Crystal structure of *N*-butylammonium phthalate monohydrate, $2(C_4H_{12}N)^+(C_8H_4O_4)^{2-}\cdot H_2O$, $C_{16}H_{30}N_2O_5$

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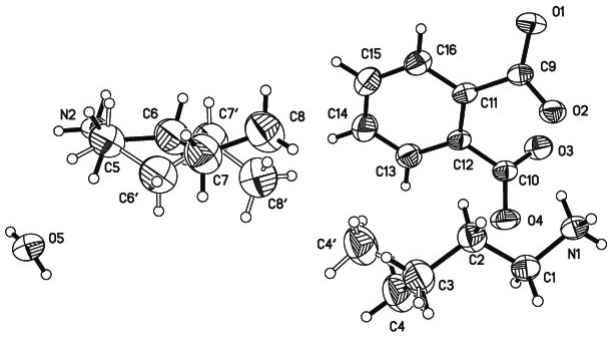

Abstract

$C_{16}H_{30}N_2O_5$, orthorhombic, *Pbca* (no. 61), a = 14.925(1) Å, b = 9.3729(8) Å, c = 27.583(3) Å, V = 3858.5 Å^3, Z = 8, $R_{gt}(F)$ = 0.0811, $wR_{ref}(F^2)$ = 0.2655, T = 298 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.370.40×0.48 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.84 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	50.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	14795, 3370
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1649
$N(param)_{refined}$:	252
Programs:	SHELXS-97 [3]

Source of material

All the reagents and solvents from commercial sources were used without further purification. A mixture of phthalic anhydride (1.52 g, 0.01 mol), butan-1-amine (0.73 g, 0.01 mol) and water (15 ml) was refluxed for 2 h. Then the above solution was kept at room temperature for 12 d. Isothermal evaporation gave colourless single crystals of the title compound, suitable for *X*-ray analysis.

Discussion

Phthalimides and *N*-substituted phthalimides are an important class of compounds because of their interesting biological activities [1]. *N*-butylammonium phthalate is an intermediate in the preparation of *N*-substituted phthalimides. In this paper, the crystal structure of the title compound is reported.

The asymmetric unit of the title compound contains two *N*-butylammonium cations, one phthalate anion and one water solvent molecule. In the anion of the title compound, the dihedral angles formed by the benzene ring and the mean planes of the two carboxylate groups are 41.5(2) and 55.8(2)°, respectively. The atom C4 of one *N*-butylammonium cation is disordered, while the atoms of C6, C7 and C8 of the another one are disordered. The bond lengths and angles are in agreement with those of the related hexamethylenediammonium phthalate trihydrate [2]. The crystal structure is stabilized by N–H···O and O–H···O hydrogen bonds.

