

catena-Poly[[aqua(3-methylbenzoato- κ^2O,O')lead(II)]- μ -3-methylbenzoato- $\kappa^4O:O,O':O'$]

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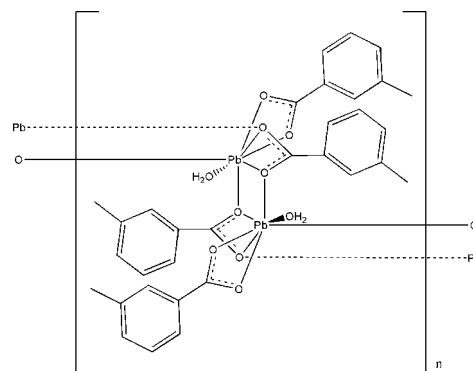
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(C-C) = 0.009$ Å; R factor = 0.035; wR factor = 0.061; data-to-parameter ratio = 20.4.

The reaction of lead(II) acetate and 3-methylbenzoic acid (MBA) in aqueous solution yielded the title polymer, $[Pb(C_8H_7O_2)_2(H_2O)]_n$. The asymmetric unit contains two Pb^{II} atoms, four MBA ligands and two water molecules. Each Pb^{II} cation is heptacoordinated and chelated by four carboxylate O atoms from two MBA ligands. The Pb atoms are bridged through the carboxylate O atoms from another two MBA ligands, leading to a central Pb_2O_2 core. The Pb–O bond lengths are in the range 2.325 (3)–2.757 (4) Å. The intra- and interdimer Pb···Pb distances are 4.2942 (3) and 4.2283 (3) Å, respectively, indicating little direct metal–metal interaction. The coordinating water molecules and carboxylate O atoms are involved in extensive O–H···O hydrogen-bonding interactions. The complex has an extended ladder-like chain structure and the chains are assembled by hydrogen bonds and π – π interactions [centroid–centroid distance = 3.6246 (3) Å] into a three-dimensional supramolecular structure.

Related literature

For general background to metal-organic frameworks and their applications, see: Hamilton *et al.* (2004); Meng *et al.* (2003); Fan & Zhu (2006); Wang *et al.* (2006); Masaoka *et al.* (2001). For related structures, see: Shi *et al.* (2007).



Experimental

Crystal data

$[Pb(C_8H_7O_2)_2(H_2O)]$
 $M_r = 495.48$
Monoclinic, $P2_1/n$
 $a = 7.1745$ (3) Å
 $b = 42.745$ (2) Å
 $c = 10.7126$ (5) Å
 $\beta = 90.765$ (1)°

$V = 3285.0$ (3) Å³
 $Z = 8$
Mo $K\alpha$ radiation
 $\mu = 10.29$ mm⁻¹
 $T = 296$ K
 $0.36 \times 0.17 \times 0.12$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Bruker, 2007)
 $T_{min} = 0.144$, $T_{max} = 0.300$

40611 measured reflections
8096 independent reflections
6265 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.056$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.061$
 $S = 1.03$
8096 reflections

397 parameters
H-atom parameters constrained
 $\Delta\rho_{max} = 0.87$ e Å⁻³
 $\Delta\rho_{min} = -1.00$ e Å⁻³

Table 1

Selected bond lengths (Å).

| | | | |
|---------------------|-----------|----------------------|-----------|
| Pb1–O4 | 2.386 (3) | Pb2–O6 | 2.325 (3) |
| Pb1–O1 | 2.424 (3) | Pb2–O8 | 2.494 (4) |
| Pb1–O3 | 2.594 (3) | Pb2–O3 ⁱⁱ | 2.538 (3) |
| Pb1–O5 | 2.603 (3) | Pb2–O7 | 2.565 (4) |
| Pb1–O2 | 2.622 (4) | Pb2–O10 | 2.665 (3) |
| Pb1–O9 | 2.724 (4) | Pb2–O4 | 2.712 (3) |
| Pb1–O6 ⁱ | 2.751 (3) | Pb2–O5 | 2.757 (4) |

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

Table 2

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-----------------------------|-------|-------------|-------------|---------------|
| O9–H9A···O8 | 0.82 | 2.03 | 2.805 (5) | 158 |
| O9–H9B···O7 ⁱ | 0.82 | 2.25 | 3.017 (5) | 156 |
| O10–H10B···O2 | 0.82 | 2.12 | 2.881 (5) | 153 |
| O10–H10A···O1 ⁱⁱ | 0.82 | 1.97 | 2.774 (5) | 166 |

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINTE* (Bruker, 2007); 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); 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: FJ2218).

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supporting information

Acta Cryst. (2009). E65, m709–m710 [doi:10.1107/S1600536809019771]

catena-Poly[[aqua(3-methylbenzoato- κ^2 O,O')lead(II)]- μ -3-methylbenzoato- κ^4 O:O,O':O']

Jun Dai, Juan Yang and Xiaobing An

S1. Comment

Interest in porous metal-organic frameworks (MOFs) has been driven by the prospect of generating a wide range of materials with useful properties for applications such as ion-exchange, nonlinear optics and catalysis (Hamilton *et al.*, 2004; Meng *et al.*, 2003; Fan *et al.* 2006). On the other hand, lead(II) compounds have been increasingly studied (Shi *et al.* 2007) owing to their possible applications in different fields, especially in environmental protection due to the toxicity of lead and in biological systems for its diverse interactions with biological molecules. As an important family of multidentate O-donor ligands, aromatic carboxylate ligands have been extensively employed in the preparation of metal-organic complexes because of their potential properties and intriguing structural topologies (Wang *et al.*, 2006; Masaoka *et al.* 2001). Herein, we report the structure of the title complex.

