Crystal structure of 2-aminobenzothiazol-3-ium-2,3,4,5-tetrabromo-6-(methoxycarbonyl)benzoate methanol monosolvate, $(C_7H_7N_2S)^+(C_9H_3Br_4O_4)^-\cdot CH_4O$, $C_{17}H_{14}Br_4N_2O_5S$

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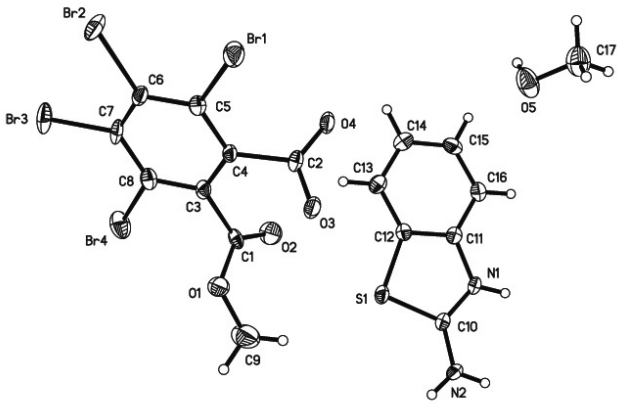

Abstract

$C_{17}H_{14}Br_4N_2O_5S$, monoclinic, $P2_1/c$ (no. 14), a = 17.352(2) Å, b = 13.689(1) Å, c = 9.5605(8) Å, β = 101.638(1)°, V = 2224.3 Å^3, Z = 4, $R_{gt}(F)$ = 0.0439, $wR_{ref}(F^2)$ = 0.0940, T = 298 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.23×0.35×0.40 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	73.62 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	50.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11065, 3920
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2227
$N(param)_{refined}$:	264
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 benzothiazol-2-amine (1.50 g, 0.01 mol) was added to the above solution and mixed for 0.5 h at room temperature. After filtration and drying, the title compound was obtained of which 20 mg was dissolved in 15 ml methanol, and the solution was kept at room temperature for 5 d. Isothermal evaporation at RT 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 benzothiazol-2-amine in methanol is expected to form 2-(benzothiazol-2-yl)-4,5,6,7-tetrabromoisoindoline-1,3-dione, but instead the title compound formed. In this paper, the crystal structure of the title compound is reported. The asymmetric unit of the title compound contains one 2-aminobenzothiazol-3-ium cation, one 2,3,4,5-tetrabromo-6-(methoxycarbonyl)benzoate anion and one methanol solvent molecule. In the anion of the title compound, the dihedral angles formed by the benzene ring and the mean planes of the carboxylate and methoxycarbonyl groups are 67.1(2)° and 65.1(2)°, respectively. All non-hydrogen atoms of the cation are essentially planar. The bond lengths and angles are in agreement with those which are related in ethylammonium 2-(methoxycarbonyl)-3,4,5,6-tetrabromobenzoate methanol solvate [2] and in 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(1)	4*e*	0.6166	0.3757	1.1594	0.035
H(2A)	4*e*	0.5490	0.3999	1.3548	0.042
H(2B)	4*e*	0.4615	0.4062	1.3212	0.042
H(5)	4*e*	0.6515	0.5289	0.5866	0.108
H(9A)	4*e*	0.2172	0.3557	0.9793	0.126
H(9B)	4*e*	0.1594	0.4280	1.0330	0.126
H(9C)	4*e*	0.2438	0.4631	1.0203	0.126
H(13)	4*e*	0.4004	0.3607	0.7066	0.044
H(14)	4*e*	0.4941	0.3454	0.5686	0.046
H(15)	4*e*	0.6252	0.3368	0.6729	0.047
H(16)	4*e*	0.6670	0.3481	0.9164	0.044
H(17A)	4*e*	0.6817	0.7028	0.6863	0.102
H(17B)	4*e*	0.7024	0.6597	0.5464	0.102
H(17C)	4*e*	0.7499	0.6265	0.6960	0.102

