# Crystal structure of ethanediammonium 3,4,5,6-tetrabromo-2-(ethoxy carbonyl)benzoate hydrate, $(C_2H_{10}N_2)(C_{10}H_5Br_4O_4)_2{\cdot}2H_2O$, $C_{22}H_{24}Br_8N_2O_{10}$

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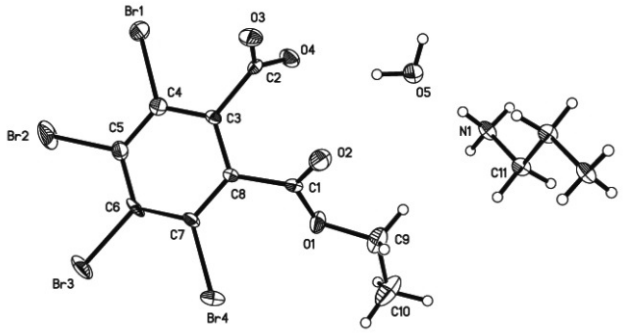


**Abstract**

$C_{11}H_{12}Br_4NO_5$, monoclinic, $P2_1/c$ (no. 14), $a$ = 17.724(2) Å, $b$ = 11.718(1) Å, $c$ = 8.1510(7) Å, $\beta$ = 100.655(1)°, $V$ = 1663.7 Å$^3$, $Z$ = 4, $R_{gt}(F)$ = 0.0471, $wR_{ref}(F^2)$ = 0.0961, $T$ = 298 K.

**Table 1.** Data collection and handling.

| | |
|---|---|
| Crystal: | colourless blocks, size 0.21×0.32×0.43 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 96.93 cm$^{-1}$ |
| Diffractometer, scan mode: | CCD area detector, $\varphi$ and $\omega$ |
| $2\theta_{max}$: | 50.04° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 8082, 2922 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 1779 |
| $N(param)_{refined}$: | 192 |
| 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, 10 mmol) and methanol (15 ml) was refluxed for 0.5 h. Then an ethanediamine water solution (65%) (0.89 g, 5 mmol) was added to the above solution and mixed for 0.5 h at room temperature. The above solution was kept at room temperature for 7 d. Isothermal evaporation gave colourless single crystals of the title compound, suitable for *X*-ray analysis.

## Discussion

Tetrabromophthalimide and its derivatives have been found to be useful flame retardants in polyesters, e.g. polybutylene terephthalate, and other resin formulations [1]. Ethanediammonium 3,4,5,6-tetrabromo-2-(methoxycarbonyl) benzoate is an intermediate of flame retardant *N*,*N*-ethylene-*bis*-(tetrabromophthalimide). In this paper, the crystal structure of ethanediammonium 3,4,5,6-tetrabromo-2-(ethoxycarbonyl) benzoate hydrate is reported.

The asymmetric unit of the title compound contains one hemiethanediammonium cation, one 3,4,5,6-tetrabromo-2-(ethoxycarbonyl)benzoate anion and water molecule. In anion, the dihedral angles formed by the benzene ring and the mean planes of the carboxylate and methoxycarbonyl groups are 81.6(2) and 51.7(2)°, respectively. The bond lengths and angles are in agreement with those found in ethaneammonium 2-(methoxycarbonyl)-3,4,5,6-tetrabromobenzoate methanol monosolvate [2] and 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* | 0.0248 | 0.8711 | 0.3291 | 0.049 |
| H(1B) | 4*e* | 0.0652 | 0.9583 | 0.2525 | 0.049 |
| H(1C) | 4*e* | –0.0181 | 0.9608 | 0.2296 | 0.049 |
| H(5C) | 4*e* | 0.0543 | 0.6697 | 0.4119 | 0.068 |
| H(5D) | 4*e* | –0.0218 | 0.6912 | 0.4139 | 0.068 |
| H(9A) | 4*e* | 0.2124 | 0.8502 | 0.7786 | 0.067 |
| H(9B) | 4*e* | 0.1695 | 0.8642 | 0.5927 | 0.067 |
| H(10A) | 4*e* | 0.3180 | 0.9414 | 0.7199 | 0.112 |
| H(10B) | 4*e* | 0.2466 | 1.0193 | 0.6571 | 0.112 |
| H(10C) | 4*e* | 0.2817 | 0.9457 | 0.5294 | 0.112 |
| H(11A) | 4*e* | 0.0803 | 1.0068 | 0.5220 | 0.034 |
| H(11B) | 4*e* | 0.0240 | 1.0958 | 0.4200 | 0.034 |

