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(4,6-Dibromo-*m*-phenylenedimethylidyne) tetraacetate

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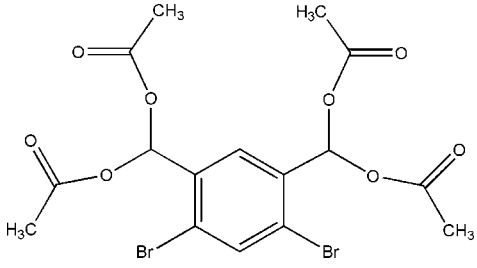
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.009 Å; R factor = 0.067; wR factor = 0.130; data-to-parameter ratio = 17.5.

The title molecule, $C_{16}H_{16}Br_2O_8$, lies on a crystallographic twofold axis. Weak intramolecular C—H···O hydrogen bonds may, in part, control the conformation of the molecule. In the crystal structure, molecules are connected into a two-dimensional network *via* weak intermolecular C—H···O hydrogen bonds.

Related literature

For related literature, see: Allen *et al.* (1987); Mitchell *et al.* (1995).



Experimental

Crystal data

$C_{16}H_{16}Br_2O_8$
M_r = 496.09
Orthorhombic, *Pbcn*
a = 20.639 (4) Å
b = 10.150 (2) Å
c = 9.0880 (18) Å
V = 1903.8 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.30 mm^{-1}
T = 298 (2) K
0.40 × 0.30 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.278, T_{max} = 0.480
(expected range = 0.245–0.423)
3687 measured reflections
1877 independent reflections
805 reflections with $I > 2\sigma(I)$
R_{int} = 0.054
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.130
S = 0.96
1877 reflections
107 parameters
29 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.58 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4A···O3	0.93	2.46	2.815 (7)	103
C5—H5A···O2	0.98	2.34	2.693 (9)	100
C7—H7C···O2^{i}	0.96	2.37	3.318 (11)	170

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); 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: LH2578).

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supporting information

Acta Cryst. (2008). E**64**, o153 [https://doi.org/10.1107/S1600536807063581]

(4,6-Dibromo-*m*-phenylenedimethylidyne) tetraacetate

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S1. Comment

The molecular structure of the title compound is shown in Fig. 1. The bond lengths and angles are within normal ranges (Allen *et al.*, 1987).

The asymmetric unit contains one half-molecule; the full molecule being generated by a crystallographic twofold rotation axis. Weak intramolecular C—H···O hydrogen bonds may, in part, control the conformation of the molecule. In the crystal structure, molecules are connected into a two-dimensional network *via* weak intermolecular C—H···O hydrogen bonds. (Fig. 2).

S2. Experimental

The title compound was prepared by a previously reported method (Mitchell *et al.*, 1995).

The crystals of the title compound, were obtained by dissolving (I) (2.00 g, 4.03 mmol) into acetone (50 ml), and evaporating the solvent slowly at room temperature for about 3 d.

S3. Refinement

H atoms were positioned geometrically, with C—H = 0.93 - 0.98Å and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C), where x = 1.5 for methyl H and x = 1.2 for all other H atoms.

