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Crystal structure of ethyl 3,5-dihydroxybenzoate hemihydrate, $C_9H_{10}O_4 \cdot \frac{1}{2}H_2O$

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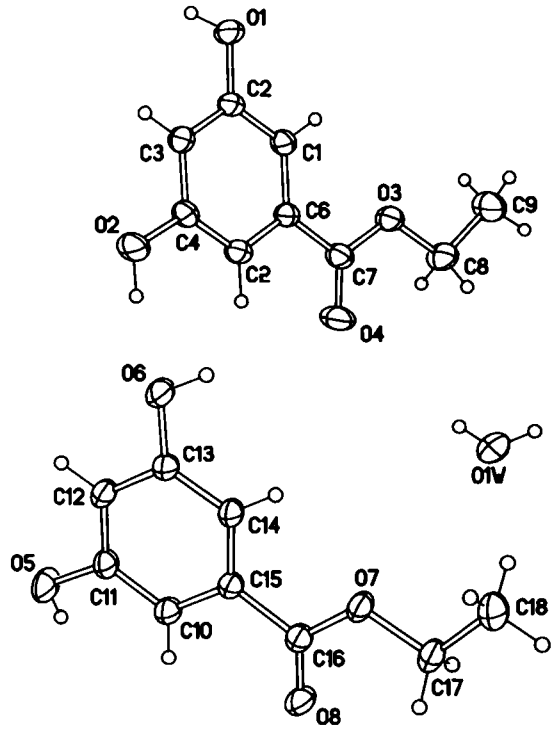

Abstract

$C_9H_{11}O_{4.5}$, monoclinic, $P12_1/c1$ (no. 14), a = 7.353(2) Å, b = 14.523(3) Å, c = 17.585(4) Å, β = 93.387(3)°, V = 1874.7 Å^3, Z = 8, $R_{gt}(F)$ = 0.040, $wR_{ref}(F^2)$ = 0.092, T = 273 K.

Source of material

A mixture of $ZnSO_4 \cdot 7H_2O$ (0.287 g, 1 mmol), 3,5-dihydroxybenzoic acid (0.156 g, 1 mmol), NaOH (0.08 g, 2 mmol) and EtOH/H_2O (1:1, *v/v*, 20 ml) was sealed in a 25 ml Teflon-lined stainless steel reactor and heated to 433 K for 60 h, and then cooled to room temperature over 3 days. Pale yellow crystals suitable for X-ray analysis were obtained.

Experimental details

While the H atoms bonded to C atoms were fixed geometrically, the positions of H atoms attached to O atoms were refined under using of restraints. More details are available in the CIF.

Discussion

One strategy in the design and synthesis of coordination architectures is the building-block approach [1-3]. In our current work, we selected the multifunctional 3,5-dihydroxybenzoic acid ligand as the main building block to construct coordination compounds.

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The title compound was obtained unexpectedly during an attempt to form an esterifiable derivative by reaction of 3,5-dihydroxybenzoic acid with $ZnSO_4$ in an ethanol-water mixture under hydrothermal condition. The X-ray analysis shows that the title structure consists of two neutral $C_9H_{10}O_4$ molecules and one lattice water molecule. In each $C_9H_{10}O_4$ unit, eight carbon and four oxygen atoms are approximately coplanar, with maximum deviations of 0.060 Å for O7 atom and 0.313 Å for O4 atom from the corresponding best planes. The dihedral angle defined by two phenyl rings is 6.24°. There are extensive O–H···O hydrogen bonds involving the hydroxy groups and water molecules. This discrete structure is further extended to form a three-dimensional network via hydrogen bonds.

Table 1. Data collection and handling.

Crystal:	pale yellow prism, size 0.20 × 0.36 × 0.38 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.10 cm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX II CCD, ω
$2\theta_{max}$:	56.32°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	12620, 4581
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2260
$N(param)_{refined}$:	265
Programs:	SHELXS-97 [4], SHELXL-97 [5], SHELXTL [6]

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

Atom	Site	x	y	z	U_{iso}
H(1A)	4e	0.8384	0.2208	1.1264	0.053
H(3A)	4e	0.6686	0.0471	0.9595	0.056
H(5A)	4e	0.6177	0.3193	0.9280	0.054
H(8A)	4e	0.6954	0.5156	1.1523	0.076
H(8B)	4e	0.9026	0.5173	1.1343	0.076
H(9A)	4e	0.8689	0.5393	1.2644	0.115
H(9B)	4e	0.9803	0.4494	1.2506	0.115
H(9C)	4e	0.7742	0.4428	1.2683	0.115
H(10A)	4e	0.3353	0.3622	0.6233	0.053
H(12A)	4e	0.4037	0.1193	0.7294	0.057
H(14A)	4e	0.1964	0.1273	0.5133	0.052
H(17A)	4e	0.1743	0.3400	0.3415	0.065
H(17B)	4e	−0.0184	0.3506	0.3752	0.065
H(18A)	4e	−0.0548	0.2660	0.2638	0.102
H(18B)	4e	−0.0917	0.2003	0.3318	0.102
H(18C)	4e	0.0953	0.1961	0.2936	0.102
H(1)	4e	0.811(2)	−0.000(1)	1.0727(8)	0.076
H(1WA)	4e	0.677(3)	0.040(2)	1.2717(8)	0.129
H(1WB)	4e	0.685(3)	0.049(2)	1.1930(9)	0.129
H(2)	4e	0.530(2)	0.210(1)	0.8333(9)	0.079
H(5)	4e	0.500(2)	0.341(1)	0.7403(9)	0.079
H(6)	4e	0.269(2)	−0.009(1)	0.5800(7)	0.081

