

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Ethanaminium 3,4,5,6-tetrabromo-2-(methoxycarbonyl)benzoate methanol monosolvate

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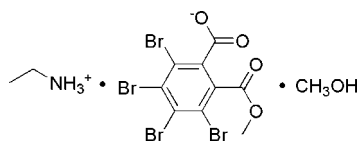
Received 10 December 2010; accepted 15 December 2010

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.011$ Å; R factor = 0.042; wR factor = 0.068; data-to-parameter ratio = 16.5.

In the crystal structure of the title compound, $\text{C}_2\text{H}_8\text{N}^+ \cdot \text{C}_9\text{H}_3\text{Br}_4\text{O}_4^- \cdot \text{CH}_4\text{O}$, intermolecular $\text{N}-\text{H} \cdots \text{O}$ and $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds link the components into chains along [001]. Additional stabilization is supplied by weak $\text{C}-\text{H} \cdots \text{O}$ and $\text{C}-\text{H} \cdots \text{Br}$ interactions.

Related literature

For a related structure, see: Liang (2008).



Experimental

Crystal data

$\text{C}_2\text{H}_8\text{N}^+ \cdot \text{C}_9\text{H}_3\text{Br}_4\text{O}_4^- \cdot \text{CH}_4\text{O}$

$M_r = 572.89$

Monoclinic, $P2_1/c$

$a = 9.4651$ (8) Å

$b = 25.6544$ (19) Å

$c = 8.3365$ (6) Å

$\beta = 112.787$ (1)°

$V = 1866.3$ (2) Å³

$Z = 4$

Mo $K\alpha$ radiation

$\mu = 8.64$ mm⁻¹

$T = 298$ K

$0.40 \times 0.35 \times 0.33$ mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (SADABS; Bruker, 1997)
 $T_{\min} = 0.130$, $T_{\max} = 0.163$

9436 measured reflections
3295 independent reflections
1454 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.086$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.068$

$S = 0.99$

3295 reflections

200 parameters

H-atom parameters constrained

$\Delta\rho_{\text{max}} = 0.49$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.51$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{N1}-\text{H1A} \cdots \text{O4}^{\text{i}}$	0.89	1.94	2.772 (9)	155
$\text{N1}-\text{H1B} \cdots \text{O5}$	0.89	1.92	2.810 (9)	174
$\text{N1}-\text{H1C} \cdots \text{O3}^{\text{ii}}$	0.89	1.99	2.871 (7)	170
$\text{N1}-\text{H1C} \cdots \text{O4}^{\text{ii}}$	0.89	2.58	3.249 (8)	132
$\text{O5}-\text{H5} \cdots \text{O3}$	0.82	1.90	2.705 (8)	165
$\text{C10}-\text{H10A} \cdots \text{Br3}^{\text{iii}}$	0.97	2.92	3.736 (8)	143
$\text{C11}-\text{H11B} \cdots \text{O2}^{\text{iv}}$	0.96	2.47	3.357 (10)	153

Symmetry codes: (i) $x, y, z + 1$; (ii) $x, -y + \frac{3}{2}, z + \frac{1}{2}$; (iii) $-x + 1, -y + 2, -z + 1$; (iv) $x - 1, y, z$.

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008) and PLATON (Spek, 2009); software used to prepare material for publication: SHELXTL.

The author thanks Shandong Provincial Natural Science Foundation, China (ZR2009BL027) for support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5189).

References

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Liang, Z.-P. (2008). *Acta Cryst.* **E64**, o2416.
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Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

supplementary materials

Acta Cryst. (2011). E67, o200 [doi:10.1107/S1600536810052591]

Ethanaminium 3,4,5,6-tetrabromo-2-(methoxycarbonyl)benzoate methanol monosolvate

J. Li

Comment

4,5,6,7-Tetrabromo-2-ethylisoindoline-1,3-dione is an important flame retardant. 2-(Methoxycarbonyl)-3,4,5,6-tetrabromobenzoic acid is an intermediate in the synthesis of this flame retardant. In this paper, the structure of the title compound is reported. The asymmetric unit of the title compound (I) contains one ethanaminium cation, one 2-(methoxycarbonyl)-3,4,5,6-tetrabromobenzoate anion and one methanol solvent molecule (Fig. 1). The bond lengths and angles agree with those in ethane-1,2-diaminium 2-(methoxycarbonyl)-3,4,5,6-tetrabromo benzoate methanol solvate (Liang, 2008). In the crystal structure, intermolecular N—H···O and O—H···O hydrogen bonds link the components of the structure into one-dimensional chains along [001](see fig. 2). Additional stabilization is supplied by weak C—H···O and C—H···Br interactions.