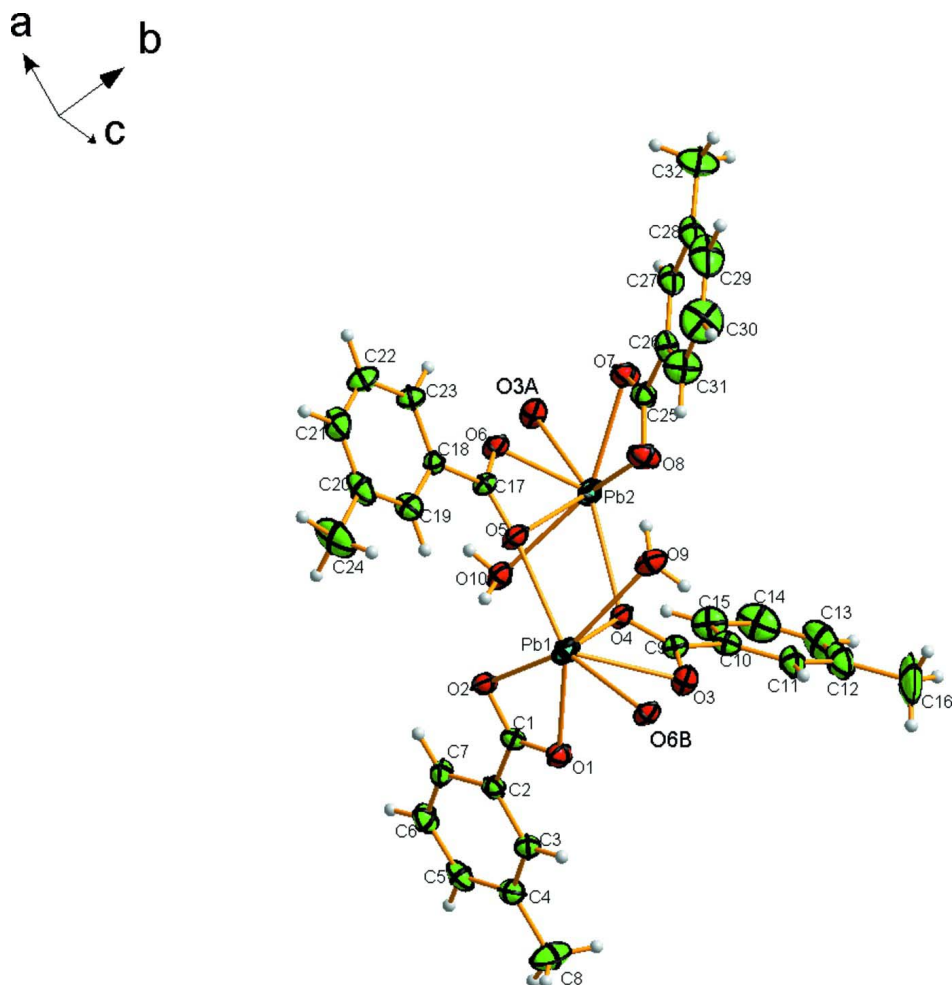
The asymmetric unit of the title complex, $[\text{Pb}_2(\text{C}_8\text{H}_7\text{O}_2)_4(\text{H}_2\text{O})_2]_n$, contains two Pb^{II} cation, four MBA ligands and two coordinating water molecule, as illustrated in Fig. 1. The two Pb atoms are connected *via* two bridging O atoms belonging to two MBA ligands, resulting the central Pb_2O_2 core tetratomic ring. The Pb—O bond lengths are in the range of 2.325 (3) to 2.757 (4) Å (Table 1). The average distance of two Pb atoms is 4.2942 Å, which leads to the weak metal-metal interactions. This coordination polymer structure presents extended ladder-like chain along the *a* axis direction. The coordinating water molecules and carboxylate O atoms are involved in extensive O—H \cdots O hydrogen-bonding interactions (Table 2). These chains are assembled by H-bonds and π - π interactions to three-dimensional supramolecular structure.

S2. Experimental

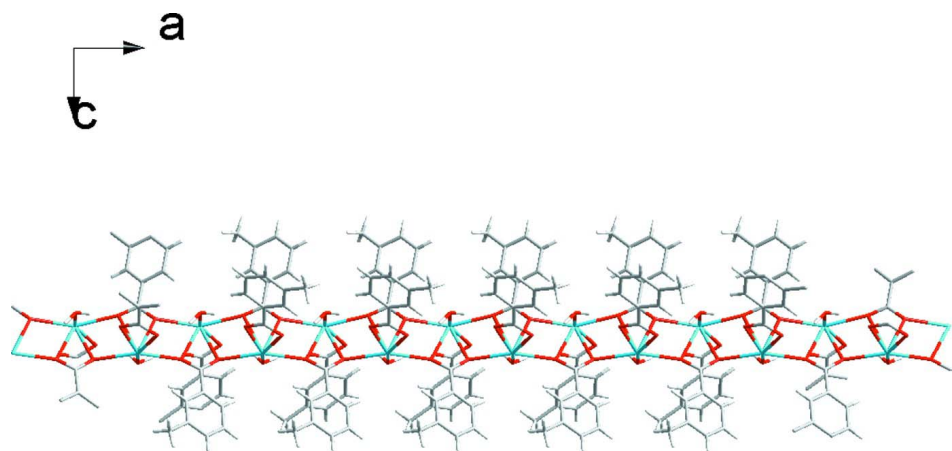
A mixture of $\text{Pb}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}$ (0.1992 g, 0.52 mmol), MBA (0.1139 g, 0.84 mmol), melamine (0.0255 g, 0.20 mmol) and distilled water (10 ml) was sealed in a 25 ml Teflon-lined stainless autoclave (Shi *et al.* 2007). The mixture was heated at 373 K for 5 days to give colorless crystals suitable for X-ray diffraction analysis.

S3. Refinement

All H atoms bounded to C atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H distances in the range of 0.93–0.96 Å. The positions of the water H atoms were found from a difference Fourier map and refined with distance restraints O—H = 0.82 Å, $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O})$.

**Figure 1**

The coordination environment around Pb(II) in the title complex with the atom-labeling scheme. Displacement ellipsoids for non-hydrogen atoms are drawn at the 30% probability level.

**Figure 2**

The extended ladder-like chain structure of the title compound.

