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Tetrakis[(4-methoxycarbonyl)anilinium] hexachloridostannate(IV) dichloride

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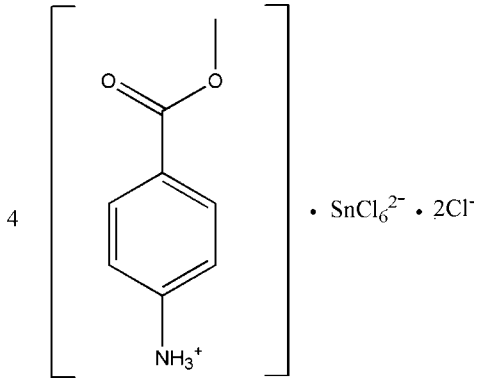
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.031; wR factor = 0.090; data-to-parameter ratio = 15.2.

The asymmetric unit of the title compound, $(C_8H_{10}NO_2)_4[SnCl_6]Cl_2$, contains two (4-methoxycarbonyl)anilinium cations, one chloride anion and one half of a hexachloridostannate(IV) dianion situated on a twofold rotation axis. All aminium H atoms are involved in N—H···Cl hydrogen bonding, which consolidate the crystal packing along with weak C—H···O interactions.

Related literature

For general background to inorganic–organic hybrid compounds, see: Zhang *et al.* (2009); Descalzo *et al.* (2006); Li *et al.* (2007), Sanchez *et al.* (2005).



Experimental

Crystal data

$(C_8H_{10}NO_2)_4[SnCl_6]Cl_2$ M_r = 1010.97
Monoclinic, $C2/c$
a = 30.748 (3) Å
b = 7.1172 (8) Å
c = 22.113 (2) Å
β = 119.424 (2)°
V = 4215.0 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.16 mm^{-1}
T = 298 K
0.50 × 0.46 × 0.46 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.594, T_{max} = 0.617
10221 measured reflections
3719 independent reflections
2969 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.090
S = 1.01
3719 reflections
245 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···Cl3	0.89	2.78	3.659 (4)	170
N2—H2B···Cl3	0.89	2.71	3.479 (3)	145
N2—H2C···Cl4	0.89	2.21	3.098 (4)	177
N1—H1B···Cl4^{i}	0.89	2.29	3.155 (4)	165
N1—H1C···Cl4^{ii}	0.89	2.22	3.092 (4)	166
N2—H2A···Cl1^{iii}	0.89	3.01	3.482 (3)	115
C3—H3···O4^{iv}	0.93	2.39	3.148 (6)	139
C15—H15···O2^{v}	0.93	2.38	3.130 (5)	138

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

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5035).

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supporting information

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Tetrakis[(4-methoxycarbonyl)anilinium] hexachloridostannate(IV) dichloride

Wenzhi Xiao, Ruiting Xue and Yansheng Yin

S1. Comment

Considerable attention has been devoted to inorganic-organic hybrid materials over recent years (Zhang *et al.*, 2009). These hybrid materials have potential applications in many areas including gas storage, separation, catalysis, magnetism, optics as well as electrical conductivity (Descalzo *et al.*, 2006; Li *et al.*, 2007; Sanchez *et al.*, 2005]. Herein we report the structure of the title compound (Fig.1.),

This title compound contains $SnCl_6$ inorganic anions, organic cations and dissociated chloride anions. The $SnCl_6$ inorganic anion adopts a regular octahedron geometry, with average Sn—Cl distance of 2.4262 Å. In the organic cation, the dihedral angle between the ester group and the phenyl ring is 14.86(0.19)°.

In the crystal structure, intermolecular N—H···Cl and C—H···O hydrogen bonds (Table 1) link cations and anions into layers with alternating inorganic and organic species.

S2. Experimental

4-Aminobenzoic acid (10 mmol) was dissolved to acid methanol solution (10 ml). Ten minutes later, a methanol solution (10 ml) of tin tetrachloride(5 mmol) was added with stirring. The mixture was stirred for 4 h. Crystals of the title compound suitable for X-ray analysis were grown from the saturation ethanol solution after about two weeks.

