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Butane-1,4-diaminium bis[3,4,5,6-tetra-chloro-2-(methoxycarbonyl)benzoate]

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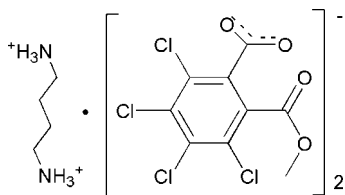
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.036; wR factor = 0.096; data-to-parameter ratio = 14.2.

In the title salt, $\text{C}_4\text{H}_{14}\text{N}_2^{2+} \cdot 2\text{C}_9\text{H}_3\text{Cl}_4\text{O}_4^-$, the cation lies on an inversion center. In the anion, the mean planes of methoxy-carbonyl and carboxylate groups form dihedral angles of 64.9 (3) and 58.5 (3)°, respectively, with the benzene ring. In the crystal, intermolecular $\text{N}-\text{H} \cdots \text{O}$ hydrogen bonds connect the components into sheets parallel to (100).

Related literature

For a related structure, see: Li (2011).



Experimental

Crystal data

$\text{C}_4\text{H}_{14}\text{N}_2^{2+} \cdot 2\text{C}_9\text{H}_3\text{Cl}_4\text{O}_4^-$ $a = 14.4243$ (13) Å
 $M_r = 724.00$ $b = 6.1041$ (6) Å
 Monoclinic, $P2_1/c$ $c = 16.9653$ (15) Å

$\beta = 97.056$ (1)°
 $V = 1482.4$ (2) Å³
 $Z = 2$
 Mo $K\alpha$ radiation

$\mu = 0.81$ mm⁻¹
 $T = 298$ K
 $0.46 \times 0.43 \times 0.40$ mm

Data collection

Bruker SMART CCD diffractometer
 Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
 $T_{\min} = 0.708$, $T_{\max} = 0.738$

7307 measured reflections
 2603 independent reflections
 1908 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.024$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.096$
 $S = 1.05$
 2603 reflections

183 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.23$ e Å⁻³
 $\Delta\rho_{\min} = -0.20$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1C···O3 ⁱ	0.89	1.85	2.735 (3)	172
N1—H1B···O4 ⁱⁱ	0.89	1.94	2.823 (3)	174
N1—H1A···O4	0.89	1.88	2.761 (3)	169

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINTE* (Bruker, 1997); data reduction: *SAINTE*; 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5244).

References

- Bruker (1997). *SMART, SAINT and SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
 Li, J. (2011). *Acta Cryst.* **E67**, o901.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

supplementary materials

Acta Cryst. (2011). E67, o1357 [doi:10.1107/S1600536811016795]

Butane-1,4-diaminium bis[3,4,5,6-tetrachloro-2-(methoxycarbonyl)benzoate]

Z. P. Liang

Comment

In the present work, the reaction of 3,4,5,6-tetrachloro-2-(methoxycarbonyl)benzoic acid and butane-1,4-diamine in methanol was expected to yield 4,5,6,7-tetrachloro-2-[4-(4,5,6,7-tetrachloro-1,3-dioxoisindolin-2-yl)butanyl]isoindoline-1,3-dione. However, the product was the title compound and this may have occurred because of the reduced time and temperature of the reaction. The asymmetric unit of the title compound (I) contains one half of a butane-1,4-diaminium cation and one 3,4,5,6-tetrachloro-2-(methoxycarbonyl)benzoate anions (Fig. 1). In the anion, the mean planes of the methoxycarbonyl and carboxyl groups are aligned at dihedral angles of 64.9 (3) and 58.5 (3)°, respectively with the benzene ring. The bond lengths and angles are in agreement with those which are related in hexane-1,6-diaminium bis[3,4,5,6-tetrachloro-2-(methoxycarbonyl)benzoate] (Li, 2011). In the crystal, intermolecular N—H...O hydrogen bonds connect the components into two-dimensional sheets parallel to (100) (Fig. 2 and Table 1).

