X-ray Structure Analysis Online

# Crystal Structure of 4′-Chlorobenzoylmethylenetriphenylphosphorane ylide, $C_{26}H_{20}ClOP$

**Seyyed Javad Sabounchei,**[*†] **Hassan Nemattalab,**[*] **and Hamid Reza Khavasi**[**]

**Faculty of Chemistry, Bu-Ali Sina University, Hamedan, 65174, Iran*
***Department of Chemistry, Shahid Beheshti University, Evin, Tehran, 1983963113, Iran*

The crystal structure of a colorless phosphorus ylide, $Ph_3PCHCOC_6H_4Cl$, was determined by the X-ray diffraction method. All atoms occupy general positions in the monoclinic space group, $C2/c$, and have the following unit-cell dimensions: $a = 16.6184(9)$Å, $b = 10.9196(6)$Å, $c = 23.5465(12)$Å, $\beta = 100.366(4)^\circ$, $Z = 8$ and $V = 4203.2(4)$Å$^3$.

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Phosphorus ylides are important reagents in organic chemistry, especially in the synthesis of naturally occurring products with biological and pharmacological activities.[1] The chemistry of organic phosphorus compounds has shown remarkable growth throughout the past 5 decades. Meanwhile, it has attracted much interest, especially concerning extensive utilization of the organophosphorus derivatives as plasticizers for synthetics, extraction agents, oxidation inhibitors for lubricants, flotation agents, complexing agents for transition metals, and insecticides.[2,3]

Preparation: This compound was prepared according to a published method.[4] Colorless crystals of $Ph_3PCHCOC_6H_4Cl$ (in Fig. 1) were obtained from a methanol solution by slow evaporation of the solvent.

Single crystal X-ray diffraction analysis was performed on a STOE IPDS-II two-circle diffractometer using graphite-monochromated Mo $K_\alpha$ X-ray radiation ($\lambda = 0.71073$ Å). Data collection was performed at 120(2)K using the $\omega$-scan technique and the STOE X-AREA software package.[5] The crystal structure was solved by direct methods and refined by using the X-STEP32 crystallographic software package.[6,7] All of the non-hydrogen atoms were refined anisotropically. All hydrogen atoms were located at ideal positions. Table 1 provides the crystallographic results and refinement information.

All hydrogen atoms were refined with the riding model to final $R$ indices of $R_1$ (0.0377) and $WR_2$ (0.0989). Selected bond lengths and angles are given in Table 2.

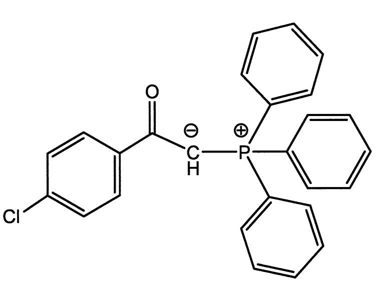

Fig. 1 Chemical structure.

Table 1 Crystal and experimental data

| | |
|---|---|
| Compound | $ClC_6H_4COCHPPh_3$ |
| Empirical formula | $C_{26}H_{20}ClOP$ |
| $Fw$ | 414.84 |
| Temperature (K) | 120(2) |
| Wavelength (Å) | 0.71073 |
| Crystal system | Monoclinic |
| Space group | $C2/c$ |
| $a$(Å) | 16.6184(9) |
| $b$(Å) | 10.9196(6) |
| $c$(Å) | 23.5465(12) |
| $\alpha$(°) | 90 |
| $\beta$(°) | 100.366(4) |
| $\gamma$(°) | 90 |
| Volume (Å$^3$) | 4203.2(4) |
| $Z$ | 8 |
| $\Delta/\sigma$ | 0.011 |
| $D_{Calc}$ (Mg/m$^3$) | 1.311 |
| Absorption coefficient (mm$^{-1}$) | 0.273 |
| $F(0\,0\,0)$ | 1728 |
| Crystal size (mm) | 0.50 × 0.16 × 0.12 |
| $\theta$ range for data collection (°) | 2.24 to 29.19 |
| Limiting indices | $-22 \leqslant h \leqslant 22$, $-14 \leqslant k \leqslant 14$, $-23 \leqslant l \leqslant 32$ |
| Reflections collected/unique ($R_{int}$) | 15184/5571 (0.0388) |
| Completeness to $\theta$ | 97.9% |
| Absorption correction | Numerical |
| Maximum and minimum transmission | 0.970 and 0.020 |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data/restraints/parameters | 5571/0/262 |
| Goodness-of-fit on $F^2$ | 1.088 |
| Final $R$ indices [$I > 2\sigma(I)$] | $R_1 = 0.0377$, $wR_2 = 0.0989$ |
| $R$ indices (all data) | $R_1 = 0.0400$, $wR_2 = 0.1005$ |
| Largest difference in peak and hole (eÅ$^{-3}$) | 0.405 and –0.412 |

Crystal data have been deposited at the Cambridge Crystallographic Data Center under the number CCDC 648308.

