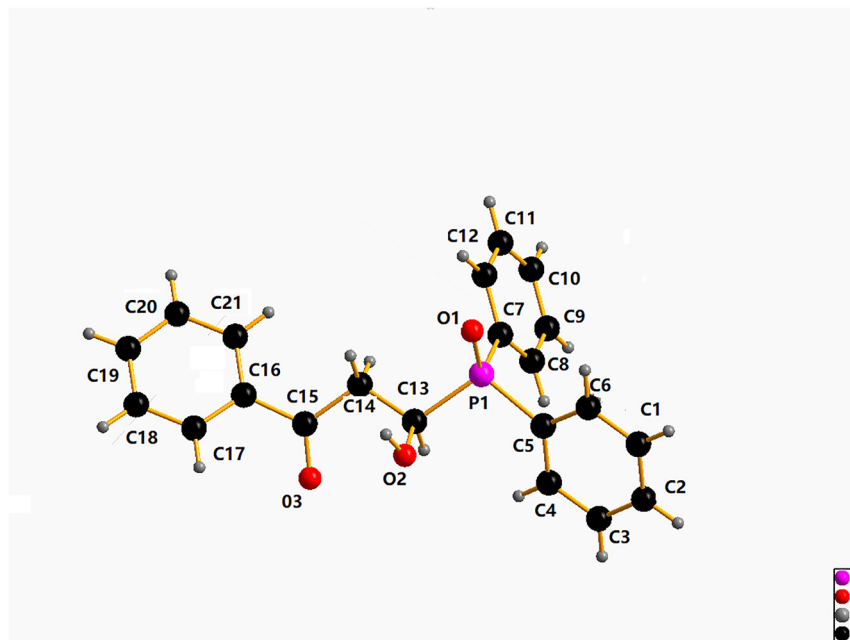


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Crystal structure of 3-(diphenylphosphoryl)-3-hydroxy-1-phenylpropan-1-one, C₂₁H₁₉O₃P



<https://doi.org/10.1515/ncrs-2024-0028>

Received January 22, 2024; accepted March 1, 2024;

published online March 18, 2024

Abstract

C₂₁H₁₉O₃P, triclinic, $P\bar{1}$ (no. 2), $a = 9.5751(18)$ Å, $b = 10.0602(19)$ Å, $c = 10.307(2)$ Å, $\alpha = 82.979(3)^\circ$, $\beta = 78.057(3)^\circ$, $\gamma = 65.622(3)^\circ$, $V = 884.0(3)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0429$, $wR_{\text{ref}}(F^2) = 0.1110$, $T = 296$ K.

CCDC no.: 2336731

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.26 × 0.24 × 0.22 mm
Wavelength:	MoKα radiation (0.71073 Å)
μ :	0.17 mm ⁻¹
Diffractometer, scan mode:	φ and ω
θ_{max} , completeness:	25.0°, 99 %
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	4537, 3084, 0.018
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2375
$N(\text{param})_{\text{refined}}$:	227
Programs:	CRYSA LIS ^{PRO} [1], OLEX2 [2], SHELX [3]

1 Source of material

3-(Diphenylphosphoryl)-3-hydroxy-1-phenylpropan-1-one was prepared by the direct difunctionalization of tertiary enamines with H-phosphine oxide and water. To the solution of (*E*)-3-(dimethylamino)-1-phenylprop-2-en-1-one (175 mg, 1 mmol), diphenylphosphine oxide (404 mg, 2 mmol), dried aluminium muriate (266 mg, 2 mmol) in the mixed solvent of tetramethylene oxide (8 ml) and pure water (1 ml) was stirred at 323 K for 3 h. After completion of reaction, the above mixture was filtrated and washed three times by ethyl acetate. The organic liquid was evaporated under vacuum to obtain the residue. The residue was purified by flash column

