Crystal structure of triethylammonium 3,5-dihydroxybenzoate, $[C_6H_{16}N][C_7H_5O_4]$

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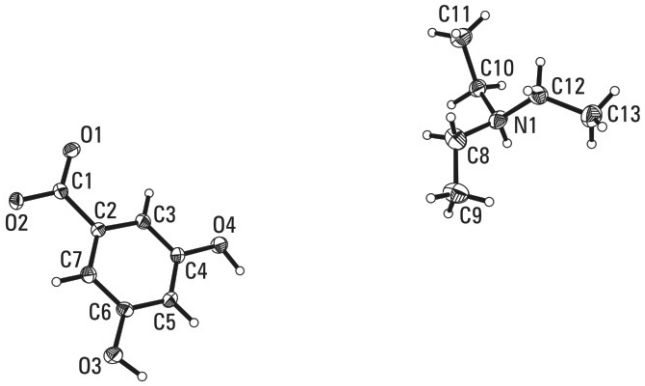
Received July 8, 2011, accepted and available on-line November 14, 2011; CCDC no. 1267/3621



Abstract

$C_{13}H_{21}NO_4$, orthorhombic, *Pbca* (no. 61), a = 13.904(2) Å, b = 14.221(2) Å, c = 14.621(1) Å, V = 2891.1 Å^3, Z = 8, $R_{gt}(F)$ = 0.054, $wR_{ref}(F^2)$ = 0.093, T = 291 K.

Source of material

The triethylamine was added slowly to a mixture of zirconocene dichloride (Cp_2ZrCl_2) (146 mg, 0.5 mmol) and 3,5-dihydroxybenzoic acid (154 mg, 1.1 mmol) dissolved in acetonitrile (15 mL). The reaction mixture was first stirred for 10 min at room temperature under anaerobic conditions and then transferred to a Teflon-lined reactor and sealed. The reactor was heated to 81 °C and kept for 1d, then cooled to room temperature. The colorless block-shaped crystals were obtained for single-crystal X-ray diffraction experiment.

Discussion

In the title crystal structure, each asymmetric unit contains a 3,5-dihydroxybenzoate anion, which transfers a proton to triethylamine to form the three-dimensional network structure. The crystal structure of triethylammonium benzoate salt is similar to that of its analogue salicylic salts [1,2]. Among the complex intermolecular interactions [3,4], the most important hydrogen bonding is N1–H1···O2 (bond length 2.744(2) Å; bond angle 174.4°), providing the main driving force for direct binding of carboxylate anion [3,5-dihydroxybenzoate]$^-$ with triethylammonium cation $[Et_3NH]^+$. In addition, the hydrogen bonds O4–H4D···O2 and O3–H3B···O1 (bond lengths 2.58(2) Å and 2.68(2) Å and bond angles 163.25° and 162.4°, respectively) are another important force to stabilize the crystal structure [5].

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.20 × 0.22 × 0.28 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.86 cm^{-1}
Diffractometer, scan mode:	Bruker Smart Apex CCD, φ/ω
$2\theta_{max}$:	52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	14753, 2838
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1673
$N(param)_{refined}$:	166
Program:	SHELXTL [6]

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

Atom	Site	x	y	z	U_{iso}
H(3A)	8*c*	1.0046	0.2857	0.6085	0.040
H(5)	8*c*	0.9983	0.3723	0.8695	0.043
H(7)	8*c*	0.9147	0.1113	0.8057	0.043
H(8A)	8*c*	0.8002	0.8215	0.5571	0.070
H(8B)	8*c*	0.6905	0.8435	0.5613	0.070
H(9A)	8*c*	0.8217	0.8565	0.7054	0.095
H(9B)	8*c*	0.7238	0.8020	0.7044	0.095
H(9C)	8*c*	0.7245	0.9120	0.7114	0.095
H(10A)	8*c*	0.8779	0.8943	0.4662	0.060
H(10B)	8*c*	0.8572	0.9964	0.4326	0.060
H(11A)	8*c*	0.7144	0.8730	0.3851	0.102
H(11B)	8*c*	0.8139	0.8505	0.3396	0.102
H(11C)	8*c*	0.7650	0.9485	0.3235	0.102
H(12A)	8*c*	0.6315	0.9777	0.5315	0.064
H(12B)	8*c*	0.6874	1.0258	0.4520	0.064
H(13A)	8*c*	0.7315	1.1251	0.5980	0.102
H(13B)	8*c*	0.6203	1.1074	0.6037	0.102
H(13C)	8*c*	0.6643	1.1572	0.5177	0.102
H(1)	8*c*	0.8111	0.9831	0.5693	0.056
H(3B)	8*c*	0.9458	0.2715	0.9784	0.048
H(4D)	8*c*	1.0490	0.4688	0.7566	0.051

