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Binary charge-transfer complexes using pyromellitic acid dianhydride featuring C—H···O hydrogen bonds

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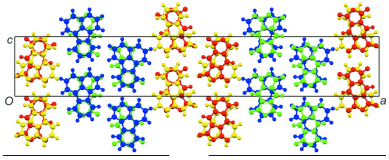
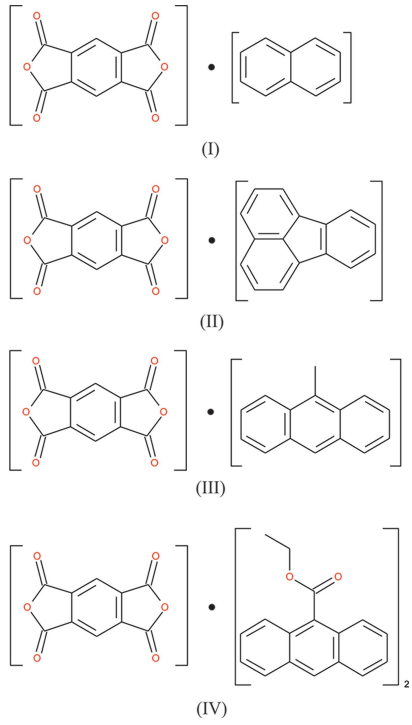
Four binary charge-transfer complexes were made using pyromellitic acid dianhydride (pmda), those being pmda–naphthalene (1/1), $C_{10}H_2O_6{\cdot}C_{10}H_8$, (I), pmda–fluoranthene (1/1), $C_{10}H_2O_6{\cdot}C_{16}H_{10}$, (II), pmda–9-methylanthracene (1/1), $C_{10}H_2O_6{\cdot}C_{15}H_{12}$, (III), and pmda–ethyl anthracene-9-carboxylate (1/2), $C_{10}H_2O_6{\cdot}2C_{17}H_{12}O_3$, (IV). All charge-transfer complexes show alternating donor and acceptor stacks, which have weak C—H···O hydrogen bonds connecting the donor and acceptor molecules. In addition, complex (I) has $Z' = 1/2$, complex (II) has a $Z' = 2$ and complex (IV) has half molecule of pyromellitic acid dianhydride in the asymmetric unit.

1. Chemical context

Crystal engineering, the conception and synthesis of molecular solid state structures, is fundamentally based upon the discernment and subsequent exploitation of intermolecular interactions. Consequently, non-covalent bonding interactions are primarily used to achieve the organization of molecules and ions in the solid state in order to produce materials with desired properties. and this understanding using a variety of intermolecular interactions is at the very heart of crystal engineering. Recently, it has been shown that one can synthesize supramolecular assemblies that contain anywhere from three to six different molecular moieties (Paul *et al.*, 2018). Supramolecular synthesis chiefly uses the hydrogen-bond interaction as the most directional of the known intermolecular interactions (Aakeröy & Beatty, 2001). An equally important interaction is that of charge transfer (CT) between an electron-rich π-system (donor) and an electron-poor π-system (acceptor) (Herbstein, 2005). Classic donor molecules (polycyclic aromatic hydrocarbons) generally have an electron-rich π-system. On the other hand, aromatic hydrocarbons with strongly polarizing groups, such as 1,3,5-trinitrobenzene (TNB), have an electron-poor π-system and are classified as the acceptor molecule (Hill *et al.*, 2018*a*,*b*). Another common acceptor molecule is pyromellitic acid dianhydride (pmda), which has electron-withdrawing O atoms of the carboxylic acid dianhydride groups. (pmda)·(pyrene) complexes have been investigated for order–disorder transitions as a function of temperature using infrared and Raman spectroscopy (Isaac *et al.*, 2018), (pmda)·(naphthalene) has been studied *via* Raman spectroscopy for having orientational disorder (Macfarlane & Ushioda, 1977), disorder in (pmda)·(perylene) *via* computer simulation (Boeyens & Levendis, 1986), and photoconductivity and magentoconductance in pmda··



(pyrene) (Kato *et al.*, 2017). To this end, we have synthesized four new charge-transfer co-crystals that show no disorder: (pmda)·(naphthalene) (I), (pmda)·(fluoranthene) (II), (pmda)·(9-methylanthracene) (III), and (pmda)$_2$·(9-ethyl ester anthracene) (IV).



2. Structural commentary

The asymmetric units and atom-labelling schemes are shown in Fig. 1, together with their displacement ellipsoids, for all

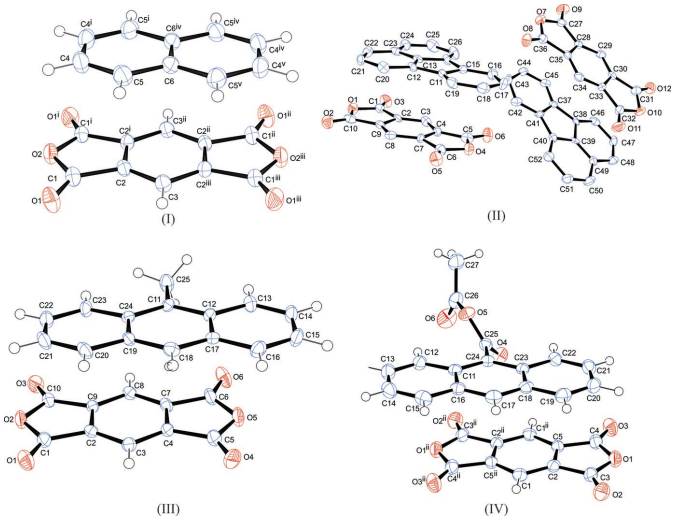


Figure 1
Perspective views of compounds (I)–(IV), showing the atom-numbering schemes. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. [Symmetry codes: (i) $x, 1-y, z$; (ii) $-x, 1-y, 1-z$; (iii) $-x, y, 1-z$; (iv) $-x, 1-y, -z$; (v) $-x, y, -z$; (vi) $x-1, y-1, z-1$.]

Table 1
Centroid distances (Å) between the pmda and the ring centroids (*Cg*) of the aromatic polycyclics.

Structure	Acceptor *Cg*	Donor *Cg*	*Cg*···*Cg*	Symmetry Operator
(I)	C1–O1 (*Cg*3)	C4–C6 (*Cg*6)	3.3724 (2)	$-x+\frac{1}{2}, y-\frac{1}{2}, -z$
(II)	O1–C10 (*Cg*5)	C11–C19 (*Cg*14)	3.3193 (5)	x, y, z
(III)	C2–C9 (*Cg*3)	C11–C24 (*Cg*10)	3.2994 (4)	$x-1, y, z$
(IV)	C1–O1 (*Cg*9)	C11–C24 (*Cg*3)	3.3280 (3)	$1-x, -y, 1-z$

charge-transfer complexes. As a result of the strong polarizing effect of the carboxylic acid dianhydride groups, pmda has an electron-poor π-system and functions as an acceptor. On the other side, the donor molecules comprising polycyclic aromatic hydrocarbons have an electron-rich π-system. The packing of the molecules of the four complexes follows a donor (D) acceptor (A) π–π interaction, which is the major driving force in the formation of these complexes, as seen in Figs. 2 and 3 (donor molecules shown in blue/yellow and acceptor in green/red), resulting in a general face-to-face π-stacking, with Table 1 summarizing the closest centroid–centroid distances between the pmda acceptor and aromatic donor systems. The intermolecular interactions of the $D\cdots A$ stacks can be quantified using Hirshfeld surface analysis as well as the resulting fingerprint plots using the programme

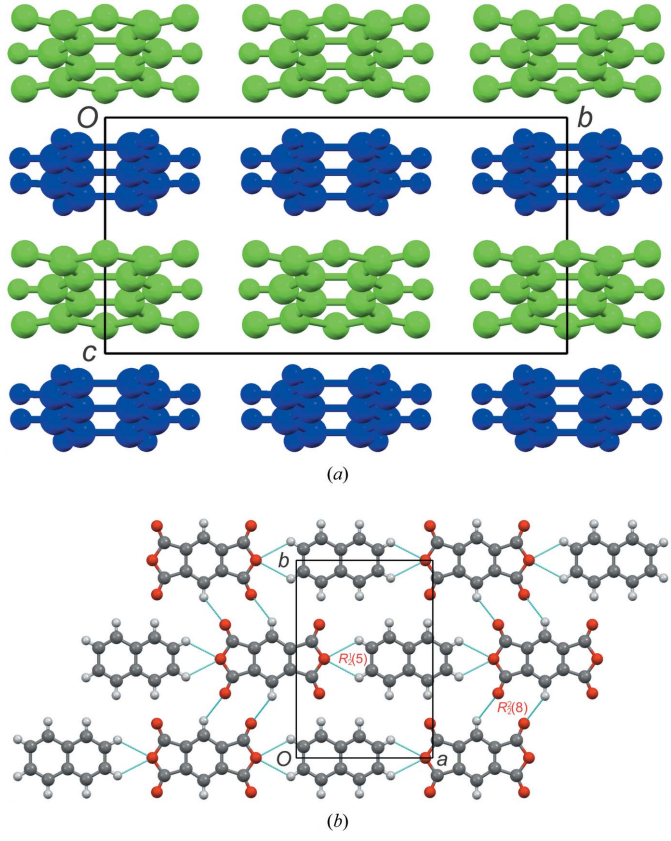


Figure 2
(*a*) A packing diagram of (I) showing the layers of donor (blue) and acceptor (green) molecules. (*b*) Hydrogen-bonding diagram for (I) showing the C—H···O hydrogen-bonded rings formed between the pmda and naphthalene molecules.

Table 2
Proportion (%) of intermolecular contacts between donor and acceptor (pmda) molecules in the Hirshfeld fingerprint plots.

Structure	C···C	H···H	C···H	O···O	O···H	C···O
(pmda)	0.2	8.0	1.0	29.9	17.9	43.0
(I)	19.8	6.6	3.9	9.5	58.4	1.7
(II*A*)	21.0	8.6	5.4	5.5	52.8	6.6
(II*B*)	20.6	11.7	6.2	7.1	48.5	5.9
(III)	20.2	9.5	4.1	4.2	56.8	5.2
(IV)	20.9	10.8	2.7	4.4	53.9	7.3

CrystalExplorer 17.5 (Spackman & McKinnon, 2002). Table 2 summarizes the percentages for all combinations of contacts between C, H and O atoms and the relevant fingerprint plots are given in the supporting information. In the paper by Chen *et al.* (2017), the authors describe that regions of blue and red triangles on the Hirshfeld surface using the shape index as evidence of π–π interactions. Fig. 4 shows such surfaces plotted for the pmda molecules in (I)–(IV), and for comparison the shape index of the pmda molecule in its unimolecular crystal structure. The red triangles show concave regions indicative of ring carbons of the π stacked molecule above it. Complexes (I)–(IV) display a high number of triangles, which reveals the increased proportion of π–π stacking observed for the four structures.. The shape index of pmda shows no such pattern [Fig. 4(*a*)]. This π stacking can be quantified by looking at the contribution of the C···C contacts contained in the fingerprint plots, which vary only slightly from 19.9 to 21.0%. The greatest contribution to the Hirshfeld surfaces are seen in the H···O contacts, which vary from 48.5 to 58.4%. In comparison, the C···C contacts only make up 0.2% in pmda···pmda and the C···O contacts have the greatest single contribution at 43%. In summary, the introduction of an aromatic polycylic changes the biggest contributor from C···O in pmda to H···O in pmda-aromatic polycyclics.

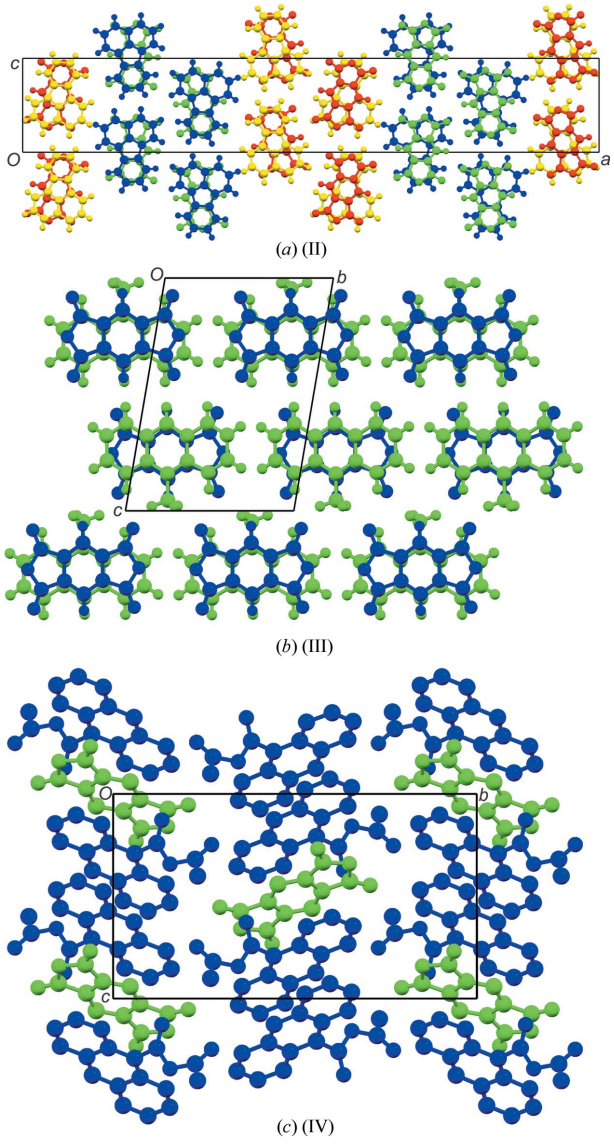


Figure 3
Packing diagrams for (II)–(IV). The donor molecules are shown in blue or yellow, and the acceptor molecules in green or red.

