Crystal structure of 2-phenoxyethyl 2-hydroxybenzoate, $C_{15}H_{14}O_4$

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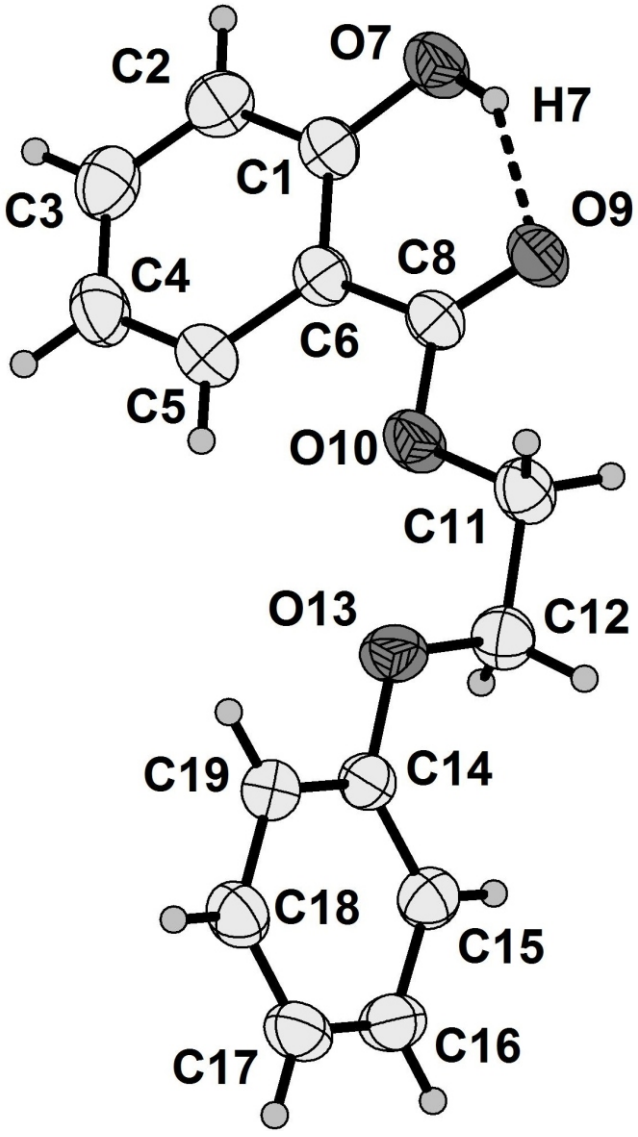

Abstract

$C_{15}H_{14}O_4$, triclinic, $P\bar{1}$ (no. 2), a = 5.3317(3) Å, b = 11.0927(7) Å, c = 11.3125(7) Å, α = 76.112(2)°, β = 82.870(2)°, γ = 80.128(2)°, V = 637.5 Å^3, Z = 2, $R_{gt}(F)$ = 0.0339, $wR_{ref}(F^2)$ = 0.0924, T = 296 K.

Source of material

2-Hydroxybenzoic acid (5.0 g, 0.04 mol) was mixed without solvent with 2-phenoxyethanol (5.5 g, 0.04 mol) and refluxed for 4 hours. The reaction solution was left at room temperature for 24 hours under stirring. The raw product was collected as white powder in 60% yield. The crystalline material was purified by washing several times with cold ethanol and *n*-hexane.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.18 0.19 0.20 mm
Wavelength:	Cu K radiation (1.54178 Å)
μ:	8.07 cm ^{1}
Diffractometer, scan mode:	Bruker X8 Proteum, φ and ω
$2\theta_{max}$:	129.56°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8787, 2091
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1977
$N(param)_{refined}$:	173
Programs:	SHELX [4], PLATON [5], DIAMOND [6]

Discussion

Phenoxyethyl benzoate has applications in the synthesis of e.g. oxazoles, imidazoles and benzoxazepines [1]. Esters are also useful as photo-removable protecting groups for carboxylic acids in organic synthesis and biochemistry [2]. This contribution is a continuation of our work to prepare and characterize 2-phenoxyethyl benzoate and their derivative thorough etherification of substituted benzoic acid with 2-phenoxyethanol [3]. The structure of 2-phenoxyethyl 2-hydroxybenzoate $C_{15}H_{14}O_4$ compound was synthesized and its crystal structure is reported herein. All bond lengths and angles are in the expected ranges. There is only one intramolecular O–H H hydrogen bond (Fig.; O7 O9 = 2.6369(12) Å) present in the title structure.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(2)	2*i*	0.6340	0.7652	0.5237	0.067
H(3)	2*i*	0.8173	0.7686	0.6949	0.072
H(4)	2*i*	0.7220	0.6331	0.8794	0.072
H(5)	2*i*	0.4470	0.4916	0.8894	0.062
H(7)	2*i*	0.2251	0.5793	0.5094	0.088
H(11A)	2*i*	0.0789	0.2159	0.7925	0.063
H(11B)	2*i*	0.1567	0.3230	0.7925	0.063
H(12A)	2*i*	0.1186	0.2963	1.0079	0.061
H(12B)	2*i*	0.2155	0.1840	0.9735	0.061
H(15)	2*i*	0.2447	0.1512	1.1834	0.059

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Table 2. continued.

