Crystal structure of *p*-carboxyanilinium 3,4,5,6-tetrabromo-2-(methoxycarbonyl)benzoate methanol monosolvate, $(C_7H_8NO_2)^+(C_9H_3Br_4O_4)^-\cdot CH_3OH$, $C_{17}H_{15}Br_4NO_7$

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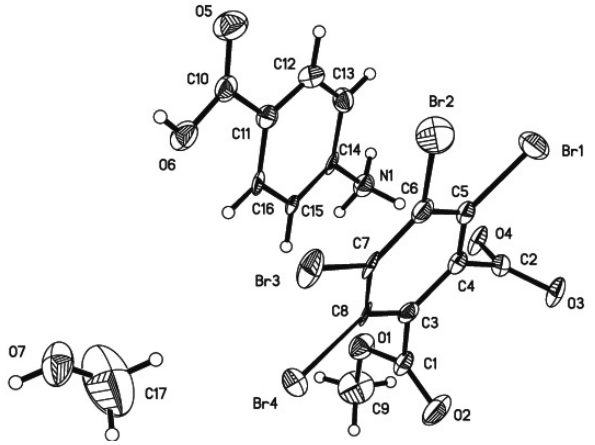

Abstract

$C_{17}H_{15}Br_4NO_7$, monoclinic, $P2_1/c$ (no. 14), a = 11.076(1) Å, b = 17.571(2) Å, c = 13.092(1) Å, β = 120.100(2)°, V = 2204.4 Å^3, Z = 4, $R_{gt}(F)$ = 0.0690, $wR_{ref}(F^2)$ = 0.1692, T = 298 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.22×0.28×0.32 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	73.40 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	50.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11118, 3890
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2108
$N(param)_{refined}$:	265
Programs:	SHELXS-97 [4]

Soure of material

All the reagents and solvents from commercial sources were used without further purification. A mixture of 4,5,6,7-tetrabromo-isobenzofuran-1,3-dione (4.64 g, 0.01 mol) and methanol (15 ml) was refluxed for 0.5 h. Then *p*-carboxyaniline (1.37 g, 0.01 mol) was added to the above solution and mixed for 0.5 h at room temperature. The above solution was kept at room temperature for 5 d. Isothermal evaporation gave colourless single crystals of the title compound, suitable for *X*-ray analysis.

Discussion

Tetrabromphthalimide and its derivatives have been found to be useful flame retardants in polyesters, e.g. polybutylene terephthalate, and other resin formulations [1]. In the present work, the reaction of tetrabromophthalic acid anhydride and *p*-carboxyaniline in methanol is expected to form 2-(4-carboxy benzenyl)-4,5,6,7-tetrabromoisoindoline-1,3-dione, but instead the title compound formed, and this may have occured because of the reduced time and temperature of the reaction. In this paper, the crystal structure of the title compound is reported.

The asymmetric unit of the title compound contains one *p*-carboxyanilinium cation, one 3,4,5,6-tetrabromo-2-(methoxy carbonyl)benzoate anion and one methanol molecule. In the anion, the dihedral angles formed by the benzene ring and the mean planes of the carboxylate and methoxycarbonyl groups are 83.3(2)° and 85.0(2)°, respectively. All non-hydrogen atoms of the cation are essentially planar with a mean deviation of 0.0159 Å. The bond lengths and angles are in agreement with those reported for the ethaneaminium-2-(methoxycarbonyl)-3,4,5,6-tetrabromobenzoate methanol monosolvate [2] and for the 2-methylanilinium 3,4,5,6-tetrabromo-2-(methoxycarbonyl) benzoate methanol monosolvate [3]. 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	x	y	z	U_{iso}
H(1A)	4*e*	0.1994	0.1774	0.5364	0.061
H(1B)	4*e*	0.1871	0.1317	0.4391	0.061
H(1C)	4*e*	0.2017	0.2138	0.4378	0.061
H(6)	4*e*	0.9699	0.1941	0.7759	0.075
H(7)	4*e*	1.1103	0.4921	0.8549	0.097
H(9A)	4*e*	0.0427	0.3880	0.5179	0.121
H(9B)	4*e*	0.1279	0.4154	0.4586	0.121
H(9C)	4*e*	0.1491	0.4553	0.5735	0.121
H(12)	4*e*	0.6186	0.0366	0.6470	0.052
H(13)	4*e*	0.3765	0.0490	0.5454	0.048
H(15)	4*e*	0.4187	0.2758	0.5572	0.036
H(16)	4*e*	0.6609	0.2619	0.6545	0.040
H(17A)	4*e*	0.9307	0.4242	0.8834	0.298
H(17B)	4*e*	0.9078	0.4572	0.7637	0.298
H(17C)	4*e*	0.9362	0.5124	0.8675	0.298