Table 3
Hydrogen-bond geometry (Å, °) for (I).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O2^{i}	0.95	2.68	3.2748 (14)	121
C3—H3···O1^{ii}	0.95	2.63	3.3463 (13)	133
C5—H5···O1^{ii}	0.95	2.69	3.4127 (14)	133

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

3. Supramolecular features

Compound (I) crystallizes in the *C*2/m space group with one quarter of the pmda and naphthalene molecules occupying a twofold axis and a mirror plane, resulting in $Z' = 0.25$ for the asymmetric unit. The donor and acceptor molecules stack along the *c*-axis direction, and in a checker board fashion along the *ab* plane [Fig. 2(*a*)]. In the direction of the *a*-axis, there is a symmetrical C4—H4···O2 interaction from both ends of the naphthalene molecule to the oxygen atoms on the pmda [Fig. 2(*b*), Table 3]. As a result of the mirror plane symmetry, this results in a very symmetrical $R_2^1(5)$ ring as

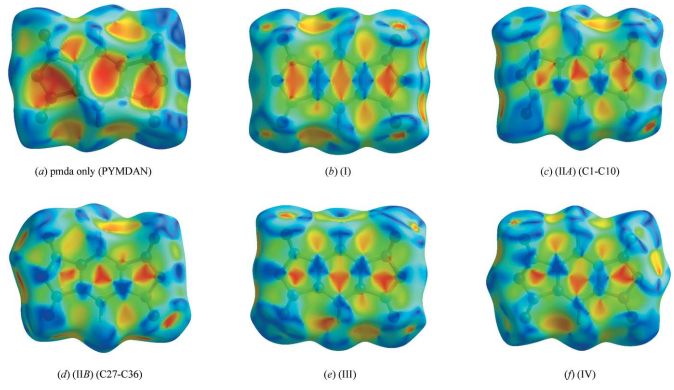


Figure 4
The molecular Hirshfeld surfaces mapped over shape index for the pmda molecule by itself (PYMDAN) and for the pmda acceptor molecule in charge transfer complexes (I)–(IV).

Table 4
Hydrogen-bond geometry (Å, °) for (II).

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···O2^{i}	0.95	2.67	3.373 (5)	132
C16—H16···O8	0.95	2.59	3.444 (5)	150
C17—H17···O3^{ii}	0.95	2.65	3.576 (5)	166
C18—H18···O1^{ii}	0.95	2.67	3.332 (5)	127
C22—H22···O4^{iii}	0.95	2.59	3.481 (5)	155
C25—H25···O11^{iii}	0.95	2.55	3.347 (5)	142
C29—H29···O12^{iv}	0.95	2.71	3.370 (5)	127
C42—H42···O6	0.95	2.49	3.413 (5)	165
C43—H43···O11^{iii}	0.95	2.58	3.293 (5)	132
C44—H44···O10^{iii}	0.95	2.52	3.428 (5)	160
C45—H45···O12^{iv}	0.95	2.57	3.429 (5)	150
C46—H46···O9^{v}	0.95	2.64	3.256 (5)	123
C48—H48···O9^{vi}	0.95	2.55	3.473 (5)	164
C50—H50···O7^{vi}	0.95	2.5	3.420 (5)	164
C52—H52···O6	0.95	2.62	3.525 (5)	159

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

Table 5
Hydrogen-bond geometry (Å, °) for (III).

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O1^{i}	0.95	2.55	3.376 (4)	145
C14—H14···O2^{ii}	0.95	2.63	3.347 (4)	133
C16—H16···O4^{iii}	0.95	2.68	3.365 (4)	130
C22—H22···O5^{iv}	0.95	2.64	3.323 (4)	130

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

Table 6
Hydrogen-bond geometry (Å, °) for (IV).

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12···O2^{i}	0.95	2.65	3.351 (2)	131
C15—H15···O3^{ii}	0.95	2.55	3.306 (2)	137
C21—H21···O4^{iii}	0.95	2.48	3.433 (2)	176

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

described using graph-set notation (Bernstein *et al.*, 1995). Along the *b*-axis, there is an additional hydrogen bonded ring, $R_2^2(8)$, resulting from C3—H3···O1 hydrogen-bond interaction [Fig. 2(*b*)].

Compound (II) crystallizes in the *P*ca2$_1$ space group with two pmda and two fluoranthene molecules in the asymmetric unit. One set of *D*/*A* pairs is shown in blue/green, and the second is shown in yellow/red. The separation of the two *D*/*A* pairs can be clearly seen in Fig. 3(*a*). Between the four unique pmda acceptor and fluoranthene donors there are numerous C—H···O interactions (Table 4). As the fluoranthene has only C and H atoms, it is the molecule that has the most weak hydrogen-bond donor groups (C—H), and the pmda, with six oxygen atoms, has numerous good hydrogen-bond acceptor atoms (O). Fig. 5(*a*) and 5(*b*) illustrate four of the hydrogen bonds emanating from the two symmetry-independent fluoranthene molecules, which form a number of hydrogen-bonded rings: $R_2^1(7)$, $R_2^2(7)$, $R_2^2(8)$ and $R_3^3(12)$.

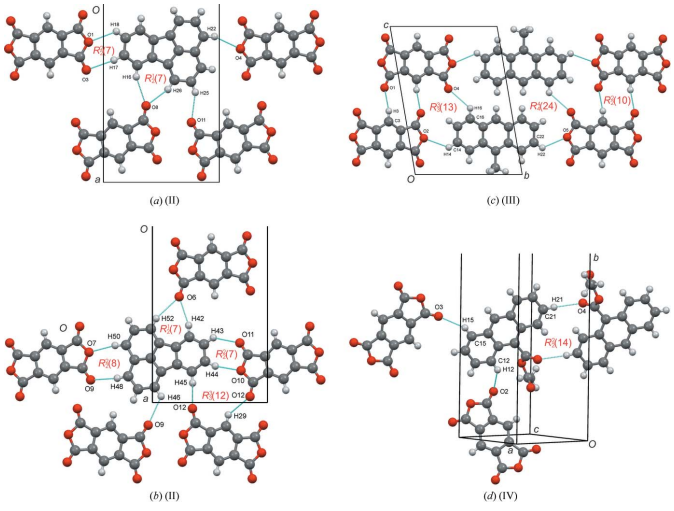


Figure 5
Hydrogen-bonding diagrams for (II)–(IV). Atom labels correspond to those given in the hydrogen-bonding tables.

Compound (III) crystallizes in the $P\overline{1}$ space group with both the pmda and 9-methylanthracene in the asymmetric unit. The packing of the structure shows the typical donor–acceptor stacking along the *a* axis [Fig. 3(*b*)] and has the closest centroid-to-centroid distance of all four charge-transfer complexes at 3.2994 (4) Å (Table 1). Perpendicular to the stacking axis, the donor and acceptor molecules form hydrogen-bonded layers using four distinct C—H···O hydrogen bonds (Table 5). The combination of these individually or in groups results in three types of hydrogen bonded rings, $R_2^2(10)$, $R_3^3(13)$ and $R_4^4(24)$, shown in Fig. 5(*c*).

Compound (IV) crystallizes in the *P*2$_1$/*c* space group with half a pmda (on a centre of inversion) and one complete 9-ethyl ester anthracene molecule in the asymmetric unit, giving a ratio of one acceptor to two donors. [Fig. 3(*c*)]. Two donor molecules form a hydrogen-bonded ring dimer [Fig. 5(*d*)], graph-set $R_2^2(14)$, *via* a C21—H21···O4 hydrogen bond Two pmda molecules are connected to the donor *via* discrete hydrogen bonds C12—H12···O2 and C15—H15···O3 (Table 6).

One of the major differences between the four complexes is the symmetry of the asymmetric unit. Pmda, being a very symmetrical molecule with point group D_{2h}, is shown to crystallize with Z' = 0.25, 0.5 and 1 in the title complexes. In the literature, the most common case is with Z' = 0.5, such as those with anthracene (ANTPML; Boeyens & Herbstein, 1965; ANTPML01 and ANTPML01; Robertson & Stezowski, 1978), acridine (BIWVUY; Karl *et al.*, 1982*b*), biphenylene (DURZAR, DURZAR01, DURZAR02; Stezowski *et al.*, 1986), chrysene (FILHIR; Bulgarovskaya *et al.*, 1987*b*) to name but a few. More unusual is the case with Z' = 0.25, seen only twice in 9,10-dibromoanthracene (FILHEN; Bulgarovskaya *et al.*, 1987*a*) and naphthalene (NAPYMA01; Le Bars-Combe *et al.*, 1979). It has also been observed were pmda is present with both Z' = 0.5 and 1, such as in RUYWIR (Kurebayashi *et al.*, 2001), 3,6-dibromocarbazole (VILFIF; Bulgarovskaya *et al.*, 1989) and *N*-methyl-3,6-dibromocarbazole (WEXKEP; Dzyabchenko *et al.*, 1994). In summary,

Table 7
Experimental details.

	(I)	(II)	(III)	(IV)
Crystal data				
Chemical formula	$C_{10}H_2O_6{\cdot}C_{10}H_8$	$C_{10}H_2O_6{\cdot}C_{16}H_{10}$	$C_{10}H_2O_6{\cdot}C_{15}H_{12}$	$C_{17}H_{12}O_3{\cdot}0.5C_{10}H_2O_6$
M_r	346.28	420.36	410.36	373.32
Crystal system, space group	Monoclinic, $C2/m$	Orthorhombic, $Pca2_1$	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/c$
Temperature (K)	173	173	173	173
a, b, c (Å)	9.1478 (4), 12.8195 (6), 6.7459 (3)	57.356 (9), 7.0172 (10), 9.3429 (13)	7.1012 (8), 9.5674 (12), 13.6147 (16)	9.1949 (7), 17.9751 (14), 10.9716 (10)
α, β, γ (°)	90, 104.202 (3), 90	90, 90, 90	99.109 (4), 99.941 (4), 92.219 (4)	90, 112.829 (2), 90
V (Å^3)	766.91 (6)	3760.3 (9)	897.53 (19)	1671.3 (2)
Z	2	8	2	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.11	0.11	0.11	0.11
Crystal size (mm)	0.40 × 0.08 × 0.05	0.5 × 0.1 × 0.1	0.19 × 0.06 × 0.05	0.55 × 0.1 × 0.06
Data collection				
Diffractometer	Bruker D8 Venture Photon CCD area detector	Bruker D8 Venture Photon CCD area detector	Bruker D8 Venture Photon CCD area detector	Bruker D8 Venture Photon CCD area detector
Absorption correction	Multi-scan *SADABS* (Krause *et al.*, 2015)	Multi-scan *SADABS* (Krause *et al.*, 2015)	Multi-scan *SADABS* (Krause *et al.*, 2015)	Multi-scan *SADABS* (Krause *et al.*, 2015)
T_{min}, T_{max}	0.9, 0.95	0.9, 0.95	0.9, 0.95	0.9, 0.95
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	3774, 967, 841	40403, 6983, 5636	20202, 3280, 2159	13071, 4035, 2731
R_{int}	0.042	0.054	0.075	0.046
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.034, 0.098, 1.04	0.045, 0.106, 1.08	0.073, 0.223, 1.02	0.043, 0.112, 1.05
No. of reflections	967	6983	3280	4035
No. of parameters	63	577	281	254
No. of restraints	0	1	0	0
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.36, −0.3	0.21, −0.17	0.67, −0.28	0.30, −0.26

Computer programs: *APEX3*, *SAINT-Plus* and *XPREP* (Bruker 2016), *SHELXS97* (Sheldrick, 2008), *SHELXL2017/1* (Sheldrick, 2015), *ORTEP-3 for Windows* and *WinGX* publication routines (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2006).

we have characterized a further new set of four CT complexes of pmda and aromatic molecules.

4. Database survey

A database survey in the Cambridge Structural Database (CSD, Version 5.39; November 2017 update; Groom *et al.*, 2016) was undertaken for any structures containing the pmda moiety. A total of 26 complexes were found, four showing polymorphism [BECNUS02 (Karl *et al.*, 1982*a*) and BECNUS10 (Bugarovskaya *et al.*, 1982); DURZAR and DURZAR01 (Stezowski *et al.*, 1986); NAPYMA01 (Le Bars-Combe *et al.*, 1979) and NAPYMA12 (Le Bars-Combe *et al.*, 1981); PYRPMA04 (Herbstein *et al.*, 1994) and PYRPMA11 (Kato *et al.*, 2017)] and one showing stoichiometric variation [VILFEB and VILFIF (Bulgarovskaya *et al.*, 1989)].

