H18B	0.9800
C1—C6	1.400 (4)	C18—H18C	0.9800
C2—H2	0.9500	C19—H19A	0.9800
C2—C3	1.393 (4)	C19—H19B	0.9800
C3—C4	1.400 (5)	C19—H19C	0.9800
C3—C7	1.500 (5)	C21—C22	1.391 (4)
C4—C5	1.385 (5)	C21—C26	1.403 (4)
C5—C6	1.401 (4)	C22—H22	0.9500
C5—C9	1.513 (5)	C22—C23	1.397 (4)
C6—H6	0.9500	C23—C24	1.408 (4)
C7—H7A	0.9800	C23—C27	1.507 (5)
C7—H7B	0.9800	C24—C25	1.386 (5)
C7—H7C	0.9800	C25—C26	1.402 (4)
C8—H8A	0.9800	C25—C29	1.511 (4)
C8—H8B	0.9800	C26—H26	0.9500
C8—H8C	0.9800	C27—H27A	0.9800
C9—H9A	0.9800	C27—H27B	0.9800
C9—H9B	0.9800	C27—H27C	0.9800
C9—H9C	0.9800	C28—H28A	0.9800
C11—C12	1.386 (5)	C28—H28B	0.9800
C11—C16	1.403 (4)	C28—H28C	0.9800
C12—H12	0.9500	C29—H29A	0.9800
C12—C13	1.403 (4)	C29—H29B	0.9800
C13—C14	1.390 (4)	C29—H29C	0.9800
C1—P1—C11	101.77 (13)	C11—C16—H16	119.3
C21—P1—C1	101.66 (14)	C15—C16—C11	121.4 (3)
C21—P1—C11	103.75 (14)	C15—C16—H16	119.3
C4—O1—C8	112.3 (3)	C13—C17—H17A	109.5
C14—O2—C18	113.3 (2)	C13—C17—H17B	109.5
C24—O3—C28	112.6 (3)	C13—C17—H17C	109.5
C2—C1—P1	123.5 (2)	H17A—C17—H17B	109.5
C2—C1—C6	119.3 (3)	H17A—C17—H17C	109.5
C6—C1—P1	117.2 (2)	H17B—C17—H17C	109.5
C1—C2—H2	119.3	O2—C18—H18A	109.5
C1—C2—C3	121.4 (3)	O2—C18—H18B	109.5
C3—C2—H2	119.3	O2—C18—H18C	109.5