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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}
Br(1)	4*e*	0.3936(1)	0.06706(5)	0.8736(1)	0.0664(8)	0.0384(5)	0.0810(8)	0.0016(5)	0.0389(7)	0.0120(5)
Br(2)	4*e*	0.7277(1)	0.10729(7)	0.9883(1)	0.0518(8)	0.0824(8)	0.0892(9)	0.0298(6)	0.0303(8)	0.0282(7)
Br(3)	4*e*	0.8226(1)	0.28064(6)	0.96415(9)	0.0325(6)	0.0956(8)	0.0530(7)	–0.0126(6)	0.0277(6)	–0.0153(6)
Br(4)	4*e*	0.5779(1)	0.40690(5)	0.7999(1)	0.0724(8)	0.0444(6)	0.0889(9)	–0.0172(5)	0.0559(8)	–0.0074(5)
N(1)	4*e*	0.2268(7)	0.1726(4)	0.4835(6)	0.046(5)	0.046(4)	0.036(4)	0.001(4)	0.026(4)	–0.002(3)
O(1)	4*e*	0.2400(7)	0.3543(3)	0.6033(5)	0.054(5)	0.058(4)	0.040(4)	0.015(3)	0.025(4)	0.007(3)
O(2)	4*e*	0.2285(9)	0.3868(4)	0.7599(6)	0.100(7)	0.084(5)	0.059(5)	0.040(5)	0.060(5)	0.016(4)
O(3)	4*e*	0.1496(6)	0.2077(4)	0.8136(5)	0.035(4)	0.073(4)	0.040(4)	–0.011(3)	0.029(3)	–0.011(3)
O(4)	4*e*	0.1276(6)	0.1842(4)	0.6371(5)	0.042(4)	0.078(4)	0.032(4)	–0.007(3)	0.028(4)	–0.012(3)
O(5)	4*e*	0.8750(7)	0.0767(4)	0.7377(7)	0.051(5)	0.060(4)	0.082(5)	0.005(4)	0.034(5)	–0.008(4)
O(6)	4*e*	0.8859(7)	0.2026(3)	0.7440(6)	0.035(4)	0.060(4)	0.059(4)	–0.002(3)	0.028(4)	–0.011(3)
O(7)	4*e*	1.0874(9)	0.4591(4)	0.8861(8)	0.060(6)	0.089(5)	0.109(7)	0.031(5)	0.054(6)	0.047(5)
C(1)	4*e*	0.278(1)	0.3509(5)	0.7148(8)	0.045(6)	0.051(5)	0.035(6)	0.000(5)	0.030(6)	–0.005(5)
C(2)	4*e*	0.195(1)	0.2023(4)	0.7436(9)	0.040(6)	0.035(4)	0.041(6)	0.002(4)	0.027(6)	0.003(4)
C(3)	4*e*	0.391(1)	0.2918(4)	0.7806(7)	0.044(6)	0.037(5)	0.023(5)	0.004(5)	0.022(5)	–0.004(4)
C(4)	4*e*	0.3499(9)	0.2187(5)	0.7942(7)	0.041(6)	0.040(5)	0.028(5)	0.001(4)	0.024(5)	–0.003(4)
C(5)	4*e*	0.453(1)	0.1645(4)	0.8572(7)	0.045(6)	0.037(5)	0.031(5)	0.003(5)	0.028(5)	0.001(4)
C(6)	4*e*	0.593(1)	0.1819(5)	0.9036(8)	0.045(6)	0.044(5)	0.035(5)	0.006(5)	0.029(5)	–0.002(4)
C(7)	4*e*	0.6315(9)	0.2559(5)	0.8899(7)	0.035(6)	0.055(5)	0.025(5)	–0.002(5)	0.027(5)	–0.008(4)
C(8)	4*e*	0.5299(9)	0.3087(5)	0.8276(7)	0.039(6)	0.044(5)	0.030(5)	–0.007(4)	0.033(5)	–0.009(4)
C(9)	4*e*	0.131(1)	0.4077(6)	0.5324(9)	0.072(9)	0.089(8)	0.054(7)	0.033(7)	0.011(7)	0.024(6)
C(10)	4*e*	0.818(1)	0.1386(5)	0.7170(8)	0.051(7)	0.045(6)	0.039(6)	–0.006(5)	0.032(6)	–0.008(5)
C(11)	4*e*	0.663(1)	0.1474(5)	0.6576(7)	0.046(6)	0.039(5)	0.032(5)	0.001(5)	0.026(5)	0.000(4)
C(12)	4*e*	0.579(1)	0.0847(5)	0.6275(8)	0.045(7)	0.039(5)	0.050(6)	0.007(5)	0.027(6)	0.000(4)
C(13)	4*e*	0.434(1)	0.0916(5)	0.5682(8)	0.041(6)	0.040(5)	0.046(6)	–0.011(5)	0.026(6)	–0.005(4)
C(14)	4*e*	0.3800(9)	0.1644(5)	0.5449(7)	0.039(6)	0.044(5)	0.028(5)	0.000(5)	0.031(5)	–0.003(4)
C(15)	4*e*	0.4593(9)	0.2277(4)	0.5747(7)	0.049(6)	0.030(4)	0.030(5)	0.000(4)	0.035(5)	0.000(4)
C(16)	4*e*	0.604(1)	0.2191(5)	0.6326(7)	0.045(6)	0.042(5)	0.036(5)	–0.005(5)	0.037(5)	–0.002(4)
C(17)	4*e*	0.958(2)	0.464(1)	0.848(2)	0.12(2)	0.19(2)	0.26(3)	0.03(2)	0.07(2)	0.13(2)

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