5. Synthesis and crystallization

All chemicals were purchased from commercial sources (Sigma Aldrich) and used as received without further purification. The pyromellitic acid dianhydride charge transfer complexes were prepared in a 10 mL ethanolic solution with a 1:1 stoichiometric ratio of the donor to the acceptor molecule which was then heated and stirred until total dissolution took place (approx. 4 h). The solution was then cooled very slowly and allowed to evaporate to obtain crystals suitable for X-ray diffraction. Detailed masses are as follows: (I): 0.100 g of pyromellitic acid dianhydride and 0.059 g of naphthalene; (II): 0.100 g of pyromellitic acid dianhydride and 0.093 g of fluoranthene; (III): 0.100 g of pyromellitic acid dianhydride and 0.088 g of 9-methylanthracene; and (IV): 0.100 g of pyromellitic acid dianhydride and 0.12 1 g of 9-ethyl ester anthracene.

6. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 7. For all compounds, the C-bound H atoms were geometrically placed (C—H bond lengths of 0.96 (methyl CH_3), and 0.95 (Ar—H) Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}$(Ar-C) or $U_{iso}(H) = 1.5U_{eq}$(methyl-C).

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Binary charge-transfer complexes using pyromellitic acid dianhydride featuring C—H···O hydrogen bonds

Tania N. Hill and Andreas Lemmerer

Computing details

For all structures, data collection: *APEX3* (Bruker, 2016); cell refinement: *SAINT-Plus* (Bruker, 2016); data reduction: *SAINT-Plus* and *XPREP* (Bruker 2016); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2017*/1 (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 2012).

Pyromellitic acid dianhydride–naphthalene (1/1) (I)

Crystal data

$C_{10}H_2O_6{\cdot}C_{10}H_8$
M_r = 346.28
Monoclinic, *C*2/*m*
Hall symbol: -C 2y
a = 9.1478 (4) Å
b = 12.8195 (6) Å
c = 6.7459 (3) Å
β = 104.202 (3)°
V = 766.91 (6) Å^3
Z = 2
F(000) = 356
D_x = 1.5 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 2019 reflections
θ = 2.8–28.2°
μ = 0.11 mm^{-1}
T = 173 K
Plate, yellow
0.40 × 0.08 × 0.05 mm

Data collection

Bruker D8 Venture Photon CCD area detector diffractometer
Graphite monochromator
ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.9, T_{max} = 0.95
3774 measured reflections
967 independent reflections
841 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 28.0°, θ_{min} = 2.8°
h = −11→12
k = −16→16
l = −8→8

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.098
S = 1.04
967 reflections
63 parameters
0 restraints
0 constraints
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0541P)^2 + 0.2771P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.3 e Å^{-3}

Extinction correction: SHELXL-2017/1 (Sheldrick 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.011 (3)

Special details

Experimental. Absorption corrections were made using the program SADABS (Sheldrick, 1996)

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.29033 (11)	0.41071 (8)	0.62113 (16)	0.0267 (3)
C2	0.13074 (10)	0.44565 (8)	0.55335 (13)	0.0221 (3)
C3	0	0.38652 (11)	0.5	0.0238 (3)
H3	0	0.312417	0.5	0.029*
O1	0.34466 (8)	0.32627 (6)	0.65041 (12)	0.0379 (3)
O2	0.37963 (11)	0.5	0.65364 (16)	0.0309 (3)
C4	0.27282 (12)	0.44493 (10)	0.10439 (16)	0.0358 (3)
H4	0.365478	0.407881	0.140233	0.043*
C5	0.13998 (12)	0.39113 (10)	0.05322 (16)	0.0324 (3)
H5	0.141317	0.317033	0.053253	0.039*
C6	0	0.44465 (12)	0	0.0262 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0203 (5)	0.0307 (6)	0.0273 (5)	0.0019 (4)	0.0023 (4)	−0.0027 (4)
C2	0.0187 (5)	0.0248 (5)	0.0219 (5)	0.0025 (3)	0.0032 (3)	0.0002 (3)
C3	0.0225 (6)	0.0209 (6)	0.0263 (7)	0	0.0030 (5)	0
O1	0.0283 (4)	0.0329 (5)	0.0476 (5)	0.0107 (3)	0.0000 (3)	−0.0026 (3)
O2	0.0181 (5)	0.0337 (6)	0.0382 (6)	0	0.0017 (4)	0
C4	0.0221 (5)	0.0545 (7)	0.0289 (6)	0.0073 (5)	0.0029 (4)	0.0021 (5)
C5	0.0294 (6)	0.0377 (6)	0.0295 (5)	0.0060 (4)	0.0059 (4)	0.0020 (4)
C6	0.0224 (6)	0.0353 (8)	0.0204 (6)	0	0.0042 (5)	0

Geometric parameters (Å, º)

C1—O1	1.1872 (12)	C4—C5	1.3659 (16)
C1—O2	1.3921 (12)	C4—C4^{i}	1.412 (3)
C1—C2	1.4878 (12)	C4—H4	0.9497
C2—C3	1.3868 (12)	C5—C6	1.4190 (13)
C2—C2^{i}	1.394 (2)	C5—H5	0.95
C3—H3	0.9499	C6—C6^{ii}	1.419 (3)
O1—C1—O2	121.20 (9)	C5—C4—C4^{i}	120.33 (7)

O1—C1—C2	131.66 (10)	C5—C4—H4	119.7
O2—C1—C2	107.13 (8)	C4^{i}—C4—H4	120
C3—C2—C2^{i}	123.13 (6)	C4—C5—C6	120.76 (12)
C3—C2—C1	129.34 (9)	C4—C5—H5	119.6
C2^{i}—C2—C1	107.52 (6)	C6—C5—H5	119.6
C2—C3—C2^{iii}	113.73 (12)	C5—C6—C5^{iv}	122.18 (15)
C2—C3—H3	123.1	C5—C6—C6^{ii}	118.91 (7)
C2^{iii}—C3—H3	123.1	C5^{iv}—C6—C6^{ii}	118.91 (7)
C1^{i}—O2—C1	110.63 (11)		
O1—C1—C2—C3	−1.92 (18)	O1—C1—O2—C1^{i}	−176.38 (6)
O2—C1—C2—C3	179.21 (8)	C2—C1—O2—C1^{i}	2.63 (15)
O1—C1—C2—C2^{i}	177.30 (11)	C4^{i}—C4—C5—C6	−0.22 (11)
O2—C1—C2—C2^{i}	−1.57 (9)	C4—C5—C6—C5^{iv}	−179.78 (11)
C2^{i}—C2—C3—C2^{iii}	0	C4—C5—C6—C6^{ii}	0.22 (11)
C1—C2—C3—C2^{iii}	179.11 (11)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O2^{v}	0.95	2.68	3.2748 (14)	121
C3—H3···O1^{vi}	0.95	2.63	3.3463 (13)	133
C5—H5···O1^{vi}	0.95	2.69	3.4127 (14)	133

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

Pyromellitic acid dianhydride–fluoranthene (1/1) (II)

Crystal data

$C_{10}H_2O_6{\cdot}C_{16}H_{10}$
M_r = 420.36
Orthorhombic, $Pca2_1$
Hall symbol: P 2c -2ac
a = 57.356 (9) Å
b = 7.0172 (10) Å
c = 9.3429 (13) Å
V = 3760.3 (9) Å^3
Z = 8
$F(000)$ = 1728
D_x = 1.485 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9936 reflections
θ = 2.9–25.0°
μ = 0.11 mm^{-1}
T = 173 K
Needle, yellow
0.5 × 0.1 × 0.1 mm

Data collection

Bruker D8 Venture Photon CCD area detector diffractometer
Graphite monochromator
ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.9, T_{max} = 0.95
40403 measured reflections
6983 independent reflections
5636 reflections with $I > 2\sigma(I)$
R_{int} = 0.054
θ_{max} = 25.5°, θ_{min} = 2.9°
h = −68→69
k = −8→8
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.106$
$S = 1.08$
6983 reflections
577 parameters
1 restraint
0 constraints

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0572P)^2 + 0.1599P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}

Special details

Experimental. Absorption corrections were made using the program SADABS (Sheldrick, 1996)
Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.82357 (7)	1.1250 (5)	0.7955 (4)	0.0292 (9)
C2	0.81768 (6)	1.0608 (5)	0.6489 (4)	0.0222 (9)
C3	0.83230 (7)	1.0187 (5)	0.5355 (4)	0.0247 (9)
H3	0.848791	1.028283	0.541393	0.03*
C4	0.82087 (6)	0.9617 (5)	0.4134 (4)	0.0223 (8)
C5	0.83073 (7)	0.9076 (5)	0.2727 (4)	0.0274 (9)
C6	0.79114 (7)	0.8856 (5)	0.2571 (4)	0.0265 (9)
C7	0.79692 (6)	0.9479 (4)	0.4036 (4)	0.0211 (9)
C8	0.78227 (6)	0.9916 (5)	0.5173 (4)	0.0239 (8)
H8	0.765778	0.98281	0.510836	0.029*
C9	0.79363 (6)	1.0491 (5)	0.6408 (4)	0.0237 (9)
C10	0.78389 (7)	1.1076 (5)	0.7808 (4)	0.0263 (9)
O1	0.80273 (5)	1.1492 (3)	0.8698 (3)	0.0313 (6)
O2	0.76441 (5)	1.1242 (4)	0.8216 (3)	0.0367 (7)
O3	0.84189 (5)	1.1546 (4)	0.8516 (3)	0.0398 (7)
O4	0.81209 (4)	0.8637 (3)	0.1825 (3)	0.0296 (6)
O5	0.77309 (5)	0.8529 (4)	0.1999 (3)	0.0362 (7)
O6	0.85035 (5)	0.8965 (4)	0.2327 (3)	0.0373 (7)
C11	0.79962 (7)	0.4589 (4)	0.4321 (4)	0.0220 (9)
C12	0.79336 (6)	0.5209 (5)	0.5780 (4)	0.0216 (8)
C13	0.81464 (6)	0.5575 (5)	0.6486 (4)	0.0209 (8)
C14	0.83381 (6)	0.5247 (5)	0.5586 (4)	0.0247 (9)
C15	0.82429 (6)	0.4634 (5)	0.4204 (4)	0.0235 (9)
C16	0.83500 (7)	0.4131 (5)	0.2922 (4)	0.0292 (9)
H16	0.851483	0.416417	0.282892	0.035*
C17	0.82101 (7)	0.3579 (5)	0.1783 (4)	0.0324 (10)
H17	0.828098	0.323881	0.09003	0.039*
C18	0.79686 (7)	0.3513 (5)	0.1905 (4)	0.0309 (10)
H18	0.787731	0.311898	0.110994	0.037*

