Crystal structure of methylammonium 2, 3, 4, 5-tetrabromo-6-(methoxy carbonyl)benzoate monohydrate $(CH_6N)(C_9H_3Br_4O_4)\cdot H_2O$, $C_{10}H_{11}Br_4NO_5$

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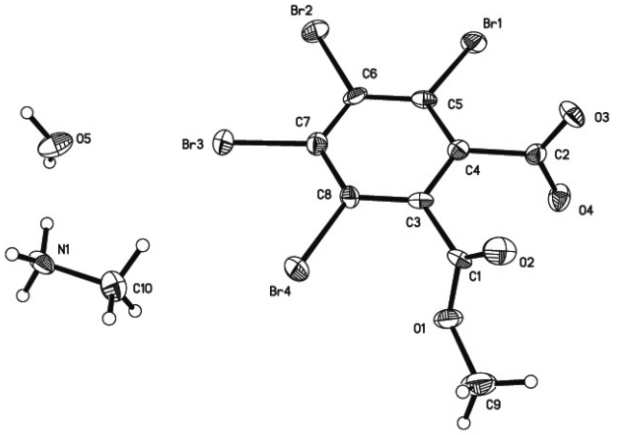

Abstract

$C_{10}H_{11}Br_4NO_5$, monoclinic, *Cc* (no. 9), a = 8.3756(7) Å, b = 23.781(2) Å, c = 8.2055(7) Å, β = 98.602(1)°, V = 1616.0Å^3, Z = 4, $R_{gt}(F)$ = 0.0422, $wR_{ref}(F^2)$ = 0.1093, T = 298 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.23×0.30×0.34 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	99.76 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	50.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4040, 2051
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1679
$N(param)_{refined}$:	185
Programs:	SHELXS-97 [4]

Source 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 a methylamine water solution (25%) (1.24 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 8 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 anhydride and methylamine in methanol is expected to form 2-methyl-4,5,6,7-tetrabromoisoindoline-1,3-dione, but instead the title compound formed, and this may have occured because of the presence of water. In this paper, the crystal structure of the title compound is reported.

The asymmetric unit of the title compound contains one methylammonium cation, one 2,3,4,5-tetrabromo-6-(methoxy carbonyl)benzoate anion and one water molecule. In the anion, the dihedral angles formed by the benzene ring and the mean planes of the carboxylate and methoxycarbonyl groups are 77.2(2) and 66.2(2)°, respectively. The bond lengths and angles are in agreement with those found in ethylammonium 2-(methoxy carbonyl)-2,3,4,5-tetrabromobenzoate methanol monosolvate [2] and in 2-methylanilinium 2,3,4,5-tetrabromo-6-(methoxy carbonyl)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*a*	0.5334	0.2848	0.5053	0.058
H(1B)	4*a*	0.3717	0.3085	0.4831	0.058
H(1C)	4*a*	0.4455	0.2890	0.6448	0.058
H(5C)	4*a*	0.2009	0.3018	0.2441	0.071
H(5D)	4*a*	0.0925	0.2932	0.3521	0.071
H(9A)	4*a*	0.8266	0.6822	0.6413	0.106
H(9B)	4*a*	0.9121	0.6302	0.5748	0.106
H(9C)	4*a*	0.8301	0.6244	0.7337	0.106
H(10A)	4*a*	0.4475	0.3883	0.5986	0.073
H(10B)	4*a*	0.5908	0.3738	0.5029	0.073
H(10C)	4*a*	0.6056	0.3610	0.6919	0.073

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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*a*	–0.0129(1)	0.68019(5)	0.2028(1)	0.0295(7)	0.0461(7)	0.0546(8)	0.0060(6)	–0.0050(6)	0.0113(6)
Br(2)	4*a*	–0.0785(2)	0.54235(6)	0.1775(2)	0.0276(8)	0.0572(9)	0.085(1)	–0.0087(7)	–0.0104(7)	–0.0066(7)
Br(3)	4*a*	0.2018(2)	0.45508(5)	0.3502(2)	0.0426(9)	0.0304(6)	0.0703(9)	–0.0056(6)	0.0057(7)	–0.0036(6)
Br(4)	4*a*	0.5287(2)	0.50713(5)	0.5782(2)	0.0376(8)	0.0348(7)	0.076(1)	0.0088(6)	–0.0069(6)	0.0082(6)
N(1)	4*a*	0.464(1)	0.3055(4)	0.552(1)	0.037(6)	0.041(6)	0.042(6)	0.017(5)	0.015(5)	0.008(4)
O(1)	4*a*	0.671(1)	0.6262(3)	0.526(1)	0.023(5)	0.058(5)	0.057(6)	–0.001(4)	0.005(4)	–0.014(4)
O(2)	4*a*	0.533(1)	0.6613(4)	0.713(1)	0.043(6)	0.071(6)	0.040(5)	0.003(5)	0.003(4)	–0.024(5)
O(3)	4*a*	0.230(1)	0.7367(3)	0.537(1)	0.049(5)	0.035(4)	0.041(5)	0.015(4)	0.021(4)	0.003(4)
O(4)	4*a*	0.403(1)	0.7352(3)	0.352(1)	0.055(6)	0.035(4)	0.044(5)	–0.009(4)	0.020(4)	–0.003(4)
O(5)	4*a*	0.172(1)	0.3131(4)	0.334(1)	0.044(6)	0.088(7)	0.050(6)	–0.014(5)	0.020(5)	–0.003(5)
C(1)	4*a*	0.538(2)	0.6361(4)	0.589(1)	0.023(6)	0.034(6)	0.030(6)	0.013(5)	0.004(5)	0.004(5)
C(2)	4*a*	0.302(1)	0.7125(5)	0.432(1)	0.027(7)	0.029(6)	0.041(7)	–0.001(5)	–0.003(6)	0.001(5)
C(3)	4*a*	0.392(1)	0.6127(5)	0.486(1)	0.017(6)	0.042(7)	0.029(6)	0.002(5)	0.003(5)	0.003(5)
C(4)	4*a*	0.274(1)	0.6500(4)	0.410(1)	0.029(7)	0.031(6)	0.027(6)	–0.001(5)	0.012(5)	0.006(5)
C(5)	4*a*	0.136(1)	0.6284(5)	0.317(1)	0.020(6)	0.045(7)	0.028(6)	0.007(5)	–0.005(5)	0.006(5)
C(6)	4*a*	0.111(2)	0.5702(5)	0.303(2)	0.021(7)	0.043(7)	0.049(8)	–0.011(5)	0.010(6)	–0.004(5)
C(7)	4*a*	0.228(2)	0.5337(5)	0.375(1)	0.036(7)	0.030(6)	0.034(7)	–0.003(5)	0.009(5)	–0.002(5)
C(8)	4*a*	0.368(2)	0.5556(4)	0.469(1)	0.032(7)	0.022(6)	0.038(7)	0.002(5)	0.007(5)	0.009(5)
C(9)	4*a*	0.823(2)	0.6421(7)	0.628(2)	0.032(9)	0.09(1)	0.09(1)	0.004(8)	–0.011(8)	–0.029(9)
C(10)	4*a*	0.533(2)	0.3621(5)	0.590(2)	0.057(9)	0.041(7)	0.047(8)	0.005(7)	0.008(6)	0.006(6)

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