S3. Refinement

All H-atoms were positioned geometrically and refined using a riding model, with C—H = 0.96 Å (methyl), 0.93 Å (aromatic), N—H =0.89 Å (ammonium) and U_{iso}(H) =1.5U_{eq}(C), U_{iso}(H) =1.2U_{eq}(C),U_{iso}(H) =1.5U_{eq}(N)

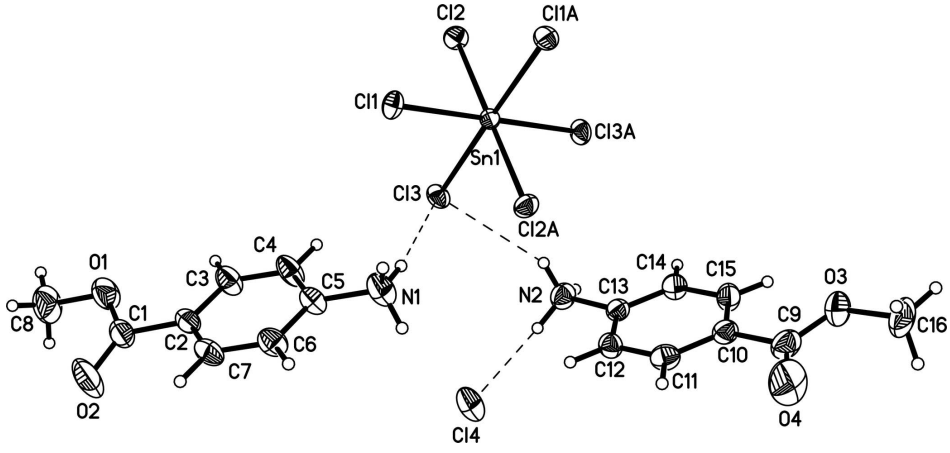

Figure 1

A portion of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme in asymmetric unit [symmetry code (A): -*x*, *y*, -*z* + 1/2]. Dashed lines denote N—H···Cl hydrogen bonds.

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Crystal data

$(C_8H_{10}NO_2)_4[SnCl_6]Cl_2$
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$b = 7.1172$ (8) Å
$c = 22.113$ (2) Å
$\beta = 119.424$ (2)°
$V = 4215.0$ (7) Å^3
$Z = 4$
$F(000) = 2040$
$D_x = 1.593$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4623 reflections
$\theta = 2.7$–$27.7°$
$\mu = 1.16$ mm^{-1}
$T = 298$ K
Block, yellow
0.50 × 0.46 × 0.46 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.594$, $T_{max} = 0.617$
10221 measured reflections
3719 independent reflections
2969 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.5°$
$h = -29 \rightarrow 36$
$k = -7 \rightarrow 8$
$l = -26 \rightarrow 25$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.090$
$S = 1.01$
3719 reflections
245 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.045P)^2 + 5.2918P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.51$ e Å^{-3}
$\Delta\rho_{min} = -0.44$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00204 (13)

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}
Sn1	0.0000	0.88525 (4)	0.2500	0.03147 (14)
Cl1	0.05414 (3)	1.12542 (12)	0.24657 (5)	0.0448 (2)
Cl2	0.04668 (3)	0.88401 (13)	0.37614 (4)	0.0446 (2)