C2—C3—C4	117.7 (3)	H18A—C18—H18B	109.5
C2—C3—C7	121.3 (3)	H18A—C18—H18C	109.5
C4—C3—C7	120.9 (3)	H18B—C18—H18C	109.5
O1—C4—C3	118.4 (3)	C15—C19—H19A	109.5
C5—C4—O1	118.8 (3)	C15—C19—H19B	109.5
C5—C4—C3	122.6 (3)	C15—C19—H19C	109.5
C4—C5—C6	118.0 (3)	H19A—C19—H19B	109.5
C4—C5—C9	121.6 (3)	H19A—C19—H19C	109.5
C6—C5—C9	120.4 (3)	H19B—C19—H19C	109.5
C1—C6—C5	120.8 (3)	C22—C21—P1	124.3 (2)
C1—C6—H6	119.6	C22—C21—C26	118.6 (3)
C5—C6—H6	119.6	C26—C21—P1	116.8 (2)
C3—C7—H7A	109.5	C21—C22—H22	119.0
C3—C7—H7B	109.5	C21—C22—C23	121.9 (3)
C3—C7—H7C	109.5	C23—C22—H22	119.0
H7A—C7—H7B	109.5	C22—C23—C24	117.7 (3)
H7A—C7—H7C	109.5	C22—C23—C27	121.2 (3)
H7B—C7—H7C	109.5	C24—C23—C27	121.1 (3)
O1—C8—H8A	109.5	O3—C24—C23	117.9 (3)
O1—C8—H8B	109.5	C25—C24—O3	119.7 (3)
O1—C8—H8C	109.5	C25—C24—C23	122.2 (3)
H8A—C8—H8B	109.5	C24—C25—C26	118.2 (3)
H8A—C8—H8C	109.5	C24—C25—C29	122.3 (3)
H8B—C8—H8C	109.5	C26—C25—C29	119.4 (3)
C5—C9—H9A	109.5	C21—C26—H26	119.3
C5—C9—H9B	109.5	C25—C26—C21	121.3 (3)
C5—C9—H9C	109.5	C25—C26—H26	119.3
H9A—C9—H9B	109.5	C23—C27—H27A	109.5
H9A—C9—H9C	109.5	C23—C27—H27B	109.5
H9B—C9—H9C	109.5	C23—C27—H27C	109.5
C12—C11—P1	125.4 (2)	H27A—C27—H27B	109.5
C12—C11—C16	118.6 (3)	H27A—C27—H27C	109.5
C16—C11—P1	115.9 (2)	H27B—C27—H27C	109.5
C11—C12—H12	119.1	O3—C28—H28A	109.5
C11—C12—C13	121.7 (3)	O3—C28—H28B	109.5
C13—C12—H12	119.1	O3—C28—H28C	109.5
C12—C13—C17	120.6 (3)	H28A—C28—H28B	109.5
C14—C13—C12	117.8 (3)	H28A—C28—H28C	109.5
C14—C13—C17	121.6 (3)	H28B—C28—H28C	109.5
O2—C14—C15	118.1 (3)	C25—C29—H29A	109.5
C13—C14—O2	119.5 (3)	C25—C29—H29B	109.5
C13—C14—C15	122.3 (3)	C25—C29—H29C	109.5
C14—C15—C19	121.0 (3)	H29A—C29—H29B	109.5
C16—C15—C14	118.1 (3)	H29A—C29—H29C	109.5
C16—C15—C19	120.9 (3)	H29B—C29—H29C	109.5

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C29-H29A\cdots O3^i$	0.98	2.58	3.524 (5)	161

Symmetry code: (i) $-x+2, y-1/2, -z+3/2$.Tris(4-methoxy-3,5-dimethylphenyl(oxo)- λ^5 -phosphane (IV)

Crystal data

 $C_{27}H_{33}O_4P$ $M_r = 452.50$ Orthorhombic, $Pbca$ $a = 11.28601$ (11) \AA $b = 11.90008$ (11) \AA $c = 36.3801$ (3) \AA $V = 4886.01$ (8) \AA^3 $Z = 8$ $F(000) = 1936$ $D_x = 1.230$ Mg m^{-3} Cu $K\alpha$ radiation, $\lambda = 1.54184$ \AA

Cell parameters from 16756 reflections

 $\theta = 4.6-80.0^\circ$ $\mu = 1.24$ mm^{-1} $T = 108$ K

Plate, clear colourless

 $0.2 \times 0.2 \times 0.04$ mm

Data collection

Rigaku Oxford Diffraction SuperNova, Dual,
Cu at zero, Pilatus 200/300K
diffractometerRadiation source: micro-focus sealed X-ray
tube, SuperNova (Cu) X-ray Source

Mirror monochromator

 ω scansAbsorption correction: multi-scan
(CrysAlis PRO; Rigaku OD, 2015) $T_{\min} = 0.755$, $T_{\max} = 1.000$

29719 measured reflections

5325 independent reflections

4821 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.033$ $\theta_{\max} = 80.3^\circ$, $\theta_{\min} = 4.6^\circ$ $h = -14 \rightarrow 13$ $k = -10 \rightarrow 15$ $l = -32 \rightarrow 46$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.039$ $wR(F^2) = 0.100$ $S = 1.05$

5325 reflections

299 parameters

0 restraints

Primary atom site location: iterative

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0444P)^2 + 2.732P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.002$ $\Delta\rho_{\max} = 0.35$ e \AA^{-3} $\Delta\rho_{\min} = -0.40$ e \AA^{-3}

Extinction correction: SHELXL2016

(Sheldrick, 2015b),

 $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.00025 (5)

Special details

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

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
P1	0.79578 (3)	0.57887 (3)	0.63306 (2)	0.01782 (10)
O1	0.82216 (9)	0.45664 (8)	0.63566 (3)	0.0241 (2)