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(1A)	8*c*		0.6308	0.4246	0.4628	0.086
H(1B)	8*c*		0.6400	0.5404	0.4975	0.086
H(1C)	8*c*		0.7182	0.4585	0.4830	0.086
H(2A)	8*c*		0.5602	0.2122	0.0607	0.101
H(2B)	8*c*		0.5871	0.3069	0.0216	0.101
H(2C)	8*c*		0.5461	0.3650	0.0651	0.101
H(5E)	8*c*		0.6279	0.5671	–0.0160	0.094
H(5F)	8*c*		0.6430	0.6711	0.0184	0.094
H(1D)	8*c*		0.7299	0.6597	0.4423	0.080
H(1E)	8*c*		0.6285	0.6500	0.4275	0.080
H(2D)	8*c*		0.6620	0.4466	0.3799	0.092
H(2E)	8*c*		0.7636	0.4653	0.3930	0.092
H(3A)	8*c*	0.594	0.7625	0.6896	0.3533	0.128
H(3B)	8*c*	0.594	0.7449	0.5624	0.3178	0.128
H(3'A)	8*c*	0.406	0.6766	0.6933	0.3474	0.128
H(3'B)	8*c*	0.406	0.7790	0.6581	0.3454	0.128
H(4A)	8*c*	0.594	0.6510	0.7448	0.3007	0.188
H(4B)	8*c*	0.594	0.6133	0.7414	0.3538	0.188
H(4C)	8*c*	0.594	0.5952	0.6132	0.3186	0.188
H(4'1)	8*c*	0.406	0.6497	0.5004	0.2947	0.188
H(4'2)	8*c*	0.406	0.7535	0.4768	0.2908	0.188
H(4'3)	8*c*	0.406	0.7104	0.6175	0.2708	0.188
H(5A)	8*c*	0.507	0.6987	0.4047	0.0681	0.129
H(5B)	8*c*	0.507	0.7121	0.2375	0.0657	0.129
H(5'A)	8*c*	0.493	0.7098	0.3550	0.0511	0.129
H(5'B)	8*c*	0.493	0.6941	0.2168	0.0806	0.129
H(6A)	8*c*	0.507	0.6031	0.3694	0.1377	0.152
H(6B)	8*c*	0.507	0.6278	0.2072	0.1368	0.152
H(7A)	8*c*	0.507	0.7410	0.4307	0.1650	0.159
H(7B)	8*c*	0.507	0.7794	0.2767	0.1542	0.159
H(8A)	8*c*	0.507	0.6300	0.3125	0.2169	0.223
H(8B)	8*c*	0.507	0.7274	0.3111	0.2382	0.223
H(8C)	8*c*	0.507	0.6890	0.1748	0.2126	0.223
H(6'1)	8*c*	0.493	0.7557	0.3948	0.1283	0.151
H(6'2)	8*c*	0.493	0.6692	0.4883	0.1209	0.151
H(7'1)	8*c*	0.493	0.6617	0.2118	0.1624	0.154
H(7'2)	8*c*	0.493	0.5817	0.3228	0.1612	0.154
H(8'1)	8*c*	0.493	0.7400	0.3422	0.2181	0.217
H(8'2)	8*c*	0.493	0.6440	0.3931	0.2333	0.217
H(8'3)	8*c*	0.493	0.7024	0.4874	0.1986	0.217
H(13)	8*c*		0.4220	0.4571	0.3293	0.088
H(14)	8*c*		0.4472	0.3282	0.2607	0.105
H(15)	8*c*		0.5221	0.1132	0.2651	0.109
H(16)	8*c*		0.5601	0.0205	0.3396	0.088

