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Tetramethyl anthracene-2,3,6,7-tetracarboxylate–tetramethyl 9,10-dihydro-9,10-dioxoanthracene-2,3,6,7-tetracarboxylate (1/1)[1]

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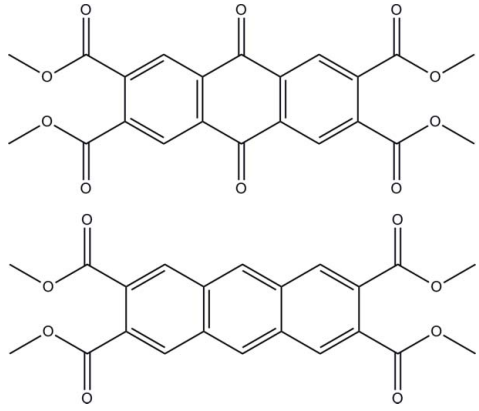
Key indicators: single-crystal X-ray study; T = 90 K, P = 0.0 kPa; mean σ(C–C) = 0.002 Å; disorder in main residue; R factor = 0.052; wR factor = 0.154; data-to-parameter ratio = 21.6.

In the title co-crystal, $C_{22}H_{16}O_{10}\cdot C_{22}H_{18}O_8$, the independent tetramethyl 9,10-dihydro-9,10-dioxoanthracene-2,3,6,7-tetracarboxylate, (I), and tetramethyl anthracene-2,3,6,7-tetracarboxylate, (II), components occupy separate crystallographic inversion centers. In (II), the dihedral angles between the mean aromatic plane and the two independent carboxylate planes are 41.32 (10) and −38.35 (10)°. The methylcarboxylate groups of (I) are disordered, with each resolvable into two groups. In the least disordered carboxylate, the apparent angles between the mean aromatic plane and the two partial carboxylate planes [site occupations = 0.510 (3) and 0.490 (3)] are 16.8 (3) and 23.3 (3)°. In the highly disordered group, the apparent angles between the mean aromatic plane and the two partial carboxylate planes [site occupations = 0.510 (3) and 0.490 (3)] are 78.3 (3) and −74.1 (3)°. In addition, this extreme disorder leads to an artificially elongated C(aromatic)—C(carboxyl) bond.

Related literature

For (I), see: Tarnchompoo *et al.* (1987). For (II), see: Luo & Hart (1988); Morris *et al.* (1994); Yanagimoto *et al.* (2006).



Experimental

Crystal data

$C_{22}H_{16}O_{10}\cdot C_{22}H_{18}O_8$
M_r = 850.71
Triclinic, $P\overline{1}$
a = 8.2110 (3) Å
b = 9.5965 (3) Å
c = 12.0886 (5) Å
α = 86.281 (2)°
β = 81.514 (2)°
γ = 81.705 (2)°
V = 931.34 (6) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 90 K
0.40 × 0.24 × 0.14 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.954, T_{max} = 0.984
12654 measured reflections
7048 independent reflections
4579 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.154
S = 1.03
7048 reflections
326 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The purchase of the diffractometer was made possible by grant No. LEQSF(1999–2000)-ENH-TR-13, administered by the Louisiana Board of Regents.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2569).

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[1] CAS 116896-77-6 and 113431-17-7.

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supporting information

Acta Cryst. (2012). E**68**, o2540–o2541 [https://doi.org/10.1107/S1600536812032424]

Tetramethyl anthracene-2,3,6,7-tetracarboxylate–tetramethyl 9,10-dihydro-9,10-dioxoanthracene-2,3,6,7-tetracarboxylate (1/1)

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S1. Comment

The room temperature crystal structure of anthracene dedrivative (II) was determined by Morris *et al.* (1994). Along with pure crystals of (II), they obtained the co-crystalled product reported here. It is presumed that the anthraquinone derivative ((I)) was formed as an air oxidation product of (II).

The structure exhibits little aromatic ring stacking, with closest contacts C4···C18(at -*x*, -*y*, -*z*) 3.486 (2) and C2···C16(at -*x*, -*y*, -*z*) 3.575 (2) Å.

S2. Experimental

The synthesis of (II) was reported by Morris *et al.* (1994). It is presumed that the anthraquinone derivative ((I)) was formed as an air oxidation product of (II).