C19	0.78599 (7)	0.4014 (5)	0.3169 (4)	0.0271 (9)
H19	0.769492	0.396593	0.325054	0.032*
C20	0.77293 (6)	0.5517 (5)	0.6511 (4)	0.0270 (9)
H20	0.758286	0.52863	0.606837	0.032*
C21	0.77417 (7)	0.6187 (5)	0.7942 (4)	0.0281 (9)
H21	0.760053	0.640805	0.844686	0.034*
C22	0.79487 (6)	0.6527 (5)	0.8623 (4)	0.0266 (9)
H22	0.794862	0.696499	0.958518	0.032*
C23	0.81630 (6)	0.6229 (5)	0.7904 (4)	0.0252 (9)
C24	0.83898 (7)	0.6531 (5)	0.8429 (5)	0.0316 (9)
H24	0.841192	0.695894	0.938428	0.038*
C25	0.85813 (7)	0.6204 (5)	0.7558 (5)	0.0357 (10)
H25	0.873296	0.643029	0.792923	0.043*
C26	0.85582 (7)	0.5545 (5)	0.6131 (5)	0.0331 (10)
H26	0.869228	0.531161	0.55609	0.04*
C27	0.95132 (7)	0.2701 (5)	0.3916 (4)	0.0273 (9)
C28	0.94915 (6)	0.3286 (4)	0.2407 (4)	0.0211 (8)
C29	0.96640 (6)	0.3586 (5)	0.1395 (4)	0.0240 (9)
H29	0.982522	0.342393	0.159271	0.029*
C30	0.95802 (6)	0.4145 (4)	0.0070 (4)	0.0214 (8)
C31	0.97109 (7)	0.4574 (5)	−0.1257 (5)	0.0284 (9)
C32	0.93229 (7)	0.5019 (5)	−0.1718 (4)	0.0268 (9)
C33	0.93452 (7)	0.4410 (5)	−0.0213 (4)	0.0236 (9)
C34	0.91744 (6)	0.4095 (5)	0.0794 (4)	0.0247 (9)
H34	0.901313	0.425949	0.059588	0.03*
C35	0.92565 (6)	0.3521 (5)	0.2117 (4)	0.0239 (9)
C36	0.91269 (7)	0.3065 (5)	0.3450 (5)	0.0299 (9)
O7	0.92884 (4)	0.2590 (3)	0.4485 (3)	0.0315 (7)
O8	0.89225 (5)	0.3043 (4)	0.3676 (3)	0.0396 (7)
O9	0.96799 (5)	0.2355 (4)	0.4632 (3)	0.0396 (7)
O10	0.95479 (5)	0.5088 (3)	−0.2297 (3)	0.0318 (7)
O11	0.91570 (5)	0.5429 (4)	−0.2403 (3)	0.0382 (7)
O12	0.99143 (5)	0.4552 (4)	−0.1519 (3)	0.0370 (7)
C37	0.94594 (6)	0.8366 (4)	0.2153 (4)	0.0224 (8)
C38	0.95615 (6)	0.8863 (4)	0.0751 (4)	0.0212 (8)
C39	0.93732 (6)	0.9377 (5)	−0.0149 (4)	0.0224 (9)
C40	0.91574 (6)	0.9237 (5)	0.0587 (4)	0.0215 (8)
C41	0.92138 (6)	0.8593 (5)	0.2046 (4)	0.0218 (8)
C42	0.90731 (7)	0.8204 (5)	0.3214 (4)	0.0277 (9)
H42	0.890861	0.833772	0.315028	0.033*
C43	0.91770 (7)	0.7615 (5)	0.4480 (4)	0.0309 (10)
H43	0.908251	0.734899	0.52909	0.037*
C44	0.94165 (7)	0.7410 (5)	0.4578 (4)	0.0302 (10)
H44	0.948373	0.701203	0.545763	0.036*
C45	0.95597 (7)	0.7771 (5)	0.3420 (4)	0.0261 (9)
H45	0.972381	0.761441	0.34947	0.031*
C46	0.97819 (7)	0.8917 (5)	0.0193 (4)	0.0278 (9)
H46	0.991274	0.855935	0.075564	0.033*

C47	0.98106 (7)	0.9521 (5)	−0.1249 (4)	0.0303 (9)
H47	0.996364	0.956701	−0.16371	0.036*
C48	0.96282 (7)	1.0037 (5)	−0.2099 (4)	0.0296 (10)
H48	0.965573	1.043925	−0.305523	0.036*
C49	0.93963 (6)	0.9974 (4)	−0.1556 (4)	0.0240 (9)
C50	0.91870 (7)	1.0472 (5)	−0.2278 (5)	0.0322 (10)
H50	0.919178	1.088363	−0.32461	0.039*
C51	0.89775 (7)	1.0355 (5)	−0.1570 (4)	0.0296 (9)
H51	0.883939	1.071225	−0.206259	0.036*
C52	0.89595 (7)	0.9724 (5)	−0.0137 (4)	0.0286 (9)
H52	0.88115	0.96405	0.031533	0.034*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.035 (2)	0.023 (2)	0.030 (2)	0.0012 (17)	0.000 (2)	−0.0011 (17)
C2	0.025 (2)	0.0174 (18)	0.024 (2)	0.0030 (15)	0.0004 (18)	−0.0001 (15)
C3	0.023 (2)	0.0215 (19)	0.029 (2)	0.0009 (16)	0.0007 (18)	−0.0023 (16)
C4	0.028 (2)	0.0169 (17)	0.022 (2)	0.0022 (16)	0.0019 (18)	−0.0020 (15)
C5	0.032 (3)	0.0204 (19)	0.030 (2)	0.0005 (16)	0.003 (2)	−0.0028 (17)
C6	0.029 (2)	0.0221 (19)	0.029 (2)	−0.0001 (16)	0.0037 (19)	−0.0039 (18)
C7	0.026 (2)	0.0146 (17)	0.023 (2)	−0.0004 (15)	−0.0003 (18)	−0.0023 (15)
C8	0.025 (2)	0.0201 (17)	0.026 (2)	−0.0004 (16)	0.0009 (19)	0.0016 (15)
C9	0.028 (2)	0.0165 (18)	0.027 (2)	0.0021 (15)	−0.0001 (18)	0.0005 (16)
C10	0.033 (2)	0.0182 (18)	0.028 (2)	−0.0013 (16)	0.004 (2)	0.0002 (16)
O1	0.0394 (17)	0.0318 (14)	0.0226 (16)	0.0010 (12)	0.0035 (14)	−0.0063 (12)
O2	0.0370 (18)	0.0329 (15)	0.0402 (19)	−0.0011 (12)	0.0127 (14)	−0.0046 (13)
O3	0.0395 (18)	0.0430 (16)	0.0368 (18)	0.0043 (13)	−0.0064 (16)	−0.0115 (14)
O4	0.0320 (16)	0.0330 (15)	0.0238 (15)	0.0015 (12)	0.0020 (13)	−0.0062 (12)
O5	0.0347 (17)	0.0394 (15)	0.0345 (17)	−0.0004 (13)	−0.0071 (15)	−0.0096 (13)
O6	0.0295 (17)	0.0480 (17)	0.0344 (18)	0.0026 (13)	0.0100 (14)	−0.0077 (14)
C11	0.028 (2)	0.0155 (17)	0.022 (2)	0.0036 (16)	0.0003 (17)	0.0015 (15)
C12	0.026 (2)	0.0149 (17)	0.024 (2)	0.0005 (15)	−0.0016 (18)	−0.0015 (15)
C13	0.026 (2)	0.0155 (18)	0.021 (2)	−0.0001 (15)	0.0011 (17)	−0.0002 (15)
C14	0.027 (2)	0.0196 (19)	0.027 (2)	0.0003 (15)	0.0003 (18)	−0.0037 (16)
C15	0.028 (2)	0.0188 (18)	0.024 (2)	0.0010 (16)	0.0018 (18)	0.0012 (15)
C16	0.036 (2)	0.0242 (19)	0.028 (2)	−0.0002 (17)	0.006 (2)	−0.0025 (16)
C17	0.053 (3)	0.026 (2)	0.018 (2)	0.0076 (19)	0.011 (2)	−0.0013 (17)
C18	0.047 (3)	0.025 (2)	0.021 (2)	0.0057 (18)	−0.004 (2)	0.0012 (16)
C19	0.032 (2)	0.0209 (18)	0.029 (2)	0.0016 (16)	−0.0035 (19)	−0.0018 (16)
C20	0.029 (2)	0.0215 (19)	0.030 (2)	0.0009 (16)	0.0007 (19)	−0.0017 (16)
C21	0.036 (2)	0.0237 (19)	0.025 (2)	0.0019 (17)	0.0102 (19)	0.0005 (16)
C22	0.036 (2)	0.0245 (19)	0.019 (2)	0.0001 (16)	0.0040 (19)	−0.0038 (16)
C23	0.033 (2)	0.0147 (17)	0.028 (2)	0.0022 (15)	−0.0033 (19)	0.0006 (16)
C24	0.038 (2)	0.028 (2)	0.029 (2)	−0.0019 (17)	−0.004 (2)	−0.0036 (17)
C25	0.028 (2)	0.035 (2)	0.044 (3)	−0.0008 (18)	−0.005 (2)	−0.007 (2)
C26	0.027 (2)	0.031 (2)	0.042 (3)	0.0008 (17)	−0.001 (2)	−0.0073 (19)
C27	0.038 (2)	0.0209 (19)	0.023 (2)	−0.0012 (17)	0.001 (2)	0.0014 (16)

C28	0.027 (2)	0.0154 (17)	0.021 (2)	−0.0006 (15)	0.0009 (17)	−0.0011 (15)
C29	0.025 (2)	0.0205 (18)	0.027 (2)	0.0029 (15)	−0.0039 (17)	0.0029 (16)
C30	0.025 (2)	0.0145 (17)	0.024 (2)	−0.0008 (15)	0.0027 (17)	−0.0032 (15)
C31	0.035 (3)	0.0181 (18)	0.032 (2)	0.0003 (16)	0.001 (2)	0.0007 (17)
C32	0.036 (2)	0.023 (2)	0.022 (2)	0.0023 (17)	0.000 (2)	0.0015 (16)
C33	0.032 (2)	0.0182 (18)	0.021 (2)	0.0026 (16)	0.0001 (18)	0.0014 (15)
C34	0.026 (2)	0.0203 (19)	0.028 (2)	0.0018 (16)	−0.0001 (19)	−0.0007 (16)
C35	0.030 (2)	0.0162 (17)	0.025 (2)	0.0009 (15)	0.0039 (18)	−0.0007 (15)
C36	0.036 (2)	0.0209 (19)	0.033 (2)	0.0020 (17)	0.005 (2)	0.0030 (17)
O7	0.0381 (16)	0.0324 (15)	0.0241 (16)	0.0020 (12)	0.0034 (13)	0.0066 (12)
O8	0.0348 (17)	0.0429 (16)	0.0411 (19)	0.0011 (13)	0.0147 (15)	0.0044 (14)
O9	0.0449 (18)	0.0443 (17)	0.0295 (16)	0.0003 (14)	−0.0095 (15)	0.0090 (14)
O10	0.0377 (17)	0.0334 (14)	0.0241 (15)	0.0025 (12)	0.0060 (14)	0.0084 (12)
O11	0.0432 (18)	0.0424 (16)	0.0291 (17)	0.0034 (13)	−0.0054 (15)	0.0109 (14)
O12	0.0331 (17)	0.0369 (15)	0.0410 (18)	0.0003 (12)	0.0123 (15)	0.0048 (13)
C37	0.029 (2)	0.0138 (16)	0.024 (2)	−0.0023 (15)	0.0005 (18)	−0.0011 (14)
C38	0.026 (2)	0.0124 (17)	0.025 (2)	−0.0031 (14)	−0.0004 (18)	−0.0006 (15)
C39	0.027 (2)	0.0136 (17)	0.027 (2)	−0.0017 (14)	−0.0008 (18)	0.0003 (15)
C40	0.029 (2)	0.0169 (18)	0.019 (2)	−0.0039 (15)	0.0007 (17)	−0.0004 (15)
C41	0.029 (2)	0.0148 (17)	0.021 (2)	−0.0038 (15)	0.0013 (17)	0.0006 (14)
C42	0.031 (2)	0.0230 (19)	0.029 (2)	−0.0005 (16)	0.0037 (19)	0.0020 (16)
C43	0.041 (3)	0.027 (2)	0.024 (2)	−0.0029 (17)	0.007 (2)	0.0011 (17)
C44	0.049 (3)	0.0235 (19)	0.018 (2)	−0.0020 (18)	−0.005 (2)	0.0035 (15)
C45	0.033 (2)	0.0187 (17)	0.027 (2)	−0.0014 (16)	−0.009 (2)	0.0002 (16)
C46	0.026 (2)	0.0207 (18)	0.036 (2)	−0.0011 (16)	−0.0002 (19)	−0.0007 (17)
C47	0.029 (2)	0.0282 (19)	0.034 (3)	−0.0030 (17)	0.011 (2)	−0.0004 (17)
C48	0.041 (3)	0.0218 (19)	0.026 (2)	−0.0064 (17)	0.010 (2)	0.0024 (16)
C49	0.031 (2)	0.0177 (18)	0.023 (2)	−0.0028 (15)	0.0043 (19)	−0.0026 (16)
C50	0.047 (3)	0.027 (2)	0.022 (2)	−0.0032 (18)	−0.001 (2)	0.0065 (17)
C51	0.030 (2)	0.033 (2)	0.026 (2)	0.0011 (17)	−0.0052 (19)	0.0033 (18)
C52	0.029 (2)	0.028 (2)	0.029 (2)	−0.0021 (16)	0.0013 (18)	0.0018 (17)

Geometric parameters (Å, º)

C1—O3	1.193 (5)	C27—O9	1.192 (4)
C1—O1	1.393 (5)	C27—O7	1.397 (4)
C1—C2	1.481 (6)	C27—C28	1.474 (5)
C2—C3	1.383 (5)	C28—C29	1.384 (5)
C2—C9	1.384 (5)	C28—C35	1.385 (5)
C3—C4	1.375 (5)	C29—C30	1.385 (5)
C3—H3	0.95	C29—H29	0.95
C4—C7	1.380 (5)	C30—C33	1.386 (5)
C4—C5	1.480 (5)	C30—C31	1.479 (5)
C5—O6	1.188 (4)	C31—O12	1.193 (4)
C5—O4	1.396 (4)	C31—O10	1.396 (5)
C6—O5	1.188 (4)	C32—O11	1.182 (4)
C6—O4	1.397 (4)	C32—O10	1.400 (5)
C6—C7	1.474 (5)	C32—C33	1.475 (5)

