Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

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

Jian Li

Department of Chemistry and Chemical Engineering, Weifang University, Weifang 261061, People's Republic of China
Correspondence e-mail: ljwfu@163.com

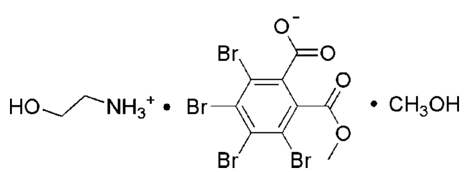
Received 19 January 2011; accepted 6 March 2011

Key indicators: single-crystal X-ray study; *T* = 298 K; mean σ(C–C) = 0.011 Å; *R* factor = 0.044; *wR* factor = 0.071; data-to-parameter ratio = 15.4.

In the title compound, $C_2H_8NO^+ \cdot C_9H_3Br_4O_4^- \cdot CH_4O$, intermolecular N—H···O and O—H···O hydrogen bonds link the components into chains along [001].

Related literature

For related structures, see: Li (2011); Liang (2008).



Experimental

Crystal data

$C_2H_8NO^+ \cdot C_9H_3Br_4O_4^- \cdot CH_4O$
M_r = 588.89
Monoclinic, $P2_1/c$
a = 9.4231 (11) Å
b = 25.475 (2) Å
c = 8.3463 (7) Å
β = 111.990 (1)°
V = 1857.8 (3) Å^3
Z = 4
Mo *Kα* radiation
μ = 8.69 mm^{-1}
T = 298 K
0.42 × 0.35 × 0.34 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.121, T_{max} = 0.156
9367 measured reflections
3269 independent reflections
1581 reflections with $I > 2\sigma(I)$
R_{int} = 0.086

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.071
S = 1.00
3269 reflections
212 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.58 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O3^{i}	0.89	2.01	2.885 (7)	168
N1—H1*B*···O6	0.89	1.86	2.740 (7)	168
N1—H1*C*···O4^{ii}	0.89	1.96	2.789 (8)	154
O5—H5···O4^{ii}	0.82	2.00	2.813 (8)	169
O6—H6···O3^{iii}	0.82	1.92	2.714 (8)	163

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

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: KJ2169).

References

Bruker (1997). *SADABS*, *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Li, J. (2011). *Acta Cryst.* E**67**, o200.
Liang, Z.-P. (2008). *Acta Cryst.* E**64**, o2416.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supplementary materials

Acta Cryst. (2011). E**67**, o866 [**doi:10.1107/S160053681100852X**]

2-Hydroxyethanaminium 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 the intermediate in the synthesis of 4,5,6,7-Tetrabromo-2-ethylisoindoline-1,3-dione. In this paper, the structure of the title compound is reported. The asymmetric unit of the title compound (I) contains one ethanolaminium 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 ethanaminium 2-(methoxycarbonyl)-3,4,5,6-tetrabromo benzoate methanol solvate (Li, 2011) and 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 and Table 1).

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. Ethanolamine (0.61 g, 0.01 mol) was added to this solution, followed by stirring for 10 min at room temperature. The solution was kept at room temperature for 5 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 = 0.82Å and $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(O, N, methyl C).

Figures

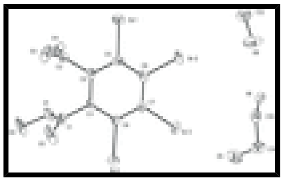


Fig. 1. The molecular structure of the title compound, drawn with 30% probability ellipsoids.

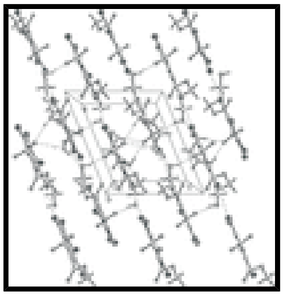


Fig. 2. The crystal packing of the title compound, viewed along the *b* axis. Hydrogen bonds are indicated by dashed lines.