Experimental

A mixture of 4,5,6,7-tetrachloroisobenzofuran-1,3-dione (2.86 g, 0.01 mol) and methanol (15 ml) was refluxed for 0.5 h. Then butane-1,4-diamine (0.44 g, 0.005 mol) was added to the above solution and mixed for 20 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 approximation with C—H = 0.96–0.97 Å and N—H = 0.89 Å $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{N, methyl C})$.

Figures

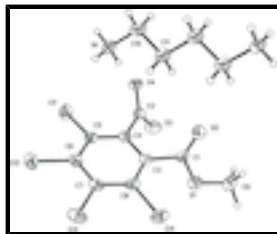


Fig. 1. The asymmetric unit of (I), drawn with 30% probability ellipsoids. The symmetry complete cation is shown with unlabeled atoms related by the symmetry operator $(-x+1, -y+2, -z+1)$.

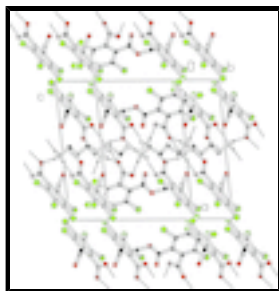
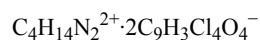


Fig. 2. The crystal packing of (I) with hydrogen bonds shown as dashed lines. Only H atoms involved in hydrogen bonds are shown.

Butane-1,4-diaminium bis[3,4,5,6-tetrachloro-2-(methoxycarbonyl)benzoate]

Crystal data



$$M_r = 724.00$$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$$a = 14.4243 (13) \text{ \AA}$$

$$b = 6.1041 (6) \text{ \AA}$$

$$c = 16.9653 (15) \text{ \AA}$$

$$\beta = 97.056 (1)^\circ$$

$$V = 1482.4 (2) \text{ \AA}^3$$

$$Z = 2$$

$$F(000) = 732$$

$$D_x = 1.622 \text{ Mg m}^{-3}$$

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

Cell parameters from 2612 reflections

$$\theta = 2.4\text{--}26.5^\circ$$

$$\mu = 0.81 \text{ mm}^{-1}$$

$$T = 298 \text{ K}$$

Block, colorless

$$0.46 \times 0.43 \times 0.40 \text{ mm}$$

Data collection

Bruker SMART CCD
diffractometer

Radiation source: fine-focus sealed tube
graphite

φ and ω scans

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

$$T_{\min} = 0.708, T_{\max} = 0.738$$

7307 measured reflections

2603 independent reflections

1908 reflections with $I > 2\sigma(I)$

$$R_{\text{int}} = 0.024$$

$$\theta_{\max} = 25.0^\circ, \theta_{\min} = 2.4^\circ$$

$$h = -9 \rightarrow 17$$

$$k = -7 \rightarrow 7$$

$$l = -18 \rightarrow 20$$

Refinement

Refinement on F^2

Least-squares matrix: full

$$R[F^2 > 2\sigma(F^2)] = 0.036$$

$$wR(F^2) = 0.096$$

$$S = 1.05$$

2603 reflections

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

$$w = 1/[\sigma^2(F_o^2) + (0.0373P)^2 + 1.0059P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} < 0.001$$

