

## 4-Bromobenzoylmethylene–triphenylphosphorane ylide

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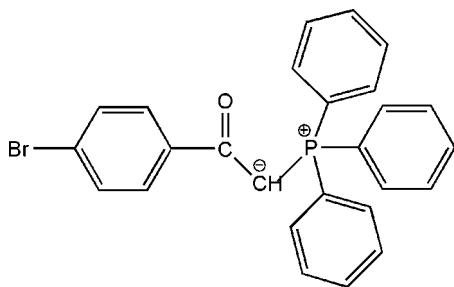
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Key indicators: single-crystal X-ray study;  $T = 128$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.027;  $wR$  factor = 0.059; data-to-parameter ratio = 17.0.

In the molecule of the title compound, (4-bromobenzoyl)-(triphenylphosphonio)methanide,  $\text{C}_{26}\text{H}_{20}\text{BrOP}$ , the geometry around the P atom is nearly tetrahedral and the O atom is oriented *cis* to the P atom. The bromophenyl ring of the benzoyl group is twisted with respect to the plane of the carbonyl group through an angle of  $3.83$  ( $3$ )°.

### Related literature

For general background, see: Allen *et al.* (1987); Dunitz (1979). For related literature, see: Bart (1969).



### Experimental

#### Crystal data

$\text{C}_{26}\text{H}_{20}\text{BrOP}$

$M_r = 459.3$

Monoclinic,  $C2/c$

$a = 16.6819$  (17) Å

$b = 10.9590$  (8) Å

$c = 23.718$  (2) Å

$\beta = 98.983$  (8)°  
 $V = 4282.8$  (7) Å<sup>3</sup>  
 $Z = 8$   
 Mo  $K\alpha$  radiation

$\mu = 2.01$  mm<sup>-1</sup>  
 $T = 128$  (2) K  
 $0.3 \times 0.2 \times 0.05$  mm

#### Data collection

Stoe IPDS II diffractometer  
 Absorption correction: numerical  
 shape of crystal determined  
 optically (*X-SHAPE*; Stoe &  
 Cie, 2005)  
 $T_{\min} = 0.62$ ,  $T_{\max} = 0.91$

16156 measured reflections  
 4447 independent reflections  
 4025 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.027$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.028$   
 $wR(F^2) = 0.059$   
 $S = 1.12$   
 4447 reflections

262 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.39$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.24$  e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å, °).

|            |             |           |             |
|------------|-------------|-----------|-------------|
| C7—O1      | 1.256 (2)   | C9—P1     | 1.8101 (18) |
| C7—C8      | 1.401 (2)   | C15—P1    | 1.8204 (18) |
| C8—P1      | 1.7194 (17) |           |             |
| C8—P1—C21  | 105.31 (8)  | C9—P1—C15 | 105.02 (8)  |
| C8—P1—C9   | 114.25 (9)  | P1—C8—C7  | 120.37 (13) |
| C21—P1—C9  | 107.98 (8)  | O1—C7—C8  | 123.21 (16) |
| C8—P1—C15  | 116.12 (9)  | C4—C7—C8  | 119.01 (15) |
| C21—P1—C15 | 107.81 (8)  | O1—C7—C4  | 117.75 (15) |

Data collection: *X-Area* (Stoe & Cie, 2005); cell refinement: *X-Area*; data reduction: *X-Red* (Stoe & Cie, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2238).

### References

- Allen, F. H., Kennard, O., Waston, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.  
 Bart, J. C. J. (1969). *J. Chem. Soc. B*, pp. 350–365.  
 Dunitz, J. D. (1979). *X-ray Analysis and the Structure of Organic Molecules*. Ithaca: Cornell University Press.  
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.  
 Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.  
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.  
 Stoe & Cie (2005). *X-Area* (Version 1.31), *X-Red* (Version 1.28b) and *X-SHAPE* (Version 2.05). Stoe & Cie, Darmstadt, Germany.

**supplementary materials**

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## 4-Bromobenzoylmethylene-triphenylphosphorane ylide

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### Comment

Phosphoranes of the type  $(C_6H_5)_3PCHCOC_6H_4Br$  (BBPPY) can coordinate to metals through either C or O atoms. The crystal and molecular structure of this ylide with space group  $P2_1/c$ , was determined successfully (Bart, 1969). The structural investigation with metrical parameters for the title compound, (I), show that how they vary with a change in delocalization in the metal derivatives, as well as in other resonance-stabilized ylides.

