Shixiong Sun*, Benbo Zhao, Ping Deng and Yuan Cheng

Crystal structure of 1-(4-methoxyphenyl)-2-phenoxyethan-1-one, $C_{15}H_{14}O_3$

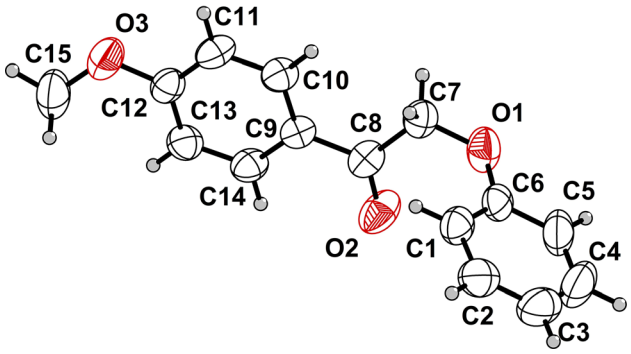

Table 1: Data collection and handling.

Crystal:	White plate
Size:	0.11 × 0.10 × 0.09 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	φ and ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6422, 2238, 0.019
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1654
$N(param)_{refined}$:	164
Programs:	SHELX [1], Olex2 [2], CrysAlis^{PRO} [3]

https://doi.org/10.1515/ncrs-2022-0595
Received December 21, 2022; accepted January 15, 2023;
published online February 13, 2023

Abstract

$C_{15}H_{14}O_3$, M = 242.26 g/mol, monoclinic, $P2_1/c$ (no. 14), a = 5.5859(10) Å, b = 24.818(4) Å, c = 9.3393(15) Å, β = 101.128(3)°, V = 1270.4(4) Å^3, Z = 4, $R_{gt}(F)$ = 0.0410, w$R_{ref}(F^2)$ = 0.1394, T = 296(2) K.

CCDC no: 2236290

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.

Source of material

The title compound was prepared according to the known procedure in one step with commercial available phenol and p-methoxyphenacyl bromide as starting material affording the product as a white solid [4–6]. The crude product was

***Corresponding author: Shixiong Sun**, School of Chemical Engineering and Technology, North University of China, Taiyuan, Shanxi Province, P. R. China; and Dezhou Industrial Technology Research, Institute of North University of China, Dezhou, Shandong Province, P. R. China, E-mail: zhaobenbo@163.com. https://orcid.org/0000-0001-5486-6537
Benbo Zhao, **Ping Deng and Yuan Cheng,** School of Chemical Engineering and Technology, North University of China, Taiyuan, Shanxi Province, P. R. China; and Dezhou Industrial Technology Research, Institute of North University of China, Dezhou, Shandong Province, P. R. China

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

Atom	x	y	z	U_{iso}*/U_{eq}
O1	0.1146 (3)	0.21897 (6)	0.37365 (17)	0.0679 (5)
O2	0.5252 (3)	0.25338 (6)	0.54377 (15)	0.0743 (5)
O3	1.0305 (3)	0.45265 (6)	0.30063 (17)	0.0743 (5)
C1	0.3987 (4)	0.15403 (7)	0.3038 (2)	0.0571 (5)
H1	0.4855	0.1804	0.2649	0.069*
C2	0.4640 (4)	0.10018 (9)	0.2986 (2)	0.0711 (6)
H2	0.5943	0.0907	0.2551	0.085*
C3	0.3401 (6)	0.06109 (10)	0.3564 (3)	0.0903 (9)
H3	0.3845	0.0251	0.3518	0.108*
C4	0.1504 (6)	0.07529 (11)	0.4209 (3)	0.0981 (10)
H4	0.0662	0.0487	0.4610	0.118*
C5	0.0811 (4)	0.12833 (10)	0.4277 (3)	0.0776 (7)
H5	−0.0479	0.1374	0.4728	0.093*
C6	0.2043 (4)	0.16796 (8)	0.3672 (2)	0.0537 (5)
C7	0.2403 (4)	0.26231 (7)	0.3223 (2)	0.0548 (5)
H7A	0.1327	0.2933	0.3044	0.066*
H7B	0.2841	0.2522	0.2304	0.066*
C8	0.4683 (4)	0.27776 (7)	0.4295 (2)	0.0493 (5)
C9	0.6146 (3)	0.32386 (6)	0.39454 (18)	0.0431 (4)
C10	0.5666 (4)	0.35008 (7)	0.2603 (2)	0.0504 (5)
H10	0.4376	0.3387	0.1883	0.060*
C11	0.7089 (4)	0.39279 (7)	0.2334 (2)	0.0559 (5)
H11	0.6748	0.4101	0.1434	0.067*
C12	0.9021 (4)	0.41018 (7)	0.3388 (2)	0.0505 (5)
C13	0.9522 (4)	0.38463 (7)	0.4725 (2)	0.0547 (5)
H13	1.0814	0.3962	0.5443	0.066*
C14	0.8095 (4)	0.34197 (7)	0.4987 (2)	0.0524 (5)
H14	0.8446	0.3248	0.5888	0.063*
C15	1.2437 (4)	0.46947 (9)	0.4003 (3)	0.0830 (8)
H15A	1.3190	0.4988	0.3586	0.125*
H15B	1.3561	0.4399	0.4197	0.125*
H15C	1.1994	0.4810	0.4898	0.125*