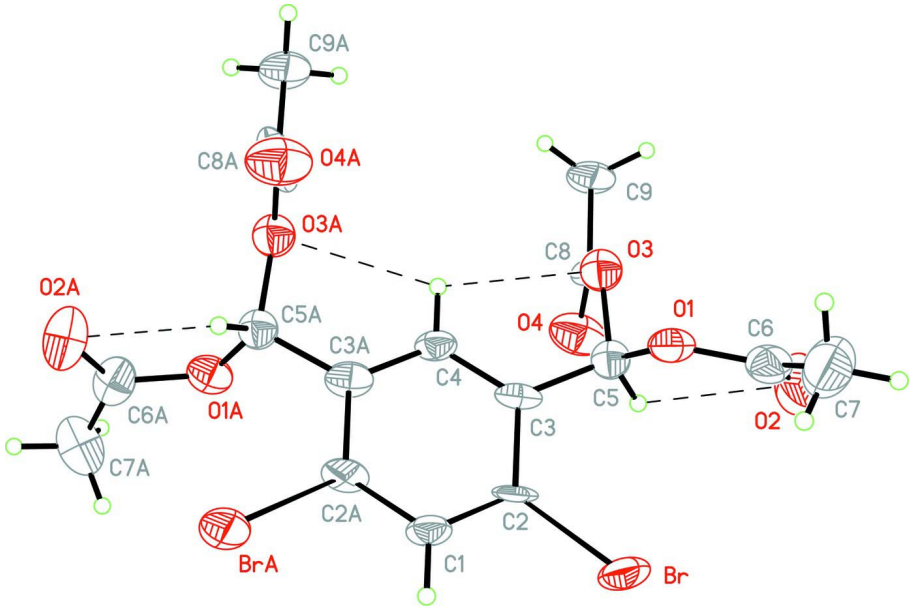

Figure 1

The molecular structure, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 40% probability level. Hydrogen bonds are shown by dashed lines. [Symmetry code: (A) -*x* + 1, *y*, -*z* + 1/2.]

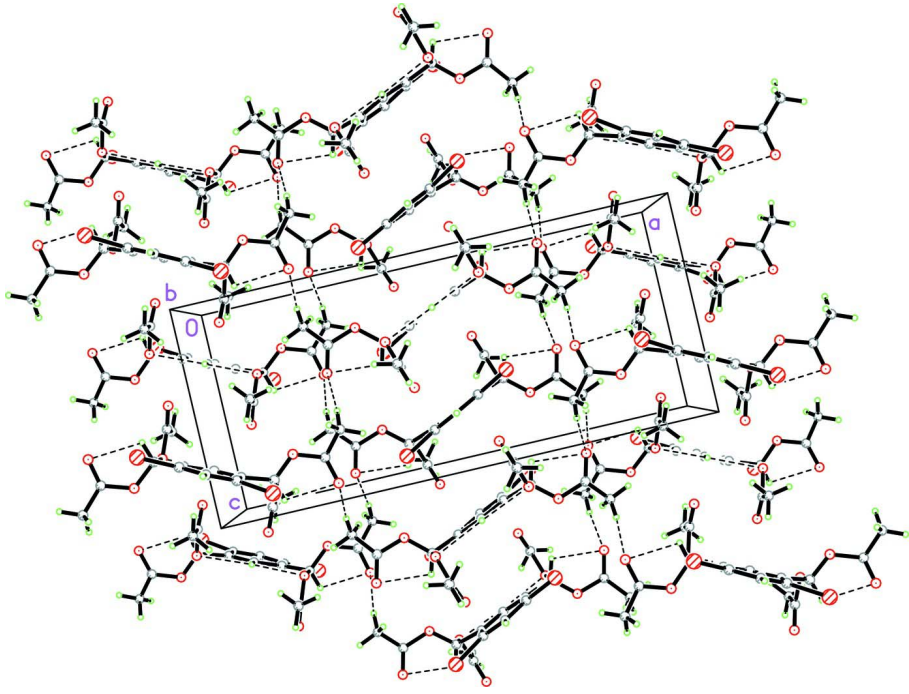

Figure 2

The packing of the title compound with C—H···O hydrogen bonds are shown by dashed lines.