In the molecule of the title compound, (I), (Fig. 1), the bond lengths and angles (Table 1) are generally within normal ranges (Allen *et al.*, 1987).

The P1—C8 [1.719 (17) Å] bond is shorter than the other P—C bonds (Table 1) and longer than the equivalent bond lengths of 1.66 Å reported for methylenetriphenylphosphorane (Bart, 1969), which shows partial double-bond character for these two bonds. The C7—O1 [1.256 (2) Å] double bond is shorter than the C=O bonds in ketons (1.331 Å; Allen *et al.*, 1987). These bond distances suggest resonance decolorization in the molecule (Fig. 2). The resonance formulation is supported by the near planarity of P1, C8, O1 and C7 in (I). The torsion angle O1—C7—C8—P1 [2.8 (3)°] also indicates the resonance.

The bromophenyl ring of the benzoyl group is twisted with respect to the plane of the carbonyl group through an angle of 3.83 (3)°. The C7—C8—P1 [120.37 (13)°] bond angle indicates a distorted trigonal arrangement about C8. The P1···O1 [2.990 (3) Å] distance is significantly shorter than the sum of the van der Waals radii of P and O (3.3 Å; Dunitz, 1979), indicating a strong intramolecular interaction between  $P^+$  and  $O^-$  charge centers, which leads to the *cis* orientation.

### Experimental

The title compound was prepared by addition of 2,4-Bromophenyl acetophenone (278 mg, 1 mmol) in chloroform (20 ml) to a solution of triphenylphosphine (262 mg, 1 mmol) in the same solvent (5 ml). The resulting pale yellow solution was stirred for 10 h, and then concentrated under reduced pressure to 5 ml, and diethyl ether (20 ml) was added. The yellow solid formed was filtered off, washed with petroleum benzene (10 ml), and then dried under reduced pressure. In order to get the final product, all of the crude solid, was transferred to an alkaline solution of NaOH (5%) and stirred at 310 K for about 14 h, yielding the white precipitate. The product was washed several times with distilled water and air dried. The resulting solid was recrystallized from an acetonitrile-diethyl ether mixture (5:15) (yield; 436 mg, 95%, m.p. 465–467 K).

### Refinement

H atoms were positioned geometrically, with C—H = 0.93 Å for aromatic and methine H, respectively, and constrained to ride on their parent atoms, with  $U_{iso}(H) = 1.2U_{eq}(C)$ .

## Figures

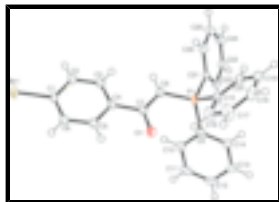


Fig. 1. The molecular structure of the title molecule with the atom-numbering scheme. Displacement ellipsoids are drawn at 30% probability level.



Fig. 2. Resonance in the BBPPY.

## (4-bromobenzoyl)(triphenylphosphonio)methanide

### Crystal data

|                                |   |
|--------------------------------|---|
| $C_{26}H_{20}BrOP$             | $Z = 8$                                 |
| $M_r = 459.3$                  | $F_{000} = 1872$                        |
| Monoclinic, $C2/c$             | $D_x = 1.425 \text{ Mg m}^{-3}$         |
| Hall symbol: $-C 2yc$          | Mo $K\alpha$ radiation                  |
| $a = 16.6819 (17) \text{ \AA}$ | $\lambda = 0.71073 \text{ \AA}$         |
| $b = 10.9590 (8) \text{ \AA}$  | $\theta = 1.7\text{--}26.8^\circ$       |
| $c = 23.718 (2) \text{ \AA}$   | $\mu = 2.01 \text{ mm}^{-1}$            |
| $\beta = 98.983 (8)^\circ$     | $T = 128 (2) \text{ K}$                 |
| $V = 4282.8 (7) \text{ \AA}^3$ | Plate, colorless                        |
|                                | $0.3 \times 0.2 \times 0.05 \text{ mm}$ |