Cl3	0.05329 (3)	0.64043 (12)	0.24497 (5)	0.0452 (2)
Cl4	0.01937 (4)	0.27991 (19)	0.05504 (6)	0.0745 (4)
N1	0.05296 (13)	0.8682 (5)	0.09640 (19)	0.0681 (11)
H1A	0.0488	0.8145	0.1295	0.102*
H1B	0.0354	0.8058	0.0568	0.102*
H1C	0.0425	0.9868	0.0908	0.102*
O1	0.28945 (11)	0.8266 (5)	0.23899 (19)	0.0801 (9)
O2	0.27319 (15)	0.8654 (6)	0.1308 (2)	0.1100 (15)
C1	0.25910 (17)	0.8534 (6)	0.1724 (3)	0.0614 (12)
C2	0.20539 (15)	0.8609 (5)	0.1534 (2)	0.0488 (9)
C3	0.19023 (15)	0.8011 (7)	0.1997 (2)	0.0596 (11)
H3	0.2138	0.7596	0.2437	0.071*
C4	0.14066 (15)	0.8028 (7)	0.1808 (2)	0.0627 (12)
H4	0.1305	0.7621	0.2119	0.075*
C5	0.10627 (15)	0.8642 (5)	0.1165 (2)	0.0510 (10)
C6	0.12064 (16)	0.9276 (6)	0.0699 (2)	0.0563 (10)
H6	0.0971	0.9721	0.0265	0.068*
C7	0.17023 (16)	0.9236 (6)	0.0889 (2)	0.0571 (11)
H7	0.1803	0.9640	0.0577	0.069*
C8	0.34155 (17)	0.8066 (9)	0.2610 (3)	0.0977 (18)
H8A	0.3466	0.6959	0.2404	0.146*
H8B	0.3601	0.7958	0.3107	0.146*
H8C	0.3528	0.9148	0.2467	0.146*
N2	−0.04641 (12)	0.3799 (4)	0.12080 (17)	0.0549 (8)
H2A	−0.0469	0.2835	0.1461	0.082*
H2B	−0.0329	0.4794	0.1481	0.082*
H2C	−0.0284	0.3495	0.1008	0.082*
O3	−0.27704 (11)	0.5652 (5)	−0.06223 (17)	0.0754 (9)
O4	−0.25318 (15)	0.6482 (7)	−0.1379 (2)	0.1235 (17)
C9	−0.24390 (17)	0.5845 (7)	−0.0834 (2)	0.0649 (12)
C10	−0.19295 (13)	0.5235 (6)	−0.03008 (18)	0.0475 (9)
C11	−0.15324 (15)	0.5639 (6)	−0.0409 (2)	0.0530 (10)
H11	−0.1591	0.6244	−0.0814	0.064*
C12	−0.10518 (13)	0.5155 (5)	0.00777 (19)	0.0460 (9)
H12	−0.0785	0.5435	0.0007	0.055*
C13	−0.09772 (13)	0.4248 (5)	0.06700 (18)	0.0412 (8)
C14	−0.13659 (14)	0.3806 (5)	0.07820 (19)	0.0484 (9)
H14	−0.1306	0.3179	0.1185	0.058*
C15	−0.18453 (14)	0.4296 (6)	0.0293 (2)	0.0538 (10)
H15	−0.2112	0.3995	0.0363	0.065*
C16	−0.32747 (17)	0.6233 (8)	−0.1111 (3)	0.100 (2)
H16A	−0.3402	0.5467	−0.1521	0.150*
H16B	−0.3274	0.7526	−0.1234	0.150*
H16C	−0.3483	0.6091	−0.0903	0.150*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.0315 (2)	0.0292 (2)	0.0350 (2)	0.000	0.01734 (15)	0.000
Cl1	0.0388 (5)	0.0417 (5)	0.0498 (5)	−0.0108 (4)	0.0187 (4)	0.0027 (4)
Cl2	0.0430 (5)	0.0531 (6)	0.0319 (5)	0.0061 (4)	0.0138 (4)	0.0043 (4)
Cl3	0.0441 (5)	0.0385 (5)	0.0611 (6)	0.0090 (4)	0.0322 (5)	0.0006 (4)
Cl4	0.0609 (7)	0.0948 (9)	0.0812 (8)	0.0232 (6)	0.0452 (6)	0.0309 (7)
N1	0.054 (2)	0.094 (3)	0.063 (2)	0.0182 (18)	0.0344 (19)	0.021 (2)
O1	0.0490 (18)	0.115 (3)	0.080 (2)	0.0101 (17)	0.0348 (18)	−0.003 (2)
O2	0.084 (3)	0.174 (4)	0.107 (3)	0.001 (2)	0.074 (3)	0.016 (3)
C1	0.063 (3)	0.055 (3)	0.086 (4)	−0.006 (2)	0.052 (3)	−0.008 (2)
C2	0.056 (2)	0.045 (2)	0.060 (3)	0.0003 (17)	0.040 (2)	−0.0021 (18)
C3	0.051 (2)	0.083 (3)	0.052 (2)	0.013 (2)	0.031 (2)	0.017 (2)
C4	0.056 (3)	0.091 (3)	0.056 (3)	0.019 (2)	0.038 (2)	0.028 (2)
C5	0.052 (2)	0.056 (3)	0.055 (2)	0.0110 (18)	0.034 (2)	0.0093 (19)
C6	0.063 (3)	0.065 (3)	0.047 (2)	0.009 (2)	0.032 (2)	0.0141 (19)
C7	0.072 (3)	0.062 (3)	0.056 (3)	0.003 (2)	0.046 (2)	0.008 (2)
C8	0.053 (3)	0.114 (4)	0.128 (5)	0.012 (3)	0.046 (3)	−0.002 (4)
N2	0.0470 (19)	0.057 (2)	0.052 (2)	0.0027 (15)	0.0173 (16)	−0.0006 (16)
O3	0.0418 (16)	0.094 (2)	0.075 (2)	0.0087 (16)	0.0164 (16)	−0.0012 (18)
O4	0.081 (3)	0.201 (5)	0.063 (2)	0.034 (3)	0.015 (2)	0.051 (3)
C9	0.055 (3)	0.073 (3)	0.047 (3)	0.008 (2)	0.010 (2)	−0.002 (2)
C10	0.046 (2)	0.053 (2)	0.039 (2)	0.0024 (17)	0.0163 (18)	−0.0014 (18)
C11	0.063 (3)	0.055 (2)	0.041 (2)	0.0008 (19)	0.025 (2)	0.0055 (18)
C12	0.046 (2)	0.046 (2)	0.051 (2)	−0.0042 (17)	0.0277 (19)	0.0003 (18)
C13	0.040 (2)	0.042 (2)	0.038 (2)	0.0015 (15)	0.0161 (16)	−0.0023 (16)
C14	0.048 (2)	0.056 (2)	0.040 (2)	0.0017 (18)	0.0207 (18)	0.0097 (17)
C15	0.042 (2)	0.070 (3)	0.048 (2)	−0.0043 (19)	0.0216 (19)	0.004 (2)
C16	0.039 (3)	0.114 (5)	0.103 (4)	0.013 (3)	0.002 (3)	−0.023 (3)