183 parameters

$$\Delta\rho_{\max} = 0.23 \text{ e } \text{\AA}^{-3}$$

0 restraints

$$\Delta\rho_{\min} = -0.20 \text{ e } \text{\AA}^{-3}$$

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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.27338 (6)	0.79357 (15)	0.17344 (4)	0.0617 (3)
Cl2	0.10875 (6)	1.10088 (15)	0.19656 (5)	0.0705 (3)
Cl3	0.01193 (5)	1.07644 (14)	0.34929 (5)	0.0642 (3)
Cl4	0.07322 (6)	0.72291 (15)	0.47687 (5)	0.0630 (3)
N1	0.46526 (16)	0.9951 (4)	0.31490 (13)	0.0465 (6)
H1A	0.4416	0.8603	0.3128	0.070*
H1B	0.5002	1.0132	0.2756	0.070*
H1C	0.4189	1.0923	0.3099	0.070*
O1	0.19109 (14)	0.3094 (3)	0.46777 (12)	0.0559 (5)
O2	0.32465 (15)	0.4957 (4)	0.48905 (13)	0.0675 (6)
O3	0.31815 (14)	0.2789 (3)	0.31198 (14)	0.0626 (6)
O4	0.41355 (12)	0.5599 (3)	0.30166 (11)	0.0459 (5)
C1	0.24890 (19)	0.4667 (5)	0.45324 (15)	0.0415 (6)
C2	0.33628 (19)	0.4761 (4)	0.30956 (14)	0.0379 (6)
C3	0.20896 (17)	0.6149 (4)	0.38674 (15)	0.0376 (6)
C4	0.25510 (17)	0.6303 (4)	0.31904 (15)	0.0358 (6)
C5	0.22239 (18)	0.7791 (4)	0.26073 (15)	0.0390 (6)
C6	0.14757 (18)	0.9170 (4)	0.26928 (16)	0.0428 (7)
C7	0.10285 (18)	0.9025 (4)	0.33718 (17)	0.0430 (7)
C8	0.13250 (18)	0.7475 (5)	0.39497 (16)	0.0415 (7)
C9	0.2212 (2)	0.1710 (6)	0.53489 (19)	0.0662 (9)
H9A	0.2630	0.0610	0.5195	0.099*
H9B	0.1679	0.1018	0.5529	0.099*
H9C	0.2529	0.2581	0.5770	0.099*
C10	0.5233 (2)	1.0276 (6)	0.39168 (18)	0.0619 (9)
H10A	0.5461	1.1772	0.3950	0.074*
H10B	0.5770	0.9309	0.3947	0.074*
C11	0.4702 (2)	0.9828 (6)	0.46041 (17)	0.0578 (8)
H11A	0.4477	0.8330	0.4572	0.069*
H11B	0.4163	1.0791	0.4572	0.069*

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0654 (5)	0.0794 (6)	0.0435 (4)	0.0200 (4)	0.0191 (4)	0.0101 (4)
C12	0.0592 (5)	0.0752 (6)	0.0774 (6)	0.0234 (5)	0.0093 (4)	0.0269 (5)
C13	0.0443 (4)	0.0644 (5)	0.0856 (6)	0.0176 (4)	0.0143 (4)	-0.0125 (4)
C14	0.0541 (5)	0.0812 (6)	0.0597 (5)	0.0003 (4)	0.0309 (4)	-0.0073 (4)
N1	0.0478 (14)	0.0430 (13)	0.0528 (14)	0.0035 (11)	0.0233 (11)	0.0023 (11)
O1	0.0553 (13)	0.0521 (12)	0.0602 (13)	-0.0123 (11)	0.0065 (10)	0.0088 (10)
O2	0.0502 (13)	0.0832 (16)	0.0665 (14)	-0.0169 (12)	-0.0032 (11)	0.0221 (12)
O3	0.0513 (13)	0.0355 (12)	0.1034 (18)	0.0027 (10)	0.0198 (12)	-0.0069 (11)
O4	0.0376 (11)	0.0456 (11)	0.0581 (12)	0.0035 (9)	0.0206 (9)	-0.0009 (9)
C1	0.0385 (16)	0.0471 (17)	0.0407 (15)	-0.0064 (13)	0.0117 (13)	-0.0019 (12)
C2	0.0390 (16)	0.0385 (16)	0.0372 (14)	0.0043 (13)	0.0087 (12)	-0.0014 (11)
C3	0.0320 (14)	0.0382 (15)	0.0433 (14)	-0.0038 (12)	0.0076 (11)	-0.0036 (12)
C4	0.0319 (14)	0.0339 (14)	0.0424 (14)	-0.0005 (11)	0.0077 (11)	-0.0051 (11)
C5	0.0352 (15)	0.0435 (15)	0.0393 (14)	0.0011 (12)	0.0083 (11)	-0.0043 (12)
C6	0.0357 (15)	0.0415 (16)	0.0502 (16)	0.0029 (13)	0.0014 (12)	0.0003 (13)
C7	0.0308 (14)	0.0415 (16)	0.0572 (17)	0.0040 (12)	0.0074 (13)	-0.0096 (13)
C8	0.0335 (14)	0.0487 (17)	0.0448 (15)	-0.0025 (13)	0.0144 (12)	-0.0093 (13)
C9	0.077 (2)	0.062 (2)	0.063 (2)	-0.0074 (19)	0.0209 (18)	0.0136 (17)
C10	0.0485 (19)	0.082 (2)	0.057 (2)	-0.0095 (17)	0.0134 (15)	0.0004 (17)
C11	0.0532 (19)	0.067 (2)	0.0557 (18)	-0.0077 (17)	0.0179 (15)	0.0026 (16)