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

Crystal data

$C_2H_8NO^+ \cdot C_9H_3Br_4O_4^- \cdot CH_4O$	$F(000) = 1128$
$M_r = 588.89$	$D_x = 2.105$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 9.4231$ (11) Å	Cell parameters from 1705 reflections
$b = 25.475$ (2) Å	$\theta = 2.9–21.8°$
$c = 8.3463$ (7) Å	$\mu = 8.69$ mm^{-1}
$\beta = 111.990$ (1)°	$T = 298$ K
$V = 1857.8$ (3) Å^3	Block, colorless
$Z = 4$	0.42 × 0.35 × 0.34 mm

Data collection

Bruker SMART CCD diffractometer	3269 independent reflections
Radiation source: fine-focus sealed tube	1581 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.086$
φ and ω scans	$\theta_{max} = 25.0°$, $\theta_{min} = 2.5°$
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)	$h = -10\rightarrow11$
$T_{min} = 0.121$, $T_{max} = 0.156$	$k = -30\rightarrow28$
9367 measured reflections	$l = -9\rightarrow9$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.044$	H-atom parameters constrained
$wR(F^2) = 0.071$	$w = 1/[\sigma^2(F_o^2) + (0.0103P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} = 0.001$
3269 reflections	$\Delta\rho_{max} = 0.55$ e Å^{-3}
212 parameters	$\Delta\rho_{min} = -0.58$ e Å^{-3}
0 restraints	Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.00077 (5)

