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4-Hydroxy-*N*,*N*-bis(2-hydroxy-3-methoxybenzyl)aniline

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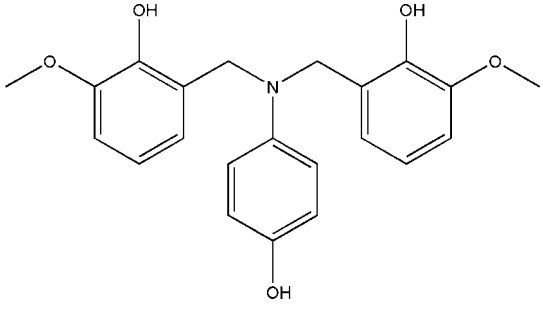
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.050; wR factor = 0.067; data-to-parameter ratio = 13.2.

The title compound, $C_{22}H_{23}NO_5$, crystallizes with two independent molecules in the asymmetric unit. In one molecule, the dihedral angle between one outer ring and the central ring is 86.37 (10)°, while that between the two outer rings is 88.99 (10)°; in the other molecule, these angles are 87.37 (9) and 88.14 (9)°, respectively. The molecules are linked into two chains by a combination of O—H···O and C—H···O hydrogen bonds.

Related literature

For related literature, see: Bernstein *et al.* (1995); Liu *et al.* (2007); Xia *et al.* (2007).



Experimental

Crystal data

$C_{22}H_{23}NO_5$
M_r = 381.41
Triclinic, $P\overline{1}$
a = 10.4059 (19) Å
b = 13.699 (2) Å
c = 15.196 (2) Å
α = 113.549 (3)°
β = 96.908 (2)°
γ = 99.792 (2)°
V = 1914.0 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
0.24 × 0.17 × 0.15 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.978, T_{max} = 0.986
10128 measured reflections
6656 independent reflections
2772 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.067
S = 0.89
6656 reflections
505 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O9^{i}	0.82	2.38	3.185 (3)	168
O1—H1···O10^{i}	0.82	2.35	2.911 (3)	127
O2—H2···N1	0.82	1.97	2.697 (3)	147
O4—H4···O5	0.82	2.19	2.640 (3)	115
O4—H4···O6^{ii}	0.82	2.49	2.941 (3)	116
O6—H6···O2^{iii}	0.82	2.10	2.913 (3)	171
O6—H6···O3^{iii}	0.82	2.50	3.008 (3)	121
O7—H7···O1^{iv}	0.82	2.11	2.835 (3)	148
O7—H7···O8	0.82	2.17	2.629 (3)	115
O9—H9···O7	0.82	2.45	3.023 (3)	128
O9—H9···N2	0.82	2.04	2.753 (3)	146
C5—H5···O7^{ii}	0.93	2.50	3.218 (4)	134

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); 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: AT2396).

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supplementary materials

Acta Cryst. (2007). E**63**, o4070 [**doi:10.1107/S1600536807044558**]

4-Hydroxy-*N*,*N*-bis(2-hydroxy-3-methoxybenzyl)aniline

Y.-F. Liu, H.-T. Xia, D.-Q. Wang, S.-P. Yang and Y.-L. Meng

Comment

We have recently reported the supramolecular structures of amine derivatives containing hydroxy and methoxy groups, for example, *N*-(2-hydroxy-3-methoxybenzyl)-4-chloroaniline (Liu *et al.*, 2007), and 6,6'-dimethoxy-2,2'-(2,2,4-trimethylimidazolidine-1,3-diyldimethylene)diphenol (Xia *et al.*, 2007). We have now continued our studies in this area with the title compound, (I), an aniline derivative containing hydroxy and methoxy groups.

