

Hua-Bin Wang, Lian-Hua Xu, Ju-Mei Shi and Jiang-Yong An*

Crystal structure of (Z)-4-((4,5,6-trimethoxy-3-oxobenzofuran-2(3H)-ylidene)methyl)phenyl diphenylphosphinate, C₃₀H₂₅O₇P

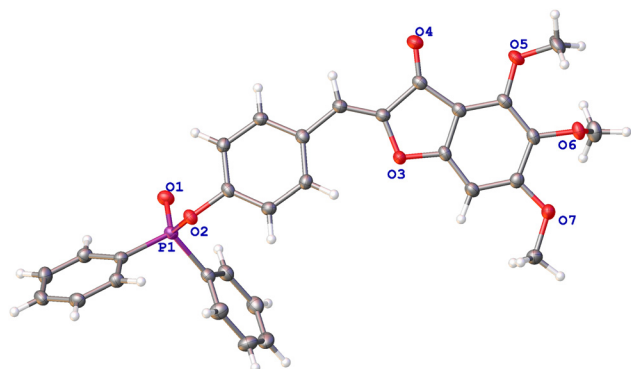


Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.16 × 0.14 × 0.12 mm
Wavelength:	Cu Kα radiation (1.54184 Å)
μ:	1.41 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
θ _{max} , completeness:	73.8°, >99 %
N(hkl) _{measured} , N(hkl) _{unique} , R _{int} :	9486, 4856, 0.030
Criterion for I _{obs} , N(hkl) _{gt} :	I _{obs} > 2σ(I _{obs}), 4444
N(param) _{refined} :	346
Programs:	CrysAlis ^{PRO} [1], Olex2 [2], SHELX [3]

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Abstract

C₃₀H₂₅O₇P, monoclinic, *P*₂/c (no. 14), *a* = 12.1091(3) Å, *b* = 8.0535(2) Å, *c* = 25.4435(6) Å, β = 92.169(2)°, *V* = 2479.49(10) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0438, *wR*_{ref}(*F*²) = 0.1219, *T* = 150 K.

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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.

1 Source of material

(Z)-2-(4-hydroxybenzylidene)-4,5,6-trimethoxybenzofuran-3(2H)-one (0.16 g, 0.5 mmol) and diphenylphosphine oxide (0.11 g, 0.55 mmol) were dissolved in 2 mL of chloroform, and

methyltriphenylphosphonium iodide (0.02 g, 0.05 mmol) and cesium carbonate (0.33 g, 1 mmol) were added to the mixed solution for reflux reaction at 298 K for about 12 h. After the reaction was completed, the solvent was evaporated under vacuum. The residue was subjected to flash column chromatography on silicagel (petroleum ether/ethyl acetate = 2:1), and recrystallized with methyl alcohol to obtain a yellow powder of the target compound.

2 Experimental details

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

3 Comment

Aurones, which have an exo-conjugated benzylidene group, are one of geometric isomers of flavones. Aurones possess a styryl motif and are the key members of benzo-fused oxacycles. They are widely present in fruits and yellow flowers and are found in secondary metabolites. They exhibit many biological activities, such as antioxidant, antiviral, and anticancer activities [4, 5]. They are also important intermediates in the synthesis of a variety of drugs. In the

*Corresponding author: Jiang-Yong An, Guizhou University of Traditional Chinese Medicine, Guiyang 550025, P.R. China, E-mail: ajy0824@163.com
Hua-Bin Wang, Lian-Hua Xu and Ju-Mei Shi, Guizhou University of Traditional Chinese Medicine, Guiyang 550025, P.R. China. <https://orcid.org/0009-0003-2897-4878> (H.-B. Wang). <https://orcid.org/0009-0002-0579-5087> (L.-H. Xu). <https://orcid.org/0009-0008-4199-9611> (J.-M. Shi)