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Br1	0.33226 (9)	0.40176 (3)	0.76997 (13)	0.0614 (3)
Br2	0.40915 (10)	0.52775 (3)	0.75108 (12)	0.0620 (3)
Br3	0.73518 (10)	0.56240 (3)	0.72387 (12)	0.0632 (3)
Br4	0.98636 (10)	0.47177 (3)	0.73227 (13)	0.0709 (3)
N1	0.4471 (6)	0.6959 (2)	0.6870 (8)	0.0456 (18)
H1A	0.4218	0.7297	0.6773	0.068*
H1B	0.3755	0.6775	0.7077	0.068*
H1C	0.4542	0.6848	0.5892	0.068*
O1	0.8414 (6)	0.3298 (2)	0.6314 (8)	0.0643 (18)
O2	1.0002 (7)	0.3545 (2)	0.8847 (9)	0.092 (2)
O3	0.6507 (6)	0.30316 (19)	0.8975 (8)	0.0702 (18)
O4	0.5049 (6)	0.30677 (18)	0.6230 (8)	0.0567 (17)
O5	0.7644 (6)	0.6848 (2)	0.6739 (8)	0.081 (2)
H5	0.6920	0.6850	0.5807	0.097*
O6	0.2414 (7)	0.6457 (2)	0.7949 (8)	0.095 (2)
H6	0.2570	0.6592	0.8891	0.114*
C1	0.8828 (10)	0.3586 (3)	0.7696 (14)	0.051 (2)
C2	0.5883 (10)	0.3264 (3)	0.7595 (14)	0.044 (2)
C3	0.7674 (8)	0.4002 (2)	0.7569 (9)	0.0337 (19)
C4	0.6248 (8)	0.3843 (3)	0.7589 (9)	0.0342 (19)
C5	0.5202 (7)	0.4236 (3)	0.7578 (9)	0.038 (2)
C6	0.5502 (8)	0.4761 (2)	0.7492 (9)	0.037 (2)
C7	0.6908 (9)	0.4915 (2)	0.7421 (10)	0.041 (2)
C8	0.7971 (8)	0.4530 (2)	0.7450 (10)	0.038 (2)
C9	0.9453 (9)	0.2862 (3)	0.6319 (12)	0.096 (3)
H9A	1.0227	0.2988	0.5928	0.145*
H9B	0.8878	0.2588	0.5563	0.145*
H9C	0.9927	0.2726	0.7471	0.145*
C10	0.5970 (9)	0.6887 (3)	0.8323 (10)	0.052 (2)
H10A	0.5915	0.7038	0.9365	0.062*
H10B	0.6174	0.6514	0.8525	0.062*
C11	0.7246 (9)	0.7134 (3)	0.7971 (11)	0.061 (3)
H11A	0.6954	0.7488	0.7547	0.074*
H11B	0.8133	0.7158	0.9040	0.074*
C12	0.1062 (10)	0.6179 (3)	0.7424 (12)	0.087 (3)
H12A	0.1093	0.5932	0.8306	0.130*
H12B	0.0927	0.5994	0.6375	0.130*
H12C	0.0224	0.6417	0.7225	0.130*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0395 (5)	0.0646 (5)	0.0851 (8)	−0.0002 (5)	0.0292 (5)	0.0080 (6)
Br2	0.0537 (6)	0.0497 (5)	0.0860 (8)	0.0231 (5)	0.0299 (6)	0.0052 (5)
Br3	0.0653 (6)	0.0307 (4)	0.0958 (9)	−0.0021 (5)	0.0328 (6)	0.0015 (5)
Br4	0.0410 (6)	0.0579 (5)	0.1211 (10)	−0.0072 (5)	0.0387 (6)	−0.0055 (6)
N1	0.050 (5)	0.036 (3)	0.052 (5)	−0.013 (3)	0.020 (4)	−0.010 (4)
O1	0.044 (4)	0.061 (4)	0.078 (5)	0.021 (3)	0.012 (4)	−0.019 (4)
O2	0.060 (5)	0.086 (4)	0.092 (6)	0.041 (4)	−0.016 (4)	−0.030 (5)
O3	0.097 (5)	0.041 (3)	0.050 (5)	−0.003 (3)	0.001 (4)	0.011 (3)
O4	0.062 (4)	0.048 (3)	0.053 (5)	−0.022 (3)	0.014 (4)	−0.006 (3)
O5	0.049 (4)	0.097 (5)	0.093 (6)	0.017 (4)	0.023 (4)	0.017 (4)
O6	0.082 (5)	0.139 (5)	0.076 (5)	−0.057 (5)	0.042 (4)	−0.023 (4)
C1	0.037 (6)	0.037 (5)	0.064 (8)	−0.002 (5)	0.002 (6)	−0.010 (5)
C2	0.035 (6)	0.030 (5)	0.066 (8)	−0.003 (4)	0.018 (6)	−0.004 (5)
C3	0.021 (4)	0.031 (4)	0.046 (6)	0.008 (4)	0.008 (4)	−0.001 (4)
C4	0.031 (5)	0.035 (4)	0.031 (6)	0.006 (4)	0.004 (4)	0.003 (4)
C5	0.027 (5)	0.045 (4)	0.042 (6)	−0.005 (4)	0.012 (4)	0.001 (4)
C6	0.038 (5)	0.025 (4)	0.048 (6)	0.004 (4)	0.014 (4)	−0.002 (4)
C7	0.044 (5)	0.025 (4)	0.053 (6)	0.006 (4)	0.019 (5)	0.010 (4)
C8	0.025 (5)	0.038 (4)	0.044 (6)	−0.001 (4)	0.005 (4)	−0.002 (4)
C9	0.078 (7)	0.082 (7)	0.114 (9)	0.044 (6)	0.019 (7)	−0.041 (6)
C10	0.067 (7)	0.033 (4)	0.052 (7)	0.010 (5)	0.019 (6)	0.005 (5)
C11	0.039 (6)	0.066 (6)	0.070 (8)	0.006 (5)	0.010 (5)	0.004 (5)
C12	0.067 (7)	0.092 (7)	0.113 (10)	−0.023 (6)	0.047 (7)	−0.027 (7)

Geometric parameters (Å, °)