recrystallized from ethyl acetate as single crystals suitable for X–ray diffraction analysis.

Experimental details

All the Friedel pairs were merged [1]. All H atoms were positioned geometrically and treated as riding, with C–H bond lengths constrained to 0.96 Å for methyl H atoms, 0.97 Å for ethyl H atoms, and with U_{iso}(H) = 1.2U_{eq}(C) for ethyl H atoms, and phenyl H atoms, and 1.5U_{eq}(C) for methyl H atoms.

Comment

The chemical structures of lignin biomass are comprised of the polyphenolic compounds with versatile linkage modes including β–O-4 (β-aryl ether), β-5 (phenylcoumaran), β-β (resinol), and β-1 (spirodienone), etc., the β–O-4 linkage mode is the most abundant component which accounts for 40 – 65% of total linkages presented in lignin biomass. 1-(4-methoxyphenyl)-2- phenoxyethan-1-one was one of the mode of β–O-4 linkage [7, 8]. Here we report the crystal structure of the title compound. In the crystal structure, the crystal packing is stabilized in layers and held together by Van der Waals. Bond lengths and angles are all in the expected ranges.

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

Research funding: This work was supported by the Applied Basic Research Programs of Shanxi Province of China (Grant No. 201901D211220).

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

References

1. Chen K., Schwarz J., Karl T. A., Chatterjee A., König B. Visible light induced redox neutral fragmentation of 1,2-diol derivatives. *Chem. Commun.* 2019, *55*, 13144–13147.
2. Zhou Y., Klinger G. E., Hegg E. L., Saffron C. M., Jackson J. E. Multiple mechanisms mapped in aryl alkyl ether cleavage via aqueous electrocatalytic hydrogenation (ECH) over skeletal nickel. *J. Am. Chem. Soc.* 2020, *142*, 4037–4050.
3. Tian H. R., Liu Y. W., Zhang Z., Liu S. M., Dang T. Y., Li X. H., Sun X. W., Lu Y., Liu S. X. A multicentre synergistic polyoxometalate- based metal-organic framework for one-step selective oxidative cleavage of β–O-4 lignin model compounds. *Green Chem.* 2020, *22*, 248–255.
4. Sheldrick G. M. *SHELXTL* – integrated space-group and crystal-structure determination. *Acta Crystallogr.* 2015, *A71*, 3–8.
5. 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.
6. Agilent Technologies. *CrysAlis*^{PRO}, Version 1.171.37.35; Agilent Technologies: Santa Clara, CA, USA, 2017.
7. Luo J., Zhang J. Aerobic oxidation of olefins and lignin model compounds using photogenerated phthalimide–N-oxyl (PINO) radical. *J. Org. Chem.* 2016, *81*, 9131–9137.
8. Tsai Y.-T., Chen C.-Y., Hsieh Y.-J., Tsai M.-L. Selective Ca alcohol oxidation of lignin substrates featuring a β–O-4 linkage by a dinuclear oxovanadium catalyst via two-electron redox processes. *Eur. J. Inorg. Chem.* 2019, *43*, 4637–4646.