### Data collection

|  |                                    |
|--|------------------------------------|
| Stoe IPDS II diffractometer  | $R_{\text{int}} = 0.027$           |
| rotation method scans  | $\theta_{\text{max}} = 26.8^\circ$ |
| Absorption correction: numerical shape of crystal determined optically | $\theta_{\text{min}} = 1.7^\circ$  |
| $T_{\text{min}} = 0.62, T_{\text{max}} = 0.91$                         | $h = -21 \rightarrow 21$           |
| 16156 measured reflections   | $k = -13 \rightarrow 13$           |
| 4447 independent reflections   | $l = -29 \rightarrow 30$           |
| 4025 reflections with $I > 2\sigma(I)$                                 |                                    |

### Refinement

|                                 |   |
|---------------------------------|---|
| Refinement on $F^2$             | H-atom parameters constrained                     |
| Least-squares matrix: full      | $w = 1/[\sigma^2(F_o^2) + (0.0207P)^2 + 5.8973P]$ |
|                                 | where $P = (F_o^2 + 2F_c^2)/3$                    |
| $R[F^2 > 2\sigma(F^2)] = 0.028$ | $(\Delta/\sigma)_{\text{max}} = 0.015$            |

$wR(F^2) = 0.059$

$S = 1.12$

4447 reflections

262 parameters

$\Delta\rho_{\max} = 0.39 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.24 \text{ e } \text{\AA}^{-3}$

Extinction correction: none

*Special details*

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

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|     | <i>x</i>      | <i>y</i>      | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|---------------|---------------|----------------------------------|
| C1  | -0.16258 (10) | -0.03950 (17) | 0.14125 (7)   | 0.0192 (3)                       |
| C2  | -0.21774 (11) | 0.05592 (17)  | 0.13165 (8)   | 0.0218 (4)                       |
| H2  | -0.223        | 0.1118        | 0.1604        | 0.026*                           |
| C3  | -0.26484 (10) | 0.06607 (16)  | 0.07828 (8)   | 0.0205 (4)                       |
| H3  | -0.3019       | 0.1298        | 0.0712        | 0.025*                           |
| C4  | -0.25771 (10) | -0.01771 (15) | 0.03477 (7)   | 0.0170 (3)                       |
| C5  | -0.20215 (10) | -0.11313 (16) | 0.04636 (8)   | 0.0206 (4)                       |
| H5  | -0.197        | -0.1697       | 0.0179        | 0.025*                           |
| C6  | -0.15460 (11) | -0.12498 (16) | 0.09947 (8)   | 0.0212 (4)                       |
| H6  | -0.118        | -0.1892       | 0.1069        | 0.025*                           |
| C7  | -0.30930 (10) | -0.01368 (16) | -0.02367 (7)  | 0.0186 (3)                       |
| C8  | -0.36518 (11) | 0.08152 (16)  | -0.03652 (7)  | 0.0197 (4)                       |
| H8  | -0.3671       | 0.1443        | -0.0104       | 0.024*                           |
| C9  | -0.48997 (10) | -0.05477 (16) | -0.11460 (7)  | 0.0188 (3)                       |
| C10 | -0.49752 (11) | -0.13549 (16) | -0.07044 (8)  | 0.0217 (4)                       |
| H10 | -0.4711       | -0.1196       | -0.0337       | 0.026*                           |
| C11 | -0.54467 (12) | -0.23993 (17) | -0.08151 (9)  | 0.0266 (4)                       |
| H11 | -0.5495       | -0.2943       | -0.0521       | 0.032*                           |
| C12 | -0.58459 (13) | -0.26341 (18) | -0.13623 (10) | 0.0308 (5)                       |
| H12 | -0.6158       | -0.3337       | -0.1434       | 0.037*                           |
| C13 | -0.57818 (13) | -0.18271 (19) | -0.18017 (9)  | 0.0319 (5)                       |
| H13 | -0.6056       | -0.1981       | -0.2167       | 0.038*                           |
| C14 | -0.53068 (12) | -0.07852 (17) | -0.16958 (8)  | 0.0255 (4)                       |
| H14 | -0.526        | -0.0245       | -0.1991       | 0.031*                           |
| C15 | -0.38403 (11) | 0.10728 (17)  | -0.16402 (7)  | 0.0199 (4)                       |
| C16 | -0.32349 (13) | 0.0264 (2)    | -0.17531 (9)  | 0.0333 (5)                       |
| H16 | -0.3085       | -0.0391       | -0.151        | 0.04*                            |
| C17 | -0.28599 (14) | 0.0444 (2)    | -0.22271 (9)  | 0.0386 (5)                       |
| H17 | -0.2455       | -0.0091       | -0.23         | 0.046*                           |
| C18 | -0.30800 (12) | 0.1410 (2)    | -0.25940 (8)  | 0.0306 (4)                       |
| H18 | -0.2821       | 0.1528        | -0.2909       | 0.037*                           |
| C19 | -0.36853 (12) | 0.21988 (18)  | -0.24907 (8)  | 0.0264 (4)                       |
| H19 | -0.384        | 0.2841        | -0.274        | 0.032*                           |