Br1—C5	1.894 (6)	C2—C4	1.514 (9)
Br2—C6	1.875 (6)	C3—C8	1.384 (8)
Br3—C7	1.872 (6)	C3—C4	1.410 (8)
Br4—C8	1.888 (6)	C4—C5	1.402 (8)
N1—C10	1.489 (8)	C5—C6	1.376 (8)
N1—H1A	0.8900	C6—C7	1.404 (8)
N1—H1B	0.8900	C7—C8	1.397 (8)
N1—H1C	0.8900	C9—H9A	0.9600
O1—C1	1.298 (9)	C9—H9B	0.9600
O1—C9	1.480 (7)	C9—H9C	0.9600
O2—C1	1.166 (9)	C10—C11	1.481 (8)
O3—C2	1.232 (9)	C10—H10A	0.9700
O4—C2	1.223 (9)	C10—H10B	0.9700
O5—C11	1.420 (8)	C11—H11A	0.9700
O5—H5	0.8200	C11—H11B	0.9700
O6—C12	1.377 (8)	C12—H12A	0.9600
O6—H6	0.8200	C12—H12B	0.9600
C1—C3	1.493 (9)	C12—H12C	0.9600

C10—N1—H1A	109.5	C6—C7—Br3	121.1 (5)
C10—N1—H1B	109.5	C3—C8—C7	121.5 (6)
H1A—N1—H1B	109.5	C3—C8—Br4	118.1 (5)
C10—N1—H1C	109.5	C7—C8—Br4	120.5 (5)
H1A—N1—H1C	109.5	O1—C9—H9A	109.5
H1B—N1—H1C	109.5	O1—C9—H9B	109.5
C1—O1—C9	116.4 (6)	H9A—C9—H9B	109.5
C11—O5—H5	109.5	O1—C9—H9C	109.5
C12—O6—H6	109.5	H9A—C9—H9C	109.5
O2—C1—O1	123.9 (8)	H9B—C9—H9C	109.5
O2—C1—C3	124.3 (9)	C11—C10—N1	112.3 (6)
O1—C1—C3	111.7 (8)	C11—C10—H10A	109.2
O4—C2—O3	126.1 (7)	N1—C10—H10A	109.2
O4—C2—C4	117.6 (9)	C11—C10—H10B	109.2
O3—C2—C4	116.2 (8)	N1—C10—H10B	109.2
C8—C3—C4	119.9 (6)	H10A—C10—H10B	107.9
C8—C3—C1	122.2 (6)	O5—C11—C10	112.3 (6)
C4—C3—C1	117.9 (6)	O5—C11—H11A	109.1
C5—C4—C3	117.8 (6)	C10—C11—H11A	109.1
C5—C4—C2	122.3 (6)	O5—C11—H11B	109.1
C3—C4—C2	119.9 (6)	C10—C11—H11B	109.1
C6—C5—C4	122.4 (6)	H11A—C11—H11B	107.9
C6—C5—Br1	120.2 (5)	O6—C12—H12A	109.5
C4—C5—Br1	117.4 (5)	O6—C12—H12B	109.5
C5—C6—C7	119.4 (6)	H12A—C12—H12B	109.5
C5—C6—Br2	121.5 (5)	O6—C12—H12C	109.5
C7—C6—Br2	119.2 (5)	H12A—C12—H12C	109.5
C8—C7—C6	119.0 (5)	H12B—C12—H12C	109.5
C8—C7—Br3	119.9 (5)		