S3. Refinement

All H atoms were placed in calculated positions, guided by difference maps, with C—H bond distances 0.95 (aromatic-H) and 0.98 (methyl-H), displacement parameters U_{iso}=1.2U_{eq} (aromatic C) and 1.5U_{eq} (methyl-C), and thereafter refined as riding.

The site occupation factor for disordered atoms O2A and O2B were constrained to be X1 and 1 - X1 respectively, and both atoms were constrained to have the same anisotropic displacement paramters; X1 was refined to a value of 0.510 (3). Likewise, site occupation factor X2 was independently refined to a value of 0.510 (3) for disordered methyl-carboxylate O4A, C10A, O5A, C11A (1 - X2 for O4B, C10B, O5B, C11B).

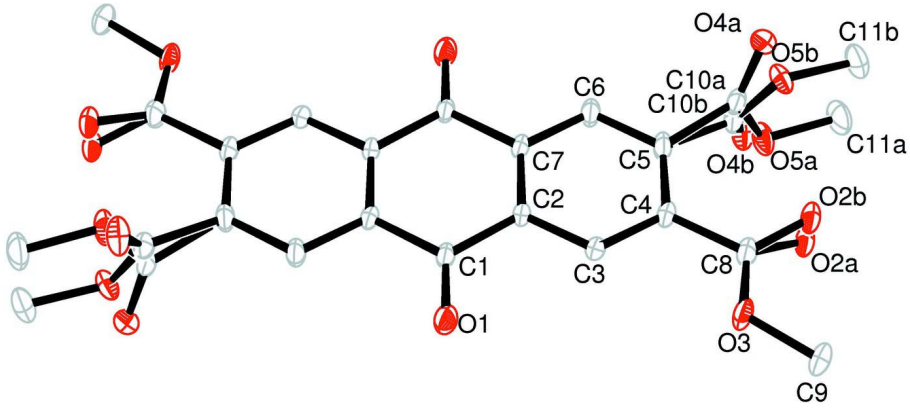

Figure 1

The molecular structure of (I) with the atom numbering scheme showing disorder. Displacement ellipsoids are drawn at the 30% probability level. H atoms were omitted for clarity.

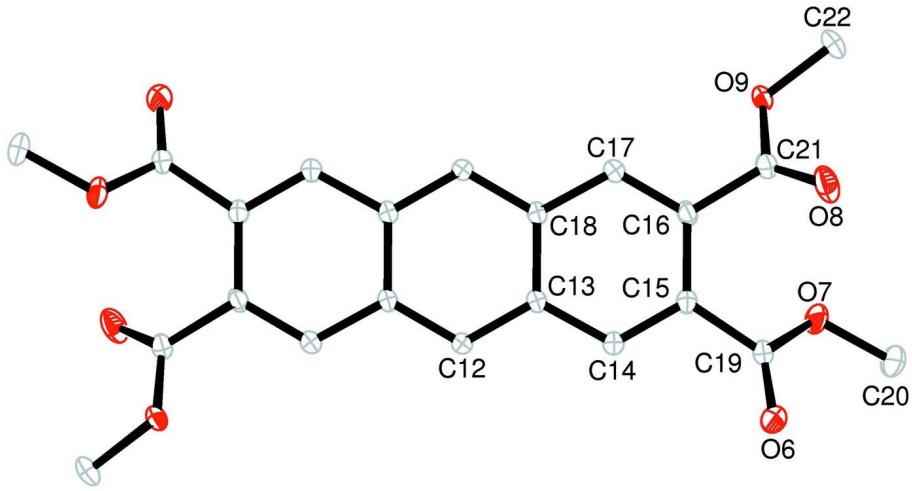

Figure 2

The molecular structure of (I) with the atom numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms were omitted for clarity.