Experimental

A mixture of 4,5,6,7-tetrabromoisobenzofuran-1,3-dione (4.64 g, 0.01 mol) and methanol (15 ml) was refluxed for 0.5 h. And then ethanamine (0.45 g, 0.01 mol) was added to the above solution, and mixed for 10 min at room temperature. The solution was kept at room temperature for 3 d. Natural evaporation gave colourless single crystals of the title compound, suitable for X-ray analysis.

Refinement

H atoms were initially located from difference maps and then refined in a riding model with C—H = 0.96–0.97 Å, N—H = 0.89 Å, O—H = 0.82 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{O, N, methyl C})$.

Figures

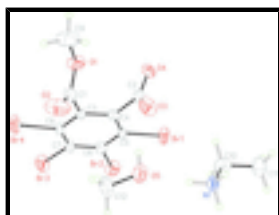


Fig. 1. The asymmetric unit of (I), drawn with 30% probability ellipsoids.

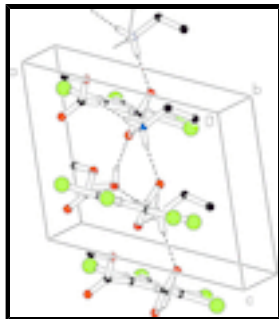
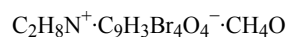


Fig. 2. The crystal packing of (I). Hydrogen bonds are indicated by dashed lines.

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Crystal data



$M_r = 572.89$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 9.4651 (8) \text{ \AA}$

$b = 25.6544 (19) \text{ \AA}$

$c = 8.3365 (6) \text{ \AA}$

$\beta = 112.787 (1)^\circ$

$V = 1866.3 (2) \text{ \AA}^3$

$Z = 4$

$F(000) = 1096$

$D_x = 2.039 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 1513 reflections

$\theta = 2.3\text{--}20.6^\circ$

$\mu = 8.64 \text{ mm}^{-1}$

$T = 298 \text{ K}$

Block, colorless

$0.40 \times 0.35 \times 0.33 \text{ mm}$

Data collection

Bruker SMART CCD
diffractometer

Radiation source: fine-focus sealed tube
graphite

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 1997)

$T_{\min} = 0.130$, $T_{\max} = 0.163$

9436 measured reflections

3295 independent reflections

1454 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.086$

$\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 2.3^\circ$

$h = -9 \rightarrow 11$

$k = -30 \rightarrow 30$

$l = -9 \rightarrow 8$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.068$

$S = 0.99$

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0024P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

3295 reflections	$(\Delta/\sigma)_{\max} = 0.001$
200 parameters	$\Delta\rho_{\max} = 0.49 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\min} = -0.51 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.32729 (8)	0.90633 (3)	0.26426 (13)	0.0702 (3)
Br2	0.40752 (9)	1.03175 (3)	0.25156 (12)	0.0705 (3)
Br3	0.73585 (10)	1.06551 (3)	0.23297 (14)	0.0807 (3)
Br4	0.98266 (9)	0.97499 (3)	0.23443 (13)	0.0773 (3)
N1	0.5436 (6)	0.7996 (2)	0.8034 (8)	0.0546 (19)
H1A	0.5376	0.8133	0.8986	0.082*
H1B	0.6186	0.8151	0.7820	0.082*
H1C	0.5631	0.7656	0.8196	0.082*
O1	0.8313 (6)	0.8333 (2)	0.1273 (8)	0.0672 (18)
O2	0.9937 (7)	0.8565 (2)	0.3871 (9)	0.101 (3)
O3	0.6431 (6)	0.8063 (2)	0.3863 (8)	0.076 (2)
O4	0.4945 (6)	0.8125 (2)	0.1079 (8)	0.0713 (19)
O5	0.7663 (7)	0.8481 (2)	0.7090 (7)	0.104 (2)
H5	0.7413	0.8383	0.6081	0.157*
C1	0.8783 (9)	0.8620 (3)	0.2667 (14)	0.050 (3)
C2	0.5820 (10)	0.8306 (3)	0.2499 (14)	0.050 (3)
C3	0.7630 (7)	0.9042 (3)	0.2521 (9)	0.0369 (19)
C4	0.6200 (8)	0.8886 (3)	0.2517 (9)	0.040 (2)
C5	0.5158 (7)	0.9276 (3)	0.2534 (9)	0.040 (2)
C6	0.5515 (7)	0.9794 (3)	0.2527 (9)	0.047 (2)
C7	0.6915 (8)	0.9947 (3)	0.2450 (10)	0.045 (2)
C8	0.7960 (7)	0.9565 (3)	0.2469 (9)	0.040 (2)
C9	0.9329 (9)	0.7886 (3)	0.1323 (11)	0.097 (3)
H9A	1.0367	0.8006	0.1685	0.146*
H9B	0.9010	0.7734	0.0185	0.146*
H9C	0.9266	0.7629	0.2131	0.146*
C10	0.3935 (9)	0.8076 (3)	0.6508 (10)	0.056 (2)
H10A	0.3730	0.8447	0.6333	0.068*
H10B	0.4018	0.7937	0.5466	0.068*