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)	8*c*	0.9304(1)	0.1147(1)	0.6261(1)	0.046(1)	0.029(1)	0.033(1)	0.0037(8)	–0.0022(8)	0.0015(9)
C(2)	8*c*	0.9555(1)	0.1870(1)	0.6959(1)	0.042(1)	0.029(1)	0.028(1)	0.0015(8)	–0.0012(8)	–0.0018(8)
C(3)	8*c*	0.9914(1)	0.2737(1)	0.6698(1)	0.041(1)	0.031(1)	0.028(1)	–0.0029(8)	0.0049(8)	0.0039(7)

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Table 3. Continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(4)	8*c*	1.0077(2)	0.3421(1)	0.7345(1)	0.048(1)	0.028(1)	0.035(1)	–0.0066(8)	–0.0023(9)	0.0016(8)
C(5)	8*c*	0.9878(1)	0.3257(1)	0.8260(1)	0.043(1)	0.031(1)	0.032(1)	–0.0011(8)	0.0003(8)	–0.0069(8)
C(6)	8*c*	0.9521(1)	0.2391(1)	0.8517(1)	0.041(1)	0.039(1)	0.026(1)	–0.0030(9)	0.0053(8)	0.0036(9)
C(7)	8*c*	0.9372(1)	0.1700(1)	0.7875(1)	0.040(1)	0.033(1)	0.035(1)	–0.0079(8)	0.0002(8)	0.0034(9)
C(8)	8*c*	0.7532(2)	0.8654(2)	0.5811(2)	0.053(2)	0.058(2)	0.065(2)	–0.003(1)	–0.006(1)	0.011(1)
C(9)	8*c*	0.7561(2)	0.8583(2)	0.6852(2)	0.061(2)	0.072(2)	0.058(2)	–0.023(1)	–0.013(1)	0.013(1)
C(10)	8*c*	0.8268(2)	0.9379(2)	0.4506(1)	0.048(1)	0.051(1)	0.053(1)	0.017(1)	–0.013(1)	–0.015(1)
C(11)	8*c*	0.7753(2)	0.8989(2)	0.3670(2)	0.076(2)	0.071(2)	0.058(2)	0.015(1)	–0.014(1)	–0.020(1)
C(12)	8*c*	0.6881(2)	1.0149(2)	0.5175(2)	0.051(1)	0.061(2)	0.048(1)	0.019(1)	–0.005(1)	–0.005(1)
C(13)	8*c*	0.6748(2)	1.1099(2)	0.5635(2)	0.064(2)	0.068(2)	0.073(2)	0.019(1)	–0.015(1)	–0.020(1)
N(1)	8*c*	0.7704(1)	0.9570(1)	0.5367(1)	0.0386(9)	0.046(1)	0.054(1)	–0.0031(8)	–0.0130(7)	–0.0128(9)
O(1)	8*c*	0.9329(1)	0.13455(9)	0.54324(9)	0.0523(9)	0.0411(8)	0.0307(8)	–0.0136(6)	–0.0034(6)	–0.0012(6)
O(2)	8*c*	0.90367(9)	0.03390(9)	0.65318(8)	0.0421(8)	0.0274(7)	0.0421(8)	–0.0074(5)	–0.0079(6)	0.0030(6)
O(3)	8*c*	0.93228(9)	0.2179(1)	0.94065(8)	0.0467(8)	0.0453(9)	0.0285(7)	–0.0141(6)	0.0150(6)	–0.0011(6)
O(4)	8*c*	1.04206(9)	0.42713(9)	0.70534(9)	0.0501(9)	0.0402(8)	0.0364(8)	–0.0182(6)	0.0030(6)	0.0020(6)

Acknowledgments. We would like to thank the National Natural Science Foundation of China (grant no. 21041004), the Program for New Century Excellent Talents in Universities of China and Natural Science Foundation of Shaanxi Province (grant no. 2007B06).

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