C7—C8	1.389 (5)	C33—C34	1.376 (5)
C8—C9	1.385 (5)	C34—C35	1.383 (5)
C8—H8	0.95	C34—H34	0.95
C9—C10	1.480 (5)	C35—C36	1.485 (5)
C10—O2	1.186 (4)	C36—O8	1.191 (4)
C10—O1	1.395 (5)	C36—O7	1.380 (5)
C11—C19	1.390 (5)	C37—C45	1.381 (5)
C11—C15	1.420 (5)	C37—C41	1.421 (5)
C11—C12	1.475 (5)	C37—C38	1.476 (5)
C12—C20	1.374 (5)	C38—C46	1.368 (5)
C12—C13	1.411 (5)	C38—C39	1.416 (5)
C13—C14	1.403 (5)	C39—C49	1.386 (5)
C13—C23	1.405 (5)	C39—C40	1.419 (5)
C14—C26	1.378 (5)	C40—C52	1.365 (5)
C14—C15	1.466 (5)	C40—C41	1.472 (5)
C15—C16	1.391 (5)	C41—C42	1.385 (5)
C16—C17	1.388 (5)	C42—C43	1.387 (5)
C16—H16	0.95	C42—H42	0.95
C17—C18	1.391 (5)	C43—C44	1.384 (5)
C17—H17	0.95	C43—H43	0.95
C18—C19	1.381 (5)	C44—C45	1.381 (5)
C18—H18	0.95	C44—H44	0.95
C19—H19	0.95	C45—H45	0.95
C20—C21	1.419 (6)	C46—C47	1.422 (6)
C20—H20	0.95	C46—H46	0.95
C21—C22	1.368 (5)	C47—C48	1.362 (5)
C21—H21	0.95	C47—H47	0.95
C22—C23	1.416 (5)	C48—C49	1.424 (5)
C22—H22	0.95	C48—H48	0.95
C23—C24	1.407 (5)	C49—C50	1.421 (5)
C24—C25	1.386 (6)	C50—C51	1.374 (5)
C24—H24	0.95	C50—H50	0.95
C25—C26	1.417 (6)	C51—C52	1.413 (6)
C25—H25	0.95	C51—H51	0.95
C26—H26	0.95	C52—H52	0.95

O3—C1—O1	121.1 (4)	O9—C27—O7	121.0 (3)
O3—C1—C2	131.3 (4)	O9—C27—C28	131.4 (4)
O1—C1—C2	107.6 (3)	O7—C27—C28	107.6 (3)
C3—C2—C9	123.3 (4)	C29—C28—C35	123.0 (3)
C3—C2—C1	129.4 (3)	C29—C28—C27	129.4 (4)
C9—C2—C1	107.2 (3)	C35—C28—C27	107.6 (3)
C4—C3—C2	114.1 (3)	C28—C29—C30	113.9 (3)
C4—C3—H3	122.9	C28—C29—H29	123
C2—C3—H3	122.9	C30—C29—H29	123
C3—C4—C7	123.4 (3)	C29—C30—C33	123.0 (4)
C3—C4—C5	129.0 (3)	C29—C30—C31	129.1 (3)
C7—C4—C5	107.7 (3)	C33—C30—C31	107.8 (3)

O6—C5—O4	121.4 (4)	O12—C31—O10	121.0 (4)
O6—C5—C4	131.2 (4)	O12—C31—C30	131.7 (4)
O4—C5—C4	107.5 (3)	O10—C31—C30	107.3 (3)
O5—C6—O4	120.3 (4)	O11—C32—O10	121.6 (4)
O5—C6—C7	132.1 (4)	O11—C32—C33	131.0 (4)
O4—C6—C7	107.6 (3)	O10—C32—C33	107.4 (3)
C4—C7—C8	122.4 (3)	C34—C33—C30	122.7 (4)
C4—C7—C6	107.8 (3)	C34—C33—C32	129.5 (4)
C8—C7—C6	129.8 (3)	C30—C33—C32	107.7 (3)
C9—C8—C7	114.7 (3)	C33—C34—C35	114.5 (3)
C9—C8—H8	122.7	C33—C34—H34	122.7
C7—C8—H8	122.7	C35—C34—H34	122.7
C2—C9—C8	122.1 (4)	C34—C35—C28	122.8 (3)
C2—C9—C10	108.1 (3)	C34—C35—C36	129.9 (3)
C8—C9—C10	129.8 (3)	C28—C35—C36	107.3 (3)
O2—C10—O1	121.2 (4)	O8—C36—O7	122.2 (4)
O2—C10—C9	131.8 (4)	O8—C36—C35	130.1 (4)
O1—C10—C9	107.0 (3)	O7—C36—C35	107.7 (3)
C1—O1—C10	110.0 (3)	C36—O7—C27	109.8 (3)
C5—O4—C6	109.4 (3)	C31—O10—C32	109.8 (3)
C19—C11—C15	120.5 (3)	C45—C37—C41	120.5 (3)
C19—C11—C12	131.6 (4)	C45—C37—C38	131.8 (3)
C15—C11—C12	107.9 (3)	C41—C37—C38	107.7 (3)
C20—C12—C13	118.5 (4)	C46—C38—C39	118.1 (4)
C20—C12—C11	135.5 (4)	C46—C38—C37	135.3 (4)
C13—C12—C11	106.0 (3)	C39—C38—C37	106.6 (3)
C14—C13—C23	124.5 (3)	C49—C39—C38	124.6 (3)
C14—C13—C12	111.5 (3)	C49—C39—C40	124.3 (3)
C23—C13—C12	124.0 (3)	C38—C39—C40	111.1 (3)
C26—C14—C13	118.1 (4)	C52—C40—C39	117.9 (3)
C26—C14—C15	135.4 (4)	C52—C40—C41	136.0 (4)
C13—C14—C15	106.5 (3)	C39—C40—C41	106.2 (3)
C16—C15—C11	120.0 (3)	C42—C41—C37	120.0 (3)
C16—C15—C14	131.9 (4)	C42—C41—C40	131.5 (3)
C11—C15—C14	108.1 (3)	C37—C41—C40	108.5 (3)
C17—C16—C15	118.4 (4)	C41—C42—C43	118.7 (3)
C17—C16—H16	120.8	C41—C42—H42	120.6
C15—C16—H16	120.8	C43—C42—H42	120.6
C16—C17—C18	121.5 (4)	C44—C43—C42	120.9 (4)
C16—C17—H17	119.3	C44—C43—H43	119.6
C18—C17—H17	119.3	C42—C43—H43	119.6
C19—C18—C17	120.7 (4)	C45—C44—C43	121.3 (4)
C19—C18—H18	119.6	C45—C44—H44	119.4
C17—C18—H18	119.6	C43—C44—H44	119.4
C18—C19—C11	118.8 (4)	C37—C45—C44	118.6 (3)
C18—C19—H19	120.6	C37—C45—H45	120.7
C11—C19—H19	120.6	C44—C45—H45	120.7
C12—C20—C21	118.6 (4)	C38—C46—C47	118.5 (4)

C12—C20—H20	120.7	C38—C46—H46	120.8
C21—C20—H20	120.7	C47—C46—H46	120.8
C22—C21—C20	122.7 (4)	C48—C47—C46	122.8 (4)
C22—C21—H21	118.7	C48—C47—H47	118.6
C20—C21—H21	118.7	C46—C47—H47	118.6
C21—C22—C23	120.4 (4)	C47—C48—C49	120.1 (4)
C21—C22—H22	119.8	C47—C48—H48	120
C23—C22—H22	119.8	C49—C48—H48	120
C13—C23—C24	116.1 (4)	C39—C49—C50	116.3 (3)
C13—C23—C22	115.9 (3)	C39—C49—C48	115.9 (4)
C24—C23—C22	128.0 (4)	C50—C49—C48	127.8 (4)
C25—C24—C23	120.2 (4)	C51—C50—C49	119.7 (4)
C25—C24—H24	119.9	C51—C50—H50	120.1
C23—C24—H24	119.9	C49—C50—H50	120.1
C24—C25—C26	122.2 (4)	C50—C51—C52	122.6 (4)
C24—C25—H25	118.9	C50—C51—H51	118.7
C26—C25—H25	118.9	C52—C51—H51	118.7
C14—C26—C25	118.9 (4)	C40—C52—C51	119.1 (4)
C14—C26—H26	120.6	C40—C52—H52	120.4
C25—C26—H26	120.6	C51—C52—H52	120.4

O3—C1—C2—C3	−0.5 (7)	O9—C27—C28—C29	0.3 (6)
O1—C1—C2—C3	179.7 (3)	O7—C27—C28—C29	−179.9 (3)
O3—C1—C2—C9	179.8 (4)	O9—C27—C28—C35	−179.4 (4)
O1—C1—C2—C9	0.0 (4)	O7—C27—C28—C35	0.4 (4)
C9—C2—C3—C4	−0.7 (5)	C35—C28—C29—C30	0.1 (5)
C1—C2—C3—C4	179.7 (3)	C27—C28—C29—C30	−179.6 (3)
C2—C3—C4—C7	0.2 (5)	C28—C29—C30—C33	1.0 (5)
C2—C3—C4—C5	179.4 (3)	C28—C29—C30—C31	−179.5 (3)
C3—C4—C5—O6	1.8 (7)	C29—C30—C31—O12	0.0 (7)
C7—C4—C5—O6	−178.9 (4)	C33—C30—C31—O12	179.5 (4)
C3—C4—C5—O4	−179.0 (3)	C29—C30—C31—O10	−179.5 (3)
C7—C4—C5—O4	0.2 (4)	C33—C30—C31—O10	0.0 (4)
C3—C4—C7—C8	0.2 (5)	C29—C30—C33—C34	−1.6 (5)
C5—C4—C7—C8	−179.1 (3)	C31—C30—C33—C34	178.8 (3)
C3—C4—C7—C6	179.2 (3)	C29—C30—C33—C32	179.4 (3)
C5—C4—C7—C6	−0.1 (4)	C31—C30—C33—C32	−0.2 (4)
O5—C6—C7—C4	179.2 (4)	O11—C32—C33—C34	2.4 (7)
O4—C6—C7—C4	−0.1 (4)	O10—C32—C33—C34	−178.6 (3)
O5—C6—C7—C8	−1.9 (7)	O11—C32—C33—C30	−178.7 (4)
O4—C6—C7—C8	178.8 (3)	O10—C32—C33—C30	0.3 (4)
C4—C7—C8—C9	−0.3 (5)	C30—C33—C34—C35	1.0 (5)
C6—C7—C8—C9	−179.0 (3)	C32—C33—C34—C35	179.7 (3)
C3—C2—C9—C8	0.7 (5)	C33—C34—C35—C28	0.2 (5)
C1—C2—C9—C8	−179.6 (3)	C33—C34—C35—C36	179.6 (3)
C3—C2—C9—C10	−179.0 (3)	C29—C28—C35—C34	−0.7 (5)
C1—C2—C9—C10	0.7 (4)	C27—C28—C35—C34	179.0 (3)
C7—C8—C9—C2	−0.2 (5)	C29—C28—C35—C36	179.8 (3)

