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2,8,14,20-*para*-Methoxytetraphenylpyrogallol[4]arene dimethylformamide hexasolvate

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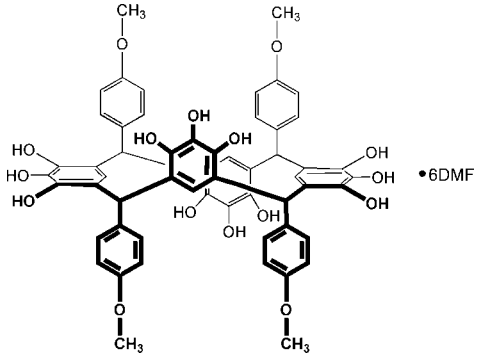
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.051; wR factor = 0.143; data-to-parameter ratio = 18.1.

The title compound, $C_{56}H_{48}O_{16}\cdot 6C_3H_7NO$, adopts a macrocyclic chair conformation in the solid state. The asymmetric unit contains one half of the molecule, which lies on a crystallographic inversion center, and three molecules of dimethylformamide. The crystal structure is stabilized by extensive hydrogen bonding.

Related literature

For related literature, see: Asfari *et al.* (2001); Bruno *et al.* (2002); Cave *et al.* (2005); Dueno *et al.* (2006); Farrugia (1997); Kass *et al.* (2006); Liu *et al.* (2005); Makeiff & Sherman (2005); Zambrano *et al.* (2006).



Experimental

Crystal data

$C_{56}H_{48}O_{16}\cdot 6C_3H_7NO$
M_r = 1415.52
Triclinic, $P\overline{1}$
a = 10.8399 (6) Å
b = 11.6307 (7) Å
c = 15.3585 (9) Å
α = 73.480 (1)°
β = 73.554 (1)°
γ = 73.003 (1)°
V = 1732.57 (17) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100 (2) K
0.44 × 0.39 × 0.25 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS* in *SAINT-Plus*; Bruker, 2003*b*)
T_{min} = 0.889, T_{max} = 0.975
18068 measured reflections
8578 independent reflections
7224 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.143
S = 1.03
8578 reflections
474 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.75 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4*B*—H4*B*1···O3*B*	0.84	2.54	2.9446 (16)	111
O4*B*—H4*B*1···O3^{i}	0.84	1.89	2.7231 (15)	175
O3*B*—H3*B*1···O4*B*	0.84	2.55	2.9446 (16)	110
O3*B*—H3*B*1···O3^{i}	0.84	1.86	2.6971 (16)	173
O2*B*—H2*B*2···O3*B*	0.84	2.26	2.6773 (15)	111
O2*B*—H2*B*2···O1^{ii}	0.84	2.02	2.7595 (18)	147
O4*A*—H4*A*1···O3*A*	0.84	2.52	2.9334 (15)	112
O4*A*—H4*A*1···O2^{iii}	0.84	1.89	2.7158 (15)	169
O3*A*—H3*A*1···O4*A*	0.84	2.54	2.9334 (15)	110
O3*A*—H3*A*1···O2^{iii}	0.84	1.86	2.7010 (16)	176
O2*A*—H2*A*2···O3*A*	0.84	2.20	2.6523 (15)	114
O2*A*—H2*A*2···O1*B*^{iii}	0.84	2.04	2.7779 (15)	147

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT-Plus* (Bruker, 2003*b*); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 2003*a*); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; 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: PV2012).

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supplementary materials

Acta Cryst. (2007). E**63**, o3452-o3453 [**doi:10.1107/S1600536807021757**]

2,8,14,20-*para*-Methoxytetraphenylpyrogallol[4]arene dimethylformamide hexasolvate

C. Zambrano, R. E. Thomas, M. Zeller, R. N. Salvatore and E. E. Dueno

Comment

Bowl-shaped compounds such as pyrogallolarenes have received considerable attention over the last two decades because of their potential use in a number of technological applications (Asfari *et al.*, 2001). The conformational preferences of pyrogallol[4]arenes are still being studied by various investigators (Makeiff, *et al.*, 2005). Our studies have shown that aryl-substituted pyrogallol[4]arenes adopt a chair (rctt) conformation (Zambrano *et al.*, 2006; Kass *et al.*, 2006), whereas the alkyl substituted analogs adopt the crown (rccc) structure (Dueno *et al.*, 2006). Here, we report the crystal structure of a compound 2,8,14,20-*para*-methoxytetraphenyl-pyrogallol[4]arene, (I), recrystallized by vapor diffusion of diethyl ether into a solution of (I) in *N*,*N*-dimethylformamide.

The molecule possesses a center of inversion, where two pairs of pyrogallol rings are clearly distinguishable from each other. One pair is comprised of pyrogallol rings perfectly coplanar to each other, but with their OH groups pointing in opposite directions (ring C8B—C13B and its symmetry inverse counterpart). In the other pair, the aromatic rings are parallel to each other and separated by a distance of 4.982 (9) Å, calculated *via* least-squares mean planes of ring C8A—C13A and its symmetry equivalent ring (C8A—C13A, symmetry op.: $-x$, $1-y$, $1-z$). These two pairs of pyrogallol groups are almost perpendicular to each other, for they exhibit a planes angle of 78.88 (12)°. Another interesting structural feature of this molecule is the positions of the *para*-methoxyphenyl substituents which are almost perfectly aligned on top of each other, as in our previously reported compounds (Zambrano, *et al.*, 2006). This *para*-methoxyphenyl substituents, are not parallel with respect to each other, but are slightly bent at an angle of 22.82 (12)°, based on least-squares mean planes of both rings carbon atoms (C1A—C6A and C1B—C6B). The centroid to centroid distance between these *para*-methoxy rings is 4.282 (9)Å with a centroid-centroid offset of 0.112 (2) Å. This separation suggests that no significant π-π interaction is present between aromatic groups (Liu *et al.*, 2005).