Atom	Site	x	y	z	U_{iso}
H(16)	2*i*	0.2611	0.0264	1.3791	0.065
H(17)	2*i*	0.0784	0.1235	1.4437	0.068
H(18)	2*i*	0.4410	0.1481	1.3117	0.067
H(19)	2*i*	0.4660	0.0222	1.1178	0.060

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(7)	2*i*	0.3213(2)	0.63209(8)	0.49707(7)	0.0718(6)	0.0562(5)	0.0467(5)	0.0186(4)	0.0180(4)	0.0027(4)
O(9)	2*i*	0.0946(2)	0.44366(8)	0.62770(7)	0.0663(5)	0.0607(5)	0.0420(4)	0.0194(4)	0.0156(4)	0.0056(4)
O(10)	2*i*	0.1679(2)	0.37273(8)	0.82435(7)	0.0676(5)	0.0501(5)	0.0427(5)	0.0196(4)	0.0158(4)	0.0012(3)
O(13)	2*i*	0.1444(2)	0.14631(8)	1.01347(7)	0.0521(5)	0.0575(5)	0.0447(5)	0.0041(4)	0.0011(4)	0.0016(4)
C(1)	2*i*	0.4246(2)	0.6269(1)	0.6013(1)	0.0491(6)	0.0412(6)	0.0473(6)	0.0024(5)	0.0084(5)	0.0086(5)
C(2)	2*i*	0.5945(2)	0.7101(1)	0.5971(1)	0.0597(7)	0.0473(6)	0.0589(7)	0.0124(5)	0.0037(6)	0.0085(5)
C(3)	2*i*	0.7043(3)	0.7121(1)	0.6994(1)	0.0566(7)	0.0578(7)	0.0740(9)	0.0157(6)	0.0043(6)	0.0246(6)
C(4)	2*i*	0.6485(3)	0.6307(1)	0.8098(1)	0.0600(7)	0.0694(8)	0.0600(8)	0.0111(6)	0.0145(6)	0.0269(6)
C(5)	2*i*	0.4837(2)	0.5466(1)	0.8155(1)	0.0569(7)	0.0544(7)	0.0449(6)	0.0071(5)	0.0094(5)	0.0128(5)
C(6)	2*i*	0.3701(2)	0.5422(1)	0.7118(1)	0.0453(6)	0.0398(5)	0.0433(6)	0.0022(4)	0.0080(5)	0.0093(4)
C(8)	2*i*	0.1991(2)	0.4503(1)	0.7146(1)	0.0484(6)	0.0414(6)	0.0402(6)	0.0030(5)	0.0095(5)	0.0061(4)
C(11)	2*i*	0.0037(2)	0.2823(1)	0.8325(1)	0.0616(7)	0.0480(6)	0.0501(7)	0.0158(5)	0.0138(5)	0.0055(5)
C(12)	2*i*	0.0713(2)	0.2292(1)	0.9644(1)	0.0531(7)	0.0463(6)	0.0510(7)	0.0075(5)	0.0074(5)	0.0049(5)
C(14)	2*i*	0.1126(2)	0.0768(1)	1.1307(1)	0.0489(6)	0.0422(6)	0.0402(6)	0.0140(5)	0.0045(5)	0.0076(4)
C(15)	2*i*	0.1057(2)	0.0915(1)	1.2090(1)	0.0488(6)	0.0491(6)	0.0507(7)	0.0094(5)	0.0026(5)	0.0111(5)
C(16)	2*i*	0.1152(2)	0.0161(1)	1.3261(1)	0.0582(7)	0.0602(7)	0.0468(7)	0.0195(6)	0.0053(5)	0.0135(5)
C(17)	2*i*	0.0870(3)	0.0732(1)	1.3651(1)	0.0711(8)	0.0585(7)	0.0415(6)	0.0204(6)	0.0072(6)	0.0028(5)
C(18)	2*i*	0.3035(2)	0.0874(1)	1.2861(1)	0.0576(7)	0.0573(7)	0.0511(7)	0.0072(6)	0.0140(6)	0.0050(5)
C(19)	2*i*	0.3183(2)	0.0127(1)	1.1699(1)	0.0454(6)	0.0564(7)	0.0474(6)	0.0095(5)	0.0046(5)	0.0103(5)

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