Compound (I) crystallizes with the two independent molecules in the asymmetric unit and $Z = 4$ in the triclinic space group $P\bar{1}$ (Fig. 1). In the molecules, the dihedral angles between the phenyl ring (C1–C5) and other two phenyl rings (C8–C13) and (C16–C21) are 86.37 (10)° and 39.09 (10)°, respectively, and the dihedral angles between the phenyl ring (C23–C28) and other two phenyl rings (C30–C35) and (C38–C43) are 48.28 (9)° and 87.37 (9)°, respectively. The C—N bonds involving atom N1 are longer than those involving atom N2 (excluding C15—N1 and C29—N2 bond). The bond angles around the N1 atom are almost in agreement with those around the N2 atom.

The molecules of (I) are linked by O—H···O and C—H···O hydrogen bonds (Table 2) into centrosymmetric tetramers, and these tetramers are further linked by O—H···O, C—H···O hydrogen bonds to form two chains. Atom O6 in the molecule at $(1 - x, 2 - y, 1 - z)$ act as a hydrogen-bond donors, respectively, to atoms O2 and O3 in the molecule at (x, y, z). At the same time, atom O4 in the molecule at (x, y, z) acts as hydrogen-bond donor to atom O6 in the molecule at $(x, -1 + y, z)$, so generating $R_4^4(26)$ and $R_4^4(24)$ (Bernstein *et al.*, 1995) tetramers centred at (1/2, 1/2, 1/2) (Fig. 2). The tetramers are linked into a $[01\bar{1}]$ chain by O—H···O and C—H···O hydrogen bonds. Atom O1 in the molecules at (x, y, z) act as donors, respectively, to atoms O9 and O10 in the molecules at $(-x, 1 - y, -z)$, atoms C5 and O7 in the molecule at (x, y, z) and $(x, -1 + y, z)$ act as hydrogen-bond donors, respectively, to atoms O7 and O1 in the molecule at $(x, -1 + y, z)$ and (x, y, z). Propagation by inversion and translation of these four interactions then generates a chain running parallel to the $[01\bar{1}]$ direction containing $R_4^4(24)$ rings and $R_4^4(30)$ rings centred at (n, n, n) (n = zero or integer), $R_4^4(26)$ and $R_4^4(24)$ rings centred at (1/2 + n, 1/2 + n, 1/2 + n) (n = zero or integer) (Fig. 3). In a similar way, the tetramers are linked into a chain by O—H···O and C—H···O hydrogen bonds parallel to the *a* axis direction. Atom O6 in the molecule at $(-1 + x, -1 + y, z)$ and $(1 - x, 2 - y, 1 - z)$ accept hydrogen bond from atom C14 in the molecule at (x, y, z), and at the same time, acts as donor to atom O3 in the molecule at (x, y, z), respectively, propagation by inversion and translation of these two interactions then generates a chain running parallel to the *a* axis direction containing $R_4^4(10)$ rings centred at (n, 1/2, 1/2) (n = zero or integer), $R_4^4(26)$ and $R_4^4(24)$ rings centred at (1/2 + n, 1/2, 1/2) (n = zero or integer) (Fig. 4).

Experimental

A solution of 3-methoxysalicylaldehyde (1.52 g, 10 mmol) and 4-hydroxy aniline (1.09 g, 10 mmol) in ethanol -chloroform (*v*/*v* = 1/1) (30 ml) was stirred for 8 h and $NaBH_4$ (0.756 g, 20 mmol) were added, the mixture solution was stirred under

room temperature for 48 h and then mixtures was filtered. The filtrate was allowed to evaporate slowly, giving single crystals of (I) suitable for X-ray structure analysis..

Refinement

All H atoms were located in difference Fourier maps and then repositioned geometrically. They were refined as riding, with C—H distances of 0.93 (aryl), 0.96 (methyl) or 0.97 Å (methylene) and O—H = 0.82 Å. U_{iso}(H) values were set at xU_{eq}(carrier atom), where x = 1.5 for methyl and hydroxy, x = 1.2 for the other H atoms.