Tetramethyl anthracene-2,3,6,7-tetracarboxylate– tetramethyl 9,10-dihydro-9,10-dioxoanthracene-2,3,6,7-tetracarboxylate (1/1)

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c = 12.0886 (5) Å
α = 86.281 (2)°
β = 81.514 (2)°
γ = 81.705 (2)°
V = 931.34 (6) Å^3
Z = 1
$F(000)$ = 442
D_x = 1.517 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6290 reflections
θ = 2.6–33.1°
μ = 0.12 mm^{-1}
T = 90 K
Plate, yellow
0.40 × 0.24 × 0.14 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: sealed tube
Horizonally mounted graphite crystal monochromator
Detector resolution: 9 pixels mm^{-1}
CCD rotation images, thick slices scans
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.954, T_{max} = 0.984
12654 measured reflections
7048 independent reflections
4579 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 33.1°, θ_{min} = 2.7°
h = −11→12
k = −14→14
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.154
S = 1.03
7048 reflections
326 parameters
0 restraints
3 constraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map	$w = 1/[\sigma^2(F_o^2) + (0.0865P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
Hydrogen site location: inferred from neighbouring sites	$(\Delta/\sigma)_{max} = 0.001$
H-atom parameters constrained	$\Delta\rho_{max} = 0.45$ e Å^{-3}
	$\Delta\rho_{min} = -0.30$ 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.17551 (13)	−0.04598 (11)	0.45522 (9)	0.0160 (2)	
C2	0.03630 (13)	−0.08672 (11)	0.40307 (9)	0.0146 (2)	
C3	0.07556 (13)	−0.17164 (11)	0.31043 (9)	0.0165 (2)	
H3	0.1885	−0.198	0.28	0.02*	
C4	−0.05059 (14)	−0.21760 (12)	0.26265 (10)	0.0185 (2)	
C5	−0.21709 (14)	−0.17679 (13)	0.30674 (11)	0.0231 (3)	
C6	−0.25640 (14)	−0.08911 (13)	0.39726 (11)	0.0228 (3)	
H6	−0.3694	−0.0592	0.4256	0.027*	
C7	−0.12956 (13)	−0.04518 (11)	0.44636 (9)	0.0160 (2)	
C8	−0.00953 (15)	−0.31246 (14)	0.16572 (11)	0.0256 (3)	
O2A	−0.1090 (3)	−0.3944 (3)	0.1466 (2)	0.0274 (4)	0.510 (3)
O2B	−0.1136 (3)	−0.3369 (3)	0.1098 (2)	0.0274 (4)	0.490 (3)
C9	0.20164 (16)	−0.42773 (13)	0.03751 (11)	0.0258 (3)	
H9A	0.1959	−0.5242	0.0682	0.039*	
H9B	0.316	−0.4188	0.0039	0.039*	
H9C	0.1277	−0.4063	−0.0198	0.039*	
C10A	−0.3594 (5)	−0.2057 (4)	0.2470 (3)	0.0179 (7)	0.490 (3)
O4A	−0.4377 (3)	−0.1268 (3)	0.18701 (17)	0.0208 (5)	0.490 (3)
O5A	−0.3849 (4)	−0.3372 (3)	0.2787 (3)	0.0264 (5)	0.490 (3)
C11A	−0.4895 (4)	−0.3982 (3)	0.2144 (3)	0.0336 (8)	0.490 (3)
H11A	−0.6012	−0.3958	0.2568	0.05*	0.490 (3)
H11B	−0.4424	−0.496	0.1996	0.05*	0.490 (3)
H11C	−0.496	−0.344	0.1433	0.05*	0.490 (3)
C10B	−0.3558 (5)	−0.2516 (4)	0.2748 (4)	0.0188 (7)	0.510 (3)
O4B	−0.4236 (3)	−0.3413 (3)	0.3294 (2)	0.0263 (5)	0.510 (3)
O5B	−0.3920 (3)	−0.1937 (3)	0.17661 (18)	0.0219 (5)	0.510 (3)
C11B	−0.4997 (3)	−0.2659 (3)	0.1233 (2)	0.0274 (6)	0.510 (3)
H11D	−0.5511	−0.3327	0.1774	0.041*	0.510 (3)
H11E	−0.4345	−0.317	0.0602	0.041*	0.510 (3)
H11F	−0.5866	−0.197	0.0959	0.041*	0.510 (3)
O1	0.31995 (10)	−0.08674 (9)	0.41989 (8)	0.0246 (2)	
O3	0.15036 (10)	−0.32984 (9)	0.12660 (7)	0.02195 (19)	
C12	−0.16785 (13)	0.04142 (11)	0.04459 (9)	0.0160 (2)	
H12	−0.2806	0.0698	0.0743	0.019*	