C7—C8—C9—C10	179.4 (3)	C27—C28—C35—C36	−0.5 (4)
C2—C9—C10—O2	177.8 (4)	C34—C35—C36—O8	2.1 (7)
C8—C9—C10—O2	−1.9 (6)	C28—C35—C36—O8	−178.4 (4)
C2—C9—C10—O1	−1.2 (4)	C34—C35—C36—O7	−179.1 (3)
C8—C9—C10—O1	179.2 (3)	C28—C35—C36—O7	0.4 (4)
O3—C1—O1—C10	179.4 (3)	O8—C36—O7—C27	178.7 (3)
C2—C1—O1—C10	−0.8 (4)	C35—C36—O7—C27	−0.2 (4)
O2—C10—O1—C1	−177.9 (3)	O9—C27—O7—C36	179.7 (3)
C9—C10—O1—C1	1.2 (4)	C28—C27—O7—C36	−0.1 (4)
O6—C5—O4—C6	179.0 (3)	O12—C31—O10—C32	−179.4 (3)
C4—C5—O4—C6	−0.3 (4)	C30—C31—O10—C32	0.2 (4)
O5—C6—O4—C5	−179.1 (3)	O11—C32—O10—C31	178.8 (3)
C7—C6—O4—C5	0.2 (4)	C33—C32—O10—C31	−0.3 (4)
C19—C11—C12—C20	−3.0 (6)	C45—C37—C38—C46	0.3 (7)
C15—C11—C12—C20	177.4 (4)	C41—C37—C38—C46	−179.6 (4)
C19—C11—C12—C13	178.7 (3)	C45—C37—C38—C39	−179.9 (3)
C15—C11—C12—C13	−0.8 (4)	C41—C37—C38—C39	0.2 (4)
C20—C12—C13—C14	−178.2 (3)	C46—C38—C39—C49	−1.4 (5)
C11—C12—C13—C14	0.4 (4)	C37—C38—C39—C49	178.8 (3)
C20—C12—C13—C23	0.4 (5)	C46—C38—C39—C40	179.7 (3)
C11—C12—C13—C23	179.0 (3)	C37—C38—C39—C40	−0.1 (4)
C23—C13—C14—C26	1.5 (5)	C49—C39—C40—C52	−0.1 (5)
C12—C13—C14—C26	−179.9 (3)	C38—C39—C40—C52	178.8 (3)
C23—C13—C14—C15	−178.4 (3)	C49—C39—C40—C41	−178.9 (3)
C12—C13—C14—C15	0.2 (4)	C38—C39—C40—C41	0.0 (4)
C19—C11—C15—C16	1.1 (5)	C45—C37—C41—C42	−0.5 (5)
C12—C11—C15—C16	−179.3 (3)	C38—C37—C41—C42	179.5 (3)
C19—C11—C15—C14	−178.7 (3)	C45—C37—C41—C40	179.9 (3)
C12—C11—C15—C14	0.9 (4)	C38—C37—C41—C40	−0.1 (4)
C26—C14—C15—C16	−0.3 (7)	C52—C40—C41—C42	2.1 (7)
C13—C14—C15—C16	179.6 (4)	C39—C40—C41—C42	−179.5 (3)
C26—C14—C15—C11	179.4 (4)	C52—C40—C41—C37	−178.3 (4)
C13—C14—C15—C11	−0.7 (4)	C39—C40—C41—C37	0.1 (4)
C11—C15—C16—C17	−0.5 (5)	C37—C41—C42—C43	0.7 (5)
C14—C15—C16—C17	179.2 (3)	C40—C41—C42—C43	−179.8 (3)
C15—C16—C17—C18	−0.3 (5)	C41—C42—C43—C44	−0.3 (5)
C16—C17—C18—C19	0.6 (5)	C42—C43—C44—C45	−0.4 (5)
C17—C18—C19—C11	0.1 (5)	C41—C37—C45—C44	−0.2 (5)
C15—C11—C19—C18	−0.9 (5)	C38—C37—C45—C44	179.9 (3)
C12—C11—C19—C18	179.6 (3)	C43—C44—C45—C37	0.6 (5)
C13—C12—C20—C21	0.0 (5)	C39—C38—C46—C47	1.2 (5)
C11—C12—C20—C21	−178.1 (3)	C37—C38—C46—C47	−179.0 (3)
C12—C20—C21—C22	−0.4 (5)	C38—C46—C47—C48	−0.4 (5)
C20—C21—C22—C23	0.5 (5)	C46—C47—C48—C49	−0.4 (5)
C14—C13—C23—C24	−1.1 (5)	C38—C39—C49—C50	−178.6 (3)
C12—C13—C23—C24	−179.5 (3)	C40—C39—C49—C50	0.2 (5)
C14—C13—C23—C22	178.1 (3)	C38—C39—C49—C48	0.6 (5)
C12—C13—C23—C22	−0.3 (5)	C40—C39—C49—C48	179.4 (3)

C21—C22—C23—C13	−0.2 (5)	C47—C48—C49—C39	0.3 (5)
C21—C22—C23—C24	179.0 (3)	C47—C48—C49—C50	179.4 (3)
C13—C23—C24—C25	0.7 (5)	C39—C49—C50—C51	0.4 (5)
C22—C23—C24—C25	−178.5 (3)	C48—C49—C50—C51	−178.7 (3)
C23—C24—C25—C26	−0.7 (6)	C49—C50—C51—C52	−1.0 (6)
C13—C14—C26—C25	−1.4 (5)	C39—C40—C52—C51	−0.5 (5)
C15—C14—C26—C25	178.5 (4)	C41—C40—C52—C51	177.8 (4)
C24—C25—C26—C14	1.1 (6)	C50—C51—C52—C40	1.0 (5)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O2^{i}	0.95	2.67	3.373 (5)	132
C16—H16···O8	0.95	2.59	3.444 (5)	150
C17—H17···O3^{ii}	0.95	2.65	3.576 (5)	166
C18—H18···O1^{ii}	0.95	2.67	3.332 (5)	127
C22—H22···O4^{iii}	0.95	2.59	3.481 (5)	155
C25—H25···O11^{iii}	0.95	2.55	3.347 (5)	142
C29—H29···O12^{iv}	0.95	2.71	3.370 (5)	127
C42—H42···O6	0.95	2.49	3.413 (5)	165
C43—H43···O11^{iii}	0.95	2.58	3.293 (5)	132
C44—H44···O10^{iii}	0.95	2.52	3.428 (5)	160
C45—H45···O12^{iv}	0.95	2.57	3.429 (5)	150
C46—H46···O9^{v}	0.95	2.64	3.256 (5)	123
C48—H48···O9^{vi}	0.95	2.55	3.473 (5)	164
C50—H50···O7^{vi}	0.95	2.5	3.420 (5)	164
C52—H52···O6	0.95	2.62	3.525 (5)	159

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

Pyromellitic acid dianhydride–9-methylanthracene (1/1) (III)

Crystal data

$C_{10}H_2O_6{\cdot}C_{15}H_{12}$
M_r = 410.36
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 7.1012 (8) Å
b = 9.5674 (12) Å
c = 13.6147 (16) Å
α = 99.109 (4)°
β = 99.941 (4)°
γ = 92.219 (4)°
V = 897.53 (19) Å^3
Z = 2
F(000) = 424
D_x = 1.518 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 4805 reflections
θ = 3.5–28.2°
μ = 0.11 mm^{-1}
T = 173 K
Needle, red
0.19 × 0.06 × 0.05 mm

Data collection

Bruker D8 Venture Photon CCD area detector diffractometer
Graphite monochromator
ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.9, T_{max} = 0.95
20202 measured reflections
3280 independent reflections

2159 reflections with $I > 2\sigma(I)$
$R_{int} = 0.075$
$\theta_{max} = 25.5°$, $\theta_{min} = 3.0°$

$h = -8 \rightarrow 8$
$k = -11 \rightarrow 11$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.073$
$wR(F^2) = 0.223$
$S = 1.02$
3280 reflections
281 parameters
0 restraints
0 constraints

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1395P)^2 + 0.5023P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.028$
$\Delta\rho_{max} = 0.67$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

Special details

Experimental. Absorption corrections were made using the program SADABS (Sheldrick, 1996)
Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C11	0.5511 (4)	0.7888 (3)	0.1593 (2)	0.0168 (7)
C12	0.6280 (4)	0.6829 (3)	0.2138 (2)	0.0186 (7)
C13	0.6688 (5)	0.5463 (3)	0.1648 (2)	0.0238 (8)
H13	0.645706	0.525778	0.093003	0.029*
C14	0.7394 (5)	0.4460 (3)	0.2181 (3)	0.0267 (8)
H14	0.764739	0.356517	0.183297	0.032*
C15	0.7760 (5)	0.4733 (4)	0.3256 (3)	0.0293 (8)
H15	0.823867	0.401744	0.362338	0.035*
C16	0.7426 (5)	0.6011 (4)	0.3757 (3)	0.0255 (8)
H16	0.769232	0.618668	0.447533	0.031*
C17	0.6678 (4)	0.7105 (3)	0.3225 (2)	0.0193 (7)
C18	0.6349 (4)	0.8429 (3)	0.3731 (2)	0.0213 (7)
H18	0.663705	0.86158	0.444956	0.026*
C19	0.5610 (4)	0.9481 (3)	0.3215 (2)	0.0170 (7)
C20	0.5291 (5)	1.0846 (3)	0.3738 (3)	0.0258 (8)
H20	0.561001	1.104084	0.445555	0.031*
C21	0.4535 (5)	1.1878 (4)	0.3228 (3)	0.0279 (8)
H21	0.434305	1.277953	0.358915	0.033*
C22	0.4041 (5)	1.1596 (3)	0.2159 (3)	0.0270 (8)
H22	0.34882	1.230449	0.180651	0.032*
C23	0.4348 (5)	1.0322 (3)	0.1632 (2)	0.0220 (7)
H23	0.402008	1.016283	0.091479	0.026*
C24	0.5155 (4)	0.9210 (3)	0.2130 (2)	0.0173 (7)
C25	0.5090 (5)	0.7591 (4)	0.0452 (2)	0.0279 (8)
H25A	0.61712	0.714047	0.020221	0.042*
H25B	0.489642	0.848319	0.019345	0.042*

H25C	0.392889	0.695435	0.022068	0.042*
O1	0.0077 (3)	1.1492 (2)	0.41401 (18)	0.0317 (6)
O2	−0.0687 (3)	1.1373 (2)	0.24510 (17)	0.0277 (6)
O3	−0.1253 (3)	1.0619 (3)	0.07623 (18)	0.0333 (7)
O4	0.2767 (4)	0.5551 (3)	0.42121 (19)	0.0380 (7)
O5	0.2349 (3)	0.4790 (2)	0.25220 (18)	0.0313 (6)
O6	0.1745 (4)	0.4690 (3)	0.08289 (19)	0.0383 (7)
C1	−0.0022 (5)	1.0820 (3)	0.3321 (2)	0.0233 (8)
C2	0.0472 (4)	0.9353 (3)	0.2990 (2)	0.0189 (7)
C3	0.1180 (4)	0.8363 (3)	0.3567 (2)	0.0202 (7)
H3	0.140718	0.85421	0.428537	0.024*
C4	0.1534 (4)	0.7083 (3)	0.3013 (2)	0.0198 (7)
C5	0.2282 (5)	0.5797 (4)	0.3378 (3)	0.0274 (8)
C6	0.1740 (5)	0.5354 (4)	0.1647 (3)	0.0267 (8)
C7	0.1187 (4)	0.6823 (3)	0.1960 (2)	0.0204 (7)
C8	0.0455 (4)	0.7800 (3)	0.1375 (2)	0.0196 (7)
H8	0.020749	0.761353	0.065698	0.023*
C9	0.0114 (4)	0.9083 (3)	0.1936 (2)	0.0180 (7)
C10	−0.0672 (5)	1.0373 (3)	0.1585 (2)	0.0236 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0162 (16)	0.0139 (15)	0.0196 (16)	−0.0028 (12)	0.0030 (12)	0.0017 (12)
C12	0.0155 (16)	0.0157 (16)	0.0255 (17)	−0.0010 (12)	0.0053 (13)	0.0052 (13)
C13	0.0258 (18)	0.0182 (17)	0.0272 (18)	0.0042 (14)	0.0053 (14)	0.0017 (14)
C14	0.0250 (19)	0.0147 (16)	0.041 (2)	0.0021 (14)	0.0061 (15)	0.0058 (15)
C15	0.0249 (19)	0.0211 (18)	0.046 (2)	0.0026 (14)	0.0057 (16)	0.0178 (16)
C16	0.0244 (18)	0.0283 (19)	0.0266 (18)	0.0021 (15)	0.0042 (14)	0.0137 (15)
C17	0.0153 (16)	0.0209 (17)	0.0241 (17)	0.0007 (13)	0.0078 (13)	0.0063 (13)
C18	0.0172 (17)	0.0283 (18)	0.0187 (16)	0.0007 (14)	0.0041 (13)	0.0038 (14)
C19	0.0134 (15)	0.0162 (16)	0.0215 (16)	0.0007 (12)	0.0047 (12)	0.0017 (12)
C20	0.0194 (17)	0.0196 (17)	0.0364 (19)	−0.0005 (14)	0.0107 (14)	−0.0069 (14)
C21	0.0229 (18)	0.0176 (17)	0.042 (2)	0.0039 (14)	0.0111 (15)	−0.0051 (15)
C22	0.0240 (18)	0.0195 (17)	0.038 (2)	0.0031 (14)	0.0062 (15)	0.0046 (15)
C23	0.0222 (17)	0.0198 (17)	0.0247 (17)	0.0056 (14)	0.0018 (13)	0.0072 (14)
C24	0.0149 (16)	0.0191 (16)	0.0195 (16)	−0.0012 (13)	0.0055 (12)	0.0061 (13)
C25	0.037 (2)	0.0235 (17)	0.0230 (18)	0.0082 (15)	0.0052 (15)	0.0019 (14)
O1	0.0327 (14)	0.0253 (13)	0.0343 (15)	0.0050 (11)	0.0070 (11)	−0.0055 (11)
O2	0.0291 (13)	0.0181 (12)	0.0371 (14)	0.0093 (10)	0.0068 (10)	0.0055 (10)
O3	0.0345 (15)	0.0393 (15)	0.0312 (14)	0.0162 (12)	0.0072 (11)	0.0169 (11)
O4	0.0389 (16)	0.0406 (15)	0.0427 (16)	0.0136 (12)	0.0101 (12)	0.0261 (13)
O5	0.0303 (14)	0.0202 (12)	0.0455 (15)	0.0106 (10)	0.0081 (11)	0.0083 (11)
O6	0.0379 (15)	0.0294 (14)	0.0431 (16)	0.0119 (12)	0.0058 (12)	−0.0082 (12)
C1	0.0192 (17)	0.0209 (17)	0.0307 (19)	0.0013 (14)	0.0068 (14)	0.0048 (15)
C2	0.0131 (16)	0.0224 (17)	0.0222 (17)	0.0022 (13)	0.0043 (12)	0.0050 (13)
C3	0.0164 (16)	0.0248 (17)	0.0200 (16)	0.0025 (13)	0.0027 (12)	0.0057 (13)
C4	0.0168 (16)	0.0191 (16)	0.0250 (17)	0.0020 (13)	0.0051 (13)	0.0069 (13)