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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*	0.20248(4)	0.70016(6)	0.33411(8)	0.0382(4)	0.0542(5)	0.0544(5)	–0.0041(4)	–0.0032(4)	0.0188(4)
Br(2)	4*e*	0.04963(4)	0.58514(6)	0.14438(8)	0.0430(5)	0.0844(6)	0.0384(5)	0.0056(4)	–0.0163(4)	0.0030(4)
Br(3)	4*e*	–0.01772(4)	0.38579(6)	0.27323(9)	0.0269(4)	0.0612(5)	0.0757(6)	–0.0056(4)	–0.0108(4)	–0.0266(4)
Br(4)	4*e*	0.06887(4)	0.30573(6)	0.59246(9)	0.0451(5)	0.0497(5)	0.0845(6)	–0.0125(4)	0.0131(4)	0.0083(4)
N(1)	4*e*	0.5702(3)	0.3753(3)	1.1065(5)	0.023(3)	0.032(3)	0.029(3)	–0.003(3)	–0.003(3)	–0.003(3)
N(2)	4*e*	0.5053(3)	0.3990(3)	1.2934(5)	0.030(3)	0.048(4)	0.023(3)	–0.001(3)	0.000(3)	–0.005(3)
O(1)	4*e*	0.1697(3)	0.4531(4)	0.8323(5)	0.035(3)	0.081(4)	0.037(3)	0.018(3)	0.006(3)	0.018(3)
O(2)	4*e*	0.2708(3)	0.3866(4)	0.7602(5)	0.042(3)	0.070(4)	0.054(3)	0.020(3)	0.006(3)	0.017(3)
O(3)	4*e*	0.2845(2)	0.6236(3)	0.7317(5)	0.024(3)	0.067(3)	0.035(3)	0.000(2)	0.001(2)	–0.018(3)
O(4)	4*e*	0.3397(2)	0.5722(3)	0.5529(4)	0.020(2)	0.056(3)	0.037(3)	–0.003(2)	0.000(2)	–0.012(2)
O(5)	4*e*	0.6459(3)	0.5703(4)	0.6456(6)	0.056(4)	0.069(4)	0.100(5)	–0.016(3)	0.039(3)	–0.035(3)
S(1)	4*e*	0.42073(9)	0.3833(1)	1.0258(2)	0.0208(9)	0.042(1)	0.033(1)	0.0010(8)	–0.0010(7)	–0.0022(8)
C(1)	4*e*	0.2110(4)	0.4296(5)	0.7379(7)	0.015(4)	0.034(4)	0.044(5)	0.006(3)	0.009(4)	0.003(4)
C(2)	4*e*	0.2827(4)	0.5855(5)	0.6124(7)	0.021(4)	0.034(4)	0.034(4)	0.002(3)	–0.004(3)	–0.001(3)
C(3)	4*e*	0.1717(3)	0.4664(5)	0.5910(6)	0.018(4)	0.035(4)	0.033(4)	0.008(3)	0.003(3)	–0.002(3)
C(4)	4*e*	0.2031(3)	0.5455(5)	0.5348(6)	0.017(3)	0.039(4)	0.027(4)	0.002(3)	0.003(3)	–0.005(3)
C(5)	4*e*	0.1650(3)	0.5827(5)	0.4017(7)	0.024(4)	0.032(4)	0.034(4)	0.001(3)	0.004(3)	0.000(3)
C(6)	4*e*	0.0985(3)	0.5360(5)	0.3251(6)	0.022(4)	0.040(4)	0.028(4)	0.006(3)	–0.002(3)	–0.004(3)
C(7)	4*e*	0.0696(3)	0.4544(5)	0.3775(7)	0.017(3)	0.039(4)	0.045(4)	0.006(3)	–0.008(3)	–0.016(4)
C(8)	4*e*	0.1060(4)	0.4204(5)	0.5137(7)	0.026(4)	0.033(4)	0.046(5)	0.002(3)	0.006(3)	–0.003(4)
C(9)	4*e*	0.2001(5)	0.4224(7)	0.9786(8)	0.062(6)	0.132(9)	0.062(6)	0.028(6)	0.021(5)	0.027(6)
C(10)	4*e*	0.5055(4)	0.3869(4)	1.1588(7)	0.027(4)	0.028(4)	0.029(4)	–0.003(3)	0.000(3)	–0.002(3)
C(11)	4*e*	0.5571(4)	0.3623(4)	0.9591(7)	0.030(4)	0.027(4)	0.032(4)	–0.001(3)	0.003(3)	0.001(3)
C(12)	4*e*	0.4776(4)	0.3662(4)	0.8962(7)	0.026(4)	0.026(4)	0.033(4)	0.001(3)	0.004(3)	0.002(3)
C(13)	4*e*	0.4536(4)	0.3589(5)	0.7490(7)	0.036(4)	0.037(4)	0.034(4)	0.003(3)	0.000(4)	0.002(3)
C(14)	4*e*	0.5094(4)	0.3491(5)	0.6675(7)	0.045(5)	0.037(4)	0.032(4)	0.005(4)	0.003(4)	–0.002(3)
C(15)	4*e*	0.5884(4)	0.3446(5)	0.7305(7)	0.042(5)	0.041(4)	0.040(5)	–0.002(4)	0.020(4)	–0.001(4)
C(16)	4*e*	0.6137(4)	0.3511(5)	0.8752(7)	0.029(4)	0.038(4)	0.042(5)	–0.003(3)	0.006(4)	–0.002(4)
C(17)	4*e*	0.6991(5)	0.6456(6)	0.6434(8)	0.056(5)	0.062(6)	0.086(7)	0.000(5)	0.013(5)	–0.012(5)

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