## supplementary materials

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|     |                |                |                |             |
|-----|----------------|----------------|----------------|-------------|
| C20 | -0.40652 (11)  | 0.20340 (17)   | -0.20119 (8)   | 0.0229 (4)  |
| H20 | -0.447         | 0.257          | -0.1942        | 0.027*      |
| C21 | -0.49912 (10)  | 0.20790 (15)   | -0.09477 (7)   | 0.0162 (3)  |
| C22 | -0.58242 (11)  | 0.18995 (16)   | -0.09849 (7)   | 0.0195 (4)  |
| H22 | -0.6044        | 0.1126         | -0.1062        | 0.023*      |
| C23 | -0.63283 (11)  | 0.28770 (17)   | -0.09072 (8)   | 0.0231 (4)  |
| H23 | -0.6884        | 0.2754         | -0.0929        | 0.028*      |
| C24 | -0.60041 (12)  | 0.40320 (17)   | -0.07974 (9)   | 0.0269 (4)  |
| H24 | -0.6343        | 0.4684         | -0.0749        | 0.032*      |
| C25 | -0.51717 (12)  | 0.42187 (17)   | -0.07595 (9)   | 0.0274 (4)  |
| H25 | -0.4955        | 0.4995         | -0.0685        | 0.033*      |
| C26 | -0.46648 (11)  | 0.32434 (17)   | -0.08331 (8)   | 0.0225 (4)  |
| H26 | -0.4108        | 0.3366         | -0.0806        | 0.027*      |
| O1  | -0.30126 (8)   | -0.09884 (12)  | -0.05778 (5)   | 0.0239 (3)  |
| P1  | -0.43030 (3)   | 0.08289 (4)    | -0.100322 (18) | 0.01607 (9) |
| Br1 | -0.094213 (11) | -0.053119 (18) | 0.213126 (8)   | 0.02670 (6) |