C5	0.0216 (18)	0.0253 (18)	0.038 (2)	0.0055 (14)	0.0085 (15)	0.0107 (16)
C6	0.0221 (18)	0.0216 (18)	0.037 (2)	0.0080 (14)	0.0069 (15)	0.0038 (16)
C7	0.0113 (15)	0.0194 (16)	0.0285 (18)	−0.0004 (12)	0.0028 (13)	−0.0003 (13)
C8	0.0165 (16)	0.0207 (17)	0.0211 (16)	0.0010 (13)	0.0031 (12)	0.0025 (13)
C9	0.0132 (15)	0.0208 (17)	0.0220 (16)	0.0039 (13)	0.0046 (12)	0.0077 (13)
C10	0.0233 (18)	0.0220 (18)	0.0277 (18)	0.0074 (14)	0.0079 (14)	0.0065 (15)

Geometric parameters (Å, º)

C11—C24	1.411 (4)	C23—H23	0.95
C11—C12	1.420 (4)	C25—H25A	0.98
C11—C25	1.509 (4)	C25—H25B	0.98
C12—C13	1.434 (4)	C25—H25C	0.98
C12—C17	1.437 (4)	O1—C1	1.186 (4)
C13—C14	1.353 (4)	O2—C1	1.389 (4)
C13—H13	0.95	O2—C10	1.398 (4)
C14—C15	1.422 (5)	O3—C10	1.188 (4)
C14—H14	0.95	O4—C5	1.191 (4)
C15—C16	1.353 (5)	O5—C6	1.393 (4)
C15—H15	0.95	O5—C5	1.399 (4)
C16—C17	1.432 (4)	O6—C6	1.193 (4)
C16—H16	0.95	C1—C2	1.479 (4)
C17—C18	1.392 (4)	C2—C3	1.380 (4)
C18—C19	1.386 (4)	C2—C9	1.394 (4)
C18—H18	0.95	C3—C4	1.389 (4)
C19—C20	1.432 (4)	C3—H3	0.95
C19—C24	1.435 (4)	C4—C7	1.392 (4)
C20—C21	1.369 (5)	C4—C5	1.482 (4)
C20—H20	0.95	C6—C7	1.491 (4)
C21—C22	1.417 (5)	C7—C8	1.382 (4)
C21—H21	0.95	C8—C9	1.392 (4)
C22—C23	1.358 (4)	C8—H8	0.95
C22—H22	0.95	C9—C10	1.486 (4)
C23—C24	1.437 (4)		
C24—C11—C12	119.3 (3)	C11—C24—C23	122.5 (3)
C24—C11—C25	120.9 (3)	C19—C24—C23	117.6 (3)
C12—C11—C25	119.8 (3)	C11—C25—H25A	109.5
C11—C12—C13	122.6 (3)	C11—C25—H25B	109.5
C11—C12—C17	120.0 (3)	H25A—C25—H25B	109.5
C13—C12—C17	117.3 (3)	C11—C25—H25C	109.5
C14—C13—C12	121.8 (3)	H25A—C25—H25C	109.5
C14—C13—H13	119.1	H25B—C25—H25C	109.5
C12—C13—H13	119.1	C1—O2—C10	110.9 (2)
C13—C14—C15	120.6 (3)	C6—O5—C5	110.2 (2)
C13—C14—H14	119.7	O1—C1—O2	121.9 (3)
C15—C14—H14	119.7	O1—C1—C2	131.2 (3)
C16—C15—C14	120.1 (3)	O2—C1—C2	106.9 (3)

C16—C15—H15	120	C3—C2—C9	122.7 (3)
C14—C15—H15	120	C3—C2—C1	129.2 (3)
C15—C16—C17	121.3 (3)	C9—C2—C1	108.1 (3)
C15—C16—H16	119.4	C2—C3—C4	114.6 (3)
C17—C16—H16	119.4	C2—C3—H3	122.7
C18—C17—C16	121.8 (3)	C4—C3—H3	122.7
C18—C17—C12	119.3 (3)	C3—C4—C7	122.4 (3)
C16—C17—C12	118.9 (3)	C3—C4—C5	129.1 (3)
C19—C18—C17	121.7 (3)	C7—C4—C5	108.4 (3)
C19—C18—H18	119.2	O4—C5—O5	121.8 (3)
C17—C18—H18	119.2	O4—C5—C4	131.3 (3)
C18—C19—C20	121.5 (3)	O5—C5—C4	106.9 (3)
C18—C19—C24	119.8 (3)	O6—C6—O5	121.4 (3)
C20—C19—C24	118.7 (3)	O6—C6—C7	130.9 (3)
C21—C20—C19	121.4 (3)	O5—C6—C7	107.7 (3)
C21—C20—H20	119.3	C8—C7—C4	123.4 (3)
C19—C20—H20	119.3	C8—C7—C6	129.8 (3)
C20—C21—C22	119.7 (3)	C4—C7—C6	106.7 (3)
C20—C21—H21	120.1	C7—C8—C9	113.8 (3)
C22—C21—H21	120.1	C7—C8—H8	123.1
C23—C22—C21	120.7 (3)	C9—C8—H8	123.1
C23—C22—H22	119.7	C8—C9—C2	123.0 (3)
C21—C22—H22	119.7	C8—C9—C10	129.7 (3)
C22—C23—C24	121.8 (3)	C2—C9—C10	107.3 (3)
C22—C23—H23	119.1	O3—C10—O2	121.5 (3)
C24—C23—H23	119.1	O3—C10—C9	131.7 (3)
C11—C24—C19	119.9 (3)	O2—C10—C9	106.8 (3)

C24—C11—C12—C13	−179.8 (3)	O2—C1—C2—C3	−179.6 (3)
C25—C11—C12—C13	0.1 (5)	O1—C1—C2—C9	−179.0 (3)
C24—C11—C12—C17	0.3 (4)	O2—C1—C2—C9	0.8 (3)
C25—C11—C12—C17	−179.8 (3)	C9—C2—C3—C4	0.9 (5)
C11—C12—C13—C14	−179.0 (3)	C1—C2—C3—C4	−178.6 (3)
C17—C12—C13—C14	0.9 (5)	C2—C3—C4—C7	−0.4 (5)
C12—C13—C14—C15	−0.1 (5)	C2—C3—C4—C5	−179.8 (3)
C13—C14—C15—C16	−0.9 (5)	C6—O5—C5—O4	−178.9 (3)
C14—C15—C16—C17	0.9 (5)	C6—O5—C5—C4	1.3 (4)
C15—C16—C17—C18	−179.3 (3)	C3—C4—C5—O4	−0.7 (6)
C15—C16—C17—C12	0.0 (5)	C7—C4—C5—O4	179.8 (4)
C11—C12—C17—C18	−1.7 (4)	C3—C4—C5—O5	179.1 (3)
C13—C12—C17—C18	178.4 (3)	C7—C4—C5—O5	−0.4 (4)
C11—C12—C17—C16	179.0 (3)	C5—O5—C6—O6	177.4 (3)
C13—C12—C17—C16	−0.9 (4)	C5—O5—C6—C7	−1.6 (4)
C16—C17—C18—C19	−179.5 (3)	C3—C4—C7—C8	−0.4 (5)
C12—C17—C18—C19	1.3 (5)	C5—C4—C7—C8	179.1 (3)
C17—C18—C19—C20	−179.4 (3)	C3—C4—C7—C6	179.9 (3)
C17—C18—C19—C24	0.5 (5)	C5—C4—C7—C6	−0.5 (3)
C18—C19—C20—C21	−179.0 (3)	O6—C6—C7—C8	2.8 (6)

C24—C19—C20—C21	1.1 (5)	O5—C6—C7—C8	−178.3 (3)
C19—C20—C21—C22	0.5 (5)	O6—C6—C7—C4	−177.6 (4)
C20—C21—C22—C23	−1.5 (5)	O5—C6—C7—C4	1.3 (4)
C21—C22—C23—C24	0.8 (5)	C4—C7—C8—C9	0.7 (5)
C12—C11—C24—C19	1.4 (4)	C6—C7—C8—C9	−179.7 (3)
C25—C11—C24—C19	−178.5 (3)	C7—C8—C9—C2	−0.2 (4)
C12—C11—C24—C23	−178.9 (3)	C7—C8—C9—C10	−179.5 (3)
C25—C11—C24—C23	1.3 (5)	C3—C2—C9—C8	−0.6 (5)
C18—C19—C24—C11	−1.8 (4)	C1—C2—C9—C8	179.0 (3)
C20—C19—C24—C11	178.0 (3)	C3—C2—C9—C10	178.8 (3)
C18—C19—C24—C23	178.4 (3)	C1—C2—C9—C10	−1.7 (3)
C20—C19—C24—C23	−1.7 (4)	C1—O2—C10—O3	177.3 (3)
C22—C23—C24—C11	−179.0 (3)	C1—O2—C10—C9	−1.4 (3)
C22—C23—C24—C19	0.8 (5)	C8—C9—C10—O3	2.7 (6)
C10—O2—C1—O1	−179.7 (3)	C2—C9—C10—O3	−176.6 (4)
C10—O2—C1—C2	0.4 (3)	C8—C9—C10—O2	−178.8 (3)
O1—C1—C2—C3	0.5 (6)	C2—C9—C10—O2	1.9 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O1^{i}	0.95	2.55	3.376 (4)	145
C14—H14···O2^{ii}	0.95	2.63	3.347 (4)	133
C16—H16···O4^{iii}	0.95	2.68	3.365 (4)	130
C22—H22···O5^{iv}	0.95	2.64	3.323 (4)	130

Symmetry codes: (i) −*x*, −*y*+2, −*z*+1; (ii) *x*+1, *y*−1, *z*; (iii) −*x*+1, −*y*+1, −*z*+1; (iv) *x*, *y*+1, *z*.

Pyromellitic acid dianhydride–ethyl anthracene-9-carboxylate (1/2) (IV)

Crystal data

$C_{17}H_{12}O_3·0.5C_{10}H_2O_6$
M_r = 373.32
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 9.1949 (7) Å
b = 17.9751 (14) Å
c = 10.9716 (10) Å
β = 112.829 (2)°
V = 1671.3 (2) Å^3
Z = 4
$F(000)$ = 772
D_x = 1.484 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2311 reflections
θ = 2.3–26.6°
μ = 0.11 mm^{-1}
T = 173 K
Plate, red
0.55 × 0.1 × 0.06 mm

Data collection

Bruker D8 Venture Photon CCD area detector diffractometer
Graphite monochromator
ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.9, T_{max} = 0.95
13071 measured reflections
4035 independent reflections
2731 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 28.0°, θ_{min} = 2.3°
h = −12→12
k = −23→21
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.112$
$S = 1.05$
4035 reflections
254 parameters
0 restraints
0 constraints

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0487P)^2 + 0.0752P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

Special details

Experimental. Absorption corrections were made using the program SADABS (Sheldrick, 1996)
Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.63812 (17)	0.54682 (8)	0.55902 (15)	0.0264 (3)
H1	0.728631	0.57749	0.597638	0.032*
C2	0.49342 (18)	0.57369 (8)	0.47345 (15)	0.0251 (3)
C3	0.44815 (19)	0.65055 (9)	0.42556 (16)	0.0306 (4)
C4	0.22896 (19)	0.57678 (9)	0.33296 (15)	0.0302 (4)
C5	0.36097 (17)	0.52887 (8)	0.41673 (14)	0.0243 (3)
O1	0.28769 (13)	0.64923 (6)	0.34113 (11)	0.0342 (3)
O2	0.52155 (15)	0.70646 (6)	0.44651 (13)	0.0415 (3)
O3	0.09445 (13)	0.56283 (7)	0.26877 (12)	0.0417 (3)
C11	0.55637 (17)	0.42627 (8)	0.88576 (14)	0.0238 (3)
C12	0.60955 (18)	0.35455 (9)	0.94068 (15)	0.0296 (4)
H12	0.539628	0.313325	0.913622	0.035*
C13	0.75932 (19)	0.34440 (9)	1.03144 (16)	0.0342 (4)
H13	0.791473	0.29626	1.067614	0.041*
C14	0.86796 (19)	0.40408 (10)	1.07291 (16)	0.0357 (4)
H14	0.971959	0.395898	1.136131	0.043*
C15	0.82309 (18)	0.47274 (10)	1.02220 (15)	0.0322 (4)
H15	0.896493	0.512595	1.050527	0.039*
C16	0.66755 (17)	0.48668 (8)	0.92684 (14)	0.0251 (3)
C17	0.62178 (18)	0.55718 (8)	0.87435 (15)	0.0278 (3)
H17	0.696566	0.596523	0.901315	0.033*
C18	0.46992 (17)	0.57207 (8)	0.78354 (14)	0.0248 (3)
C19	0.4253 (2)	0.64542 (9)	0.73299 (16)	0.0319 (4)
H19	0.500385	0.684592	0.760919	0.038*
C20	0.2764 (2)	0.65969 (9)	0.64539 (16)	0.0339 (4)
H20	0.248288	0.708695	0.612195	0.041*
C21	0.1630 (2)	0.60214 (9)	0.60322 (16)	0.0323 (4)
H21	0.059209	0.612936	0.542045	0.039*
C22	0.20050 (17)	0.53167 (9)	0.64893 (15)	0.0271 (3)