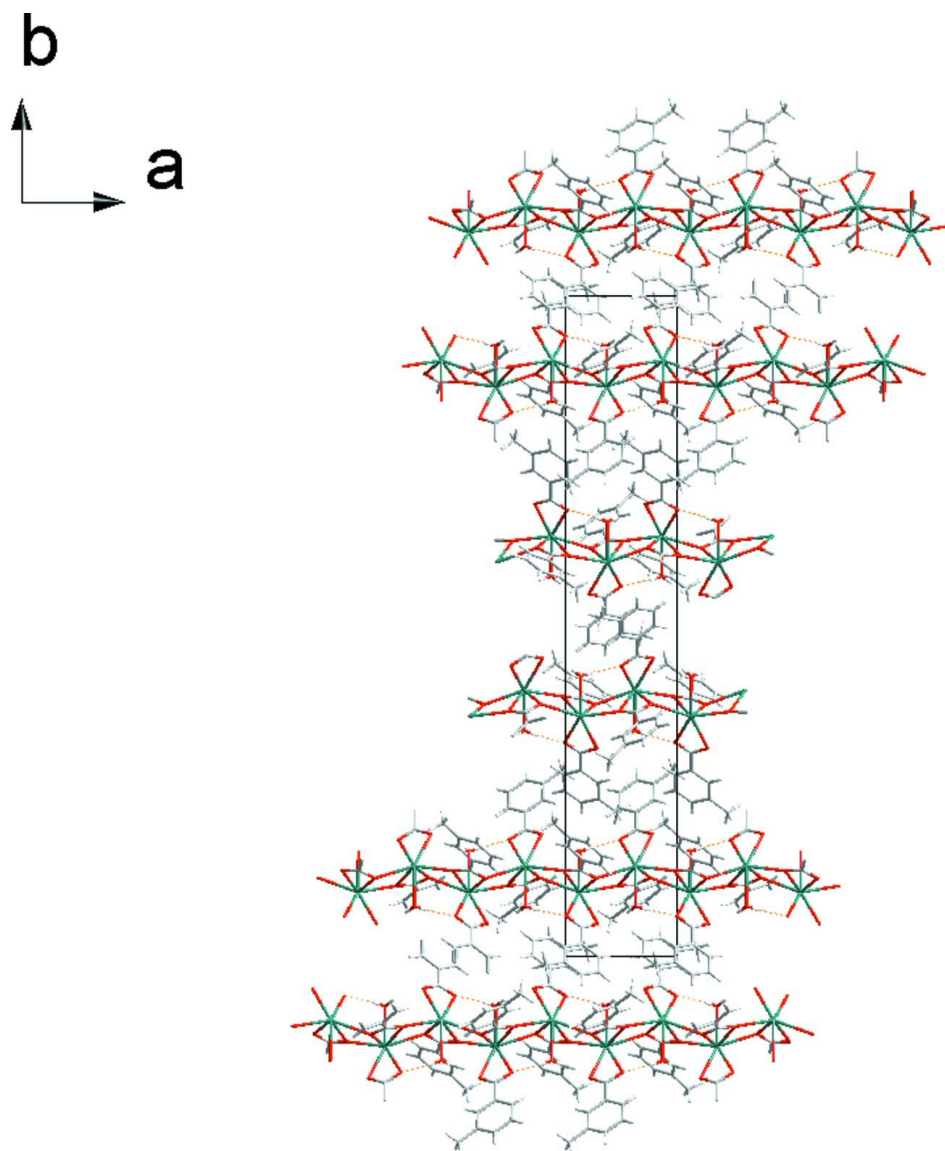
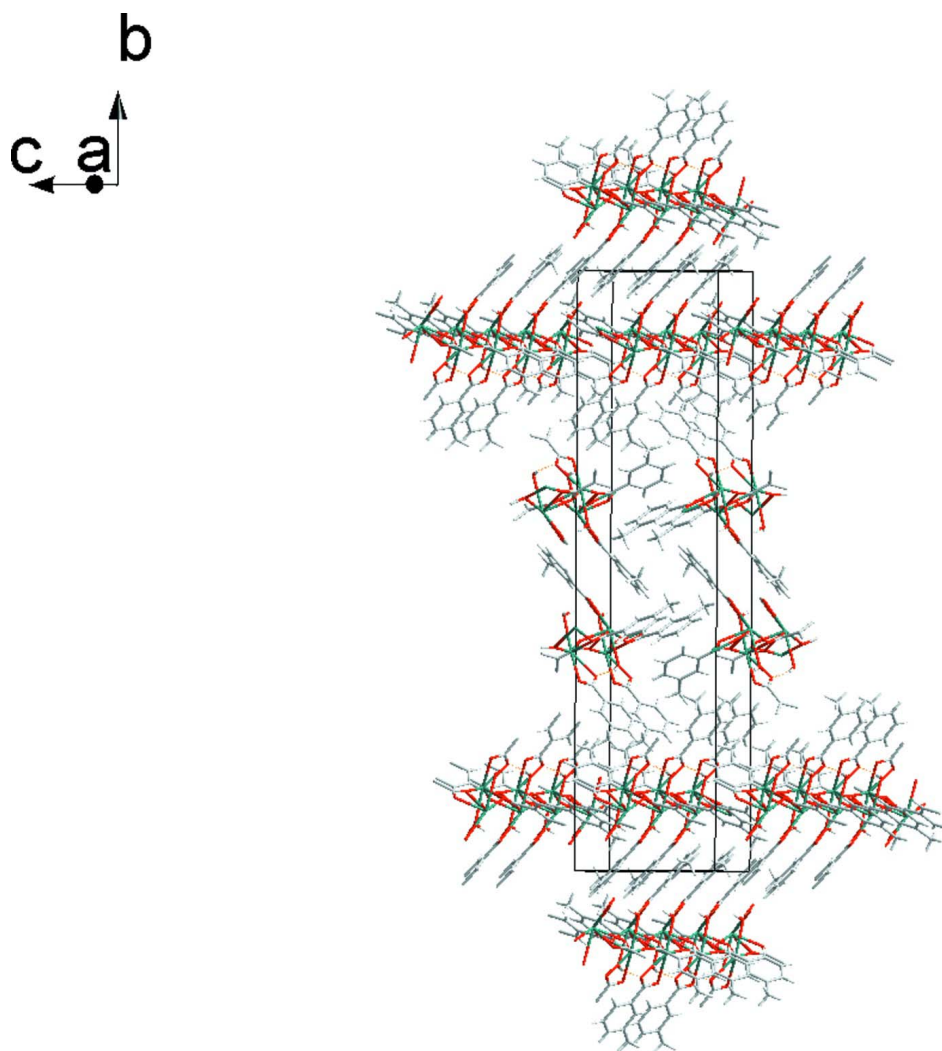


Figure 3

The two-dimensional layer structure of the title compound.