### Atomic displacement parameters ( $\text{\AA}^2$ )

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$     | $U^{23}$      |
|-----|-------------|-------------|-------------|--------------|--------------|---------------|
| C1  | 0.0132 (8)  | 0.0252 (9)  | 0.0184 (8)  | -0.0027 (7)  | -0.0002 (7)  | 0.0059 (7)    |
| C2  | 0.0185 (8)  | 0.0251 (9)  | 0.0217 (9)  | 0.0012 (7)   | 0.0029 (7)   | -0.0032 (7)   |
| C3  | 0.0168 (8)  | 0.0206 (9)  | 0.0238 (9)  | 0.0041 (7)   | 0.0019 (7)   | -0.0009 (7)   |
| C4  | 0.0128 (8)  | 0.0178 (8)  | 0.0206 (8)  | 0.0005 (6)   | 0.0030 (7)   | 0.0013 (7)    |
| C5  | 0.0191 (8)  | 0.0189 (9)  | 0.0239 (9)  | 0.0019 (7)   | 0.0038 (7)   | -0.0013 (7)   |
| C6  | 0.0166 (8)  | 0.0196 (9)  | 0.0270 (9)  | 0.0029 (7)   | 0.0018 (7)   | 0.0036 (7)    |
| C7  | 0.0172 (8)  | 0.0192 (8)  | 0.0196 (8)  | 0.0024 (7)   | 0.0035 (7)   | 0.0003 (7)    |
| C8  | 0.0201 (8)  | 0.0209 (9)  | 0.0173 (8)  | 0.0048 (7)   | 0.0007 (7)   | -0.0029 (7)   |
| C9  | 0.0188 (8)  | 0.0165 (8)  | 0.0218 (8)  | 0.0050 (7)   | 0.0059 (7)   | -0.0024 (7)   |
| C10 | 0.0217 (9)  | 0.0220 (9)  | 0.0227 (9)  | 0.0069 (7)   | 0.0071 (7)   | 0.0001 (7)    |
| C11 | 0.0280 (10) | 0.0193 (9)  | 0.0353 (11) | 0.0059 (7)   | 0.0134 (9)   | 0.0034 (8)    |
| C12 | 0.0335 (11) | 0.0168 (9)  | 0.0437 (12) | -0.0006 (8)  | 0.0109 (10)  | -0.0078 (8)   |
| C13 | 0.0382 (11) | 0.0265 (10) | 0.0297 (10) | -0.0024 (9)  | 0.0013 (9)   | -0.0098 (8)   |
| C14 | 0.0339 (10) | 0.0207 (9)  | 0.0219 (9)  | 0.0009 (8)   | 0.0042 (8)   | -0.0016 (7)   |
| C15 | 0.0182 (8)  | 0.0250 (9)  | 0.0166 (8)  | -0.0006 (7)  | 0.0035 (7)   | -0.0023 (7)   |
| C16 | 0.0340 (11) | 0.0404 (12) | 0.0278 (10) | 0.0145 (9)   | 0.0120 (9)   | 0.0065 (9)    |
| C17 | 0.0334 (11) | 0.0527 (14) | 0.0335 (11) | 0.0175 (10)  | 0.0175 (10)  | 0.0046 (10)   |
| C18 | 0.0278 (10) | 0.0449 (12) | 0.0210 (9)  | -0.0017 (9)  | 0.0098 (8)   | -0.0014 (9)   |
| C19 | 0.0305 (10) | 0.0293 (10) | 0.0198 (9)  | -0.0027 (8)  | 0.0050 (8)   | 0.0007 (8)    |
| C20 | 0.0233 (9)  | 0.0257 (9)  | 0.0199 (9)  | 0.0020 (7)   | 0.0047 (8)   | -0.0028 (7)   |
| C21 | 0.0179 (8)  | 0.0181 (8)  | 0.0131 (7)  | 0.0045 (6)   | 0.0037 (6)   | 0.0017 (6)    |
| C22 | 0.0196 (8)  | 0.0194 (9)  | 0.0200 (8)  | 0.0002 (7)   | 0.0045 (7)   | 0.0012 (7)    |
| C23 | 0.0164 (8)  | 0.0270 (10) | 0.0270 (9)  | 0.0030 (7)   | 0.0073 (8)   | 0.0025 (7)    |
| C24 | 0.0270 (10) | 0.0214 (9)  | 0.0350 (10) | 0.0096 (8)   | 0.0134 (9)   | 0.0042 (8)    |
| C25 | 0.0271 (10) | 0.0168 (9)  | 0.0404 (11) | 0.0004 (7)   | 0.0117 (9)   | -0.0003 (8)   |
| C26 | 0.0178 (8)  | 0.0226 (9)  | 0.0282 (9)  | 0.0008 (7)   | 0.0069 (8)   | 0.0020 (7)    |
| O1  | 0.0240 (7)  | 0.0242 (7)  | 0.0228 (6)  | 0.0074 (5)   | 0.0010 (5)   | -0.0053 (5)   |
| P1  | 0.0158 (2)  | 0.0174 (2)  | 0.0152 (2)  | 0.00392 (16) | 0.00301 (17) | -0.00050 (16) |

Br1                    0.02069 (10)      0.03603 (11)      0.02137 (9)      0.00387 (8)      -0.00301 (7)      0.00497 (8)