The asymmetric unit of (I) contains three molecules of DMF, which are part of an intricate network of hydrogen bonds with the OH groups of the pyrogallolarene macrocycle as the H donor groups (Table 1). H-bonding is also found between the methoxy-O atom and a pyrogallol-OH group (Figure 2). All H-bond values are consistent with the literature (Cave *et al.*, 2005).

Experimental

A 50 ml round bottom flask was charged with 2.0 g (16 mmol) pyrogallol and 11 ml 95% ethanol·The reaction vessel was cooled in an ice bath to 273 K and 2 ml of concentrated HCl was added in one portion. *Para*-anisaldehyde (2.0 g, 16 mmol) was then added dropwise over a period of ten minutes. The reaction vessel was allowed to warm slowly to room temperature and then maintained at 353 K for 12 h, the red powder that separated was collected by filtration and washed with cold 1:1 ethanol-water until the material was pale pink, and neutral to pH paper. Drying under vacuum at 313 K for 12 h afforded 2.6 g (2.8 mmol) of 2,8,14,20-*para*-methoxytetra(phenyl)pyrogallol[4]arene, yield, 70% mp 632–633 K. Single crystals suitable for X-ray diffraction analysis were grown from a solution in DMF by vapor diffusion of ether to yield large blocks of colorless crystals.

Refinement

Hydrogen atoms were treated as riding, with O—H = 0.84, C—H = 0.98 for the methoxy methyl group, C—H = 0.95 for phenyl groups and C—H = 1.00Å for the bridging unit. U_{iso}(H) = 1.5 U_{eq}(C/O) was used for the methyl and hydroxyl group hydrogen atoms and 1.2 U_{eq}(C) for all other H atoms. The methyl as well as the hydroxyl groups were allowed to rotate to best fit the experimental electron density.

Figures

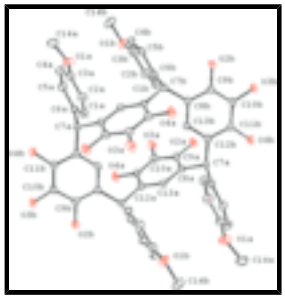

Fig. 1. An *ORTEP* view of (I) (Farrugia, 1997); displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity.

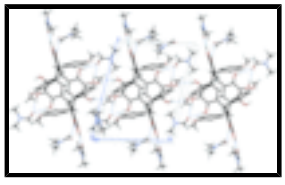

Fig. 2. Mercury packing diagram (Bruno *et al.*, 2002) of (I), viewed down the *a* axis, dashed lines indicate hydrogen bonds.

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Crystal data

$C_{56}H_{48}O_{16}\cdot 6C_3H_7NO$	$Z = 1$
$M_r = 1415.52$	$F_{000} = 752$
Triclinic, $P\overline{1}$	$D_x = 1.357$ Mg m^{-3}
$a = 10.8399$ (6) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 11.6307$ (7) Å	Cell parameters from 5991 reflections
$c = 15.3585$ (9) Å	$\theta = 2.3$–$30.5°$
$\alpha = 73.480$ (1)°	$\mu = 0.10$ mm^{-1}
$\beta = 73.554$ (1)°	$T = 100$ (2) K
$\gamma = 73.003$ (1)°	Block, pink
$V = 1732.57$ (17) Å^3	0.44 × 0.39 × 0.25 mm

Data collection

Bruker SMART APEX CCD diffractometer	8578 independent reflections
Radiation source: fine-focus sealed tube	7224 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.018$

T = 100(2) K	θ_{max} = 28.3º
ω scans	θ_{min} = 1.9º
Absorption correction: multi-scan (SADABS in SAINT-Plus; Bruker, 2003b)	h = −13→14
T_{min} = 0.889, T_{max} = 0.975	k = −15→15
18068 measured reflections	l = −20→20

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.051	H-atom parameters constrained
$wR(F^2)$ = 0.143	$w = 1/[\sigma^2(F_o^2) + (0.0768P)^2 + 0.8303P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max}$ = 0.001
8578 reflections	$\Delta\rho_{max}$ = 0.75 e Å^{-3}
474 parameters	$\Delta\rho_{min}$ = −0.48 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6658 (2)	0.6549 (2)	1.01615 (13)	0.0441 (5)
H1	0.7124	0.6711	1.0536	0.053*
C2	0.6637 (3)	0.5315 (2)	0.91362 (17)	0.0663 (8)
H2A	0.5810	0.5926	0.9063	0.099*
H2B	0.6448	0.4501	0.9426	0.099*
H2C	0.7232	0.5295	0.8524	0.099*
C3	0.8567 (3)	0.4911 (3)	0.97997 (19)	0.0827 (10)
H3A	0.8927	0.5254	1.0162	0.124*
H3B	0.9143	0.4923	0.9178	0.124*
H3C	0.8520	0.4060	1.0116	0.124*
C4	0.42933 (16)	1.11176 (14)	0.19126 (11)	0.0250 (3)
H4	0.4498	1.1682	0.1339	0.030*