O2	0.27342 (9)	0.65494 (10)	0.63075 (3)	0.0305 (2)
O3	0.93140 (10)	0.76996 (11)	0.48735 (3)	0.0350 (3)
O4	1.03456 (9)	0.84000 (9)	0.75134 (3)	0.0258 (2)
C1	0.63848 (12)	0.60698 (11)	0.63380 (3)	0.0186 (3)
C2	0.59134 (12)	0.70862 (11)	0.62114 (3)	0.0194 (3)
H2	0.643113	0.766482	0.613037	0.023*
C3	0.46935 (12)	0.72582 (11)	0.62030 (4)	0.0209 (3)
C4	0.39523 (12)	0.63846 (12)	0.63188 (4)	0.0220 (3)
C5	0.43964 (12)	0.53723 (12)	0.64567 (4)	0.0239 (3)
C6	0.56272 (12)	0.52309 (11)	0.64628 (4)	0.0209 (3)
H6	0.595138	0.454888	0.655395	0.025*
C7	0.41966 (13)	0.83624 (12)	0.60764 (4)	0.0275 (3)
H7A	0.483170	0.892330	0.606736	0.041*
H7B	0.358199	0.861138	0.624844	0.041*
H7C	0.385164	0.827349	0.583095	0.041*
C8	0.22242 (15)	0.62574 (16)	0.59606 (5)	0.0385 (4)
H8A	0.260981	0.669074	0.576526	0.058*
H8B	0.137480	0.642838	0.596364	0.058*
H8C	0.233832	0.545291	0.591516	0.058*
C9	0.35923 (14)	0.44588 (15)	0.65990 (5)	0.0372 (4)
H9A	0.375698	0.432767	0.685999	0.056*
H9B	0.373292	0.376517	0.646050	0.056*
H9C	0.276449	0.468978	0.656900	0.056*
C11	0.84536 (12)	0.63908 (12)	0.58997 (4)	0.0207 (3)
C12	0.89015 (12)	0.74785 (12)	0.58695 (4)	0.0233 (3)
H12	0.899650	0.792197	0.608462	0.028*
C13	0.92131 (13)	0.79276 (13)	0.55278 (4)	0.0267 (3)
C14	0.90563 (13)	0.72557 (13)	0.52171 (4)	0.0267 (3)
C15	0.85914 (13)	0.61662 (13)	0.52372 (4)	0.0267 (3)
C16	0.83036 (13)	0.57450 (12)	0.55830 (4)	0.0241 (3)
H16	0.799917	0.500336	0.560367	0.029*
C17	0.96890 (17)	0.91105 (14)	0.54979 (4)	0.0359 (4)
H17A	1.055600	0.908816	0.548469	0.054*
H17B	0.944584	0.954406	0.571416	0.054*
H17C	0.937404	0.946656	0.527556	0.054*
C18	1.05371 (17)	0.75999 (18)	0.47752 (5)	0.0439 (4)
H18A	1.069584	0.805621	0.455644	0.066*
H18B	1.072181	0.681140	0.472270	0.066*
H18C	1.103135	0.786379	0.497918	0.066*
C19	0.83794 (16)	0.54874 (16)	0.48934 (4)	0.0367 (4)
H19A	0.789789	0.592591	0.472108	0.055*
H19B	0.796155	0.479203	0.495677	0.055*
H19C	0.914092	0.530334	0.477881	0.055*
C21	0.86395 (12)	0.66018 (11)	0.66909 (3)	0.0192 (3)
C22	0.80829 (11)	0.75201 (11)	0.68533 (4)	0.0198 (3)
H22	0.731414	0.773357	0.677308	0.024*
C23	0.86343 (12)	0.81313 (11)	0.71315 (4)	0.0210 (3)
C24	0.97618 (12)	0.77906 (12)	0.72445 (4)	0.0213 (3)