**Figure 4**

The three-dimensional structure of the title compound.

***catena*-Poly[[aqua(3-methylbenzoato- κ^2 O,O')lead(II)]- μ -3-methylbenzoato- κ^4 O:O,O':O']**

Crystal data

[Pb(C₈H₇O₂)₂(H₂O)]

$M_r = 495.48$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 7.1745$ (3) Å

$b = 42.745$ (2) Å

$c = 10.7126$ (5) Å

$\beta = 90.765$ (1)°

$V = 3285.0$ (3) Å³

$Z = 8$

$F(000) = 1872$

$D_x = 2.004$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 7418 reflections

$\theta = 2.4$ – 25.2 °

$\mu = 10.29$ mm⁻¹

$T = 296$ K

Block, colourless

$0.36 \times 0.17 \times 0.12$ mm

Data collection

Bruker APEXII CCD area-detector
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
 φ and ω scans
Absorption correction: multi-scan
(*SADABS*; Bruker, 2007)
 $T_{\min} = 0.144$, $T_{\max} = 0.300$

40611 measured reflections
8096 independent reflections
6265 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.056$
 $\theta_{\text{max}} = 28.2^\circ$, $\theta_{\text{min}} = 1.9^\circ$
 $h = -9 \rightarrow 9$
 $k = -56 \rightarrow 56$
 $l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.061$
 $S = 1.03$
8096 reflections
397 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
 $w = 1/[\sigma^2(F_o^2) + (0.0182P)^2 + 3.7836P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.87 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -1.00 \text{ e } \text{\AA}^{-3}$
Extinction correction: *SHELXL97* (Sheldrick,
2008), $F_c^* = kFc[1 + 0.001x \text{Fc}^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.082

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

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|-------------|---------------|---------------|----------------------------------|
| Pb1 | 0.12243 (2) | 0.097018 (5) | 0.352944 (18) | 0.03251 (6) |
| Pb2 | 0.63079 (2) | 0.136517 (5) | 0.498730 (18) | 0.03285 (6) |
| O1 | 0.0014 (5) | 0.06035 (8) | 0.5043 (3) | 0.0425 (9) |
| O2 | 0.2904 (5) | 0.04888 (8) | 0.4562 (3) | 0.0422 (9) |
| O3 | -0.0208 (5) | 0.12954 (8) | 0.5322 (3) | 0.0417 (9) |
| O4 | 0.2740 (4) | 0.11793 (8) | 0.5341 (3) | 0.0382 (8) |
| O5 | 0.4697 (5) | 0.10648 (9) | 0.2967 (3) | 0.0431 (9) |
| O6 | 0.7648 (4) | 0.11863 (8) | 0.3157 (3) | 0.0361 (8) |
| O7 | 0.7820 (5) | 0.18516 (8) | 0.4032 (4) | 0.0456 (9) |
| O8 | 0.4875 (5) | 0.17631 (9) | 0.3567 (4) | 0.0532 (11) |
| O9 | 0.1259 (5) | 0.15838 (9) | 0.2844 (4) | 0.0536 (11) |
| H9A | 0.2336 | 0.1648 | 0.2856 | 0.064* |
| H9B | 0.0480 | 0.1635 | 0.3359 | 0.064* |
| O10 | 0.6329 (5) | 0.07555 (8) | 0.5507 (3) | 0.0435 (9) |
| H10B | 0.5587 | 0.0665 | 0.5043 | 0.052* |
| H10A | 0.7351 | 0.0704 | 0.5243 | 0.052* |
| C1 | 0.1464 (7) | 0.04397 (11) | 0.5191 (5) | 0.0350 (12) |
| C2 | 0.1449 (7) | 0.01859 (11) | 0.6140 (5) | 0.0327 (11) |
| C3 | -0.0085 (7) | 0.01413 (12) | 0.6892 (5) | 0.0419 (13) |
| H3A | -0.1126 | 0.0268 | 0.6775 | 0.050* |
| C4 | -0.0118 (8) | -0.00864 (13) | 0.7814 (5) | 0.0449 (14) |
| C5 | 0.1437 (9) | -0.02741 (13) | 0.7950 (6) | 0.0501 (15) |
| H5A | 0.1450 | -0.0428 | 0.8563 | 0.060* |
| C6 | 0.2948 (8) | -0.02395 (13) | 0.7213 (6) | 0.0483 (15) |