supplementary materials

C11	0.2643 (8)	0.7820 (3)	0.6773 (10)	0.081 (3)
H11A	0.2763	0.7449	0.6766	0.121*
H11B	0.1697	0.7919	0.5854	0.121*
H11C	0.2630	0.7926	0.7872	0.121*
C12	0.8988 (9)	0.8774 (3)	0.7572 (12)	0.104 (4)
H12A	0.9141	0.8959	0.8627	0.155*
H12B	0.8899	0.9019	0.6667	0.155*
H12C	0.9844	0.8548	0.7757	0.155*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0399 (5)	0.0808 (7)	0.1016 (9)	-0.0038 (5)	0.0403 (6)	0.0061 (6)
Br2	0.0615 (6)	0.0617 (6)	0.0956 (8)	0.0286 (5)	0.0386 (6)	0.0063 (6)
Br3	0.0898 (7)	0.0337 (5)	0.1331 (10)	-0.0058 (5)	0.0592 (7)	-0.0010 (6)
Br4	0.0455 (6)	0.0699 (7)	0.1306 (10)	-0.0122 (5)	0.0496 (6)	-0.0070 (6)
N1	0.061 (5)	0.042 (4)	0.072 (6)	-0.011 (3)	0.039 (4)	0.000 (4)
O1	0.057 (4)	0.063 (4)	0.077 (5)	0.027 (3)	0.022 (4)	-0.014 (4)
O2	0.068 (4)	0.114 (6)	0.085 (6)	0.048 (4)	-0.011 (4)	-0.033 (4)
O3	0.101 (5)	0.044 (4)	0.070 (5)	-0.006 (3)	0.020 (4)	0.019 (4)
O4	0.094 (5)	0.062 (4)	0.062 (5)	-0.038 (3)	0.035 (4)	-0.021 (4)
O5	0.101 (5)	0.150 (6)	0.074 (6)	-0.064 (4)	0.048 (4)	-0.018 (4)
C1	0.036 (6)	0.042 (6)	0.072 (9)	0.003 (5)	0.020 (6)	-0.009 (6)
C2	0.047 (6)	0.036 (6)	0.075 (9)	-0.006 (5)	0.035 (6)	-0.005 (6)
C3	0.031 (4)	0.028 (4)	0.057 (6)	-0.003 (4)	0.023 (4)	-0.005 (5)
C4	0.043 (5)	0.032 (5)	0.043 (6)	0.007 (4)	0.017 (5)	-0.002 (4)
C5	0.026 (4)	0.038 (5)	0.060 (6)	-0.007 (4)	0.021 (4)	-0.004 (5)
C6	0.035 (5)	0.045 (6)	0.058 (6)	0.014 (4)	0.016 (5)	-0.001 (4)
C7	0.032 (5)	0.033 (5)	0.068 (6)	-0.003 (4)	0.019 (5)	0.003 (4)
C8	0.016 (4)	0.041 (5)	0.062 (6)	-0.001 (4)	0.014 (4)	-0.002 (5)
C9	0.090 (7)	0.080 (7)	0.106 (9)	0.045 (6)	0.022 (6)	-0.025 (6)
C10	0.077 (6)	0.046 (6)	0.040 (6)	0.018 (5)	0.015 (6)	0.009 (5)
C11	0.052 (6)	0.097 (7)	0.085 (8)	-0.013 (5)	0.017 (6)	0.016 (6)
C12	0.082 (7)	0.100 (8)	0.144 (10)	-0.050 (6)	0.060 (7)	-0.030 (7)

Geometric parameters (\AA , $^\circ$)

Br1—C5	1.902 (6)	C3—C4	1.411 (8)
Br2—C6	1.911 (6)	C4—C5	1.410 (8)
Br3—C7	1.876 (6)	C5—C6	1.370 (8)
Br4—C8	1.871 (6)	C6—C7	1.408 (7)
N1—C10	1.509 (8)	C7—C8	1.389 (8)
N1—H1A	0.8900	C9—H9A	0.9600
N1—H1B	0.8900	C9—H9B	0.9600
N1—H1C	0.8900	C9—H9C	0.9600
O1—C1	1.301 (9)	C10—C11	1.478 (8)
O1—C9	1.487 (7)	C10—H10A	0.9700
O2—C1	1.171 (9)	C10—H10B	0.9700
O3—C2	1.227 (9)	C11—H11A	0.9600

