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2,8,14,20-Tetraphenylpyrogallol[4]arene dimethylformamide octasolvate

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
Disorder in solvent or counterion
R factor = 0.058
wR factor = 0.169
Data-to-parameter ratio = 18.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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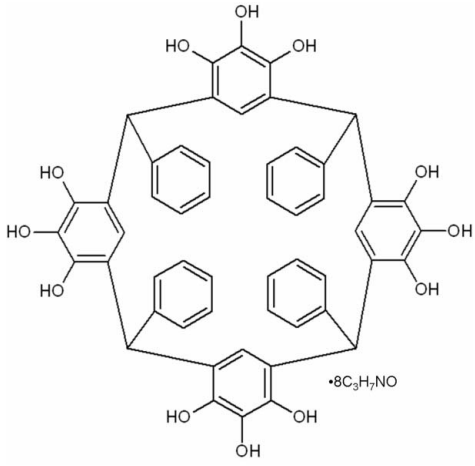
The title compound, $C_{52}H_{40}O_{12}\cdot 8C_3H_7NO$, contains substituted pyrogallolarene molecules accompanied by dimethylformamide solvent molecules. The complete main molecule displays a chair structure and has inversion symmetry. A network of O—H···O hydrogen bonds helps to consolidate the crystal packing.

Comment

Bowl-shaped compounds such as pyrogallolarenes have received considerable attention over the past 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 & Sherman, 2005, and references therein). Our investigations have shown that aryl-substituted pyrogallol[4]arenes adopt a chair (*rctt*) conformation (Zambrano *et al.*, 2005), whereas the alkyl-substituted analogs adopt the crown (*rccc*) structure (Dueno *et al.*, 2006). We report here the crystal structure of the title compound, (I),



(I)

The X-ray analysis of (I) shows (Fig. 1) that the geometry of the main molecule is consistent with the chair (*rctt*) structure, where two opposite pyrogallol rings are essentially coplanar but have their –OH groups pointing away from each other. The other two pyrogallol fragments are perpendicular to the aforementioned rings, where each set of three –OH groups is also pointing in opposite directions. The complete molecule is generated by inversion symmetry from the asymmetric unit. The asymmetric unit of (I) also contains four independent molecules of dimethylformamide (DMF) (*i.e.* eight DMF molecules per main molecule), one of which is disordered.

A network of O—H···O hydrogen bonds (Table 1) helps to consolidate the crystal packing. These include bonds from –OH donors to DMF-O-atom acceptors and two intramolecular bonds (O1*A*—H1*A*···O2*A* and O1*B*—H1*B*···O2*B*). All geometrical values are consistent with literature data (Cave *et al.*, 2005).

Experimental

A 50 ml round-bottomed flask was charged with pyrogallol (2.0 g, 16 mmol) and 95% ethanol (11 ml). The reaction vessel was cooled in an ice-bath to 273 K and concentrated HCl (2 ml) was added in one portion. Benzaldehyde (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-tetra(phenyl)pyrogallol[4]arene (yield 70%, m.p. 632–633 K). Crystals of (I) were grown by vapor diffusion of diethyl ether into a dimethylformamide solution.

Crystal data

$C_{52}H_{40}O_{12}\cdot 8C_3H_7NO$
M_r = 1441.61
Triclinic, $P\overline{1}$
a = 12.199 (4) Å
b = 12.397 (4) Å
c = 13.712 (4) Å
α = 105.282 (5)°
β = 101.099 (5)°
γ = 106.854 (5)°
V = 1831.5 (9) Å^3
Z = 1
D_x = 1.307 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100 (2) K
Plate, colorless
0.5 × 0.4 × 0.07 mm

Data collection

Bruker SMART APEX CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS* in *SAINT-Plus*; Bruker, 2002)
T_{min} = 0.768, T_{max} = 1
18122 measured reflections
8997 independent reflections
5924 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 28.3°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.169
S = 1.04
8997 reflections
501 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0871P)^2 + 0.3671P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1*A*—H1*A*···O2	0.84	2.05	2.805 (5)	149
O1*A*—H1*A*···O2*A*	0.84	2.21	2.671 (2)	115
O2*A*—H2*A*···O1^{iii}	0.84	1.82	2.661 (2)	175
O3*A*—H3*A*···O1^{iii}	0.84	1.90	2.739 (2)	180
O1*B*—H1*B*···O4^{ii}	0.84	1.95	2.702 (2)	149
O1*B*—H1*B*···O2*B*	0.84	2.29	2.706 (2)	111
O2*B*—H2*B*···O3^{i}	0.84	1.88	2.722 (2)	177
O3*B*—H3*B*···O3^{i}	0.84	1.90	2.735 (2)	174

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

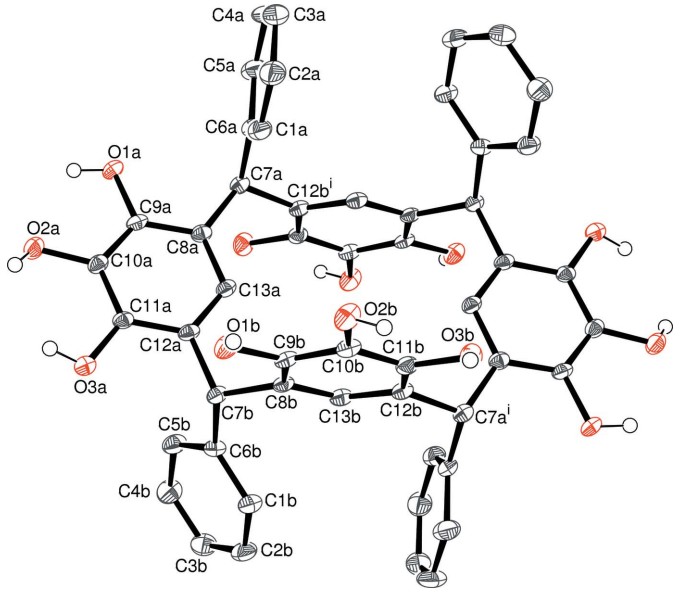


Figure 1
View of the main molecule of (I) showing 50% displacement ellipsoids (arbitrary spheres for the O-bound H atoms, other H atoms omitted for clarity). [Symmetry code: (i) $2-x, 1-y, 2-z$.]

One of the four solvent DMF molecules is flip-disordered with a refined occupancy ratio of 0.863 (3):0.137 (3). Equivalent atoms within the disordered molecule were set to have identical anisotropic displacement parameters and overlapping atoms (O2, O2*C*, N2, N2*C*, C5, C5*C*) were restrained to be isotropic within a standard deviation of 0.1 Å^2. All H atoms were positioned geometrically (C—H = 0.95–1.00 Å, O—H = 0.84 Å) and were refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C, hydroxyl O).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT-Plus* (Bruker, 2002); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 2003); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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