C5	0.47278 (18)	0.90179 (15)	0.28231 (12)	0.0310 (3)
H5A	0.4014	0.9384	0.3289	0.047*
H5B	0.5534	0.8674	0.3067	0.047*
H5C	0.4473	0.8361	0.2681	0.047*
C6	0.59696 (19)	0.95763 (18)	0.11855 (13)	0.0367 (4)
H6A	0.6050	1.0292	0.0667	0.055*
H6B	0.5708	0.8965	0.0992	0.055*
H6C	0.6821	0.9211	0.1367	0.055*
C7	−0.04528 (16)	0.06013 (14)	0.13697 (10)	0.0246 (3)
H7	−0.1108	0.0223	0.1815	0.030*
C8	0.1655 (2)	0.1142 (2)	0.10120 (13)	0.0458 (5)
H8A	0.1418	0.1525	0.0407	0.069*
H8B	0.2481	0.0510	0.0933	0.069*
H8C	0.1773	0.1772	0.1271	0.069*
C9	0.08011 (18)	−0.00344 (16)	0.25792 (11)	0.0311 (3)
H9A	0.0044	−0.0390	0.2937	0.047*
H9B	0.0879	0.0567	0.2885	0.047*
H9C	0.1611	−0.0691	0.2545	0.047*
C1A	0.08449 (15)	0.54655 (13)	0.73022 (10)	0.0221 (3)
H1A	0.0029	0.5568	0.7142	0.026*
C2A	0.10938 (15)	0.64325 (14)	0.75351 (11)	0.0253 (3)
H2A1	0.0452	0.7191	0.7529	0.030*
C3A	0.22786 (15)	0.62924 (14)	0.77765 (10)	0.0244 (3)
C4A	0.32319 (15)	0.51968 (15)	0.77624 (11)	0.0261 (3)
H4A	0.4054	0.5103	0.7912	0.031*
C5A	0.29646 (15)	0.42339 (14)	0.75247 (10)	0.0235 (3)
H5A1	0.3616	0.3483	0.7517	0.028*
C6A	0.17776 (14)	0.43408 (13)	0.72994 (9)	0.0184 (3)
C7A	0.15459 (13)	0.32867 (12)	0.70084 (9)	0.0168 (3)
H7A	0.2082	0.2503	0.7322	0.020*
C8A	0.20160 (13)	0.33775 (12)	0.59585 (9)	0.0168 (3)
C9A	0.21157 (14)	0.23746 (12)	0.55967 (10)	0.0188 (3)
C10A	0.24456 (14)	0.24523 (13)	0.46370 (10)	0.0197 (3)
C11A	0.27271 (14)	0.35332 (13)	0.40240 (9)	0.0189 (3)
C12A	0.26516 (13)	0.45511 (12)	0.43692 (9)	0.0167 (3)
C13A	0.22852 (13)	0.44541 (12)	0.53301 (9)	0.0167 (3)
H13A	0.2216	0.5147	0.5566	0.020*
C14A	0.34920 (19)	0.70947 (19)	0.84427 (14)	0.0371 (4)
H14A	0.3467	0.6388	0.8974	0.056*
H14B	0.3425	0.7832	0.8658	0.056*
H14C	0.4326	0.6930	0.7987	0.056*
C1B	0.19028 (14)	0.75328 (13)	0.44920 (10)	0.0209 (3)
H1B	0.1108	0.7616	0.4309	0.025*
C2B	0.19295 (15)	0.82921 (13)	0.50364 (10)	0.0227 (3)
H2B1	0.1157	0.8890	0.5222	0.027*
C3B	0.30867 (15)	0.81782 (13)	0.53097 (10)	0.0212 (3)
C4B	0.42235 (15)	0.73175 (13)	0.50256 (10)	0.0213 (3)
H4B	0.5020	0.7244	0.5203	0.026*
C5B	0.41803 (14)	0.65622 (13)	0.44765 (10)	0.0193 (3)

H5B1	0.4958	0.5976	0.4281	0.023*
C6B	0.30300 (14)	0.66457 (12)	0.42082 (9)	0.0174 (3)
C7B	0.29994 (13)	0.57199 (12)	0.36881 (9)	0.0164 (3)
H7B	0.3914	0.5472	0.3314	0.020*
C8B	0.20893 (14)	0.62977 (12)	0.30071 (9)	0.0175 (3)
C9B	0.25441 (14)	0.69388 (13)	0.21136 (9)	0.0190 (3)
C10B	0.16993 (14)	0.74998 (13)	0.14914 (9)	0.0203 (3)
C11B	0.03791 (14)	0.74173 (13)	0.17682 (9)	0.0196 (3)
C12B	−0.01042 (13)	0.67990 (12)	0.26735 (9)	0.0177 (3)
C13B	0.07631 (13)	0.62514 (12)	0.32698 (9)	0.0174 (3)
H13B	0.0437	0.5828	0.3882	0.021*
C14B	0.41800 (18)	0.88299 (17)	0.61660 (14)	0.0351 (4)
H14D	0.4500	0.7970	0.6468	0.053*
H14E	0.3988	0.9356	0.6608	0.053*
H14F	0.4859	0.9083	0.5625	0.053*
N1	0.72534 (19)	0.56446 (18)	0.97160 (11)	0.0471 (4)
N2	0.49696 (13)	0.99649 (12)	0.19751 (9)	0.0254 (3)
N3	0.06071 (13)	0.05763 (12)	0.16428 (9)	0.0256 (3)
O1	0.55477 (17)	0.71894 (15)	1.01234 (10)	0.0533 (4)
O2	0.34118 (13)	1.15300 (11)	0.25381 (8)	0.0315 (3)
O3	−0.06606 (12)	0.10807 (11)	0.05755 (8)	0.0312 (3)
O1A	0.24155 (12)	0.72893 (11)	0.80213 (9)	0.0330 (3)
O2A	0.18662 (11)	0.12992 (9)	0.61961 (7)	0.0235 (2)
H2A2	0.2089	0.0738	0.5900	0.035*
O3A	0.24647 (13)	0.14031 (10)	0.43813 (8)	0.0291 (3)
H3A1	0.2755	0.1483	0.3804	0.044*
O4A	0.31128 (12)	0.36441 (10)	0.30769 (7)	0.0251 (2)
H4A1	0.3096	0.2997	0.2945	0.038*
O1B	0.30071 (11)	0.89459 (10)	0.58718 (8)	0.0264 (2)
O2B	0.38426 (10)	0.70251 (11)	0.18480 (7)	0.0253 (2)
H2B2	0.4065	0.7207	0.1265	0.038*
O3B	0.22783 (11)	0.81115 (12)	0.06305 (7)	0.0305 (3)
H3B1	0.1726	0.8390	0.0289	0.046*
O4B	−0.05048 (11)	0.79371 (11)	0.11931 (7)	0.0266 (2)
H4B1	−0.0101	0.8211	0.0654	0.040*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0588 (13)	0.0467 (11)	0.0240 (8)	−0.0203 (10)	−0.0031 (8)	−0.0009 (8)
C2	0.107 (2)	0.0506 (14)	0.0404 (12)	−0.0024 (14)	−0.0303 (13)	−0.0113 (10)
C3	0.0444 (14)	0.117 (3)	0.0488 (14)	0.0006 (15)	0.0047 (11)	0.0060 (15)
C4	0.0340 (8)	0.0234 (7)	0.0213 (7)	−0.0108 (6)	−0.0083 (6)	−0.0042 (5)
C5	0.0343 (9)	0.0232 (8)	0.0297 (8)	−0.0071 (6)	−0.0024 (7)	−0.0009 (6)
C6	0.0358 (9)	0.0375 (9)	0.0307 (9)	−0.0056 (7)	0.0024 (7)	−0.0108 (7)
C7	0.0279 (7)	0.0250 (7)	0.0201 (7)	−0.0068 (6)	−0.0046 (6)	−0.0035 (6)
C8	0.0402 (10)	0.0713 (14)	0.0295 (9)	−0.0312 (10)	−0.0048 (8)	−0.0003 (9)
C9	0.0398 (9)	0.0301 (8)	0.0246 (7)	−0.0091 (7)	−0.0144 (7)	0.0003 (6)