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

C11—C5	1.735 (3)	C3—C8	1.389 (4)
C12—C6	1.711 (3)	C3—C4	1.400 (3)
C13—C7	1.719 (3)	C4—C5	1.382 (4)
C14—C8	1.725 (3)	C5—C6	1.390 (4)
N1—C10	1.473 (4)	C6—C7	1.390 (4)
N1—H1A	0.8900	C7—C8	1.391 (4)
N1—H1B	0.8900	C9—H9A	0.9600
N1—H1C	0.8900	C9—H9B	0.9600
O1—C1	1.314 (3)	C9—H9C	0.9600
O1—C9	1.441 (4)	C10—C11	1.497 (4)
O2—C1	1.196 (3)	C10—H10A	0.9700
O3—C2	1.234 (3)	C10—H10B	0.9700
O4—C2	1.248 (3)	C11—C11 ⁱ	1.518 (6)
C1—C3	1.504 (4)	C11—H11A	0.9700
C2—C4	1.526 (3)	C11—H11B	0.9700
C10—N1—H1A	109.5	C6—C7—C8	119.6 (2)
C10—N1—H1B	109.5	C6—C7—C13	119.9 (2)
H1A—N1—H1B	109.5	C8—C7—C13	120.6 (2)
C10—N1—H1C	109.5	C3—C8—C7	120.3 (2)
H1A—N1—H1C	109.5	C3—C8—C14	120.6 (2)
H1B—N1—H1C	109.5	C7—C8—C14	119.1 (2)

C1—O1—C9	116.1 (2)	O1—C9—H9A	109.5
O2—C1—O1	125.0 (3)	O1—C9—H9B	109.5
O2—C1—C3	122.2 (3)	H9A—C9—H9B	109.5
O1—C1—C3	112.7 (2)	O1—C9—H9C	109.5
O3—C2—O4	126.8 (3)	H9A—C9—H9C	109.5
O3—C2—C4	115.5 (2)	H9B—C9—H9C	109.5
O4—C2—C4	117.7 (2)	N1—C10—C11	112.0 (2)
C8—C3—C4	120.5 (2)	N1—C10—H10A	109.2
C8—C3—C1	120.7 (2)	C11—C10—H10A	109.2
C4—C3—C1	118.6 (2)	N1—C10—H10B	109.2
C5—C4—C3	118.5 (2)	C11—C10—H10B	109.2
C5—C4—C2	121.9 (2)	H10A—C10—H10B	107.9
C3—C4—C2	119.6 (2)	C10—C11—C11 ⁱ	112.1 (3)
C4—C5—C6	121.6 (2)	C10—C11—H11A	109.2
C4—C5—C11	120.1 (2)	C11 ⁱ —C11—H11A	109.2
C6—C5—C11	118.3 (2)	C10—C11—H11B	109.2
C5—C6—C7	119.5 (2)	C11 ⁱ —C11—H11B	109.2
C5—C6—C12	120.8 (2)	H11A—C11—H11B	107.9
C7—C6—C12	119.7 (2)		
C9—O1—C1—O2	3.3 (4)	C4—C5—C6—C7	-1.5 (4)
C9—O1—C1—C3	-175.2 (2)	C11—C5—C6—C7	177.3 (2)
O2—C1—C3—C8	-112.4 (3)	C4—C5—C6—C12	179.6 (2)
O1—C1—C3—C8	66.2 (3)	C11—C5—C6—C12	-1.6 (3)
O2—C1—C3—C4	62.3 (4)	C5—C6—C7—C8	-1.2 (4)
O1—C1—C3—C4	-119.2 (3)	C12—C6—C7—C8	177.8 (2)
C8—C3—C4—C5	-0.4 (4)	C5—C6—C7—C13	178.6 (2)
C1—C3—C4—C5	-175.1 (2)	C12—C6—C7—C13	-2.5 (3)
C8—C3—C4—C2	-177.3 (2)	C4—C3—C8—C7	-2.2 (4)
C1—C3—C4—C2	8.0 (4)	C1—C3—C8—C7	172.4 (2)
O3—C2—C4—C5	-119.9 (3)	C4—C3—C8—C14	178.0 (2)
O4—C2—C4—C5	61.3 (3)	C1—C3—C8—C14	-7.5 (3)
O3—C2—C4—C3	56.9 (3)	C6—C7—C8—C3	3.0 (4)
O4—C2—C4—C3	-121.9 (3)	C13—C7—C8—C3	-176.8 (2)
C3—C4—C5—C6	2.3 (4)	C6—C7—C8—C14	-177.2 (2)
C2—C4—C5—C6	179.1 (2)	C13—C7—C8—C14	3.1 (3)
C3—C4—C5—C11	-176.52 (19)	N1—C10—C11—C11 ⁱ	-179.8 (3)
C2—C4—C5—C11	0.3 (4)		