| | | | | |
|------|--------------|---------------|-------------|-------------|
| H6A | 0.3966 | -0.0372 | 0.7319 | 0.058* |
| C7 | 0.2988 (8) | -0.00088 (12) | 0.6303 (5) | 0.0416 (13) |
| H7A | 0.4032 | 0.0016 | 0.5807 | 0.050* |
| C8 | -0.1813 (10) | -0.01275 (18) | 0.8623 (7) | 0.080 (2) |
| H8A | -0.1591 | -0.0294 | 0.9210 | 0.121* |
| H8B | -0.2874 | -0.0178 | 0.8107 | 0.121* |
| H8C | -0.2050 | 0.0063 | 0.9066 | 0.121* |
| C9 | 0.1354 (7) | 0.13040 (11) | 0.5855 (4) | 0.0297 (11) |
| C10 | 0.1578 (7) | 0.14561 (12) | 0.7091 (5) | 0.0367 (12) |
| C11 | 0.0214 (9) | 0.16600 (13) | 0.7525 (6) | 0.0514 (15) |
| H11A | -0.0821 | 0.1705 | 0.7024 | 0.062* |
| C12 | 0.0388 (12) | 0.17949 (16) | 0.8690 (7) | 0.072 (2) |
| C13 | 0.1933 (15) | 0.1719 (2) | 0.9412 (7) | 0.093 (3) |
| H13A | 0.2058 | 0.1805 | 1.0205 | 0.111* |
| C14 | 0.3266 (12) | 0.1524 (2) | 0.8998 (7) | 0.081 (2) |
| H14A | 0.4297 | 0.1481 | 0.9505 | 0.097* |
| C15 | 0.3122 (9) | 0.13865 (15) | 0.7832 (5) | 0.0563 (17) |
| H15A | 0.4040 | 0.1251 | 0.7551 | 0.068* |
| C16 | -0.1079 (14) | 0.2018 (2) | 0.9145 (9) | 0.131 (4) |
| H16A | -0.0742 | 0.2091 | 0.9965 | 0.197* |
| H16B | -0.2258 | 0.1912 | 0.9175 | 0.197* |
| H16C | -0.1171 | 0.2193 | 0.8586 | 0.197* |
| C17 | 0.6265 (6) | 0.10896 (11) | 0.2509 (5) | 0.0296 (11) |
| C18 | 0.6596 (7) | 0.10073 (11) | 0.1176 (5) | 0.0308 (11) |
| C19 | 0.5301 (8) | 0.08355 (12) | 0.0511 (5) | 0.0425 (13) |
| H19A | 0.4204 | 0.0776 | 0.0896 | 0.051* |
| C20 | 0.5586 (10) | 0.07500 (14) | -0.0711 (6) | 0.0558 (17) |
| C21 | 0.7238 (11) | 0.08378 (15) | -0.1256 (6) | 0.0634 (19) |
| H21A | 0.7476 | 0.0777 | -0.2072 | 0.076* |
| C22 | 0.8544 (10) | 0.10140 (15) | -0.0611 (6) | 0.0612 (18) |
| H22A | 0.9636 | 0.1076 | -0.0999 | 0.073* |
| C23 | 0.8229 (8) | 0.10979 (13) | 0.0606 (5) | 0.0420 (13) |
| H23A | 0.9110 | 0.1215 | 0.1045 | 0.050* |
| C24 | 0.4110 (11) | 0.05680 (17) | -0.1440 (7) | 0.090 (3) |
| H24A | 0.4543 | 0.0528 | -0.2269 | 0.135* |
| H24B | 0.2980 | 0.0688 | -0.1483 | 0.135* |
| H24C | 0.3875 | 0.0373 | -0.1027 | 0.135* |
| C25 | 0.6358 (8) | 0.19171 (13) | 0.3421 (5) | 0.0413 (13) |
| C26 | 0.6358 (8) | 0.21833 (13) | 0.2526 (5) | 0.0437 (14) |
| C27 | 0.7935 (9) | 0.23678 (13) | 0.2416 (5) | 0.0500 (15) |
| H27A | 0.8980 | 0.2324 | 0.2908 | 0.060* |
| C28 | 0.7986 (11) | 0.26149 (15) | 0.1590 (6) | 0.0643 (19) |
| C29 | 0.6435 (15) | 0.26682 (19) | 0.0853 (7) | 0.091 (3) |
| H29A | 0.6446 | 0.2832 | 0.0283 | 0.109* |
| C30 | 0.4891 (14) | 0.2487 (2) | 0.0936 (8) | 0.095 (3) |
| H30A | 0.3868 | 0.2526 | 0.0417 | 0.114* |
| C31 | 0.4833 (10) | 0.22464 (17) | 0.1785 (7) | 0.069 (2) |
| H31A | 0.3758 | 0.2126 | 0.1857 | 0.083* |