C1A	0.0214 (7)	0.0223 (7)	0.0233 (7)	−0.0036 (5)	−0.0063 (5)	−0.0063 (5)
C2A	0.0257 (7)	0.0213 (7)	0.0288 (7)	−0.0029 (6)	−0.0051 (6)	−0.0086 (6)
C3A	0.0276 (7)	0.0258 (7)	0.0229 (7)	−0.0104 (6)	−0.0025 (6)	−0.0087 (6)
C4A	0.0229 (7)	0.0309 (8)	0.0280 (7)	−0.0067 (6)	−0.0077 (6)	−0.0092 (6)
C5A	0.0229 (7)	0.0238 (7)	0.0243 (7)	−0.0023 (6)	−0.0070 (6)	−0.0075 (6)
C6A	0.0214 (6)	0.0198 (6)	0.0142 (6)	−0.0057 (5)	−0.0029 (5)	−0.0041 (5)
C7A	0.0190 (6)	0.0158 (6)	0.0153 (6)	−0.0034 (5)	−0.0048 (5)	−0.0024 (5)
C8A	0.0166 (6)	0.0168 (6)	0.0167 (6)	−0.0028 (5)	−0.0042 (5)	−0.0039 (5)
C9A	0.0194 (6)	0.0163 (6)	0.0195 (6)	−0.0039 (5)	−0.0041 (5)	−0.0023 (5)
C10A	0.0235 (7)	0.0162 (6)	0.0214 (7)	−0.0046 (5)	−0.0059 (5)	−0.0063 (5)
C11A	0.0212 (6)	0.0188 (6)	0.0169 (6)	−0.0039 (5)	−0.0044 (5)	−0.0047 (5)
C12A	0.0167 (6)	0.0156 (6)	0.0177 (6)	−0.0028 (5)	−0.0049 (5)	−0.0032 (5)
C13A	0.0164 (6)	0.0167 (6)	0.0173 (6)	−0.0032 (5)	−0.0047 (5)	−0.0039 (5)
C14A	0.0369 (9)	0.0446 (10)	0.0408 (10)	−0.0185 (8)	−0.0078 (8)	−0.0177 (8)
C1B	0.0210 (7)	0.0203 (7)	0.0219 (7)	−0.0037 (5)	−0.0075 (5)	−0.0037 (5)
C2B	0.0242 (7)	0.0189 (7)	0.0248 (7)	−0.0012 (5)	−0.0073 (6)	−0.0061 (5)
C3B	0.0274 (7)	0.0169 (6)	0.0208 (6)	−0.0054 (5)	−0.0077 (5)	−0.0040 (5)
C4B	0.0222 (7)	0.0201 (7)	0.0235 (7)	−0.0057 (5)	−0.0086 (5)	−0.0035 (5)
C5B	0.0197 (6)	0.0167 (6)	0.0206 (6)	−0.0041 (5)	−0.0045 (5)	−0.0028 (5)
C6B	0.0215 (6)	0.0150 (6)	0.0157 (6)	−0.0060 (5)	−0.0044 (5)	−0.0012 (5)
C7B	0.0175 (6)	0.0166 (6)	0.0150 (6)	−0.0046 (5)	−0.0036 (5)	−0.0026 (5)
C8B	0.0206 (6)	0.0163 (6)	0.0161 (6)	−0.0042 (5)	−0.0051 (5)	−0.0031 (5)
C9B	0.0195 (6)	0.0203 (6)	0.0177 (6)	−0.0064 (5)	−0.0034 (5)	−0.0037 (5)
C10B	0.0244 (7)	0.0207 (6)	0.0148 (6)	−0.0076 (5)	−0.0037 (5)	−0.0001 (5)
C11B	0.0224 (7)	0.0198 (6)	0.0161 (6)	−0.0036 (5)	−0.0063 (5)	−0.0024 (5)
C12B	0.0196 (6)	0.0171 (6)	0.0165 (6)	−0.0044 (5)	−0.0037 (5)	−0.0040 (5)
C13B	0.0206 (6)	0.0160 (6)	0.0157 (6)	−0.0049 (5)	−0.0042 (5)	−0.0025 (5)
C14B	0.0370 (9)	0.0328 (9)	0.0455 (10)	−0.0007 (7)	−0.0216 (8)	−0.0190 (8)
N1	0.0497 (10)	0.0558 (11)	0.0290 (8)	−0.0091 (8)	−0.0030 (7)	−0.0075 (7)
N2	0.0300 (7)	0.0242 (6)	0.0215 (6)	−0.0087 (5)	−0.0025 (5)	−0.0044 (5)
N3	0.0282 (7)	0.0284 (7)	0.0192 (6)	−0.0086 (5)	−0.0058 (5)	−0.0009 (5)
O1	0.0629 (10)	0.0495 (9)	0.0272 (7)	−0.0027 (7)	0.0004 (6)	0.0019 (6)
O2	0.0435 (7)	0.0260 (6)	0.0260 (6)	−0.0065 (5)	−0.0055 (5)	−0.0107 (5)
O3	0.0335 (6)	0.0380 (7)	0.0199 (5)	−0.0065 (5)	−0.0083 (5)	−0.0023 (5)
O1A	0.0337 (6)	0.0313 (6)	0.0425 (7)	−0.0105 (5)	−0.0085 (5)	−0.0176 (5)
O2A	0.0331 (6)	0.0155 (5)	0.0211 (5)	−0.0083 (4)	−0.0021 (4)	−0.0039 (4)
O3A	0.0482 (7)	0.0205 (5)	0.0219 (5)	−0.0136 (5)	−0.0038 (5)	−0.0076 (4)
O4A	0.0392 (6)	0.0203 (5)	0.0163 (5)	−0.0078 (5)	−0.0037 (4)	−0.0060 (4)
O1B	0.0314 (6)	0.0217 (5)	0.0310 (6)	−0.0013 (4)	−0.0142 (5)	−0.0110 (4)
O2B	0.0210 (5)	0.0356 (6)	0.0181 (5)	−0.0118 (4)	−0.0031 (4)	0.0002 (4)
O3B	0.0273 (6)	0.0433 (7)	0.0171 (5)	−0.0147 (5)	−0.0058 (4)	0.0068 (5)
O4B	0.0242 (5)	0.0346 (6)	0.0181 (5)	−0.0085 (5)	−0.0081 (4)	0.0040 (4)