C9—O1—C1—O2	6.2 (13)	C4—C5—C6—C7	−0.4 (11)
C9—O1—C1—C3	−178.2 (6)	Br1—C5—C6—C7	179.3 (5)
O2—C1—C3—C8	63.9 (13)	C4—C5—C6—Br2	−179.1 (5)
O1—C1—C3—C8	−111.7 (8)	Br1—C5—C6—Br2	0.5 (9)
O2—C1—C3—C4	−115.6 (10)	C5—C6—C7—C8	−0.4 (11)
O1—C1—C3—C4	68.9 (9)	Br2—C6—C7—C8	178.4 (6)
C8—C3—C4—C5	−3.3 (11)	C5—C6—C7—Br3	178.2 (6)
C1—C3—C4—C5	176.1 (7)	Br2—C6—C7—Br3	−3.0 (9)
C8—C3—C4—C2	175.8 (8)	C4—C3—C8—C7	2.7 (12)
C1—C3—C4—C2	−4.7 (11)	C1—C3—C8—C7	−176.7 (7)
O4—C2—C4—C5	77.2 (10)	C4—C3—C8—Br4	−177.1 (5)
O3—C2—C4—C5	−105.4 (9)	C1—C3—C8—Br4	3.4 (11)
O4—C2—C4—C3	−101.9 (9)	C6—C7—C8—C3	−0.8 (12)
O3—C2—C4—C3	75.5 (10)	Br3—C7—C8—C3	−179.5 (6)
C3—C4—C5—C6	2.2 (11)	C6—C7—C8—Br4	179.0 (5)
C2—C4—C5—C6	−176.9 (8)	Br3—C7—C8—Br4	0.4 (9)
C3—C4—C5—Br1	−177.5 (5)	N1—C10—C11—O5	−73.5 (8)
C2—C4—C5—Br1	3.4 (10)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O3^{i}	0.89	2.01	2.885 (7)	168
N1—H1B···O6	0.89	1.86	2.740 (7)	168
N1—H1C···O4^{ii}	0.89	1.96	2.789 (8)	154
O5—H5···O4^{ii}	0.82	2.00	2.813 (8)	169
O6—H6···O3^{iii}	0.82	1.92	2.714 (8)	163

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

Fig. 1

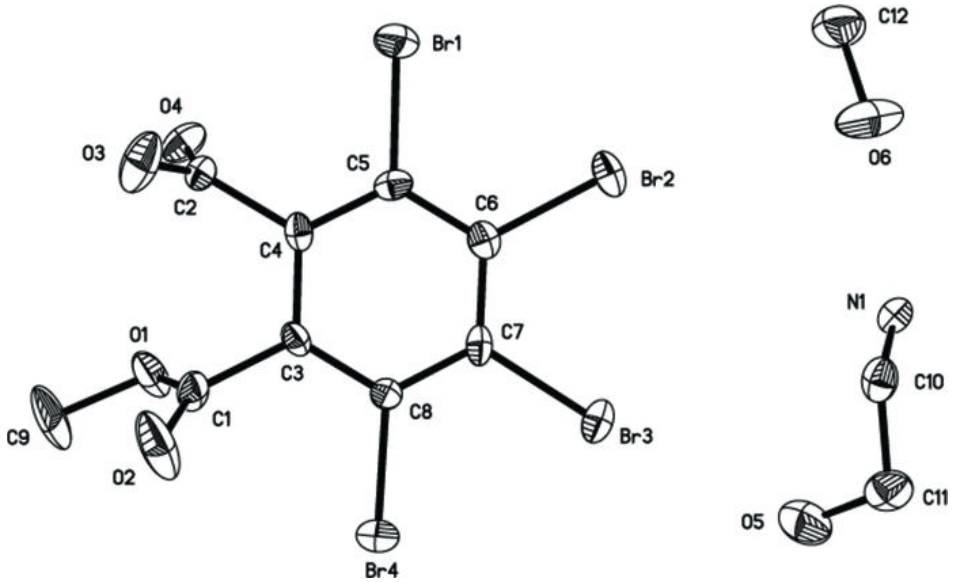

Br1
O4
O3
C2
C5
C6
Br2
C4
C12
O6
N1
C10
O1
C3
C7
C9
C1
C8
Br3
O2
C11
O5
Br4

Fig. 2