H22	0.122112	0.493954	0.619804	0.032*
C23	0.35631 (16)	0.51312 (8)	0.74042 (14)	0.0223 (3)
C24	0.40167 (16)	0.44101 (8)	0.79185 (14)	0.0230 (3)
C25	0.28407 (17)	0.38021 (8)	0.74148 (15)	0.0250 (3)
C26	0.18246 (19)	0.27453 (9)	0.81897 (17)	0.0346 (4)
C27	0.0880 (2)	0.26785 (10)	0.9011 (2)	0.0451 (5)
H27A	0.023888	0.222539	0.8766	0.068*
H27B	0.018793	0.311258	0.886488	0.068*
H27C	0.158797	0.265338	0.99463	0.068*
O4	0.21058 (13)	0.36619 (6)	0.62747 (11)	0.0335 (3)
O5	0.26000 (12)	0.34368 (6)	0.84255 (11)	0.0313 (3)
O6	0.20212 (17)	0.22943 (7)	0.74753 (14)	0.0532 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0272 (8)	0.0265 (8)	0.0271 (8)	−0.0014 (6)	0.0121 (6)	−0.0029 (6)
C2	0.0308 (8)	0.0239 (8)	0.0242 (7)	0.0019 (6)	0.0145 (6)	−0.0001 (6)
C3	0.0382 (9)	0.0265 (9)	0.0318 (8)	0.0056 (7)	0.0185 (7)	0.0034 (7)
C4	0.0342 (9)	0.0329 (9)	0.0239 (8)	0.0073 (7)	0.0116 (7)	0.0017 (7)
C5	0.0256 (8)	0.0275 (8)	0.0213 (7)	0.0036 (6)	0.0108 (6)	0.0006 (6)
O1	0.0379 (7)	0.0295 (6)	0.0346 (6)	0.0106 (5)	0.0136 (5)	0.0061 (5)
O2	0.0503 (7)	0.0221 (6)	0.0567 (8)	0.0005 (5)	0.0259 (6)	0.0035 (5)
O3	0.0299 (6)	0.0495 (8)	0.0363 (7)	0.0073 (6)	0.0027 (5)	0.0015 (6)
C11	0.0262 (8)	0.0277 (8)	0.0206 (7)	0.0000 (6)	0.0125 (6)	−0.0014 (6)
C12	0.0322 (8)	0.0274 (9)	0.0297 (8)	0.0015 (7)	0.0127 (7)	0.0026 (6)
C13	0.0383 (9)	0.0332 (9)	0.0318 (9)	0.0100 (8)	0.0144 (7)	0.0045 (7)
C14	0.0275 (8)	0.0492 (11)	0.0265 (8)	0.0072 (8)	0.0061 (7)	−0.0015 (7)
C15	0.0258 (8)	0.0411 (10)	0.0284 (8)	−0.0051 (7)	0.0091 (7)	−0.0076 (7)
C16	0.0264 (8)	0.0289 (8)	0.0223 (7)	−0.0021 (6)	0.0119 (6)	−0.0040 (6)
C17	0.0288 (8)	0.0273 (9)	0.0290 (8)	−0.0079 (7)	0.0132 (7)	−0.0058 (6)
C18	0.0313 (8)	0.0230 (8)	0.0242 (7)	−0.0002 (6)	0.0156 (6)	−0.0032 (6)
C19	0.0414 (9)	0.0232 (8)	0.0372 (9)	−0.0030 (7)	0.0218 (8)	−0.0019 (7)
C20	0.0465 (10)	0.0250 (8)	0.0367 (9)	0.0085 (8)	0.0232 (8)	0.0056 (7)
C21	0.0346 (9)	0.0344 (9)	0.0289 (8)	0.0098 (7)	0.0134 (7)	0.0014 (7)
C22	0.0266 (8)	0.0275 (8)	0.0276 (8)	0.0017 (6)	0.0112 (6)	−0.0021 (6)
C23	0.0256 (7)	0.0240 (8)	0.0204 (7)	0.0013 (6)	0.0123 (6)	−0.0012 (6)
C24	0.0242 (7)	0.0243 (8)	0.0228 (7)	−0.0019 (6)	0.0118 (6)	−0.0022 (6)
C25	0.0241 (7)	0.0227 (8)	0.0289 (8)	0.0002 (6)	0.0110 (6)	−0.0003 (6)
C26	0.0334 (9)	0.0278 (9)	0.0362 (9)	−0.0067 (7)	0.0065 (7)	0.0033 (7)
C27	0.0367 (10)	0.0422 (10)	0.0581 (12)	−0.0048 (8)	0.0203 (9)	0.0160 (9)
O4	0.0363 (6)	0.0304 (6)	0.0289 (6)	−0.0059 (5)	0.0072 (5)	−0.0030 (5)
O5	0.0362 (6)	0.0282 (6)	0.0317 (6)	−0.0117 (5)	0.0158 (5)	−0.0025 (5)
O6	0.0713 (10)	0.0330 (7)	0.0551 (9)	−0.0128 (7)	0.0244 (8)	−0.0085 (6)

Geometric parameters (Å, º)

C1—C2	1.384 (2)	C17—C18	1.391 (2)
C1—C5^{i}	1.386 (2)	C17—H17	0.95
C1—H1	0.95	C18—C19	1.428 (2)
C2—C5	1.390 (2)	C18—C23	1.433 (2)
C2—C3	1.479 (2)	C19—C20	1.357 (2)
C3—O2	1.1824 (19)	C19—H19	0.95
C3—O1	1.4054 (19)	C20—C21	1.413 (2)
C4—O3	1.1885 (19)	C20—H20	0.95
C4—O1	1.399 (2)	C21—C22	1.357 (2)
C4—C5	1.481 (2)	C21—H21	0.95
C11—C24	1.420 (2)	C22—C23	1.434 (2)
C11—C12	1.427 (2)	C22—H22	0.95
C11—C16	1.439 (2)	C23—C24	1.411 (2)
C12—C13	1.363 (2)	C24—C25	1.485 (2)
C12—H12	0.95	C25—O4	1.1954 (18)
C13—C14	1.415 (2)	C25—O5	1.3786 (18)
C13—H13	0.95	C26—O6	1.189 (2)
C14—C15	1.351 (2)	C26—O5	1.4061 (19)
C14—H14	0.95	C26—C27	1.479 (2)
C15—C16	1.429 (2)	C27—H27A	0.98
C15—H15	0.95	C27—H27B	0.98
C16—C17	1.388 (2)	C27—H27C	0.98
C2—C1—C5^{i}	113.92 (14)	C18—C17—H17	119
C2—C1—H1	123	C17—C18—C19	120.89 (14)
C5^{i}—C1—H1	123	C17—C18—C23	119.65 (13)
C1—C2—C5	123.11 (14)	C19—C18—C23	119.45 (14)
C1—C2—C3	129.11 (15)	C20—C19—C18	120.57 (15)
C5—C2—C3	107.78 (13)	C20—C19—H19	119.7
O2—C3—O1	121.11 (14)	C18—C19—H19	119.7
O2—C3—C2	131.61 (16)	C19—C20—C21	120.45 (15)
O1—C3—C2	107.28 (13)	C19—C20—H20	119.8
O3—C4—O1	121.42 (14)	C21—C20—H20	119.8
O3—C4—C5	131.21 (16)	C22—C21—C20	120.88 (15)
O1—C4—C5	107.36 (13)	C22—C21—H21	119.6
C1^{i}—C5—C2	122.97 (14)	C20—C21—H21	119.6
C1^{i}—C5—C4	129.23 (14)	C21—C22—C23	121.16 (15)
C2—C5—C4	107.80 (14)	C21—C22—H22	119.4
C4—O1—C3	109.78 (11)	C23—C22—H22	119.4
C24—C11—C12	123.88 (14)	C24—C23—C18	118.87 (13)
C24—C11—C16	118.45 (13)	C24—C23—C22	123.63 (13)
C12—C11—C16	117.66 (13)	C18—C23—C22	117.48 (13)
C13—C12—C11	120.88 (15)	C23—C24—C11	121.26 (13)
C13—C12—H12	119.6	C23—C24—C25	117.89 (13)
C11—C12—H12	119.6	C11—C24—C25	120.82 (13)
C12—C13—C14	121.36 (15)	O4—C25—O5	122.63 (14)

C12—C13—H13	119.3	O4—C25—C24	125.38 (14)
C14—C13—H13	119.3	O5—C25—C24	111.86 (12)
C15—C14—C13	119.70 (15)	O6—C26—O5	121.88 (16)
C15—C14—H14	120.1	O6—C26—C27	128.43 (16)
C13—C14—H14	120.1	O5—C26—C27	109.59 (15)
C14—C15—C16	121.38 (15)	C26—C27—H27A	109.5
C14—C15—H15	119.3	C26—C27—H27B	109.5
C16—C15—H15	119.3	H27A—C27—H27B	109.5
C17—C16—C15	121.34 (14)	C26—C27—H27C	109.5
C17—C16—C11	119.67 (13)	H27A—C27—H27C	109.5
C15—C16—C11	118.99 (14)	H27B—C27—H27C	109.5
C16—C17—C18	122.10 (14)	C25—O5—C26	120.09 (12)
C16—C17—H17	119		

C5^{i}—C1—C2—C5	−0.1 (2)	C11—C16—C17—C18	0.8 (2)
C5^{i}—C1—C2—C3	179.81 (14)	C16—C17—C18—C19	178.85 (14)
C1—C2—C3—O2	0.7 (3)	C16—C17—C18—C23	−0.9 (2)
C5—C2—C3—O2	−179.41 (17)	C17—C18—C19—C20	−179.35 (15)
C1—C2—C3—O1	−179.62 (14)	C23—C18—C19—C20	0.4 (2)
C5—C2—C3—O1	0.30 (16)	C18—C19—C20—C21	0.3 (2)
C1—C2—C5—C1^{i}	0.1 (3)	C19—C20—C21—C22	−0.2 (2)
C3—C2—C5—C1^{i}	−179.82 (13)	C20—C21—C22—C23	−0.5 (2)
C1—C2—C5—C4	179.33 (14)	C17—C18—C23—C24	0.2 (2)
C3—C2—C5—C4	−0.59 (16)	C19—C18—C23—C24	−179.56 (13)
O3—C4—C5—C1^{i}	1.0 (3)	C17—C18—C23—C22	178.68 (13)
O1—C4—C5—C1^{i}	179.84 (15)	C19—C18—C23—C22	−1.0 (2)
O3—C4—C5—C2	−178.20 (17)	C21—C22—C23—C24	179.59 (14)
O1—C4—C5—C2	0.68 (16)	C21—C22—C23—C18	1.1 (2)
O3—C4—O1—C3	178.52 (15)	C18—C23—C24—C11	0.6 (2)
C5—C4—O1—C3	−0.50 (15)	C22—C23—C24—C11	−177.83 (13)
O2—C3—O1—C4	179.89 (15)	C18—C23—C24—C25	−177.47 (12)
C2—C3—O1—C4	0.14 (16)	C22—C23—C24—C25	4.1 (2)
C24—C11—C12—C13	−179.39 (14)	C12—C11—C24—C23	−179.58 (13)
C16—C11—C12—C13	1.7 (2)	C16—C11—C24—C23	−0.7 (2)
C11—C12—C13—C14	−1.0 (2)	C12—C11—C24—C25	−1.6 (2)
C12—C13—C14—C15	0.2 (2)	C16—C11—C24—C25	177.35 (13)
C13—C14—C15—C16	−0.1 (2)	C23—C24—C25—O4	50.9 (2)
C14—C15—C16—C17	−179.71 (15)	C11—C24—C25—O4	−127.22 (17)
C14—C15—C16—C11	0.8 (2)	C23—C24—C25—O5	−125.00 (14)
C24—C11—C16—C17	0.0 (2)	C11—C24—C25—O5	56.92 (17)
C12—C11—C16—C17	178.95 (13)	O4—C25—O5—C26	18.8 (2)
C24—C11—C16—C15	179.41 (13)	C24—C25—O5—C26	−165.19 (13)
C12—C11—C16—C15	−1.6 (2)	O6—C26—O5—C25	37.5 (2)
C15—C16—C17—C18	−178.63 (14)	C27—C26—O5—C25	−146.03 (14)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···O2^{ii}	0.95	2.65	3.351 (2)	131
C15—H15···O3^{iii}	0.95	2.55	3.306 (2)	137
C21—H21···O4^{iv}	0.95	2.48	3.433 (2)	176

Symmetry codes: (ii) −*x*+1, *y*−1/2, −*z*+3/2; (iii) *x*+1, *y*, *z*+1; (iv) −*x*, −*y*+1, −*z*+1.