(4,6-Dibromo-*m*-phenylenedimethylidyne) tetraacetate

Crystal data

$C_{16}H_{16}Br_2O_8$
$M_r = 496.09$
Orthorhombic, *Pbcn*
Hall symbol: -P 2n 2ab
$a = 20.639$ (4) Å
$b = 10.150$ (2) Å
$c = 9.0880$ (18) Å
$V = 1903.8$ (6) Å^3
$Z = 4$
$F(000) = 984$
$D_x = 1.731$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10$–$14°$
$\mu = 4.30$ mm^{-1}
$T = 298$ K
Plate, colourless
0.40 × 0.30 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.278$, $T_{max} = 0.480$
3687 measured reflections
1877 independent reflections
805 reflections with $I > 2\sigma(I)$
$R_{int} = 0.054$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.0°$
$h = 0 \rightarrow 25$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 11$
3 standard reflections every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.067$
$wR(F^2) = 0.130$
$S = 0.96$
1877 reflections
107 parameters
29 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.02P)^2 + 0.0P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.58$ e Å^{-3}
$\Delta\rho_{min} = -0.49$ e Å^{-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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br	0.61944 (4)	0.39329 (6)	0.10603 (9)	0.0671 (3)
O1	0.6622 (2)	0.6733 (4)	0.2433 (6)	0.0456 (11)
O2	0.7386 (3)	0.6717 (7)	0.0680 (7)	0.0852 (19)
O3	0.6065 (2)	0.8406 (5)	0.1438 (5)	0.049

O4	0.5702 (3)	0.8387 (5)	−0.0882 (6)	0.0750 (17)
C1	0.5000	0.4229 (8)	0.2500	0.042 (2)
H1A	0.5000	0.3313	0.2500	0.051*
C2	0.5520 (3)	0.4888 (6)	0.1917 (7)	0.0390 (16)
C3	0.5535 (3)	0.6316 (6)	0.1962 (7)	0.0414 (17)
C4	0.5000	0.6958 (9)	0.2500	0.037 (2)
H4A	0.5000	0.7874	0.2500	0.044*
C5	0.6135 (3)	0.6973 (7)	0.1474 (7)	0.041
H5A	0.6256	0.6652	0.0494	0.049*
C6	0.7244 (4)	0.6543 (8)	0.1961 (9)	0.052 (2)
C7	0.7671 (4)	0.6004 (9)	0.3066 (9)	0.084 (3)
H7A	0.8098	0.5906	0.2661	0.126*
H7B	0.7513	0.5159	0.3375	0.126*
H7C	0.7688	0.6588	0.3896	0.126*
C8	0.5877 (3)	0.8970 (7)	0.0164 (10)	0.063 (2)
C9	0.5899 (4)	1.0491 (6)	0.0331 (9)	0.060 (2)
H9A	0.5761	1.0896	−0.0571	0.090*
H9B	0.6334	1.0762	0.0552	0.090*
H9C	0.5616	1.0757	0.1116	0.090*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.0785 (5)	0.0252 (4)	0.0977 (6)	0.0028 (4)	0.0283 (5)	−0.0106 (5)
O1	0.055 (3)	0.033 (2)	0.049 (3)	0.001 (2)	−0.007 (2)	−0.009 (3)
O2	0.064 (4)	0.107 (5)	0.084 (4)	−0.006 (4)	0.024 (3)	−0.001 (4)
O3	0.049	0.049	0.049	0.000	0.000	0.000
O4	0.095 (4)	0.051 (3)	0.080 (4)	−0.008 (3)	−0.023 (3)	0.005 (4)
C1	0.058 (5)	0.019 (4)	0.049 (5)	0.000	0.010 (5)	0.000
C2	0.051 (3)	0.013 (3)	0.053 (4)	−0.004 (3)	−0.011 (3)	−0.016 (3)
C3	0.058 (4)	0.022 (3)	0.045 (4)	−0.005 (3)	−0.008 (3)	−0.010 (3)
C4	0.051 (5)	0.021 (4)	0.040 (5)	0.000	0.005 (4)	0.000
C5	0.041	0.041	0.041	0.000	0.000	0.000
C6	0.058 (5)	0.052 (5)	0.046 (5)	−0.010 (4)	0.010 (4)	−0.018 (4)
C7	0.067 (5)	0.095 (8)	0.090 (7)	0.020 (6)	0.000 (5)	−0.012 (6)
C8	0.037 (3)	0.059 (5)	0.094 (6)	0.009 (4)	−0.011 (4)	−0.055 (6)
C9	0.077 (5)	0.018 (3)	0.084 (5)	−0.005 (4)	0.004 (5)	−0.001 (5)