C13	−0.12949 (13)	−0.04723 (11)	−0.04583 (9)	0.0151 (2)
C14	−0.25614 (13)	−0.09921 (12)	−0.09405 (10)	0.0178 (2)
H14	−0.3693	−0.0725	−0.0644	0.021*
C15	−0.21832 (13)	−0.18678 (12)	−0.18222 (9)	0.0163 (2)
C16	−0.04783 (13)	−0.22772 (11)	−0.22822 (9)	0.0147 (2)
C17	0.07649 (13)	−0.17920 (11)	−0.18403 (9)	0.0155 (2)
H17	0.1889	−0.2062	−0.2154	0.019*
C18	0.04030 (13)	−0.08847 (11)	−0.09143 (9)	0.0145 (2)
C19	−0.36209 (14)	−0.23079 (12)	−0.22755 (10)	0.0192 (2)
C20	−0.46657 (16)	−0.28536 (17)	−0.38791 (12)	0.0338 (3)
H20A	−0.4857	−0.3776	−0.3528	0.051*
H20B	−0.4317	−0.2946	−0.4685	0.051*
H20C	−0.5695	−0.2192	−0.3756	0.051*
C21	−0.00127 (13)	−0.33641 (12)	−0.31496 (9)	0.0167 (2)
C22	0.19237 (15)	−0.41180 (12)	−0.47134 (10)	0.0221 (2)
H22A	0.2222	−0.5027	−0.4325	0.033*
H22B	0.2911	−0.3838	−0.5185	0.033*
H22C	0.1069	−0.4203	−0.5183	0.033*
O6	−0.48764 (10)	−0.25750 (10)	−0.16986 (8)	0.0285 (2)
O7	−0.33807 (10)	−0.23321 (10)	−0.33907 (7)	0.0266 (2)
O8	−0.06719 (12)	−0.44073 (9)	−0.31555 (8)	0.0276 (2)
O9	0.12862 (9)	−0.30601 (8)	−0.38957 (7)	0.01791 (17)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0173 (5)	0.0168 (5)	0.0150 (5)	−0.0030 (4)	−0.0027 (4)	−0.0062 (4)
C2	0.0171 (5)	0.0146 (5)	0.0131 (5)	−0.0021 (4)	−0.0038 (4)	−0.0047 (4)
C3	0.0179 (5)	0.0170 (5)	0.0150 (5)	0.0007 (4)	−0.0041 (4)	−0.0058 (4)
C4	0.0210 (5)	0.0191 (5)	0.0164 (5)	−0.0013 (4)	−0.0041 (4)	−0.0086 (4)
C5	0.0193 (5)	0.0290 (6)	0.0238 (6)	−0.0032 (4)	−0.0061 (4)	−0.0145 (5)
C6	0.0173 (5)	0.0296 (6)	0.0235 (6)	−0.0025 (4)	−0.0039 (4)	−0.0153 (5)
C7	0.0178 (5)	0.0155 (5)	0.0157 (5)	−0.0017 (4)	−0.0032 (4)	−0.0069 (4)
C8	0.0228 (6)	0.0311 (6)	0.0244 (6)	0.0008 (5)	−0.0061 (5)	−0.0164 (5)
O2A	0.0273 (6)	0.0296 (11)	0.0287 (12)	−0.0019 (9)	−0.0094 (8)	−0.0177 (7)
O2B	0.0273 (6)	0.0296 (11)	0.0287 (12)	−0.0019 (9)	−0.0094 (8)	−0.0177 (7)
C9	0.0321 (6)	0.0267 (6)	0.0190 (6)	−0.0009 (5)	−0.0019 (5)	−0.0130 (5)
C10A	0.0212 (13)	0.0160 (17)	0.018 (2)	−0.0030 (14)	−0.0031 (13)	−0.0074 (12)
O4A	0.0196 (9)	0.0201 (11)	0.0237 (10)	−0.0005 (8)	−0.0080 (7)	−0.0024 (8)
O5A	0.0400 (15)	0.0175 (10)	0.0285 (14)	−0.0119 (9)	−0.0197 (12)	0.0009 (10)
C11A	0.0434 (17)	0.0230 (13)	0.0428 (18)	−0.0121 (11)	−0.0256 (14)	−0.0032 (12)
C10B	0.0191 (12)	0.022 (2)	0.0170 (18)	−0.0014 (15)	−0.0046 (12)	−0.0077 (13)
O4B	0.0326 (11)	0.0298 (11)	0.0209 (12)	−0.0155 (8)	−0.0054 (10)	−0.0043 (10)
O5B	0.0216 (10)	0.0268 (12)	0.0208 (10)	−0.0071 (9)	−0.0112 (7)	−0.0003 (8)
C11B	0.0249 (12)	0.0382 (14)	0.0236 (13)	−0.0110 (10)	−0.0096 (10)	−0.0052 (10)
O1	0.0178 (4)	0.0311 (5)	0.0257 (5)	−0.0031 (3)	−0.0004 (3)	−0.0152 (4)
O3	0.0260 (4)	0.0240 (4)	0.0168 (4)	−0.0049 (3)	0.0000 (3)	−0.0115 (3)
C12	0.0122 (5)	0.0204 (5)	0.0159 (5)	−0.0019 (4)	−0.0008 (4)	−0.0067 (4)