C25	1.03682 (12)	0.69022 (12)	0.70769 (4)	0.0218 (3)
C26	0.97866 (12)	0.63058 (11)	0.68014 (4)	0.0214 (3)
H26	1.017338	0.568957	0.668667	0.026*
C27	0.80642 (13)	0.91664 (12)	0.72900 (4)	0.0259 (3)
H27A	0.765918	0.897242	0.751965	0.039*
H27B	0.748824	0.946793	0.711416	0.039*
H27C	0.867385	0.973307	0.733934	0.039*
C28	0.99864 (14)	0.81185 (15)	0.78793 (4)	0.0311 (3)
H28A	0.913403	0.825199	0.790633	0.047*
H28B	1.042074	0.858623	0.805539	0.047*
H28C	1.015864	0.732445	0.792677	0.047*
C29	1.16351 (13)	0.66525 (13)	0.71787 (4)	0.0280 (3)
H29A	1.215520	0.722386	0.707106	0.042*
H29B	1.185603	0.590996	0.708450	0.042*
H29C	1.171810	0.666109	0.744687	0.042*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.01791 (16)	0.01810 (17)	0.01746 (16)	0.00087 (12)	0.00009 (11)	-0.00058 (11)
O1	0.0246 (5)	0.0209 (5)	0.0269 (5)	0.0024 (4)	-0.0006 (4)	-0.0009 (4)
O2	0.0181 (5)	0.0398 (6)	0.0336 (5)	0.0018 (4)	-0.0010 (4)	0.0048 (5)
O3	0.0380 (6)	0.0477 (7)	0.0194 (5)	-0.0067 (5)	0.0043 (4)	0.0048 (5)
O4	0.0252 (5)	0.0319 (5)	0.0203 (4)	-0.0070 (4)	-0.0027 (4)	-0.0028 (4)
C1	0.0198 (6)	0.0206 (6)	0.0154 (5)	0.0004 (5)	-0.0007 (4)	-0.0015 (4)
C2	0.0209 (6)	0.0199 (6)	0.0172 (6)	-0.0008 (5)	0.0004 (5)	-0.0003 (5)
C3	0.0229 (6)	0.0226 (6)	0.0173 (6)	0.0022 (5)	-0.0008 (5)	-0.0008 (5)
C4	0.0175 (6)	0.0290 (7)	0.0196 (6)	0.0011 (5)	-0.0015 (5)	-0.0007 (5)
C5	0.0228 (6)	0.0258 (7)	0.0233 (6)	-0.0038 (5)	-0.0025 (5)	0.0017 (5)
C6	0.0224 (6)	0.0200 (6)	0.0205 (6)	-0.0003 (5)	-0.0038 (5)	0.0007 (5)
C7	0.0237 (7)	0.0268 (7)	0.0321 (7)	0.0050 (6)	-0.0006 (6)	0.0037 (6)
C8	0.0268 (8)	0.0465 (10)	0.0423 (9)	-0.0063 (7)	-0.0138 (7)	0.0080 (8)
C9	0.0240 (7)	0.0357 (9)	0.0518 (10)	-0.0061 (6)	-0.0033 (7)	0.0145 (7)
C11	0.0179 (6)	0.0248 (6)	0.0194 (6)	0.0018 (5)	0.0004 (5)	-0.0009 (5)
C12	0.0246 (7)	0.0262 (7)	0.0192 (6)	-0.0005 (5)	0.0023 (5)	-0.0021 (5)
C13	0.0257 (7)	0.0298 (7)	0.0245 (7)	-0.0006 (6)	0.0032 (5)	0.0007 (6)
C14	0.0251 (7)	0.0379 (8)	0.0171 (6)	-0.0003 (6)	0.0027 (5)	0.0025 (6)
C15	0.0239 (7)	0.0362 (8)	0.0200 (6)	-0.0006 (6)	0.0000 (5)	-0.0033 (6)
C16	0.0225 (6)	0.0281 (7)	0.0215 (6)	-0.0009 (5)	0.0003 (5)	-0.0027 (5)
C17	0.0473 (10)	0.0324 (8)	0.0279 (7)	-0.0073 (7)	0.0089 (7)	0.0018 (6)
C18	0.0421 (9)	0.0629 (12)	0.0269 (8)	-0.0124 (9)	0.0125 (7)	-0.0025 (8)
C19	0.0417 (9)	0.0469 (10)	0.0216 (7)	-0.0088 (8)	0.0017 (6)	-0.0075 (7)
C21	0.0203 (6)	0.0204 (6)	0.0168 (6)	-0.0011 (5)	0.0002 (5)	0.0019 (5)
C22	0.0186 (6)	0.0224 (6)	0.0182 (6)	-0.0008 (5)	0.0005 (5)	0.0015 (5)
C23	0.0222 (6)	0.0229 (6)	0.0178 (6)	-0.0020 (5)	0.0017 (5)	0.0016 (5)
C24	0.0233 (6)	0.0231 (6)	0.0175 (6)	-0.0060 (5)	-0.0008 (5)	0.0013 (5)
C25	0.0206 (6)	0.0240 (6)	0.0210 (6)	-0.0016 (5)	-0.0017 (5)	0.0048 (5)
C26	0.0215 (6)	0.0217 (6)	0.0210 (6)	0.0016 (5)	-0.0005 (5)	0.0014 (5)

