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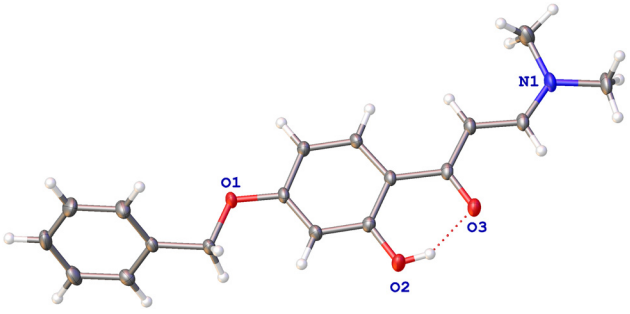
Crystal structure of (*E*)-1-(4-(benzyloxy)-2-hydroxyphenyl)-3-(dimethylamino)prop-2-en-1-one, $C_{18}H_{19}NO_3$



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Abstract

$C_{18}H_{19}NO_3$, monoclinic, $P2_1/c$ (no. 14), a = 8.9529(3) Å, b = 13.5984(4) Å, c = 12.8403(5) Å, β = 101.572(4)°, $R_{gt}(F)$ = 0.0458, $wR_{ref}(F^2)$ = 0.1395, V = 1532.54(9) Å^3, Z = 4, 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.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.16 × 0.12 × 0.11 mm
Wavelength:	Cu *K*α radiation (1.54184 Å)
μ:	0.71 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	73.9°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5775, 3005, 0.022
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2636
$N(param)_{refined}$:	203
Programs:	CrysAlis^{PRO} [1], Olex2 [2], SHELX [3]

1 Source of material

In our research of the identification of anti-rot genes of *Pinellia ternata* in Guizhou Province, we selected the hydroxyl aromatic keton derivate present in pinellia ternata as the raw material to synthesize the title compound. 1-(4-(benzyloxy)-2-hydroxyphenyl)ethan-1-one (0.53 g, 2.2 mmol) and dimethylformamide dimethylacetal (3.0 mL) were combined. The colorless solution heated to 383 K. Thin layer chromatography tracked the progress of the reaction until the 1-(4-(benzyloxy)-2-hydroxyphenyl)ethan-1-one is complete and the reaction is over. The red homogeneous solution was concentrated in vacuo to afford a solid. The solid was recrystallized with ethanol.

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

Enaminone compounds and their derivatives are widely used in the field of medicinal chemistry and organic synthesis, used as intermediates to synthesize many drugs [4–5]. The current research shows that they can be used as potential anticonvulsants, their proposed mechanisms of action, and as potential modulators of multidrug resistance (MDR) [6]. They also have anti-inflammatory and antiepileptic effects [7]. Furthermore, in our research of the identification of anti-rot genes of *Pinellia ternata* based on functional genomics, including collecting pinellia masters from Hezhang, Dafang and Zunyi in Guizhou Province, screening the pathogen of pinellia tuber rot disease, identifying the physiological species and cloning of anti-rot disease

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.59658 (11)	0.36854 (7)	0.80351 (7)	0.0329 (3)
O2	0.94962 (12)	0.23739 (7)	0.60716 (8)	0.0405 (3)
H2	0.983560	0.248510	0.551918	0.061*
O3	0.98543 (11)	0.32366 (7)	0.44442 (7)	0.0370 (3)
N1	0.90290 (14)	0.53123 (9)	0.21174 (9)	0.0363 (3)
C1	0.54004 (15)	0.30160 (9)	0.96297 (10)	0.0293 (3)
C2	0.56413 (19)	0.37971 (11)	1.03297 (12)	0.0425 (4)
H2A	0.641197	0.426542	1.028186	0.051*
C3	0.4773 (2)	0.39057 (12)	1.11002 (13)	0.0533 (5)
H3	0.494671	0.444810	1.157508	0.064*
C4	0.3655 (2)	0.32267 (13)	1.11797 (13)	0.0497 (4)
H4	0.305568	0.330246	1.170622	0.060*
C5	0.34130 (18)	0.24390 (12)	1.04927 (13)	0.0452 (4)
H5	0.265144	0.196655	1.054888	0.054*
C6	0.42817 (17)	0.23360 (10)	0.97187 (11)	0.0365 (3)
H6	0.410728	0.179313	0.924452	0.044*
C7	0.63385 (16)	0.28980 (10)	0.87935 (10)	0.0332 (3)
H7A	0.743693	0.292019	0.912274	0.040*
H7B	0.611756	0.225563	0.843292	0.040*
C8	0.67371 (14)	0.37029 (9)	0.72193 (9)	0.0276 (3)
C9	0.63841 (16)	0.44930 (10)	0.65164 (10)	0.0309 (3)
H9	0.566609	0.497615	0.662824	0.037*
C10	0.70896 (15)	0.45629 (9)	0.56598 (10)	0.0300 (3)
H10	0.684784	0.510183	0.518549	0.036*
C11	0.81508 (14)	0.38657 (9)	0.54655 (9)	0.0261 (3)
C12	0.77920 (15)	0.30066 (10)	0.70699 (10)	0.0306 (3)
H12	0.804145	0.247941	0.755904	0.037*
C13	0.84929 (15)	0.30808 (10)	0.61943 (10)	0.0291 (3)
C14	0.89004 (15)	0.39047 (9)	0.45371 (10)	0.0286 (3)
C15	0.85265 (15)	0.46604 (10)	0.37617 (10)	0.0302 (3)
H15	0.781635	0.515998	0.384167	0.036*
C16	0.92111 (15)	0.46544 (10)	0.28956 (10)	0.0314 (3)
H16	0.988501	0.412391	0.284892	0.038*
C17	0.97747 (19)	0.51927 (13)	0.12228 (11)	0.0453 (4)
H17A	1.044001	0.575814	0.118424	0.068*
H17B	0.900313	0.514994	0.056520	0.068*
H17C	1.038502	0.458904	0.131426	0.068*
C18	0.8057 (2)	0.61622 (14)	0.21072 (13)	0.0518 (4)
H18A	0.699063	0.594993	0.199826	0.078*
H18B	0.818772	0.660362	0.152927	0.078*
H18C	0.833273	0.650932	0.278704	0.078*