Geometric parameters (Å, °)

C1—O1	1.224 (3)	C8A—C13A	1.3968 (18)
C1—N1	1.324 (3)	C9A—O2A	1.3736 (16)
C1—H1	0.9500	C9A—C10A	1.3976 (19)
C2—N1	1.438 (3)	C10A—O3A	1.3773 (16)

C2—H2A	0.9800	C10A—C11A	1.3959 (19)
C2—H2B	0.9800	C11A—O4A	1.3740 (16)
C2—H2C	0.9800	C11A—C12A	1.4019 (18)
C3—N1	1.451 (3)	C12A—C13A	1.3957 (18)
C3—H3A	0.9800	C12A—C7B	1.5336 (18)
C3—H3B	0.9800	C13A—H13A	0.9500
C3—H3C	0.9800	C14A—O1A	1.422 (2)
C4—O2	1.241 (2)	C14A—H14A	0.9800
C4—N2	1.318 (2)	C14A—H14B	0.9800
C4—H4	0.9500	C14A—H14C	0.9800
C5—N2	1.462 (2)	C1B—C2B	1.389 (2)
C5—H5A	0.9800	C1B—C6B	1.402 (2)
C5—H5B	0.9800	C1B—H1B	0.9500
C5—H5C	0.9800	C2B—C3B	1.392 (2)
C6—N2	1.460 (2)	C2B—H2B1	0.9500
C6—H6A	0.9800	C3B—O1B	1.3788 (17)
C6—H6B	0.9800	C3B—C4B	1.391 (2)
C6—H6C	0.9800	C4B—C5B	1.397 (2)
C7—O3	1.2429 (19)	C4B—H4B	0.9500
C7—N3	1.320 (2)	C5B—C6B	1.3897 (19)
C7—H7	0.9500	C5B—H5B1	0.9500
C8—N3	1.453 (2)	C6B—C7B	1.5243 (18)
C8—H8A	0.9800	C7B—C8B	1.5218 (18)
C8—H8B	0.9800	C7B—H7B	1.0000
C8—H8C	0.9800	C8B—C9B	1.3899 (19)
C9—N3	1.456 (2)	C8B—C13B	1.3916 (19)
C9—H9A	0.9800	C9B—O2B	1.3755 (17)
C9—H9B	0.9800	C9B—C10B	1.403 (2)
C9—H9C	0.9800	C10B—O3B	1.3801 (16)
C1A—C2A	1.388 (2)	C10B—C11B	1.397 (2)
C1A—C6A	1.400 (2)	C11B—O4B	1.3786 (17)
C1A—H1A	0.9500	C11B—C12B	1.4015 (19)
C2A—C3A	1.388 (2)	C12B—C13B	1.3907 (19)
C2A—H2A1	0.9500	C12B—C7A^{i}	1.5237 (19)
C3A—O1A	1.3740 (18)	C13B—H13B	0.9500
C3A—C4A	1.388 (2)	C14B—O1B	1.4244 (19)
C4A—C5A	1.398 (2)	C14B—H14D	0.9800
C4A—H4A	0.9500	C14B—H14E	0.9800
C5A—C6A	1.387 (2)	C14B—H14F	0.9800
C5A—H5A1	0.9500	O2A—H2A2	0.8400
C6A—C7A	1.5235 (18)	O3A—H3A1	0.8400
C7A—C12B^{i}	1.5237 (19)	O4A—H4A1	0.8400
C7A—C8A	1.5298 (18)	O2B—H2B2	0.8400
C7A—H7A	1.0000	O3B—H3B1	0.8400
C8A—C9A	1.3936 (19)	O4B—H4B1	0.8400
O1—C1—N1	124.4 (2)	O4A—C11A—C12A	117.35 (12)
O1—C1—H1	117.8	C10A—C11A—C12A	120.05 (12)
N1—C1—H1	117.8	C13A—C12A—C11A	118.47 (12)