Geometric parameters (Å, º)

Br—C2	1.866 (6)	C3—C5	1.476 (9)
O1—C5	1.353 (7)	C4—C3^{i}	1.372 (8)
O1—C6	1.366 (8)	C4—H4A	0.9300
O2—C6	1.213 (9)	C5—H5A	0.9800
O3—C8	1.349 (9)	C6—C7	1.445 (10)
O3—C5	1.461 (9)	C7—H7A	0.9600
O4—C8	1.177 (8)	C7—H7B	0.9600
C1—C2	1.371 (7)	C7—H7C	0.9600

C1—C2^{i}	1.371 (7)	C8—C9	1.552 (8)
C1—H1A	0.9300	C9—H9A	0.9600
C2—C3	1.450 (8)	C9—H9B	0.9600
C3—C4	1.372 (8)	C9—H9C	0.9600
C5—O1—C6	121.4 (6)	C3—C5—H5A	109.6
C8—O3—C5	118.0 (5)	O2—C6—O1	120.5 (7)
C2—C1—C2^{i}	121.6 (8)	O2—C6—C7	125.0 (7)
C2—C1—H1A	119.2	O1—C6—C7	114.1 (6)
C2^{i}—C1—H1A	119.2	C6—C7—H7A	109.5
C1—C2—C3	119.6 (6)	C6—C7—H7B	109.5
C1—C2—Br	119.4 (4)	H7A—C7—H7B	109.5
C3—C2—Br	121.0 (5)	C6—C7—H7C	109.5
C4—C3—C2	117.9 (6)	H7A—C7—H7C	109.5
C4—C3—C5	124.6 (6)	H7B—C7—H7C	109.5
C2—C3—C5	117.5 (6)	O4—C8—O3	124.6 (7)
C3^{i}—C4—C3	123.3 (8)	O4—C8—C9	126.0 (8)
C3^{i}—C4—H4A	118.4	O3—C8—C9	109.3 (6)
C3—C4—H4A	118.4	C8—C9—H9A	109.5
O1—C5—O3	105.5 (5)	C8—C9—H9B	109.5
O1—C5—C3	110.4 (5)	H9A—C9—H9B	109.5
O3—C5—C3	112.0 (5)	C8—C9—H9C	109.5
O1—C5—H5A	109.6	H9A—C9—H9C	109.5
O3—C5—H5A	109.6	H9B—C9—H9C	109.5
C2^{i}—C1—C2—C3	2.3 (4)	C8—O3—C5—O1	147.6 (5)
C2^{i}—C1—C2—Br	−177.1 (5)	C8—O3—C5—C3	−92.3 (7)
C1—C2—C3—C4	−4.6 (9)	C4—C3—C5—O1	109.4 (6)
Br—C2—C3—C4	174.8 (3)	C2—C3—C5—O1	−68.3 (8)
C1—C2—C3—C5	173.3 (5)	C4—C3—C5—O3	−7.8 (8)
Br—C2—C3—C5	−7.3 (9)	C2—C3—C5—O3	174.5 (6)
C2—C3—C4—C3^{i}	2.2 (4)	C5—O1—C6—O2	7.5 (12)
C5—C3—C4—C3^{i}	−175.5 (7)	C5—O1—C6—C7	−166.4 (6)
C6—O1—C5—O3	−96.1 (7)	C5—O3—C8—O4	7.9 (10)
C6—O1—C5—C3	142.8 (6)	C5—O3—C8—C9	−174.6 (6)

Symmetry code: (i) −*x*+1, *y*, −*z*+1/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4*A*···O3	0.93	2.46	2.815 (7)	103
C5—H5*A*···O2	0.98	2.34	2.693 (9)	100
C7—H7*C*···O2^{ii}	0.96	2.37	3.318 (11)	170

Symmetry code: (ii) −*x*+3/2, −*y*+3/2, *z*+1/2.