| | | | | |
|------|-------------|--------------|------------|-----------|
| C32 | 0.9668 (12) | 0.28192 (18) | 0.1483 (8) | 0.107 (3) |
| H32A | 0.9438 | 0.2978 | 0.0865 | 0.161* |
| H32B | 1.0717 | 0.2695 | 0.1242 | 0.161* |
| H32C | 0.9928 | 0.2916 | 0.2274 | 0.161* |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|--------------|--------------|--------------|--------------|--------------|--------------|
| Pb1 | 0.02451 (10) | 0.04213 (12) | 0.03088 (11) | -0.00082 (8) | 0.00003 (7) | -0.00184 (9) |
| Pb2 | 0.02385 (9) | 0.04156 (12) | 0.03319 (11) | 0.00000 (8) | 0.00229 (8) | -0.00581 (9) |
| O1 | 0.034 (2) | 0.042 (2) | 0.052 (2) | 0.0036 (17) | 0.0058 (18) | 0.0068 (18) |
| O2 | 0.0285 (19) | 0.049 (2) | 0.050 (2) | -0.0036 (16) | 0.0087 (17) | 0.0043 (18) |
| O3 | 0.034 (2) | 0.053 (2) | 0.038 (2) | 0.0047 (17) | -0.0067 (17) | -0.0090 (18) |
| O4 | 0.0249 (18) | 0.049 (2) | 0.041 (2) | -0.0005 (16) | 0.0056 (16) | -0.0044 (18) |
| O5 | 0.0280 (19) | 0.059 (2) | 0.042 (2) | -0.0028 (17) | 0.0080 (17) | -0.0055 (19) |
| O6 | 0.0273 (18) | 0.050 (2) | 0.031 (2) | -0.0020 (16) | -0.0003 (15) | -0.0068 (17) |
| O7 | 0.037 (2) | 0.042 (2) | 0.058 (3) | -0.0016 (17) | -0.0059 (19) | 0.0046 (19) |
| O8 | 0.039 (2) | 0.052 (2) | 0.068 (3) | -0.0060 (19) | -0.012 (2) | 0.005 (2) |
| O9 | 0.035 (2) | 0.069 (3) | 0.057 (3) | -0.007 (2) | 0.0047 (19) | 0.001 (2) |
| O10 | 0.033 (2) | 0.053 (2) | 0.045 (2) | 0.0014 (17) | 0.0021 (17) | -0.0025 (18) |
| C1 | 0.032 (3) | 0.034 (3) | 0.039 (3) | -0.005 (2) | 0.000 (2) | -0.004 (2) |
| C2 | 0.036 (3) | 0.030 (3) | 0.033 (3) | -0.005 (2) | -0.001 (2) | -0.004 (2) |
| C3 | 0.034 (3) | 0.041 (3) | 0.051 (4) | -0.003 (2) | 0.002 (3) | -0.006 (3) |
| C4 | 0.047 (3) | 0.044 (3) | 0.044 (4) | -0.010 (3) | 0.003 (3) | 0.001 (3) |
| C5 | 0.069 (4) | 0.034 (3) | 0.047 (4) | -0.003 (3) | -0.007 (3) | 0.005 (3) |
| C6 | 0.052 (4) | 0.038 (3) | 0.055 (4) | 0.008 (3) | -0.002 (3) | 0.005 (3) |
| C7 | 0.041 (3) | 0.037 (3) | 0.047 (4) | 0.008 (2) | 0.004 (3) | -0.005 (3) |
| C8 | 0.069 (5) | 0.098 (6) | 0.074 (5) | -0.007 (4) | 0.018 (4) | 0.030 (4) |
| C9 | 0.028 (3) | 0.035 (3) | 0.026 (3) | -0.002 (2) | -0.002 (2) | 0.002 (2) |
| C10 | 0.041 (3) | 0.039 (3) | 0.031 (3) | -0.007 (2) | -0.004 (2) | 0.004 (2) |
| C11 | 0.064 (4) | 0.046 (3) | 0.044 (4) | 0.005 (3) | 0.003 (3) | -0.008 (3) |
| C12 | 0.111 (6) | 0.056 (4) | 0.049 (4) | -0.004 (4) | 0.008 (4) | -0.013 (4) |
| C13 | 0.151 (9) | 0.081 (6) | 0.045 (5) | -0.023 (6) | -0.015 (5) | -0.022 (4) |
| C14 | 0.105 (7) | 0.093 (6) | 0.044 (4) | -0.022 (5) | -0.029 (4) | 0.003 (4) |
| C15 | 0.058 (4) | 0.073 (4) | 0.038 (4) | -0.003 (3) | -0.016 (3) | 0.004 (3) |
| C16 | 0.178 (10) | 0.098 (7) | 0.119 (8) | 0.041 (7) | 0.035 (7) | -0.058 (6) |
| C17 | 0.025 (2) | 0.034 (3) | 0.030 (3) | 0.002 (2) | 0.002 (2) | 0.001 (2) |
| C18 | 0.031 (3) | 0.027 (3) | 0.034 (3) | 0.004 (2) | 0.005 (2) | 0.002 (2) |
| C19 | 0.056 (4) | 0.041 (3) | 0.030 (3) | -0.006 (3) | -0.001 (3) | 0.001 (2) |
| C20 | 0.086 (5) | 0.044 (4) | 0.037 (4) | -0.009 (3) | -0.008 (3) | -0.008 (3) |
| C21 | 0.106 (6) | 0.052 (4) | 0.033 (4) | -0.005 (4) | 0.020 (4) | -0.005 (3) |
| C22 | 0.073 (5) | 0.066 (4) | 0.046 (4) | -0.005 (4) | 0.026 (3) | 0.005 (3) |
| C23 | 0.043 (3) | 0.045 (3) | 0.038 (3) | -0.002 (3) | 0.003 (3) | 0.004 (3) |
| C24 | 0.139 (8) | 0.079 (5) | 0.052 (5) | -0.039 (5) | -0.021 (5) | -0.021 (4) |
| C25 | 0.039 (3) | 0.040 (3) | 0.046 (4) | 0.000 (3) | -0.002 (3) | -0.005 (3) |
| C26 | 0.053 (4) | 0.038 (3) | 0.040 (3) | 0.007 (3) | -0.004 (3) | -0.007 (3) |
| C27 | 0.065 (4) | 0.043 (3) | 0.042 (4) | 0.007 (3) | 0.005 (3) | 0.000 (3) |
| C28 | 0.101 (6) | 0.045 (4) | 0.047 (4) | 0.013 (4) | 0.018 (4) | 0.002 (3) |

| | | | | | | |
|-----|-----------|-----------|-----------|------------|------------|-----------|
| C29 | 0.148 (9) | 0.069 (5) | 0.057 (5) | 0.025 (6) | 0.020 (6) | 0.025 (4) |
| C30 | 0.107 (7) | 0.104 (7) | 0.074 (6) | 0.021 (6) | -0.022 (5) | 0.034 (5) |
| C31 | 0.064 (5) | 0.077 (5) | 0.065 (5) | 0.000 (4) | -0.014 (4) | 0.011 (4) |
| C32 | 0.132 (8) | 0.070 (5) | 0.120 (8) | -0.018 (5) | 0.045 (6) | 0.028 (5) |

Geometric parameters (Å, °)