N1—C2—H2A	109.5	C13A—C12A—C7B	122.12 (12)
N1—C2—H2B	109.5	C11A—C12A—C7B	119.40 (12)
H2A—C2—H2B	109.5	C12A—C13A—C8A	122.58 (12)
N1—C2—H2C	109.5	C12A—C13A—H13A	118.7
H2A—C2—H2C	109.5	C8A—C13A—H13A	118.7
H2B—C2—H2C	109.5	O1A—C14A—H14A	109.5
N1—C3—H3A	109.5	O1A—C14A—H14B	109.5
N1—C3—H3B	109.5	H14A—C14A—H14B	109.5
H3A—C3—H3B	109.5	O1A—C14A—H14C	109.5
N1—C3—H3C	109.5	H14A—C14A—H14C	109.5
H3A—C3—H3C	109.5	H14B—C14A—H14C	109.5
H3B—C3—H3C	109.5	C2B—C1B—C6B	121.00 (13)
O2—C4—N2	125.68 (15)	C2B—C1B—H1B	119.5
O2—C4—H4	117.2	C6B—C1B—H1B	119.5
N2—C4—H4	117.2	C1B—C2B—C3B	120.04 (13)
N2—C5—H5A	109.5	C1B—C2B—H2B1	120.0
N2—C5—H5B	109.5	C3B—C2B—H2B1	120.0
H5A—C5—H5B	109.5	O1B—C3B—C4B	124.09 (13)
N2—C5—H5C	109.5	O1B—C3B—C2B	115.86 (13)
H5A—C5—H5C	109.5	C4B—C3B—C2B	120.04 (13)
H5B—C5—H5C	109.5	C3B—C4B—C5B	119.19 (13)
N2—C6—H6A	109.5	C3B—C4B—H4B	120.4
N2—C6—H6B	109.5	C5B—C4B—H4B	120.4
H6A—C6—H6B	109.5	C6B—C5B—C4B	121.76 (13)
N2—C6—H6C	109.5	C6B—C5B—H5B1	119.1
H6A—C6—H6C	109.5	C4B—C5B—H5B1	119.1
H6B—C6—H6C	109.5	C5B—C6B—C1B	117.96 (12)
O3—C7—N3	125.13 (15)	C5B—C6B—C7B	119.63 (12)
O3—C7—H7	117.4	C1B—C6B—C7B	122.23 (12)
N3—C7—H7	117.4	C8B—C7B—C6B	111.47 (11)
N3—C8—H8A	109.5	C8B—C7B—C12A	112.53 (11)
N3—C8—H8B	109.5	C6B—C7B—C12A	110.97 (11)
H8A—C8—H8B	109.5	C8B—C7B—H7B	107.2
N3—C8—H8C	109.5	C6B—C7B—H7B	107.2
H8A—C8—H8C	109.5	C12A—C7B—H7B	107.2
H8B—C8—H8C	109.5	C9B—C8B—C13B	117.81 (12)
N3—C9—H9A	109.5	C9B—C8B—C7B	120.86 (12)
N3—C9—H9B	109.5	C13B—C8B—C7B	121.27 (12)
H9A—C9—H9B	109.5	O2B—C9B—C8B	118.78 (12)
N3—C9—H9C	109.5	O2B—C9B—C10B	120.17 (12)
H9A—C9—H9C	109.5	C8B—C9B—C10B	121.04 (13)
H9B—C9—H9C	109.5	O3B—C10B—C11B	125.18 (13)
C2A—C1A—C6A	121.08 (14)	O3B—C10B—C9B	114.94 (13)
C2A—C1A—H1A	119.5	C11B—C10B—C9B	119.88 (12)
C6A—C1A—H1A	119.5	O4B—C11B—C10B	123.10 (12)
C3A—C2A—C1A	120.07 (14)	O4B—C11B—C12B	117.05 (12)
C3A—C2A—H2A1	120.0	C10B—C11B—C12B	119.85 (13)
C1A—C2A—H2A1	120.0	C13B—C12B—C11B	118.63 (13)
O1A—C3A—C4A	124.20 (14)	C13B—C12B—C7A^{i}	120.78 (12)