Geometric parameters (Å, º)

Sn1—Cl1	2.4131 (8)	C8—H8A	0.9600
Sn1—Cl1^{i}	2.4131 (8)	C8—H8B	0.9600
Sn1—Cl2^{i}	2.4305 (9)	C8—H8C	0.9600
Sn1—Cl2	2.4305 (9)	N2—C13	1.471 (4)
Sn1—Cl3	2.4315 (8)	N2—H2A	0.8900
Sn1—Cl3^{i}	2.4315 (8)	N2—H2B	0.8900
N1—C5	1.473 (5)	N2—H2C	0.8900
N1—H1A	0.8900	O3—C9	1.320 (5)
N1—H1B	0.8900	O3—C16	1.448 (5)
N1—H1C	0.8900	O4—C9	1.185 (5)
O1—C1	1.314 (6)	C9—C10	1.489 (5)
O1—C8	1.434 (5)	C10—C15	1.380 (5)
O2—C1	1.197 (5)	C10—C11	1.384 (5)
C1—C2	1.492 (5)	C11—C12	1.377 (5)
C2—C7	1.373 (6)	C11—H11	0.9300

C2—C3	1.384 (5)	C12—C13	1.375 (5)
C3—C4	1.368 (5)	C12—H12	0.9300
C3—H3	0.9300	C13—C14	1.371 (5)
C4—C5	1.362 (5)	C14—C15	1.378 (5)
C4—H4	0.9300	C14—H14	0.9300
C5—C6	1.380 (5)	C15—H15	0.9300
C6—C7	1.369 (5)	C16—H16A	0.9600
C6—H6	0.9300	C16—H16B	0.9600
C7—H7	0.9300	C16—H16C	0.9600