C27	0.0244 (7)	0.0282 (7)	0.0250 (7)	-0.0010 (6)	0.0022 (5)	-0.0057 (5)
C28	0.0300 (8)	0.0436 (9)	0.0198 (6)	-0.0032 (7)	-0.0021 (6)	-0.0019 (6)
C29	0.0236 (7)	0.0293 (7)	0.0311 (7)	0.0011 (6)	-0.0073 (6)	0.0023 (6)

Geometric parameters (Å, °)

P1—O1	1.4878 (10)	C13—C17	1.511 (2)
P1—C1	1.8066 (14)	C14—C15	1.401 (2)
P1—C11	1.8121 (14)	C15—C16	1.393 (2)
P1—C21	1.8018 (13)	C15—C19	1.508 (2)
O2—C4	1.3893 (16)	C16—H16	0.9500
O2—C8	1.430 (2)	C17—H17A	0.9800
O3—C14	1.3880 (17)	C17—H17B	0.9800
O3—C18	1.431 (2)	C17—H17C	0.9800
O4—C24	1.3844 (16)	C18—H18A	0.9800
O4—C28	1.4314 (17)	C18—H18B	0.9800
C1—C2	1.3993 (18)	C18—H18C	0.9800
C1—C6	1.3906 (19)	C19—H19A	0.9800
C2—H2	0.9500	C19—H19B	0.9800
C2—C3	1.3923 (19)	C19—H19C	0.9800
C3—C4	1.399 (2)	C21—C22	1.3921 (18)
C3—C7	1.5010 (19)	C21—C26	1.4006 (18)
C4—C5	1.398 (2)	C22—H22	0.9500
C5—C6	1.3995 (19)	C22—C23	1.3930 (19)
C5—C9	1.508 (2)	C23—C24	1.3974 (19)
C6—H6	0.9500	C23—C27	1.5046 (19)
C7—H7A	0.9800	C24—C25	1.399 (2)
C7—H7B	0.9800	C25—C26	1.3927 (19)
C7—H7C	0.9800	C25—C29	1.5066 (19)
C8—H8A	0.9800	C26—H26	0.9500
C8—H8B	0.9800	C27—H27A	0.9800
C8—H8C	0.9800	C27—H27B	0.9800
C9—H9A	0.9800	C27—H27C	0.9800
C9—H9B	0.9800	C28—H28A	0.9800
C9—H9C	0.9800	C28—H28B	0.9800
C11—C12	1.394 (2)	C28—H28C	0.9800
C11—C16	1.3952 (19)	C29—H29A	0.9800
C12—H12	0.9500	C29—H29B	0.9800
C12—C13	1.398 (2)	C29—H29C	0.9800
C13—C14	1.396 (2)		
O1—P1—C1	112.13 (6)	C16—C15—C19	121.30 (14)
O1—P1—C11	112.32 (6)	C11—C16—H16	119.4
O1—P1—C21	113.16 (6)	C15—C16—C11	121.29 (14)
C1—P1—C11	104.08 (6)	C15—C16—H16	119.4
C21—P1—C1	108.02 (6)	C13—C17—H17A	109.5
C21—P1—C11	106.57 (6)	C13—C17—H17B	109.5
C4—O2—C8	112.96 (12)	C13—C17—H17C	109.5