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
P1	0.63366 (3)	0.03586 (5)	0.60793 (2)	0.01921 (13)
O1	0.70156 (10)	−0.06835 (16)	0.57449 (5)	0.0256 (3)
O2	0.54951 (9)	0.16054 (15)	0.57747 (4)	0.0218 (3)
O3	0.77334 (10)	0.79398 (15)	0.45619 (4)	0.0234 (3)
O4	0.78414 (11)	0.73968 (17)	0.31908 (5)	0.0306 (3)
O5	0.90997 (11)	1.07609 (16)	0.31257 (5)	0.0286 (3)
O6	0.99292 (10)	1.33768 (15)	0.38214 (5)	0.0250 (3)
O7	0.95914 (11)	1.31630 (15)	0.48297 (5)	0.0280 (3)
C1	0.53877 (13)	−0.0766 (2)	0.64672 (6)	0.0199 (3)
C2	0.44494 (14)	−0.0043 (2)	0.66727 (6)	0.0232 (3)
H2	0.427731	0.108475	0.659686	0.028*
C3	0.37682 (14)	−0.0963 (2)	0.69867 (7)	0.0261 (4)
H3	0.313541	−0.046297	0.712938	0.031*
C4	0.40105 (15)	−0.2619 (2)	0.70929 (7)	0.0271 (4)
H4	0.354364	−0.325106	0.730839	0.032*
C5	0.49304 (15)	−0.3346 (2)	0.68852 (7)	0.0282 (4)
H5	0.509066	−0.448033	0.695633	0.034*
C6	0.56221 (14)	−0.2431 (2)	0.65733 (7)	0.0238 (3)
H6	0.625438	−0.293797	0.643215	0.029*
C7	0.71694 (14)	0.1664 (2)	0.65107 (6)	0.0222 (3)
C8	0.66994 (16)	0.2789 (2)	0.68507 (7)	0.0303 (4)
H8	0.591784	0.287135	0.686065	0.036*
C9	0.73592 (17)	0.3794 (3)	0.71761 (7)	0.0337 (4)
H9	0.703159	0.456244	0.740684	0.040*
C10	0.85001 (16)	0.3668 (3)	0.71616 (7)	0.0325 (4)
H10	0.895451	0.435619	0.738237	0.039*
C11	0.89785 (15)	0.2551 (3)	0.68293 (7)	0.0310 (4)
H11	0.976048	0.247315	0.682142	0.037*
C12	0.83181 (15)	0.1536 (2)	0.65047 (7)	0.0261 (4)
H12	0.864989	0.075565	0.627922	0.031*
C13	0.58997 (13)	0.2588 (2)	0.53680 (6)	0.0200 (3)
C14	0.61479 (14)	0.4245 (2)	0.54650 (6)	0.0224 (3)
H14	0.605564	0.470164	0.580477	0.027*
C15	0.65297 (14)	0.5227 (2)	0.50654 (7)	0.0223 (3)
H15	0.670463	0.635949	0.513134	0.027*
C16	0.66599 (13)	0.4557 (2)	0.45625 (6)	0.0207 (3)
C17	0.60064 (14)	0.1895 (2)	0.48750 (6)	0.0220 (3)
H17	0.582581	0.076257	0.481204	0.026*
C18	0.63824 (14)	0.2889 (2)	0.44755 (6)	0.0217 (3)
H18	0.645362	0.242965	0.413458	0.026*
C19	0.70633 (14)	0.5502 (2)	0.41180 (6)	0.0223 (3)
H19	0.698145	0.497213	0.378523	0.027*
C20	0.75283 (14)	0.6998 (2)	0.41130 (6)	0.0218 (3)
C21	0.79387 (14)	0.7896 (2)	0.36446 (6)	0.0224 (3)
C22	0.84293 (14)	0.9402 (2)	0.38666 (6)	0.0214 (3)
C23	0.82794 (13)	0.9347 (2)	0.44048 (6)	0.0208 (3)
C24	0.86276 (14)	1.0549 (2)	0.47576 (6)	0.0226 (3)
H24	0.849906	1.047031	0.512277	0.027*
C25	0.91804 (14)	1.1886 (2)	0.45435 (7)	0.0226 (3)
C26	0.93652 (13)	1.2004 (2)	0.39963 (7)	0.0223 (3)
C27	0.89739 (13)	1.0784 (2)	0.36542 (6)	0.0220 (3)
C28	0.93971 (16)	1.3168 (2)	0.53816 (7)	0.0288 (4)
H28A	0.860075	1.323111	0.543440	0.043*
H28B	0.976451	1.413004	0.554663	0.043*
H28C	0.969328	1.214584	0.554145	0.043*

Table 2: (continued)

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C29	1.11083 (15)	1.3163 (3)	0.38630 (8)	0.0329 (4)
H29A	1.133217	1.231970	0.361058	0.049*
H29B	1.132469	1.280527	0.422035	0.049*
H29C	1.147143	1.421846	0.378635	0.049*
C30	0.91090 (17)	1.2300 (3)	0.28411 (7)	0.0351 (5)
H30A	0.882380	1.211660	0.248019	0.053*
H30B	0.986740	1.272191	0.283450	0.053*
H30C	0.864209	1.311168	0.301444	0.053*

study of safety evaluation of heavy metals in Guizhou's main wild vegetables and promoting the development of Guizhou's wild vegetable industry, we find that auronones are also present in Guizhou's wild vegetables. Organophosphorus compounds have extensive applications in pharmaceuticals, ligand scaffolds, and organic synthesis [6, 7]. Therefore, synthesis and modification of auronones and organophosphorus have been a hot topic in the field of medicine and pharmacy.

The basic structure of the title compound is auronone, containing a auronone and a diphenylphosphine oxide fragment. The key lengths and angles obtained from the title structure are within the normal range and are consistent with those previously reported in similar structures [8–11]. The keto group was confirmed by the distance of 1.224(2) Å (C21–O4), the C19=C20 double bond adopts a Z-configuration and the bond distance is 1.330(2) Å, which was defined by the dihedral angle of 1.3(3)° for O3–C20–C19–C16. The three methoxy groups on the benzene ring, of which one methoxy group (C-25) has the methoxy group forming a smaller angle with the ring (C24–C25–O7–C28 [−3.0(2)°]). The other methoxy group (C-27) is turned out of the plane of the ring (C22–C27–O5–C30 [149.0(16)°]). The third methoxy group (C-26) is turned out from the plane of the ring (C27–C26–O6–C29 [−95.5(19)°]). The P1–O2 = 1.6086(12) Å, phenyl–C [P1–C7, C1 = 1.8007(18) & 1.7887(17) Å] and oxide–O1 [P1–O1 = 1.4686(12) Å] bond lengths are in the normal ranges.

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