C13	0.0145 (5)	0.0173 (5)	0.0140 (5)	−0.0025 (4)	−0.0014 (4)	−0.0053 (4)
C14	0.0144 (5)	0.0226 (5)	0.0174 (5)	−0.0038 (4)	−0.0014 (4)	−0.0076 (4)
C15	0.0156 (5)	0.0192 (5)	0.0153 (5)	−0.0047 (4)	−0.0016 (4)	−0.0051 (4)
C16	0.0172 (5)	0.0152 (5)	0.0120 (5)	−0.0029 (4)	−0.0009 (4)	−0.0039 (4)
C17	0.0146 (5)	0.0178 (5)	0.0140 (5)	−0.0019 (4)	−0.0008 (4)	−0.0046 (4)
C18	0.0146 (5)	0.0167 (5)	0.0129 (5)	−0.0028 (4)	−0.0016 (4)	−0.0046 (4)
C19	0.0170 (5)	0.0224 (5)	0.0196 (6)	−0.0032 (4)	−0.0029 (4)	−0.0098 (4)
C20	0.0235 (6)	0.0548 (9)	0.0280 (7)	−0.0077 (6)	−0.0098 (5)	−0.0189 (6)
C21	0.0187 (5)	0.0183 (5)	0.0137 (5)	−0.0024 (4)	−0.0030 (4)	−0.0041 (4)
C22	0.0260 (6)	0.0225 (5)	0.0171 (6)	−0.0007 (4)	0.0006 (4)	−0.0104 (4)
O6	0.0196 (4)	0.0419 (5)	0.0265 (5)	−0.0119 (4)	0.0014 (3)	−0.0144 (4)
O7	0.0222 (4)	0.0437 (5)	0.0180 (4)	−0.0107 (4)	−0.0054 (3)	−0.0107 (4)
O8	0.0393 (5)	0.0219 (4)	0.0228 (5)	−0.0140 (4)	0.0054 (4)	−0.0093 (3)
O9	0.0181 (4)	0.0202 (4)	0.0160 (4)	−0.0039 (3)	0.0007 (3)	−0.0089 (3)

Geometric parameters (Å, º)

C1—O1	1.2166 (13)	O5B—C11B	1.442 (3)
C1—C2	1.4934 (14)	C11B—H11D	0.98
C1—C7^{i}	1.4935 (14)	C11B—H11E	0.98
C2—C7	1.3956 (15)	C11B—H11F	0.98
C2—C3	1.3994 (14)	C12—C13	1.3998 (14)
C3—C4	1.3938 (15)	C12—C18^{ii}	1.4021 (14)
C3—H3	0.95	C12—H12	0.95
C4—C5	1.4015 (16)	C13—C14	1.4270 (14)
C4—C8	1.4995 (15)	C13—C18	1.4305 (15)
C5—C6	1.3951 (16)	C14—C15	1.3718 (15)
C5—C10A	1.527 (4)	C14—H14	0.95
C5—C10B	1.537 (4)	C15—C16	1.4364 (15)
C6—C7	1.3987 (15)	C15—C19	1.4957 (15)
C6—H6	0.95	C16—C17	1.3691 (14)
C7—C1^{i}	1.4935 (14)	C16—C21	1.4974 (14)
C8—O2B	1.221 (3)	C17—C18	1.4329 (14)
C8—O2A	1.267 (3)	C17—H17	0.95
C8—O3	1.3187 (14)	C18—C12^{ii}	1.4021 (14)
C9—O3	1.4501 (13)	C19—O6	1.2064 (14)
C9—H9A	0.98	C19—O7	1.3346 (15)
C9—H9B	0.98	C20—O7	1.4446 (13)
C9—H9C	0.98	C20—H20A	0.98
C10A—O4A	1.199 (4)	C20—H20B	0.98
C10A—O5A	1.331 (4)	C20—H20C	0.98
O5A—C11A	1.444 (3)	C21—O8	1.2056 (13)
C11A—H11A	0.98	C21—O9	1.3445 (13)
C11A—H11B	0.98	C22—O9	1.4512 (13)
C11A—H11C	0.98	C22—H22A	0.98
C10B—O4B	1.201 (4)	C22—H22B	0.98
C10B—O5B	1.333 (4)	C22—H22C	0.98