| | | | |
|----------------------|-----------|----------|------------|
| Pb1—O4 | 2.386 (3) | C10—C15 | 1.387 (7) |
| Pb1—O1 | 2.424 (3) | C10—C11 | 1.395 (7) |
| Pb1—O3 | 2.594 (3) | C11—C12 | 1.380 (8) |
| Pb1—O5 | 2.603 (3) | C11—H11A | 0.9300 |
| Pb1—O2 | 2.622 (4) | C12—C13 | 1.381 (11) |
| Pb1—O9 | 2.724 (4) | C12—C16 | 1.505 (10) |
| Pb1—O6 ⁱ | 2.751 (3) | C13—C14 | 1.348 (11) |
| Pb2—O6 | 2.325 (3) | C13—H13A | 0.9300 |
| Pb2—O8 | 2.494 (4) | C14—C15 | 1.384 (9) |
| Pb2—O3 ⁱⁱ | 2.538 (3) | C14—H14A | 0.9300 |
| Pb2—O7 | 2.565 (4) | C15—H15A | 0.9300 |
| Pb2—O10 | 2.665 (3) | C16—H16A | 0.9600 |
| Pb2—O4 | 2.712 (3) | C16—H16B | 0.9600 |
| Pb2—O5 | 2.757 (4) | C16—H16C | 0.9600 |
| O1—C1 | 1.263 (6) | C17—C18 | 1.493 (7) |
| O2—C1 | 1.258 (6) | C18—C19 | 1.376 (7) |
| O3—C9 | 1.251 (5) | C18—C23 | 1.384 (7) |
| O3—Pb2 ⁱ | 2.538 (3) | C19—C20 | 1.377 (8) |
| O4—C9 | 1.261 (5) | C19—H19A | 0.9300 |
| O5—C17 | 1.238 (5) | C20—C21 | 1.380 (9) |
| O6—C17 | 1.273 (5) | C20—C24 | 1.521 (8) |
| O6—Pb1 ⁱⁱ | 2.751 (3) | C21—C22 | 1.381 (9) |
| O7—C25 | 1.261 (6) | C21—H21A | 0.9300 |
| O8—C25 | 1.263 (6) | C22—C23 | 1.373 (8) |
| O9—H9A | 0.8200 | C22—H22A | 0.9300 |
| O9—H9B | 0.8200 | C23—H23A | 0.9300 |
| O10—H10B | 0.8200 | C24—H24A | 0.9600 |
| O10—H10A | 0.8200 | C24—H24B | 0.9600 |
| C1—C2 | 1.487 (7) | C24—H24C | 0.9600 |
| C2—C3 | 1.386 (7) | C25—C26 | 1.488 (8) |
| C2—C7 | 1.392 (7) | C26—C31 | 1.370 (8) |
| C3—C4 | 1.387 (7) | C26—C27 | 1.386 (8) |
| C3—H3A | 0.9300 | C27—C28 | 1.378 (8) |
| C4—C5 | 1.380 (8) | C27—H27A | 0.9300 |
| C4—C8 | 1.513 (8) | C28—C29 | 1.375 (11) |
| C5—C6 | 1.358 (8) | C28—C32 | 1.495 (10) |
| C5—H5A | 0.9300 | C29—C30 | 1.355 (11) |
| C6—C7 | 1.388 (7) | C29—H29A | 0.9300 |
| C6—H6A | 0.9300 | C30—C31 | 1.375 (10) |
| C7—H7A | 0.9300 | C30—H30A | 0.9300 |
| C8—H8A | 0.9600 | C31—H31A | 0.9300 |

| | | | |
|---------------------------|-------------|---------------|-----------|
| C8—H8B | 0.9600 | C32—H32A | 0.9600 |
| C8—H8C | 0.9600 | C32—H32B | 0.9600 |
| C9—C10 | 1.482 (7) | C32—H32C | 0.9600 |
| O4—Pb1—O1 | 82.05 (12) | C4—C8—H8B | 109.5 |
| O4—Pb1—O3 | 51.51 (11) | H8A—C8—H8B | 109.5 |
| O1—Pb1—O3 | 72.68 (12) | C4—C8—H8C | 109.5 |
| O4—Pb1—O5 | 72.72 (11) | H8A—C8—H8C | 109.5 |
| O1—Pb1—O5 | 127.44 (12) | H8B—C8—H8C | 109.5 |
| O3—Pb1—O5 | 118.56 (11) | O3—C9—O4 | 119.7 (5) |
| O4—Pb1—O2 | 75.46 (12) | O3—C9—C10 | 120.4 (4) |
| O1—Pb1—O2 | 51.48 (11) | O4—C9—C10 | 119.9 (4) |
| O3—Pb1—O2 | 107.02 (11) | C15—C10—C11 | 120.2 (5) |
| O5—Pb1—O2 | 77.50 (11) | C15—C10—C9 | 119.5 (5) |
| O4—Pb1—O9 | 81.54 (12) | C11—C10—C9 | 120.2 (5) |
| O1—Pb1—O9 | 143.85 (12) | C12—C11—C10 | 120.5 (6) |
| O3—Pb1—O9 | 71.86 (12) | C12—C11—H11A | 119.8 |
| O5—Pb1—O9 | 77.05 (11) | C10—C11—H11A | 119.8 |
| O2—Pb1—O9 | 149.74 (11) | C11—C12—C13 | 118.1 (7) |
| O4—Pb1—O6 ⁱ | 113.97 (10) | C11—C12—C16 | 120.2 (8) |
| O1—Pb1—O6 ⁱ | 88.41 (11) | C13—C12—C16 | 121.8 (7) |
| O3—Pb1—O6 ⁱ | 63.23 (10) | C14—C13—C12 | 121.9 (7) |
| O5—Pb1—O6 ⁱ | 143.91 (11) | C14—C13—H13A | 119.0 |
| O2—Pb1—O6 ⁱ | 138.35 (10) | C12—C13—H13A | 119.0 |
| O9—Pb1—O6 ⁱ | 69.48 (10) | C13—C14—C15 | 121.0 (7) |
| O6—Pb2—O8 | 83.21 (13) | C13—C14—H14A | 119.5 |
| O6—Pb2—O3 ⁱⁱ | 70.29 (11) | C15—C14—H14A | 119.5 |
| O8—Pb2—O3 ⁱⁱ | 124.28 (12) | C14—C15—C10 | 118.4 (7) |
| O6—Pb2—O7 | 75.42 (12) | C14—C15—H15A | 120.8 |
| O8—Pb2—O7 | 51.46 (12) | C10—C15—H15A | 120.8 |
| O3 ⁱⁱ —Pb2—O7 | 74.37 (12) | C12—C16—H16A | 109.5 |
| O6—Pb2—O10 | 81.57 (11) | C12—C16—H16B | 109.5 |
| O8—Pb2—O10 | 142.70 (12) | H16A—C16—H16B | 109.5 |
| O3 ⁱⁱ —Pb2—O10 | 81.54 (11) | C12—C16—H16C | 109.5 |
| O7—Pb2—O10 | 151.05 (11) | H16A—C16—H16C | 109.5 |
| O6—Pb2—O4 | 115.06 (11) | H16B—C16—H16C | 109.5 |
| O8—Pb2—O4 | 84.44 (12) | O5—C17—O6 | 121.2 (5) |
| O3 ⁱⁱ —Pb2—O4 | 151.11 (11) | O5—C17—C18 | 121.1 (4) |
| O7—Pb2—O4 | 134.31 (11) | O6—C17—C18 | 117.7 (4) |
| O10—Pb2—O4 | 71.75 (10) | C19—C18—C23 | 119.4 (5) |
| O6—Pb2—O5 | 50.10 (10) | C19—C18—C17 | 120.4 (5) |
| O8—Pb2—O5 | 70.96 (12) | C23—C18—C17 | 120.2 (5) |
| O3 ⁱⁱ —Pb2—O5 | 117.25 (10) | C18—C19—C20 | 121.8 (6) |
| O7—Pb2—O5 | 103.86 (11) | C18—C19—H19A | 119.1 |
| O10—Pb2—O5 | 73.13 (11) | C20—C19—H19A | 119.1 |
| O4—Pb2—O5 | 65.60 (10) | C19—C20—C21 | 118.0 (6) |
| C1—O1—Pb1 | 98.1 (3) | C19—C20—C24 | 120.8 (6) |
| C1—O2—Pb1 | 88.9 (3) | C21—C20—C24 | 121.2 (6) |