Figures

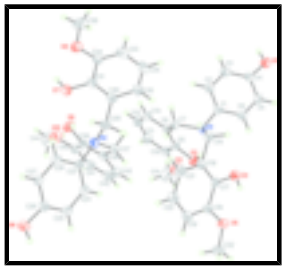

Fig. 1. The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are at the 30% probability level.

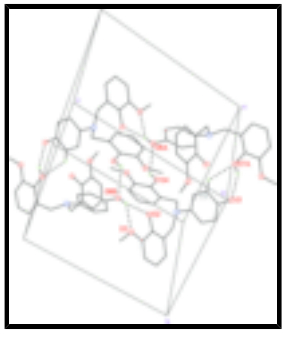

Fig. 2. A larger portion of the crystal structure of (I), showing the formation of a hydrogen-bonded tetramers built from C—H···O and O—H···O. For clarity, H atoms not involved in the hydrogen bonding have been omitted. Dashed lines indicate hydrogen bonds. [Symmetry codes: (A) x, 1 − y, z, (B) 1 − x, 2 − y, 1 − z].

Fig. 3. A larger portion of the crystal structure of (I), showing the formation of a hydrogen-bonded chains built from C—H···O and O—H···O. For clarity, H atoms not involved in the hydrogen bonding have been omitted. Dashed lines indicate hydrogen bonds.

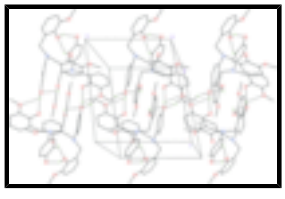

Fig. 4. A larger portion of the crystal structure of (I), showing the formation of a hydrogen-bonded chains built from C—H···O and O—H···O. For clarity, H atoms not involved in the hydrogen bonding have been omitted. Dashed lines indicate hydrogen bonds.

4-Hydroxy-*N*,*N*-bis(2-hydroxy-3-methoxybenzyl)aniline

Crystal data

$C_{22}H_{23}NO_5$	Z = 4
M_r = 381.41	F_{000} = 808
Triclinic, $P\overline{1}$	D_x = 1.324 Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation

	λ = 0.71073 Å
a = 10.4059 (19) Å	Cell parameters from 1304 reflections
b = 13.699 (2) Å	θ = 2.3–27.8º
c = 15.196 (2) Å	μ = 0.09 mm^{-1}
α = 113.549 (3)º	*T* = 298 (2) K
β = 96.908 (2)º	Block, colourless
γ = 99.792 (2)º	0.24 × 0.17 × 0.15 mm
V = 1914.0 (5) $Å^3$	

Data collection

Siemens SMART 1000 CCD area-detector diffractometer	6656 independent reflections
Radiation source: fine-focus sealed tube	2772 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.041
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.5º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −12→12
T_{min} = 0.978, T_{max} = 0.986	*k* = −16→7
10128 measured reflections	*l* = −18→18