Symmetry codes: (i) $-x+1, -y+2, -z+1$.

Hydrogen-bond geometry (Å, °)

<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—H1C \cdots O3 ⁱⁱ	0.89	1.85	2.735 (3)	172
N1—H1B \cdots O4 ⁱⁱⁱ	0.89	1.94	2.823 (3)	174
N1—H1A \cdots O4	0.89	1.88	2.761 (3)	169

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

Fig. 1

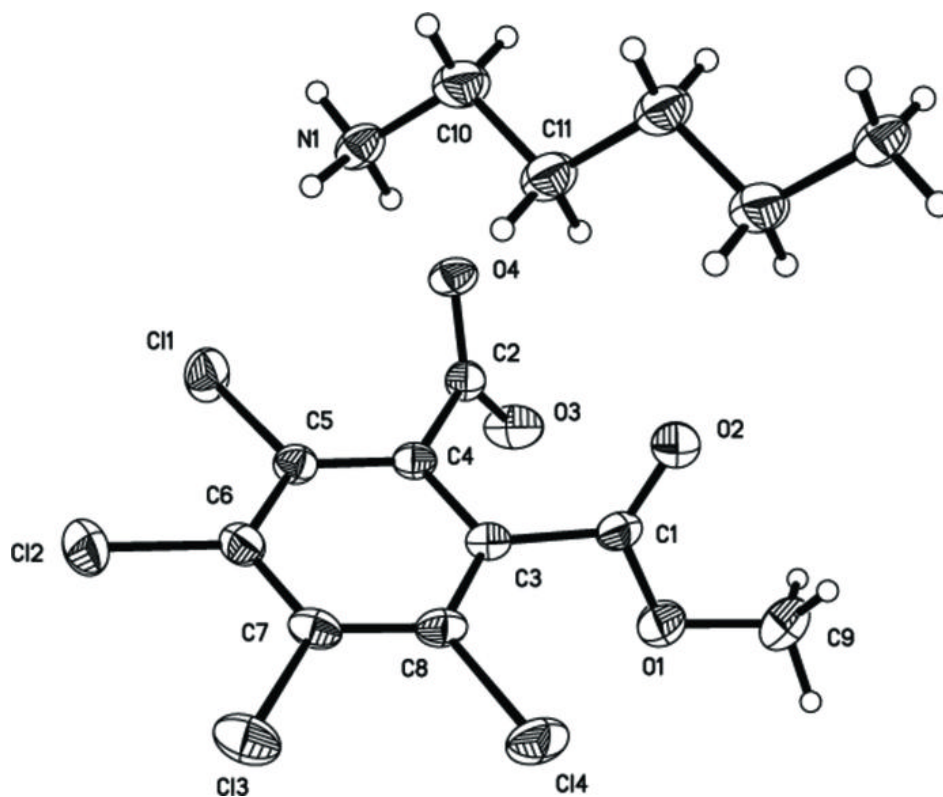


Fig. 2