O1A—C3A—C2A	115.68 (14)	C11B—C12B—C7A^{i}	120.58 (12)
C4A—C3A—C2A	120.12 (14)	C12B—C13B—C8B	122.76 (13)
C3A—C4A—C5A	118.97 (14)	C12B—C13B—H13B	118.6
C3A—C4A—H4A	120.5	C8B—C13B—H13B	118.6
C5A—C4A—H4A	120.5	O1B—C14B—H14D	109.5
C6A—C5A—C4A	122.06 (14)	O1B—C14B—H14E	109.5
C6A—C5A—H5A1	119.0	H14D—C14B—H14E	109.5
C4A—C5A—H5A1	119.0	O1B—C14B—H14F	109.5
C5A—C6A—C1A	117.67 (13)	H14D—C14B—H14F	109.5
C5A—C6A—C7A	120.40 (12)	H14E—C14B—H14F	109.5
C1A—C6A—C7A	121.84 (12)	C1—N1—C2	122.4 (2)
C6A—C7A—C12B^{i}	111.99 (11)	C1—N1—C3	121.5 (2)
C6A—C7A—C8A	111.96 (11)	C2—N1—C3	116.0 (2)
C12B^{i}—C7A—C8A	110.42 (11)	C4—N2—C6	120.99 (14)
C6A—C7A—H7A	107.4	C4—N2—C5	121.72 (13)
C12B^{i}—C7A—H7A	107.4	C6—N2—C5	117.27 (14)
C8A—C7A—H7A	107.4	C7—N3—C8	121.29 (14)
C9A—C8A—C13A	117.73 (12)	C7—N3—C9	122.09 (14)
C9A—C8A—C7A	119.38 (12)	C8—N3—C9	116.61 (14)
C13A—C8A—C7A	122.79 (12)	C3A—O1A—C14A	116.79 (13)
O2A—C9A—C8A	119.20 (12)	C9A—O2A—H2A2	109.5
O2A—C9A—C10A	119.68 (12)	C10A—O3A—H3A1	109.5
C8A—C9A—C10A	121.11 (12)	C11A—O4A—H4A1	109.5
O3A—C10A—C11A	125.35 (13)	C3B—O1B—C14B	116.51 (12)
O3A—C10A—C9A	114.62 (12)	C9B—O2B—H2B2	109.5
C11A—C10A—C9A	120.02 (12)	C10B—O3B—H3B1	109.5
O4A—C11A—C10A	122.58 (12)	C11B—O4B—H4B1	109.5
C6A—C1A—C2A—C3A	−0.4 (2)	C4B—C5B—C6B—C7B	−174.18 (12)
C1A—C2A—C3A—O1A	−178.20 (14)	C2B—C1B—C6B—C5B	−1.0 (2)
C1A—C2A—C3A—C4A	1.7 (2)	C2B—C1B—C6B—C7B	174.18 (13)
O1A—C3A—C4A—C5A	178.30 (14)	C5B—C6B—C7B—C8B	−147.78 (12)
C2A—C3A—C4A—C5A	−1.6 (2)	C1B—C6B—C7B—C8B	37.16 (17)
C3A—C4A—C5A—C6A	0.2 (2)	C5B—C6B—C7B—C12A	85.95 (15)
C4A—C5A—C6A—C1A	1.0 (2)	C1B—C6B—C7B—C12A	−89.11 (15)
C4A—C5A—C6A—C7A	177.66 (13)	C13A—C12A—C7B—C8B	−119.55 (14)
C2A—C1A—C6A—C5A	−0.9 (2)	C11A—C12A—C7B—C8B	61.78 (16)
C2A—C1A—C6A—C7A	−177.51 (13)	C13A—C12A—C7B—C6B	6.13 (18)
C5A—C6A—C7A—C12B^{i}	146.61 (13)	C11A—C12A—C7B—C6B	−172.53 (12)
C1A—C6A—C7A—C12B^{i}	−36.85 (17)	C6B—C7B—C8B—C9B	85.55 (15)
C5A—C6A—C7A—C8A	−88.73 (15)	C12A—C7B—C8B—C9B	−149.04 (13)
C1A—C6A—C7A—C8A	87.80 (16)	C6B—C7B—C8B—C13B	−91.47 (15)
C6A—C7A—C8A—C9A	167.71 (12)	C12A—C7B—C8B—C13B	33.94 (17)
C12B^{i}—C7A—C8A—C9A	−66.78 (16)	C13B—C8B—C9B—O2B	178.24 (12)
C6A—C7A—C8A—C13A	−15.94 (18)	C7B—C8B—C9B—O2B	1.1 (2)
C12B^{i}—C7A—C8A—C13A	109.57 (14)	C13B—C8B—C9B—C10B	−1.2 (2)
C13A—C8A—C9A—O2A	179.57 (12)	C7B—C8B—C9B—C10B	−178.36 (13)
C7A—C8A—C9A—O2A	−3.90 (19)	O2B—C9B—C10B—O3B	0.1 (2)

C13A—C8A—C9A—C10A	−1.4 (2)	C8B—C9B—C10B—O3B	179.62 (13)
C7A—C8A—C9A—C10A	175.16 (13)	O2B—C9B—C10B—C11B	−179.47 (13)
O2A—C9A—C10A—O3A	1.1 (2)	C8B—C9B—C10B—C11B	0.0 (2)
C8A—C9A—C10A—O3A	−177.96 (13)	O3B—C10B—C11B—O4B	0.9 (2)
O2A—C9A—C10A—C11A	−178.68 (13)	C9B—C10B—C11B—O4B	−179.53 (13)
C8A—C9A—C10A—C11A	2.3 (2)	O3B—C10B—C11B—C12B	−178.10 (14)
O3A—C10A—C11A—O4A	−2.7 (2)	C9B—C10B—C11B—C12B	1.5 (2)
C9A—C10A—C11A—O4A	177.09 (13)	O4B—C11B—C12B—C13B	179.29 (12)
O3A—C10A—C11A—C12A	178.87 (13)	C10B—C11B—C12B—C13B	−1.7 (2)
C9A—C10A—C11A—C12A	−1.4 (2)	O4B—C11B—C12B—C7A^{i}	0.19 (19)
O4A—C11A—C12A—C13A	−178.86 (12)	C10B—C11B—C12B—C7A^{i}	179.25 (12)
C10A—C11A—C12A—C13A	−0.3 (2)	C11B—C12B—C13B—C8B	0.4 (2)
O4A—C11A—C12A—C7B	−0.15 (19)	C7A^{i}—C12B—C13B—C8B	179.48 (12)
C10A—C11A—C12A—C7B	178.40 (12)	C9B—C8B—C13B—C12B	1.1 (2)
C11A—C12A—C13A—C8A	1.2 (2)	C7B—C8B—C13B—C12B	178.16 (12)
C7B—C12A—C13A—C8A	−177.46 (12)	O1—C1—N1—C2	−0.4 (3)
C9A—C8A—C13A—C12A	−0.4 (2)	O1—C1—N1—C3	−179.3 (2)
C7A—C8A—C13A—C12A	−176.78 (12)	O2—C4—N2—C6	178.74 (16)
C6B—C1B—C2B—C3B	−0.1 (2)	O2—C4—N2—C5	0.5 (3)
C1B—C2B—C3B—O1B	−178.13 (13)	O3—C7—N3—C8	0.6 (3)
C1B—C2B—C3B—C4B	1.0 (2)	O3—C7—N3—C9	−178.20 (16)
O1B—C3B—C4B—C5B	178.18 (13)	C4A—C3A—O1A—C14A	−11.8 (2)
C2B—C3B—C4B—C5B	−0.9 (2)	C2A—C3A—O1A—C14A	168.08 (15)
C3B—C4B—C5B—C6B	−0.2 (2)	C4B—C3B—O1B—C14B	0.2 (2)
C4B—C5B—C6B—C1B	1.1 (2)	C2B—C3B—O1B—C14B	179.30 (14)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4B—H4B1···O3B	0.84	2.54	2.9446 (16)	111
O4B—H4B1···O3^{ii}	0.84	1.89	2.7231 (15)	175
O3B—H3B1···O4B	0.84	2.55	2.9446 (16)	110
O3B—H3B1···O3^{ii}	0.84	1.86	2.6971 (16)	173
O2B—H2B2···O3B	0.84	2.26	2.6773 (15)	111
O2B—H2B2···O1^{iii}	0.84	2.02	2.7595 (18)	147
O4A—H4A1···O3A	0.84	2.52	2.9334 (15)	112
O4A—H4A1···O2^{iv}	0.84	1.89	2.7158 (15)	169
O3A—H3A1···O4A	0.84	2.54	2.9334 (15)	110
O3A—H3A1···O2^{iv}	0.84	1.86	2.7010 (16)	176
O2A—H2A2···O3A	0.84	2.20	2.6523 (15)	114
O2A—H2A2···O1B^{iv}	0.84	2.04	2.7779 (15)	147