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.050	H-atom parameters constrained
$wR(F^2)$ = 0.067	$w = 1/[\sigma^2(F_o^2)]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.89	$(\Delta/\sigma)_{max}$ = 0.012
6656 reflections	$\Delta\rho_{max}$ = 0.15 e $Å^{-3}$
505 parameters	$\Delta\rho_{min}$ = −0.17 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}
N1	0.3458 (2)	0.5001 (2)	0.18136 (16)	0.0403 (7)
N2	0.4358 (2)	0.9610 (2)	0.19252 (16)	0.0401 (7)
O1	0.1094 (2)	0.05510 (17)	−0.04810 (14)	0.0731 (8)
H1	0.0418	0.0440	−0.0879	0.110*
O2	0.24705 (18)	0.55357 (17)	0.34615 (12)	0.0549 (7)
H2	0.3008	0.5341	0.3113	0.082*
O3	0.0467 (2)	0.5905 (2)	0.42750 (15)	0.0678 (8)
O4	0.5303 (2)	0.49388 (16)	0.35279 (14)	0.0595 (7)
H4	0.5355	0.4929	0.4067	0.089*
O5	0.6420 (2)	0.64758 (17)	0.53099 (15)	0.0599 (7)
O6	0.6991 (2)	1.39497 (17)	0.44554 (14)	0.0664 (7)
H6	0.7205	1.4046	0.5027	0.100*
O7	0.30272 (19)	0.95072 (16)	−0.00158 (13)	0.0532 (6)
H7	0.2383	0.9564	−0.0344	0.080*
O8	0.1087 (2)	0.80271 (18)	−0.14366 (15)	0.0647 (7)
O9	0.17730 (18)	0.98333 (16)	0.17445 (13)	0.0548 (6)
H9	0.2501	0.9817	0.1591	0.082*
O10	−0.0183 (2)	1.0235 (2)	0.25875 (15)	0.0716 (8)
C1	0.2808 (3)	0.3875 (3)	0.1176 (2)	0.0420 (9)
C2	0.1742 (3)	0.3542 (3)	0.0402 (2)	0.0552 (10)
H2A	0.1425	0.4065	0.0252	0.066*
C3	0.1133 (3)	0.2432 (3)	−0.0159 (2)	0.0610 (11)
H3	0.0402	0.2219	−0.0669	0.073*
C4	0.1614 (3)	0.1659 (3)	0.0042 (2)	0.0514 (10)
C5	0.2668 (3)	0.1982 (3)	0.0823 (2)	0.0586 (10)
H5	0.2980	0.1459	0.0974	0.070*
C6	0.3257 (3)	0.3077 (3)	0.1376 (2)	0.0512 (9)
H6A	0.3972	0.3286	0.1895	0.061*
C7	0.2828 (3)	0.5848 (2)	0.1707 (2)	0.0451 (9)
H7A	0.2599	0.5679	0.1016	0.054*
H7B	0.3468	0.6552	0.2033	0.054*
C8	0.1581 (3)	0.5936 (2)	0.2131 (2)	0.0393 (8)
C9	0.1497 (3)	0.5826 (2)	0.2980 (2)	0.0425 (9)
C10	0.0415 (3)	0.6043 (3)	0.3434 (2)	0.0489 (9)
C11	−0.0580 (3)	0.6366 (3)	0.3011 (2)	0.0575 (10)
H11	−0.1302	0.6515	0.3304	0.069*
C12	−0.0498 (3)	0.6467 (3)	0.2150 (2)	0.0593 (11)
H12	−0.1176	0.6676	0.1862	0.071*
C13	0.0569 (3)	0.6264 (3)	0.1709 (2)	0.0544 (10)
H13	0.0616	0.6344	0.1133	0.065*
C14	−0.0543 (3)	0.6161 (3)	0.4813 (2)	0.0928 (15)
H14A	−0.0568	0.6912	0.4987	0.139*
H14B	−0.0358	0.6054	0.5398	0.139*
H14C	−0.1389	0.5691	0.4418	0.139*
C15	0.4914 (3)	0.5294 (2)	0.18503 (19)	0.0474 (9)