O1—C1—C2	121.43 (9)	C8—O3—C9	115.83 (9)
O1—C1—C7^{i}	121.49 (9)	C13—C12—C18^{ii}	120.19 (10)
C2—C1—C7^{i}	117.08 (9)	C13—C12—H12	119.9
C7—C2—C3	120.05 (9)	C18^{ii}—C12—H12	119.9
C7—C2—C1	121.57 (9)	C12—C13—C14	121.62 (9)
C3—C2—C1	118.37 (9)	C12—C13—C18	119.87 (9)
C4—C3—C2	120.16 (10)	C14—C13—C18	118.51 (9)
C4—C3—H3	119.9	C15—C14—C13	121.55 (10)
C2—C3—H3	119.9	C15—C14—H14	119.2
C3—C4—C5	119.74 (10)	C13—C14—H14	119.2
C3—C4—C8	120.53 (10)	C14—C15—C16	119.90 (9)
C5—C4—C8	119.72 (10)	C14—C15—C19	116.52 (10)
C6—C5—C4	120.10 (10)	C16—C15—C19	123.55 (9)
C6—C5—C10A	117.66 (17)	C17—C16—C15	119.93 (9)
C4—C5—C10A	121.49 (17)	C17—C16—C21	118.53 (9)
C6—C5—C10B	117.84 (17)	C15—C16—C21	121.13 (9)
C4—C5—C10B	120.72 (16)	C16—C17—C18	121.24 (10)
C5—C6—C7	120.05 (10)	C16—C17—H17	119.4
C5—C6—H6	120	C18—C17—H17	119.4
C7—C6—H6	120	C12^{ii}—C18—C13	119.93 (9)
C2—C7—C6	119.86 (10)	C12^{ii}—C18—C17	121.20 (10)
C2—C7—C1^{i}	121.33 (9)	C13—C18—C17	118.86 (9)
C6—C7—C1^{i}	118.81 (9)	O6—C19—O7	123.75 (10)
O2B—C8—O3	121.61 (16)	O6—C19—C15	123.71 (11)
O2A—C8—O3	123.52 (14)	O7—C19—C15	112.51 (10)
O2B—C8—C4	122.73 (15)	O7—C20—H20A	109.5
O2A—C8—C4	121.19 (15)	O7—C20—H20B	109.5
O3—C8—C4	112.55 (9)	H20A—C20—H20B	109.5
O3—C9—H9A	109.5	O7—C20—H20C	109.5
O3—C9—H9B	109.5	H20A—C20—H20C	109.5
H9A—C9—H9B	109.5	H20B—C20—H20C	109.5
O3—C9—H9C	109.5	O8—C21—O9	124.17 (10)
H9A—C9—H9C	109.5	O8—C21—C16	124.51 (10)
H9B—C9—H9C	109.5	O9—C21—C16	111.26 (9)
O4A—C10A—O5A	126.1 (4)	O9—C22—H22A	109.5
O4A—C10A—C5	128.3 (3)	O9—C22—H22B	109.5
O5A—C10A—C5	105.6 (3)	H22A—C22—H22B	109.5
C10A—O5A—C11A	115.2 (3)	O9—C22—H22C	109.5
O4B—C10B—O5B	126.2 (3)	H22A—C22—H22C	109.5
O4B—C10B—C5	126.9 (3)	H22B—C22—H22C	109.5
O5B—C10B—C5	106.9 (3)	C19—O7—C20	115.38 (10)
C10B—O5B—C11B	115.1 (2)	C21—O9—C22	115.34 (8)