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

Fig. 1

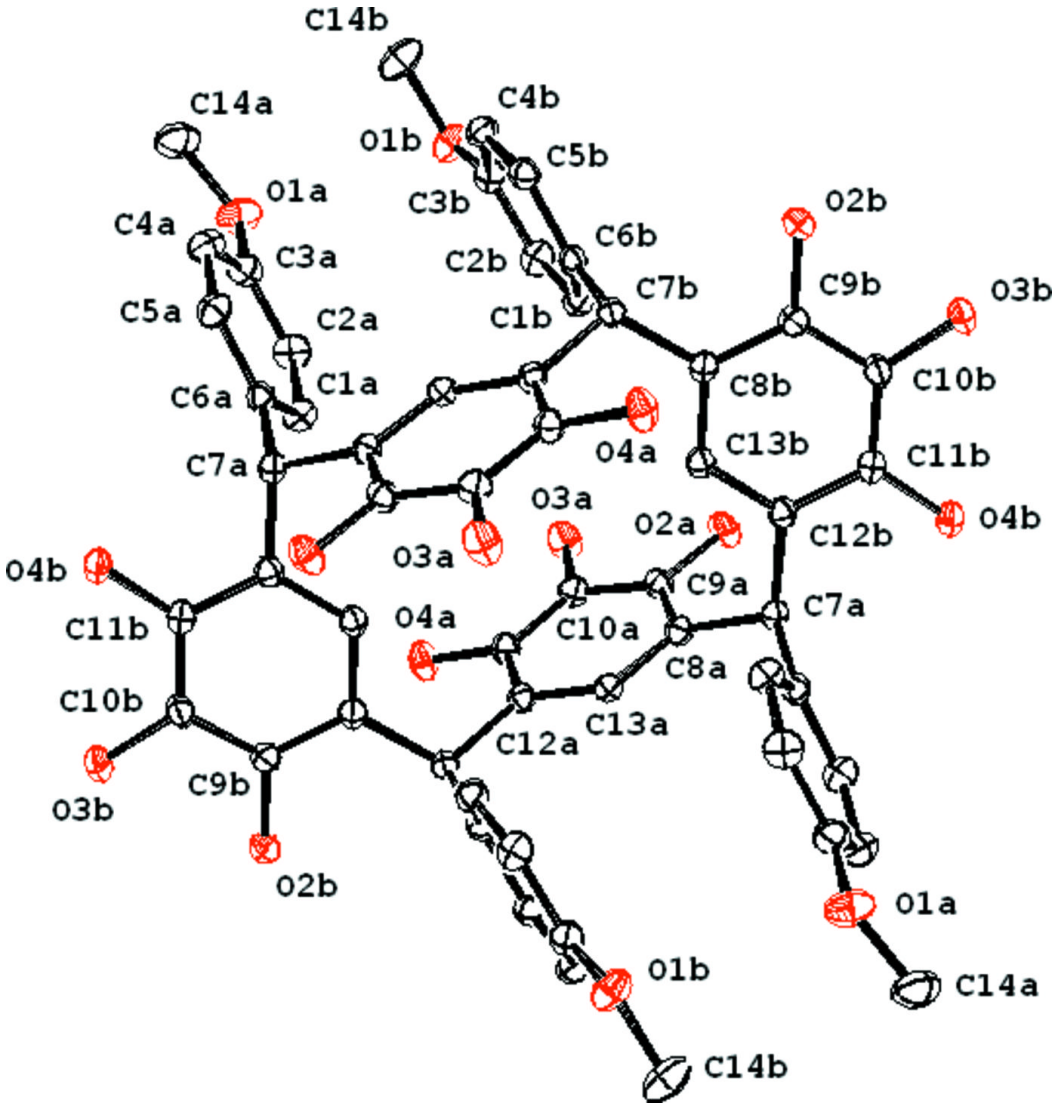
C14b
C4b
C14a
O1b
C5b
O1a
C3b
O2b
C4a
C6b
C3a
C2b
C7b
C9b
O3b
C5a
C2a
C1b
C10b
C1a
C8b
C6a
O4a
C13b
C11b
C7a
O3a
O2a
C12b
O4b
O3a
O4b
C9a
C7a
C11b
O4a
C10a
C8a
C10b
C13a
C12a
C9b
O3b
O2b
O1a
O1b
C14a
C14b

Fig. 2