O4—C2	1.241 (9)	C11—H11B	0.9600
O5—C12	1.381 (7)	C11—H11C	0.9600
O5—H5	0.8200	C12—H12A	0.9600
C1—C3	1.507 (9)	C12—H12B	0.9600
C2—C4	1.530 (9)	C12—H12C	0.9600
C3—C8	1.381 (7)		
C10—N1—H1A	109.5	C6—C7—Br3	120.5 (5)
C10—N1—H1B	109.5	C3—C8—C7	121.2 (6)
H1A—N1—H1B	109.5	C3—C8—Br4	118.6 (5)
C10—N1—H1C	109.5	C7—C8—Br4	120.2 (5)
H1A—N1—H1C	109.5	O1—C9—H9A	109.5
H1B—N1—H1C	109.5	O1—C9—H9B	109.5
C1—O1—C9	114.7 (6)	H9A—C9—H9B	109.5
C12—O5—H5	109.5	O1—C9—H9C	109.5
O2—C1—O1	125.5 (8)	H9A—C9—H9C	109.5
O2—C1—C3	124.2 (9)	H9B—C9—H9C	109.5
O1—C1—C3	110.4 (7)	C11—C10—N1	112.1 (6)
O3—C2—O4	126.6 (8)	C11—C10—H10A	109.2
O3—C2—C4	117.4 (8)	N1—C10—H10A	109.2
O4—C2—C4	116.0 (8)	C11—C10—H10B	109.2
C8—C3—C4	120.3 (6)	N1—C10—H10B	109.2
C8—C3—C1	122.3 (6)	H10A—C10—H10B	107.9
C4—C3—C1	117.4 (6)	C10—C11—H11A	109.5
C5—C4—C3	118.2 (6)	C10—C11—H11B	109.5
C5—C4—C2	121.9 (7)	H11A—C11—H11B	109.5
C3—C4—C2	119.9 (6)	C10—C11—H11C	109.5
C6—C5—C4	121.0 (6)	H11A—C11—H11C	109.5
C6—C5—Br1	121.0 (5)	H11B—C11—H11C	109.5
C4—C5—Br1	118.0 (5)	O5—C12—H12A	109.5
C5—C6—C7	120.6 (6)	O5—C12—H12B	109.5
C5—C6—Br2	120.3 (5)	H12A—C12—H12B	109.5
C7—C6—Br2	119.0 (6)	O5—C12—H12C	109.5
C8—C7—C6	118.8 (6)	H12A—C12—H12C	109.5
C8—C7—Br3	120.8 (5)	H12B—C12—H12C	109.5
C9—O1—C1—O2	3.5 (13)	C2—C4—C5—Br1	2.8 (10)
C9—O1—C1—C3	-176.8 (6)	C4—C5—C6—C7	1.9 (11)
O2—C1—C3—C8	64.3 (12)	Br1—C5—C6—C7	179.9 (5)
O1—C1—C3—C8	-115.3 (8)	C4—C5—C6—Br2	179.1 (5)
O2—C1—C3—C4	-112.9 (10)	Br1—C5—C6—Br2	-2.9 (9)
O1—C1—C3—C4	67.5 (9)	C5—C6—C7—C8	-3.2 (11)
C8—C3—C4—C5	-2.7 (10)	Br2—C6—C7—C8	179.6 (6)
C1—C3—C4—C5	174.6 (7)	C5—C6—C7—Br3	177.0 (6)
C8—C3—C4—C2	177.5 (7)	Br2—C6—C7—Br3	-0.2 (8)
C1—C3—C4—C2	-5.3 (11)	C4—C3—C8—C7	1.4 (11)
O3—C2—C4—C5	-104.5 (9)	C1—C3—C8—C7	-175.7 (8)
O4—C2—C4—C5	77.2 (10)	C4—C3—C8—Br4	-176.4 (5)
O3—C2—C4—C3	75.3 (10)	C1—C3—C8—Br4	6.4 (11)
O4—C2—C4—C3	-103.0 (8)	C6—C7—C8—C3	1.5 (11)

supplementary materials

C3—C4—C5—C6	1.0 (10)	Br3—C7—C8—C3	-178.7 (5)
C2—C4—C5—C6	-179.1 (7)	C6—C7—C8—Br4	179.3 (5)
C3—C4—C5—Br1	-177.0 (5)	Br3—C7—C8—Br4	-0.9 (9)

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1A \cdots O4 ⁱ	0.89	1.94	2.772 (9)	155
N1—H1B \cdots O5	0.89	1.92	2.810 (9)	174
N1—H1C \cdots O3 ⁱⁱ	0.89	1.99	2.871 (7)	170
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C11—H11B \cdots O2 ^{iv}	0.96	2.47	3.357 (10)	153

Symmetry codes: (i) $x, y, z+1$; (ii) $x, -y+3/2, z+1/2$; (iii) $-x+1, -y+2, -z+1$; (iv) $x-1, y, z$.

Fig. 2