Cl1—Sn1—Cl1^{i}	89.79 (5)	C2—C7—H7	119.5
Cl1—Sn1—Cl2^{i}	89.66 (3)	O1—C8—H8A	109.5
Cl1^{i}—Sn1—Cl2^{i}	90.63 (3)	O1—C8—H8B	109.5
Cl1—Sn1—Cl2	90.63 (3)	H8A—C8—H8B	109.5
Cl1^{i}—Sn1—Cl2	89.66 (3)	O1—C8—H8C	109.5
Cl2^{i}—Sn1—Cl2	179.58 (4)	H8A—C8—H8C	109.5
Cl1—Sn1—Cl3	90.88 (3)	H8B—C8—H8C	109.5
Cl1^{i}—Sn1—Cl3	179.00 (3)	C13—N2—H2A	109.5
Cl2^{i}—Sn1—Cl3	88.64 (3)	C13—N2—H2B	109.5
Cl2—Sn1—Cl3	91.06 (3)	H2A—N2—H2B	109.5
Cl1—Sn1—Cl3^{i}	179.00 (3)	C13—N2—H2C	109.5
Cl1^{i}—Sn1—Cl3^{i}	90.88 (3)	H2A—N2—H2C	109.5
Cl2^{i}—Sn1—Cl3^{i}	91.06 (3)	H2B—N2—H2C	109.5
Cl2—Sn1—Cl3^{i}	88.64 (3)	C9—O3—C16	115.8 (4)
Cl3—Sn1—Cl3^{i}	88.45 (4)	O4—C9—O3	124.0 (4)
C5—N1—H1A	109.5	O4—C9—C10	123.4 (5)
C5—N1—H1B	109.5	O3—C9—C10	112.6 (4)
H1A—N1—H1B	109.5	C15—C10—C11	119.7 (3)
C5—N1—H1C	109.5	C15—C10—C9	121.8 (4)
H1A—N1—H1C	109.5	C11—C10—C9	118.5 (4)
H1B—N1—H1C	109.5	C12—C11—C10	120.8 (4)
C1—O1—C8	117.0 (4)	C12—C11—H11	119.6
O2—C1—O1	123.1 (4)	C10—C11—H11	119.6
O2—C1—C2	123.4 (5)	C13—C12—C11	118.3 (3)
O1—C1—C2	113.5 (4)	C13—C12—H12	120.9
C7—C2—C3	119.2 (4)	C11—C12—H12	120.9
C7—C2—C1	120.1 (4)	C14—C13—C12	121.8 (3)
C3—C2—C1	120.6 (4)	C14—C13—N2	119.2 (3)
C4—C3—C2	120.1 (4)	C12—C13—N2	118.9 (3)
C4—C3—H3	120.0	C13—C14—C15	119.5 (4)
C2—C3—H3	120.0	C13—C14—H14	120.3
C5—C4—C3	119.9 (4)	C15—C14—H14	120.3
C5—C4—H4	120.0	C14—C15—C10	119.8 (4)
C3—C4—H4	120.0	C14—C15—H15	120.1
C4—C5—C6	121.0 (4)	C10—C15—H15	120.1
C4—C5—N1	119.9 (3)	O3—C16—H16A	109.5
C6—C5—N1	119.0 (4)	O3—C16—H16B	109.5
C7—C6—C5	118.8 (4)	H16A—C16—H16B	109.5

C7—C6—H6	120.6	O3—C16—H16C	109.5
C5—C6—H6	120.6	H16A—C16—H16C	109.5
C6—C7—C2	121.0 (3)	H16B—C16—H16C	109.5
C6—C7—H7	119.5		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···Cl3	0.89	2.78	3.659 (4)	170
N2—H2*B*···Cl3	0.89	2.71	3.479 (3)	145
N2—H2*C*···Cl4	0.89	2.21	3.098 (4)	177
N1—H1*B*···Cl4^{ii}	0.89	2.29	3.155 (4)	165
N1—H1*C*···Cl4^{iii}	0.89	2.22	3.092 (4)	166
N2—H2*A*···Cl1^{iv}	0.89	3.01	3.482 (3)	115
C3—H3···O4^{v}	0.93	2.39	3.148 (6)	139
C15—H15···O2^{vi}	0.93	2.38	3.130 (5)	138

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