C14—O3—C18	113.31 (13)	H17A—C17—H17B	109.5
C24—O4—C28	113.56 (11)	H17A—C17—H17C	109.5
C2—C1—P1	121.92 (10)	H17B—C17—H17C	109.5
C6—C1—P1	118.43 (10)	O3—C18—H18A	109.5
C6—C1—C2	119.62 (12)	O3—C18—H18B	109.5
C1—C2—H2	119.7	O3—C18—H18C	109.5
C3—C2—C1	120.69 (12)	H18A—C18—H18B	109.5
C3—C2—H2	119.7	H18A—C18—H18C	109.5
C2—C3—C4	118.37 (12)	H18B—C18—H18C	109.5
C2—C3—C7	120.33 (13)	C15—C19—H19A	109.5
C4—C3—C7	121.29 (12)	C15—C19—H19B	109.5
O2—C4—C3	118.53 (13)	C15—C19—H19C	109.5
O2—C4—C5	119.15 (13)	H19A—C19—H19B	109.5
C5—C4—C3	122.28 (13)	H19A—C19—H19C	109.5
C4—C5—C6	117.72 (13)	H19B—C19—H19C	109.5
C4—C5—C9	121.92 (13)	C22—C21—P1	122.53 (10)
C6—C5—C9	120.35 (13)	C22—C21—C26	119.52 (12)
C1—C6—C5	121.25 (13)	C26—C21—P1	117.93 (10)
C1—C6—H6	119.4	C21—C22—H22	119.4
C5—C6—H6	119.4	C21—C22—C23	121.11 (12)
C3—C7—H7A	109.5	C23—C22—H22	119.4
C3—C7—H7B	109.5	C22—C23—C24	117.98 (12)
C3—C7—H7C	109.5	C22—C23—C27	120.98 (12)
H7A—C7—H7B	109.5	C24—C23—C27	120.94 (12)
H7A—C7—H7C	109.5	O4—C24—C23	119.30 (12)
H7B—C7—H7C	109.5	O4—C24—C25	118.09 (12)
O2—C8—H8A	109.5	C23—C24—C25	122.43 (12)
O2—C8—H8B	109.5	C24—C25—C29	120.40 (13)
O2—C8—H8C	109.5	C26—C25—C24	117.92 (12)
H8A—C8—H8B	109.5	C26—C25—C29	121.58 (13)
H8A—C8—H8C	109.5	C21—C26—H26	119.5
H8B—C8—H8C	109.5	C25—C26—C21	120.94 (13)
C5—C9—H9A	109.5	C25—C26—H26	119.5
C5—C9—H9B	109.5	C23—C27—H27A	109.5
C5—C9—H9C	109.5	C23—C27—H27B	109.5
H9A—C9—H9B	109.5	C23—C27—H27C	109.5
H9A—C9—H9C	109.5	H27A—C27—H27B	109.5
H9B—C9—H9C	109.5	H27A—C27—H27C	109.5
C12—C11—P1	123.18 (10)	H27B—C27—H27C	109.5
C12—C11—C16	119.37 (13)	O4—C28—H28A	109.5
C16—C11—P1	117.33 (11)	O4—C28—H28B	109.5
C11—C12—H12	119.5	O4—C28—H28C	109.5
C11—C12—C13	121.08 (13)	H28A—C28—H28B	109.5
C13—C12—H12	119.5	H28A—C28—H28C	109.5
C12—C13—C17	120.64 (13)	H28B—C28—H28C	109.5
C14—C13—C12	117.98 (14)	C25—C29—H29A	109.5
C14—C13—C17	121.38 (13)	C25—C29—H29B	109.5
O3—C14—C13	119.00 (14)	C25—C29—H29C	109.5

O3—C14—C15	118.55 (13)	H29A—C29—H29B	109.5
C13—C14—C15	122.38 (13)	H29A—C29—H29C	109.5
C14—C15—C19	120.79 (13)	H29B—C29—H29C	109.5
C16—C15—C14	117.89 (13)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C2—H2 \cdots O1 ⁱ	0.95	2.44	3.1533 (16)	132
C7—H7A \cdots O1 ⁱ	0.98	2.55	3.4033 (18)	145

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