*Geometric parameters (Å, °)*

|           |             |             |             |
|-----------|-------------|-------------|-------------|
| C1—C6     | 1.385 (3)   | C13—H13     | 0.93        |
| C1—C2     | 1.388 (2)   | C14—H14     | 0.93        |
| C1—Br1    | 1.9032 (17) | C15—C20     | 1.387 (3)   |
| C2—C3     | 1.386 (2)   | C15—C16     | 1.401 (3)   |
| C2—H2     | 0.93        | C15—P1      | 1.8204 (18) |
| C3—C4     | 1.400 (2)   | C16—C17     | 1.383 (3)   |
| C3—H3     | 0.93        | C16—H16     | 0.93        |
| C4—C5     | 1.396 (2)   | C17—C18     | 1.383 (3)   |
| C4—C7     | 1.514 (2)   | C17—H17     | 0.93        |
| C5—C6     | 1.386 (2)   | C18—C19     | 1.380 (3)   |
| C5—H5     | 0.93        | C18—H18     | 0.93        |
| C6—H6     | 0.93        | C19—C20     | 1.396 (3)   |
| C7—O1     | 1.256 (2)   | C19—H19     | 0.93        |
| C7—C8     | 1.401 (2)   | C20—H20     | 0.93        |
| C8—P1     | 1.7194 (17) | C21—C22     | 1.393 (2)   |
| C8—H8     | 0.93        | C21—C26     | 1.397 (3)   |
| C9—C10    | 1.392 (3)   | C21—P1      | 1.8054 (17) |
| C9—C14    | 1.397 (3)   | C22—C23     | 1.392 (2)   |
| C9—P1     | 1.8101 (18) | C22—H22     | 0.93        |
| C10—C11   | 1.390 (3)   | C23—C24     | 1.385 (3)   |
| C10—H10   | 0.93        | C23—H23     | 0.93        |
| C11—C12   | 1.387 (3)   | C24—C25     | 1.393 (3)   |
| C11—H11   | 0.93        | C24—H24     | 0.93        |
| C12—C13   | 1.384 (3)   | C25—C26     | 1.391 (3)   |
| C12—H12   | 0.93        | C25—H25     | 0.93        |
| C13—C14   | 1.390 (3)   | C26—H26     | 0.93        |
| C6—C1—C2  | 121.84 (16) | C16—C15—P1  | 118.53 (14) |
| C6—C1—Br1 | 118.47 (13) | C17—C16—C15 | 119.76 (19) |
| C2—C1—Br1 | 119.68 (14) | C17—C16—H16 | 120.1       |
| C3—C2—C1  | 118.49 (17) | C15—C16—H16 | 120.1       |
| C3—C2—H2  | 120.8       | C16—C17—C18 | 120.7 (2)   |
| C1—C2—H2  | 120.8       | C16—C17—H17 | 119.6       |
| C2—C3—C4  | 121.27 (16) | C18—C17—H17 | 119.6       |
| C2—C3—H3  | 119.4       | C19—C18—C17 | 119.83 (19) |
| C4—C3—H3  | 119.4       | C19—C18—H18 | 120.1       |
| C5—C4—C3  | 118.45 (16) | C17—C18—H18 | 120.1       |
| C5—C4—C7  | 117.86 (15) | C18—C19—C20 | 120.08 (18) |
| C3—C4—C7  | 123.65 (15) | C18—C19—H19 | 120         |
| C6—C5—C4  | 121.16 (17) | C20—C19—H19 | 120         |
| C6—C5—H5  | 119.4       | C15—C20—C19 | 120.21 (18) |
| C4—C5—H5  | 119.4       | C15—C20—H20 | 119.9       |
| C1—C6—C5  | 118.79 (16) | C19—C20—H20 | 119.9       |
| C1—C6—H6  | 120.6       | C22—C21—C26 | 119.74 (16) |
| C5—C6—H6  | 120.6       | C22—C21—P1  | 121.86 (13) |
| O1—C7—C8  | 123.21 (16) | C26—C21—P1  | 118.31 (13) |