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[†] To whom correspondence should be addressed.
E-mail: jsabounchei@yahoo.co.uk

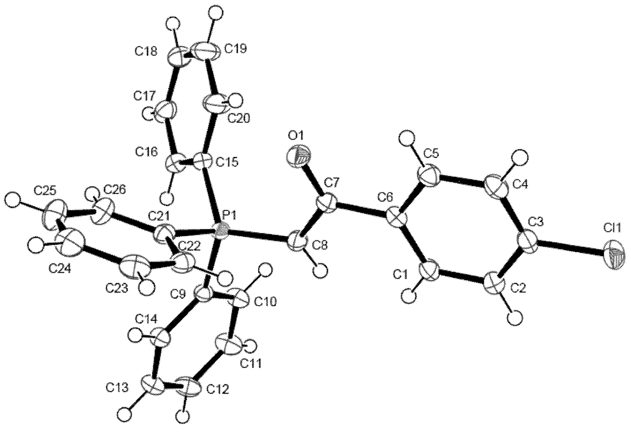
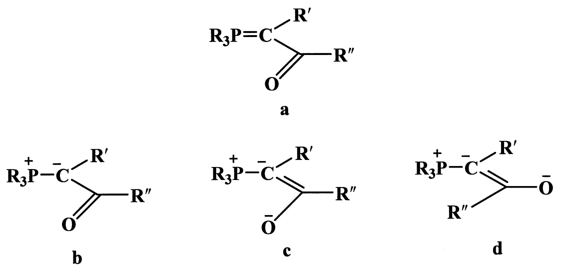


Fig. 2 ORTEP drawing, showing the atom-numbering scheme.

An ORTEP plot (Fig. 2) shows that in the molecule of the title compound, 4′-chlorobenzoylmethylenetriphenylphosphorane ylide, $C_{26}H_{20}ClOP$, the geometry around the P atom is nearly tetrahedral, and the O atom is oriented *cis* to the P atom. The chlorophenyl ring of the benzoyl group is twisted with respect to the plane of the carbonyl group through an angle of –3.99(18)°. The P-C(8) (1.7198(13)) and C(8)-C (7, $sp^2$) (1.4031(16)) bond lengths are shorter than the ($P^+$-C ($sp^3$) (1.800) and (1.511) normal values, respectively. This is due to the ylidic resonanace and intermediate between common values for single and double bonds (for example P-C = 1.80 and P = C = 1.66 Å). The CO bond also is longer (1.2580) (15) than the normal value (1.210). This bond distance suggests resonance delocalization in these molecules (scheme 1).



Scheme (1)

## Acknowledgments

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Table 2 Selected key bond lengths (Å), bond angles (°) and torsion angles (°)

| | |
|---|---|
| C(7)-O(1) | 1.2580(15) |
| C(7)-C(8) | 1.4031(16) |
| C(6)-C(7) | 1.5110(16) |
| C(8)-P(1) | 1.7198(13) |
| C(8)-H(8) | 0.9300 |
| C(9)-P(1) | 1.8037(12) |
| C(15)-P(1) | 1.8177(12) |
| C(21)-P(1) | 1.8097(13) |
| *Bond angles* | |
| O(1)-C(7)-C(8) | 123.04(11) |
| O(1)-C(7)-C(6) | 118.03(11) |
| C(8)-C(7)-C(6) | 118.91(11) |
| C(7)-C(8)-P(1) | 120.51(9) |
| C(7)-C(8)-H(8) | 119.7 |
| P(1)-C(8)-H(8) | 119.7 |
| C(8)-P(1)-C(9) | 105.03(6) |
| C(8)-P(1)-C(21) | 114.26(6) |
| C(9)-P(1)-C(21) | 107.84(5) |
| C(8)-P(1)-C(15) | 116.07(6) |
| C(9)-P(1)-C(15) | 108.13(6) |
| C(21)-P(1)-C(15) | 105.18(6) |
| *Torsion angles* | |
| O(1)-C(7)-C(8)-P(1) | -3.98(18) |
| C(1)-C(6)-C(7)-C(8) | -4.42(18) |
| C(1)-C(6)-C(7)-O(1) | 173.84(12) |
| C(5)-C(6)-C(7)-O(1) | -3.69(17) |

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