O1—C1—C2—C7	177.20 (11)	C10A—C5—C10B—O4B	−161.3 (11)
C7^{i}—C1—C2—C7	−1.92 (18)	C6—C5—C10B—O5B	110.9 (3)
O1—C1—C2—C3	−1.13 (17)	C4—C5—C10B—O5B	−82.3 (3)
C7^{i}—C1—C2—C3	179.75 (10)	C10A—C5—C10B—O5B	16.0 (6)
C7—C2—C3—C4	−1.43 (17)	O4B—C10B—O5B—C11B	−12.7 (5)

C1—C2—C3—C4	176.91 (10)	C5—C10B—O5B—C11B	169.9 (2)
C2—C3—C4—C5	0.90 (18)	O2B—C8—O3—C9	−23.4 (2)
C2—C3—C4—C8	−178.00 (11)	O2A—C8—O3—C9	14.7 (2)
C3—C4—C5—C6	0.8 (2)	C4—C8—O3—C9	176.06 (10)
C8—C4—C5—C6	179.69 (12)	C18^{ii}—C12—C13—C14	178.77 (11)
C3—C4—C5—C10A	170.7 (2)	C18^{ii}—C12—C13—C18	−0.79 (18)
C8—C4—C5—C10A	−10.4 (3)	C12—C13—C14—C15	−179.59 (11)
C3—C4—C5—C10B	−165.8 (2)	C18—C13—C14—C15	−0.04 (17)
C8—C4—C5—C10B	13.2 (3)	C13—C14—C15—C16	−0.19 (18)
C4—C5—C6—C7	−1.9 (2)	C13—C14—C15—C19	−178.28 (10)
C10A—C5—C6—C7	−172.2 (2)	C14—C15—C16—C17	−0.04 (17)
C10B—C5—C6—C7	165.0 (2)	C19—C15—C16—C17	177.91 (11)
C3—C2—C7—C6	0.28 (17)	C14—C15—C16—C21	172.59 (11)
C1—C2—C7—C6	−178.01 (11)	C19—C15—C16—C21	−9.46 (17)
C3—C2—C7—C1^{i}	−179.70 (10)	C15—C16—C17—C18	0.50 (17)
C1—C2—C7—C1^{i}	2.00 (18)	C21—C16—C17—C18	−172.32 (10)
C5—C6—C7—C2	1.40 (19)	C12—C13—C18—C12^{ii}	0.79 (18)
C5—C6—C7—C1^{i}	−178.62 (12)	C14—C13—C18—C12^{ii}	−178.78 (10)
C3—C4—C8—O2B	−167.8 (2)	C12—C13—C18—C17	−179.96 (10)
C5—C4—C8—O2B	13.3 (3)	C14—C13—C18—C17	0.48 (16)
C3—C4—C8—O2A	154.37 (19)	C16—C17—C18—C12^{ii}	178.53 (11)
C5—C4—C8—O2A	−24.5 (2)	C16—C17—C18—C13	−0.72 (17)
C3—C4—C8—O3	−7.51 (18)	C14—C15—C19—O6	−37.99 (17)
C5—C4—C8—O3	173.58 (12)	C16—C15—C19—O6	143.99 (13)
C6—C5—C10A—O4A	71.8 (4)	C14—C15—C19—O7	140.16 (11)
C4—C5—C10A—O4A	−98.4 (4)	C16—C15—C19—O7	−37.85 (16)
C10B—C5—C10A—O4A	167.6 (11)	C17—C16—C21—O8	134.54 (13)
C6—C5—C10A—O5A	−105.8 (3)	C15—C16—C21—O8	−38.19 (17)
C4—C5—C10A—O5A	84.1 (3)	C17—C16—C21—O9	−42.72 (14)
C10B—C5—C10A—O5A	−9.9 (6)	C15—C16—C21—O9	144.55 (11)
O4A—C10A—O5A—C11A	15.1 (6)	O6—C19—O7—C20	−6.45 (18)
C5—C10A—O5A—C11A	−167.3 (3)	C15—C19—O7—C20	175.39 (10)
C6—C5—C10B—O4B	−66.5 (4)	O8—C21—O9—C22	−2.47 (17)
C4—C5—C10B—O4B	100.4 (4)	C16—C21—O9—C22	174.80 (9)

Symmetry codes: (i) −*x*, −*y*, −*z*+1; (ii) −*x*, −*y*, −*z*.