## supplementary materials

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|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| O1—C7—C4        | 117.75 (15)  | C23—C22—C21     | 120.03 (17)  |
| C8—C7—C4        | 119.01 (15)  | C23—C22—H22     | 120          |
| C7—C8—P1        | 120.37 (13)  | C21—C22—H22     | 120          |
| C7—C8—H8        | 119.8        | C24—C23—C22     | 120.13 (17)  |
| P1—C8—H8        | 119.8        | C24—C23—H23     | 119.9        |
| C10—C9—C14      | 119.74 (17)  | C22—C23—H23     | 119.9        |
| C10—C9—P1       | 120.05 (14)  | C23—C24—C25     | 120.17 (17)  |
| C14—C9—P1       | 120.18 (14)  | C23—C24—H24     | 119.9        |
| C11—C10—C9      | 119.75 (18)  | C25—C24—H24     | 119.9        |
| C11—C10—H10     | 120.1        | C26—C25—C24     | 119.92 (17)  |
| C9—C10—H10      | 120.1        | C26—C25—H25     | 120          |
| C12—C11—C10     | 120.27 (18)  | C24—C25—H25     | 120          |
| C12—C11—H11     | 119.9        | C25—C26—C21     | 120.01 (17)  |
| C10—C11—H11     | 119.9        | C25—C26—H26     | 120          |
| C13—C12—C11     | 120.26 (18)  | C21—C26—H26     | 120          |
| C13—C12—H12     | 119.9        | C8—P1—C21       | 105.31 (8)   |
| C11—C12—H12     | 119.9        | C8—P1—C9        | 114.25 (9)   |
| C12—C13—C14     | 119.86 (19)  | C21—P1—C9       | 107.98 (8)   |
| C12—C13—H13     | 120.1        | C8—P1—C15       | 116.12 (9)   |
| C14—C13—H13     | 120.1        | C21—P1—C15      | 107.81 (8)   |
| C13—C14—C9      | 120.11 (18)  | C9—P1—C15       | 105.02 (8)   |
| C13—C14—H14     | 119.9        | P1—C8—C7        | 120.37 (13)  |
| C9—C14—H14      | 119.9        | O1—C7—C8        | 123.21 (16)  |
| C20—C15—C16     | 119.38 (17)  | C4—C7—C8        | 119.01 (15)  |
| C20—C15—P1      | 122.09 (14)  | O1—C7—C4        | 117.75 (15)  |
| C6—C1—C2—C3     | -1.1 (3)     | C18—C19—C20—C15 | -0.5 (3)     |
| Br1—C1—C2—C3    | 177.82 (13)  | C26—C21—C22—C23 | -0.1 (3)     |
| C1—C2—C3—C4     | 0.2 (3)      | P1—C21—C22—C23  | 176.30 (14)  |
| C2—C3—C4—C5     | 0.4 (3)      | C21—C22—C23—C24 | 0.5 (3)      |
| C2—C3—C4—C7     | 177.96 (17)  | C22—C23—C24—C25 | -0.5 (3)     |
| C3—C4—C5—C6     | -0.3 (3)     | C23—C24—C25—C26 | 0.1 (3)      |
| C7—C4—C5—C6     | -178.00 (16) | C24—C25—C26—C21 | 0.3 (3)      |
| C2—C1—C6—C5     | 1.2 (3)      | C22—C21—C26—C25 | -0.3 (3)     |
| Br1—C1—C6—C5    | -177.73 (13) | P1—C21—C26—C25  | -176.85 (15) |
| C4—C5—C6—C1     | -0.5 (3)     | C7—C8—P1—C21    | 172.54 (15)  |
| C5—C4—C7—O1     | 2.5 (2)      | C7—C8—P1—C9     | 54.22 (18)   |
| C3—C4—C7—O1     | -175.06 (17) | C7—C8—P1—C15    | -68.29 (18)  |
| C5—C4—C7—C8     | -179.62 (17) | C22—C21—P1—C8   | -119.40 (15) |
| C3—C4—C7—C8     | 2.9 (3)      | C26—C21—P1—C8   | 57.05 (16)   |
| O1—C7—C8—P1     | 2.8 (3)      | C22—C21—P1—C9   | 3.05 (16)    |
| C4—C7—C8—P1     | -174.97 (13) | C26—C21—P1—C9   | 179.50 (14)  |
| C14—C9—C10—C11  | 0.8 (3)      | C22—C21—P1—C15  | 116.03 (15)  |
| P1—C9—C10—C11   | 178.81 (14)  | C26—C21—P1—C15  | -67.52 (16)  |
| C9—C10—C11—C12  | -0.5 (3)     | C10—C9—P1—C8    | 17.02 (17)   |
| C10—C11—C12—C13 | -0.4 (3)     | C14—C9—P1—C8    | -164.99 (14) |
| C11—C12—C13—C14 | 0.8 (3)      | C10—C9—P1—C21   | -99.76 (15)  |
| C12—C13—C14—C9  | -0.5 (3)     | C14—C9—P1—C21   | 78.22 (16)   |
| C10—C9—C14—C13  | -0.3 (3)     | C10—C9—P1—C15   | 145.40 (14)  |
| P1—C9—C14—C13   | -178.34 (15) | C14—C9—P1—C15   | -36.62 (16)  |