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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* | 0.21005(5) | 0.24913(6) | 0.37686(9) | 0.0417(5) | 0.0347(4) | 0.0437(4) | 0.0017(4) | 0.0017(4) | –0.0155(4) |
| Br(2) | 4*e* | 0.39857(6) | 0.22815(8) | 0.4900(1) | 0.0498(7) | 0.0781(7) | 0.0791(6) | 0.0400(5) | –0.0059(5) | –0.0350(5) |
| Br(3) | 4*e* | 0.49529(5) | 0.43259(7) | 0.7112(1) | 0.0156(5) | 0.0684(6) | 0.0690(5) | 0.0077(4) | 0.0009(4) | 0.0144(4) |
| Br(4) | 4*e* | 0.40531(5) | 0.65723(7) | 0.8105(1) | 0.0345(6) | 0.0482(5) | 0.0964(7) | –0.0106(4) | –0.0205(5) | –0.0154(5) |
| N(1) | 4*e* | 0.0250(3) | 0.9444(4) | 0.3007(6) | 0.022(4) | 0.045(4) | 0.032(3) | 0.003(3) | 0.007(3) | 0.005(3) |
| O(1) | 4*e* | 0.2537(3) | 0.7468(4) | 0.6191(5) | 0.039(3) | 0.026(3) | 0.047(3) | 0.008(2) | 0.019(3) | –0.001(2) |
| O(2) | 4*e* | 0.1715(3) | 0.6460(4) | 0.7396(6) | 0.037(4) | 0.040(3) | 0.068(3) | –0.006(3) | 0.027(3) | –0.014(3) |
| O(3) | 4*e* | 0.1080(3) | 0.4099(4) | 0.6083(5) | 0.031(3) | 0.040(3) | 0.042(3) | –0.011(2) | 0.011(3) | 0.004(2) |
| O(4) | 4*e* | 0.1120(3) | 0.5165(4) | 0.3841(5) | 0.028(3) | 0.042(3) | 0.040(3) | 0.000(2) | –0.006(2) | 0.008(2) |
| O(5) | 4*e* | 0.0221(3) | 0.7220(4) | 0.4211(6) | 0.034(4) | 0.034(3) | 0.101(4) | –0.002(2) | 0.012(3) | 0.002(3) |
| C(1) | 4*e* | 0.2267(4) | 0.6516(5) | 0.6722(7) | 0.017(4) | 0.028(4) | 0.020(4) | –0.005(3) | 0.001(3) | –0.003(3) |
| C(2) | 4*e* | 0.1419(4) | 0.4616(5) | 0.5116(8) | 0.026(5) | 0.016(4) | 0.032(4) | 0.000(3) | 0.002(4) | –0.009(3) |
| C(3) | 4*e* | 0.2292(4) | 0.4580(5) | 0.5518(7) | 0.015(4) | 0.024(4) | 0.020(3) | –0.002(3) | 0.002(3) | 0.004(3) |
| C(4) | 4*e* | 0.2679(4) | 0.3640(5) | 0.5060(7) | 0.031(5) | 0.029(4) | 0.022(4) | 0.000(3) | 0.004(3) | –0.001(3) |
| C(5) | 4*e* | 0.3470(4) | 0.3571(5) | 0.5492(8) | 0.031(5) | 0.032(4) | 0.030(4) | 0.007(3) | 0.001(4) | –0.002(3) |
| C(6) | 4*e* | 0.3887(4) | 0.4453(6) | 0.6398(8) | 0.005(4) | 0.047(4) | 0.036(4) | 0.002(3) | 0.005(3) | 0.013(4) |
| C(7) | 4*e* | 0.3503(4) | 0.5419(5) | 0.6805(7) | 0.017(4) | 0.029(4) | 0.034(4) | –0.001(3) | –0.007(3) | 0.004(3) |
| C(8) | 4*e* | 0.2714(4) | 0.5492(5) | 0.6348(7) | 0.017(4) | 0.022(4) | 0.024(3) | –0.001(3) | –0.001(3) | –0.002(3) |
| C(9) | 4*e* | 0.2194(5) | 0.8532(6) | 0.663(1) | 0.064(7) | 0.034(5) | 0.070(6) | 0.008(4) | 0.014(5) | –0.007(4) |
| C(10) | 4*e* | 0.2709(6) | 0.9481(6) | 0.640(1) | 0.12(1) | 0.031(5) | 0.087(6) | –0.005(5) | 0.041(6) | –0.010(5) |
| C(11) | 4*e* | 0.0301(4) | 1.0161(5) | 0.4521(6) | 0.028(5) | 0.039(4) | 0.018(3) | –0.001(3) | 0.007(3) | –0.001(3) |

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