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U _{iso} */U _{eq}
C1	0.9401 (3)	0.3534 (2)	0.2388 (2)	0.0540 (6)
H1	0.8790	0.4494	0.2163	0.065*
C2	1.1008 (3)	0.3052 (3)	0.2162 (3)	0.0655 (7)
H2A	1.1469	0.3691	0.1776	0.079*
C3	1.1921 (3)	0.1653 (3)	0.2498 (2)	0.0611 (6)
H3	1.2999	0.1337	0.2330	0.073*
C4	1.1246 (3)	0.0708 (3)	0.3085 (2)	0.0605 (6)
H4	1.1868	−0.0241	0.3332	0.073*
C5	0.9652 (2)	0.1166 (2)	0.3310 (2)	0.0530 (6)
H5	0.9203	0.0521	0.3705	0.064*
C6	0.8703 (2)	0.2587 (2)	0.29503 (19)	0.0424 (5)
C7	0.6226 (2)	0.2664 (2)	0.17208 (19)	0.0444 (5)
C8	0.7182 (4)	0.1382 (3)	0.1130 (3)	0.0798 (8)
H8	0.8063	0.0762	0.1477	0.096*
C9	0.6858 (5)	0.0993 (3)	0.0027 (3)	0.1022 (11)
H9	0.7517	0.0106	−0.0351	0.123*
C10	0.5603 (4)	0.1876 (4)	−0.0514 (3)	0.0767 (8)
H10	0.5397	0.1609	−0.1263	0.092*
C11	0.4659 (3)	0.3146 (4)	0.0049 (3)	0.0882 (9)
H11	0.3793	0.3768	−0.0316	0.106*
C12	0.4959 (3)	0.3545 (3)	0.1169 (3)	0.0796 (8)
H12	0.4285	0.4426	0.1550	0.096*
C13	0.5986 (2)	0.2329 (2)	0.46332 (18)	0.0414 (5)
H13	0.6556	0.1273	0.4537	0.050*
C14	0.4267 (2)	0.2676 (2)	0.4849 (2)	0.0471 (5)
H14A	0.4026	0.2398	0.4081	0.057*
H14B	0.3689	0.3721	0.4934	0.057*
C15	0.3756 (2)	0.1885 (3)	0.6080 (2)	0.0497 (5)
C16	0.2070 (2)	0.2409 (2)	0.6651 (2)	0.0475 (5)
C17	0.1631 (3)	0.2244 (3)	0.8000 (2)	0.0696 (7)
H17	0.2384	0.1881	0.8543	0.084*
C18	0.0089 (4)	0.2610 (3)	0.8552 (3)	0.0903 (10)
H18	−0.0200	0.2516	0.9466	0.108*
C19	−0.1011 (3)	0.3110 (3)	0.7759 (4)	0.0916 (10)
H19	−0.2045	0.3311	0.8130	0.110*
C20	−0.0607 (3)	0.3318 (3)	0.6424 (4)	0.0826 (9)
H20	−0.1368	0.3675	0.5889	0.099*
C21	0.0933 (3)	0.2999 (3)	0.5867 (3)	0.0643 (6)
H21	0.1203	0.3183	0.4964	0.077*
O1	0.58533 (17)	0.48743 (14)	0.32087 (14)	0.0548 (4)
O2	0.64383 (15)	0.27337 (15)	0.56942 (13)	0.0473 (4)
H2	0.5784	0.3528	0.5953	0.071*
O3	0.46963 (19)	0.0841 (2)	0.65904 (18)	0.0780 (6)
P1	0.66182 (6)	0.32594 (5)	0.31350 (5)	0.04139 (18)

chromatography on silicagel (ethyl acetate/petroleum ether = 1:2) to obtain white solid target compound in 95 % yield.

2 Experimental details

The carbon-bound hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

3 Comment

α-Hydroxylphosphine oxide is an important organophosphorus product, and widely used in the synthesis of α-aminophosphine oxides [4, 5], and exhibits various intriguing biological activities. This compound was synthesized by the reaction between (*E*)-3-(dimethylamino)-1-phenylprop-2-en-1-one and diphenylphosphine oxide in the presence of water.

In the molecules forming the title crystal structure, the key bond lengths and angles are within normal ranges, and are consistent with those previously reported in similar structures [6]. The C–P bond is confirmed by the distance of P1–C1, P1–C7, P2–C13. Their lengths are 1.799(2) Å, 1.803(2) Å, and 1.832(2) Å respectively. The bond angles for O1–P1–C6, O1–P1–C13, O1–P1–C7 are 112.35(9) Å, 112.22(9) Å, and 110.99(9) Å, which is consistent with O=P–C group. The hydroxyl and carbonyl groups are also found by the distance of C13–O2, C15=O3. The bond lengths are 1.414(2) Å and 1.212(2) Å.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: The National Natural Science Foundation of China (No. 82360679, No. 31660443). The Natural Science Foundation of Guizhou Province (No. ZK [2022]59). The Young Talents Program of Zunyi Medical College (No. 18zy-009).

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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