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

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	8*c*		0.6663(2)	0.4948(3)	0.4730(1)	0.049(2)	0.052(2)	0.070(2)	–0.004(1)	0.002(2)	–0.008(2)
N(2)	8*c*		0.5824(2)	0.2978(3)	0.0536(1)	0.068(2)	0.052(2)	0.082(2)	0.006(2)	0.002(2)	0.002(2)
O(1)	8*c*		0.5505(2)	0.0008(3)	0.4347(1)	0.071(2)	0.043(2)	0.093(2)	–0.004(1)	–0.006(2)	0.007(1)
O(2)	8*c*		0.5913(2)	0.2200(3)	0.45430(9)	0.071(2)	0.050(2)	0.073(2)	–0.006(1)	–0.016(1)	0.001(1)
O(3)	8*c*		0.4006(2)	0.3436(3)	0.4519(1)	0.074(2)	0.067(2)	0.088(2)	–0.008(2)	0.026(2)	–0.007(2)
O(4)	8*c*		0.4717(2)	0.5282(3)	0.4217(1)	0.100(2)	0.046(2)	0.099(2)	–0.003(2)	0.008(2)	–0.009(2)
O(5)	8*c*		0.6639(2)	0.5952(3)	0.0059(1)	0.058(2)	0.072(2)	0.104(2)	0.006(1)	–0.004(2)	–0.014(2)
C(1)	8*c*		0.6823(3)	0.5944(5)	0.4333(1)	0.062(2)	0.063(3)	0.076(3)	–0.005(2)	–0.001(2)	–0.002(2)
C(2)	8*c*		0.7080(3)	0.5163(5)	0.3875(2)	0.074(3)	0.079(3)	0.077(3)	–0.004(2)	–0.001(2)	0.003(3)
C(3)	8*c*		0.7196(4)	0.6161(7)	0.3445(2)	0.114(5)	0.112(5)	0.093(4)	–0.007(4)	0.002(3)	0.014(4)
C(4)	8*c*	0.594	0.6379(8)	0.685(1)	0.3280(4)	0.125(9)	0.15(1)	0.105(8)	–0.010(7)	–0.021(6)	0.029(7)
C(4')	8*c*	0.406	0.707(1)	0.546(2)	0.2958(6)	0.13(1)	0.15(2)	0.11(1)	–0.01(1)	–0.021(9)	0.03(1)
C(5)	8*c*		0.6721(4)	0.3132(7)	0.0759(2)	0.100(4)	0.110(5)	0.113(5)	–0.012(3)	–0.026(3)	0.013(4)
C(6)	8*c*	0.507	0.651(2)	0.302(3)	0.1303(7)	0.13(1)	0.13(1)	0.12(2)	–0.03(1)	–0.03(1)	0.02(1)
C(7)	8*c*	0.507	0.727(1)	0.330(2)	0.1638(6)	0.13(1)	0.15(1)	0.13(1)	–0.01(1)	–0.02(1)	0.01(1)
C(8)	8*c*	0.507	0.690(1)	0.277(3)	0.2124(7)	0.15(1)	0.16(2)	0.14(1)	–0.02(1)	–0.02(1)	0.03(1)
C(6')	8*c*	0.493	0.692(1)	0.391(2)	0.1226(6)	0.12(1)	0.13(2)	0.13(1)	–0.01(1)	–0.02(1)	0.01(1)
C(7')	8*c*	0.493	0.646(2)	0.312(2)	0.1626(7)	0.12(2)	0.14(1)	0.12(2)	–0.01(1)	–0.01(1)	0.01(2)
C(8')	8*c*	0.493	0.687(1)	0.391(3)	0.2075(8)	0.15(1)	0.15(2)	0.13(1)	0.00(1)	–0.00(1)	0.01(1)
C(9)	8*c*		0.5547(2)	0.1317(4)	0.4269(1)	0.045(2)	0.045(2)	0.068(2)	–0.001(2)	0.002(2)	–0.004(2)
C(10)	8*c*		0.4470(2)	0.4024(4)	0.4203(1)	0.047(2)	0.047(2)	0.070(3)	0.005(2)	–0.006(2)	0.000(2)
C(11)	8*c*		0.5177(2)	0.1873(4)	0.3802(1)	0.052(2)	0.045(2)	0.066(2)	–0.004(2)	–0.002(2)	–0.008(2)
C(12)	8*c*		0.4731(2)	0.3172(4)	0.3770(1)	0.052(2)	0.045(2)	0.064(2)	–0.007(2)	–0.005(2)	–0.003(2)
C(13)	8*c*		0.4496(3)	0.3684(5)	0.3317(2)	0.081(3)	0.064(3)	0.076(3)	0.003(2)	–0.015(2)	–0.001(2)
C(14)	8*c*		0.4656(3)	0.2927(6)	0.2906(2)	0.105(4)	0.087(4)	0.072(3)	0.002(3)	–0.017(3)	–0.004(3)
C(15)	8*c*		0.5091(4)	0.1637(6)	0.2933(2)	0.110(4)	0.086(4)	0.075(3)	0.002(3)	–0.005(3)	–0.018(3)
C(16)	8*c*		0.5331(3)	0.1098(5)	0.3377(2)	0.079(3)	0.063(3)	0.078(3)	0.006(2)	–0.001(2)	–0.011(2)

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