| | | | |
|--------------------------|-------------|---------------|-----------|
| C9—O3—Pb2 ⁱ | 159.3 (3) | C20—C21—C22 | 121.2 (6) |
| C9—O3—Pb1 | 89.5 (3) | C20—C21—H21A | 119.4 |
| Pb2 ⁱ —O3—Pb1 | 110.94 (12) | C22—C21—H21A | 119.4 |
| C9—O4—Pb1 | 99.2 (3) | C23—C22—C21 | 119.9 (6) |
| C9—O4—Pb2 | 133.5 (3) | C23—C22—H22A | 120.1 |
| Pb1—O4—Pb2 | 114.63 (13) | C21—C22—H22A | 120.1 |
| C17—O5—Pb1 | 169.4 (3) | C22—C23—C18 | 119.8 (6) |
| C17—O5—Pb2 | 84.1 (3) | C22—C23—H23A | 120.1 |
| Pb1—O5—Pb2 | 106.44 (12) | C18—C23—H23A | 120.1 |
| C17—O6—Pb2 | 103.8 (3) | C20—C24—H24A | 109.5 |
| C17—O6—Pb1 ⁱⁱ | 133.6 (3) | C20—C24—H24B | 109.5 |
| Pb2—O6—Pb1 ⁱⁱ | 112.57 (13) | H24A—C24—H24B | 109.5 |
| C25—O7—Pb2 | 91.9 (3) | C20—C24—H24C | 109.5 |
| C25—O8—Pb2 | 95.2 (3) | H24A—C24—H24C | 109.5 |
| Pb1—O9—H9A | 109.2 | H24B—C24—H24C | 109.5 |
| Pb1—O9—H9B | 93.9 | O7—C25—O8 | 121.1 (5) |
| H9A—O9—H9B | 123.4 | O7—C25—C26 | 119.8 (5) |
| Pb2—O10—H10B | 109.3 | O8—C25—C26 | 119.1 (5) |
| Pb2—O10—H10A | 101.0 | C31—C26—C27 | 119.0 (6) |
| H10B—O10—H10A | 103.9 | C31—C26—C25 | 121.1 (6) |
| O2—C1—O1 | 121.4 (5) | C27—C26—C25 | 119.8 (5) |
| O2—C1—C2 | 120.1 (5) | C28—C27—C26 | 121.3 (6) |
| O1—C1—C2 | 118.5 (5) | C28—C27—H27A | 119.4 |
| C3—C2—C7 | 118.7 (5) | C26—C27—H27A | 119.4 |
| C3—C2—C1 | 120.7 (5) | C29—C28—C27 | 117.9 (7) |
| C7—C2—C1 | 120.6 (5) | C29—C28—C32 | 120.4 (7) |
| C2—C3—C4 | 122.1 (5) | C27—C28—C32 | 121.7 (7) |
| C2—C3—H3A | 118.9 | C30—C29—C28 | 121.5 (7) |
| C4—C3—H3A | 118.9 | C30—C29—H29A | 119.2 |
| C5—C4—C3 | 117.5 (5) | C28—C29—H29A | 119.2 |
| C5—C4—C8 | 121.8 (6) | C29—C30—C31 | 120.2 (8) |
| C3—C4—C8 | 120.7 (5) | C29—C30—H30A | 119.9 |
| C6—C5—C4 | 121.7 (6) | C31—C30—H30A | 119.9 |
| C6—C5—H5A | 119.1 | C26—C31—C30 | 120.0 (7) |
| C4—C5—H5A | 119.1 | C26—C31—H31A | 120.0 |
| C5—C6—C7 | 120.7 (5) | C30—C31—H31A | 120.0 |
| C5—C6—H6A | 119.6 | C28—C32—H32A | 109.5 |
| C7—C6—H6A | 119.6 | C28—C32—H32B | 109.5 |
| C6—C7—C2 | 119.3 (5) | H32A—C32—H32B | 109.5 |
| C6—C7—H7A | 120.4 | C28—C32—H32C | 109.5 |
| C2—C7—H7A | 120.4 | H32A—C32—H32C | 109.5 |
| C4—C8—H8A | 109.5 | H32B—C32—H32C | 109.5 |

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

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> |
|-------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| O9—H9A \cdots O8 | 0.82 | 2.03 | 2.805 (5) | 158 |

| | | | | |
|-----------------------------|------|------|-----------|-----|
| O9—H9B···O7 ⁱ | 0.82 | 2.25 | 3.017 (5) | 156 |
| O10—H10B···O2 | 0.82 | 2.12 | 2.881 (5) | 153 |
| O10—H10A···O1 ⁱⁱ | 0.82 | 1.97 | 2.774 (5) | 166 |

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