related genes, we find that enaminone compounds are also present in Guizhou's *Pinellia ternata*. Enaminone compounds are also used as raw material to synthesize flavonoids in pinellia ternata.

The structure of the title compound is an enaminone, containing a benzene ring, a hydroxyl group, a ketone and an enamine. The bond lengths and angles derived from the title structure are within normal ranges and consistent with those reported previously in similar structures [8–10]. The keto group was confirmed by the distance of 1.2688(16) Å (C14–O3), the C15=C16 double bond adopts an *E*-configuration and the bond distance is 1.3739(16) Å, and the phenyl hydroxyl group was identified by the distance of 1.3464(15) Å (C13–O2), the dihedral angle of –178.07(11)° for O1–C9–C8–C7 respectively.

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References

1. *CrysAlis^{Pro} software*; Rigaku Oxford Diffraction: Japan, **2015**.
2. Dolomanov O. V., Bourhis L. J., Gildea R. J., Howard J. A. K., Puschmann H. OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* **2009**, *42*, 339–341.
3. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* **2015**, *C71*, 3–8.
4. Zhang L., Dong J. H., Xu X. X., Liu Q. Chemistry of ketene *N,S*-acetals: an overview. *Chem. Rev.* **2016**, *116*, 287–322.
5. Gao Y., Hu C. F., Wen C. P., Wan J. P. Tunable synthesis of disulfide-functionalized enaminones and 1,4-thiazines via the reactions of enaminones and β-aminoethanethiol. *ACS Omega* **2017**, *2*, 7784–7789.
6. Edafiogho I. O., Kombian S. B., Ananthalakshmi K. V., Salama N. N., Eddington N. D., Wilson T. L., Alexander M. S., Jackson P. L., Hanson C. D., Scott K. R. Enaminones: Exploring additional therapeutic activities. *J. Pharm. Sci.* **2007**, *96*, 2509–2531.
7. Malawska B. New anticonvulsant agents. *Curr. Top. Med. Chem.* **2005**, *5*, 69–85.
8. Safavora A. S., Brito I., Cisterna J., Cárdenas A., Huseynov E. Z., Khalilov A. N., Naghiyev F. N., Askerov R. K., Maharramov A. M. Crystal structure of ethyl (*Z*)-(4-oxo-4-phenylbut-2-en-2-yl)glycinate, $C_{14}H_{17}NO_3$. *Z. Kristallogr. N. Cryst. Struct.* **2019**, *234*, 1183–1185.
9. Ghorab M. M., Alsaid M. S., Ghabbour H. A., Fun H. K. Crystal structure of (*E*)-1-(3,4-dimethoxyphenyl)-3-(dimethylamino)prop-2-en-1-one, $C_{13}H_{17}NO_3$. *Z. Kristallogr. N. Cryst. Struct.* **2016**, *231*, 437–438.
10. Tang B., Liu H., Li F., Wang Y., Zhang H. Single-benzene solid emitters with lasing properties based on aggregation-induced emissions. *Chem. Commun.* **2016**, *52*, 6577–6580.