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}
C(1)	4e	0.7869(2)	0.2107(1)	1.07749(8)	0.059(1)	0.0394(9)	0.0337(7)	−0.0016(7)	−0.0050(7)	−0.0017(7)
C(2)	4e	0.7635(2)	0.1222(1)	1.04986(8)	0.059(1)	0.0375(9)	0.0346(8)	0.0044(7)	−0.0039(7)	0.0042(7)
C(3)	4e	0.6851(2)	0.1069(1)	0.97755(8)	0.066(1)	0.0349(8)	0.0386(8)	−0.0031(8)	−0.0031(8)	−0.0031(7)
C(4)	4e	0.6316(2)	0.1809(1)	0.93240(7)	0.055(1)	0.047(1)	0.0325(8)	0.0012(8)	−0.0070(7)	−0.0010(7)
C(5)	4e	0.6543(2)	0.2697(1)	0.95863(7)	0.057(1)	0.0401(9)	0.0364(8)	0.0030(8)	−0.0038(7)	0.0044(7)
C(6)	4e	0.7329(2)	0.28424(9)	1.03158(7)	0.050(1)	0.0344(9)	0.0351(8)	−0.0008(7)	−0.0001(7)	0.0014(7)
C(7)	4e	0.7557(2)	0.3807(1)	1.05778(8)	0.065(1)	0.044(1)	0.0374(9)	−0.0021(8)	−0.0039(8)	0.0027(8)
C(8)	4e	0.8109(3)	0.4838(1)	1.16051(8)	0.098(1)	0.037(1)	0.054(1)	−0.0041(9)	−0.0126(9)	−0.0056(8)
C(9)	4e	0.8632(3)	0.4783(1)	1.24326(8)	0.123(2)	0.056(1)	0.050(1)	−0.010(1)	−0.013(1)	−0.0053(9)
C(10)	4e	0.3265(2)	0.2984(1)	0.62278(7)	0.058(1)	0.0343(8)	0.0388(8)	−0.0023(7)	−0.0040(7)	0.0012(7)
C(11)	4e	0.3821(2)	0.2471(1)	0.68654(7)	0.060(1)	0.042(1)	0.0317(8)	−0.0032(8)	−0.0069(7)	−0.0027(7)
C(12)	4e	0.3672(2)	0.1529(1)	0.68621(8)	0.068(1)	0.0405(9)	0.0337(8)	0.0016(8)	−0.0070(7)	0.0061(7)
C(13)	4e	0.2977(2)	0.1083(1)	0.62150(8)	0.064(1)	0.0331(9)	0.0347(8)	−0.0002(8)	−0.0032(7)	0.0001(7)
C(14)	4e	0.2427(2)	0.15750(9)	0.55691(7)	0.060(1)	0.0377(9)	0.0303(7)	−0.0009(8)	−0.0070(7)	−0.0002(6)
C(15)	4e	0.2577(2)	0.25244(9)	0.55826(7)	0.0463(9)	0.0349(8)	0.0324(7)	0.0006(7)	−0.0029(6)	0.0014(6)
C(16)	4e	0.1999(2)	0.3091(1)	0.49046(7)	0.048(1)	0.0392(9)	0.0373(8)	−0.0003(7)	−0.0026(7)	0.0021(7)
C(17)	4e	0.0753(2)	0.3068(1)	0.36321(7)	0.070(1)	0.054(1)	0.0363(8)	0.0033(9)	−0.0087(8)	0.0088(8)
C(18)	4e	−0.0008(2)	0.2359(1)	0.30814(8)	0.093(1)	0.065(1)	0.045(1)	−0.000(1)	−0.0136(9)	−0.0048(9)
O(1)	4e	0.8188(2)	0.05105(7)	1.09672(6)	0.110(1)	0.0369(6)	0.0406(6)	0.0096(7)	−0.0137(6)	0.0003(5)
O(1W)	4e	0.6257(2)	0.0609(1)	1.23074(7)	0.132(1)	0.0694(9)	0.0564(8)	0.0361(9)	0.0036(8)	0.0197(7)
O(2)	4e	0.5553(2)	0.16044(8)	0.86177(6)	0.104(1)	0.0512(8)	0.0388(6)	−0.0012(7)	−0.0237(6)	−0.0017(5)
O(3)	4e	0.7959(2)	0.38972(7)	1.13174(5)	0.0815(8)	0.0359(6)	0.0399(6)	−0.0035(5)	−0.0064(5)	−0.0013(5)
O(4)	4e	0.7406(2)	0.44729(7)	1.01649(6)	0.152(1)	0.0405(7)	0.0450(6)	−0.0066(7)	−0.0148(7)	0.0085(6)
O(5)	4e	0.4525(2)	0.28795(7)	0.75249(6)	0.107(1)	0.0499(7)	0.0382(6)	−0.0168(7)	−0.0221(6)	0.0002(5)
O(6)	4e	0.2871(2)	0.01409(7)	0.62501(5)	0.129(1)	0.0331(7)	0.0378(6)	−0.0016(7)	−0.0143(7)	0.0025(5)
O(7)	4e	0.1409(1)	0.25778(6)	0.43131(5)	0.0733(7)	0.0416(6)	0.0343(5)	−0.0002(6)	−0.0137(5)	0.0064(5)
O(8)	4e	0.2050(2)	0.39201(7)	0.48907(5)	0.0837(8)	0.0347(6)	0.0482(6)	−0.0010(6)	−0.0113(6)	0.0074(5)

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