## supplementary materials

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|                 |              |                |              |
|-----------------|--------------|----------------|--------------|
| C20—C15—C16—C17 | 1.1 (3)      | C20—C15—P1—C8  | -121.99 (16) |
| P1—C15—C16—C17  | -178.53 (18) | C16—C15—P1—C8  | 57.58 (18)   |
| C15—C16—C17—C18 | -0.4 (4)     | C20—C15—P1—C21 | -4.19 (18)   |
| C16—C17—C18—C19 | -0.6 (4)     | C16—C15—P1—C21 | 175.38 (16)  |
| C17—C18—C19—C20 | 1.1 (3)      | C20—C15—P1—C9  | 110.76 (16)  |
| C16—C15—C20—C19 | -0.6 (3)     | C16—C15—P1—C9  | -69.66 (17)  |
| P1—C15—C20—C19  | 178.96 (14)  |                |              |

Fig. 1

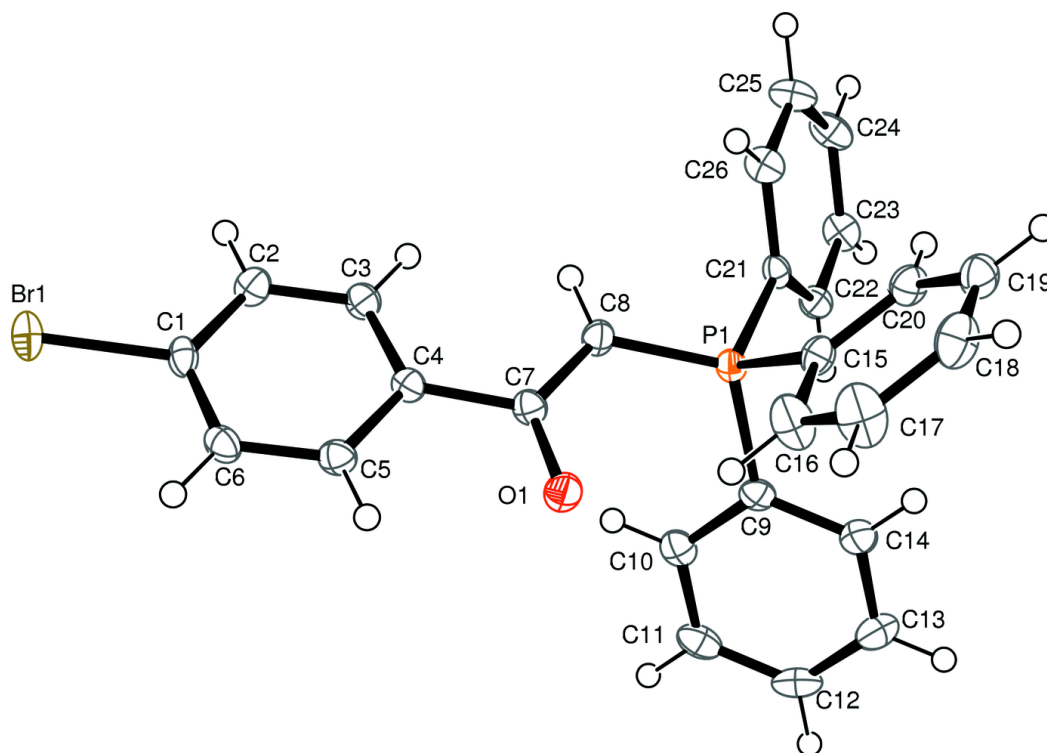


Fig. 2