H15A	0.5058	0.5520	0.1334	0.057*
H15B	0.5272	0.4655	0.1736	0.057*
C16	0.5636 (3)	0.6209 (3)	0.2829 (2)	0.0416 (8)
C17	0.5774 (3)	0.5989 (3)	0.3648 (2)	0.0426 (9)
C18	0.6368 (3)	0.6832 (3)	0.4588 (2)	0.0460 (9)
C19	0.6838 (3)	0.7870 (3)	0.4686 (2)	0.0573 (10)
H19	0.7231	0.8430	0.5306	0.069*
C20	0.6738 (3)	0.8101 (3)	0.3875 (3)	0.0610 (10)
H20	0.7071	0.8810	0.3949	0.073*
C21	0.6141 (3)	0.7272 (3)	0.2955 (2)	0.0532 (10)
H21	0.6076	0.7429	0.2412	0.064*
C22	0.7085 (3)	0.7266 (3)	0.62790 (19)	0.0703 (12)
H22A	0.7983	0.7573	0.6272	0.105*
H22B	0.7101	0.6915	0.6714	0.105*
H22C	0.6620	0.7839	0.6501	0.105*
C23	0.5141 (3)	1.0693 (3)	0.2602 (2)	0.0379 (8)
C24	0.5500 (3)	1.1011 (2)	0.3610 (2)	0.0441 (9)
H24	0.5304	1.0499	0.3862	0.053*
C25	0.6153 (3)	1.2095 (3)	0.4240 (2)	0.0488 (9)
H25	0.6394	1.2304	0.4910	0.059*
C26	0.6440 (3)	1.2852 (3)	0.3870 (2)	0.0470 (9)
C27	0.6139 (3)	1.2538 (3)	0.2871 (2)	0.0444 (9)
H27	0.6378	1.3042	0.2618	0.053*
C28	0.5477 (3)	1.1467 (3)	0.2252 (2)	0.0446 (9)
H28	0.5253	1.1263	0.1581	0.054*
C29	0.4974 (3)	0.9035 (2)	0.10890 (18)	0.0453 (9)
H29A	0.5642	0.8715	0.1301	0.054*
H29B	0.5413	0.9557	0.0873	0.054*
C30	0.3925 (3)	0.8142 (3)	0.0244 (2)	0.0408 (9)
C31	0.2957 (3)	0.8415 (3)	−0.0254 (2)	0.0421 (8)
C32	0.1927 (3)	0.7609 (3)	−0.1006 (2)	0.0476 (9)
C33	0.1868 (3)	0.6525 (3)	−0.1228 (2)	0.0561 (10)
H33	0.1175	0.5985	−0.1711	0.067*
C34	0.2835 (3)	0.6227 (3)	−0.0739 (2)	0.0563 (10)
H34	0.2798	0.5492	−0.0897	0.068*
C35	0.3846 (3)	0.7035 (3)	−0.0020 (2)	0.0499 (9)
H35	0.4498	0.6834	0.0301	0.060*
C36	0.0044 (3)	0.7281 (3)	−0.2222 (2)	0.0889 (14)
H36A	0.0411	0.6820	−0.2740	0.133*
H36B	−0.0468	0.7678	−0.2461	0.133*
H36C	−0.0521	0.6836	−0.1999	0.133*
C37	0.3824 (3)	0.8887 (2)	0.2352 (2)	0.0482 (9)
H37A	0.4550	0.8861	0.2800	0.058*
H37B	0.3482	0.8153	0.1829	0.058*
C38	0.2732 (3)	0.9204 (2)	0.2897 (2)	0.0412 (8)
C39	0.1764 (3)	0.9610 (3)	0.2540 (2)	0.0416 (9)
C40	0.0678 (3)	0.9796 (3)	0.2994 (2)	0.0495 (9)
C41	0.0546 (3)	0.9549 (3)	0.3772 (2)	0.0598 (10)
H41	−0.0182	0.9663	0.4067	0.072*

C42	0.1512 (4)	0.9124 (3)	0.4117 (2)	0.0624 (11)
H42	0.1422	0.8945	0.4640	0.075*
C43	0.2600 (3)	0.8967 (3)	0.3690 (2)	0.0542 (10)
H43	0.3251	0.8700	0.3937	0.065*
C44	−0.1383 (3)	1.0354 (3)	0.2931 (2)	0.1023 (16)
H44A	−0.1878	0.9654	0.2849	0.154*
H44B	−0.1907	1.0632	0.2564	0.154*
H44C	−0.1175	1.0857	0.3614	0.154*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0402 (17)	0.0338 (17)	0.0422 (16)	0.0135 (14)	0.0027 (13)	0.0116 (14)
N2	0.0455 (17)	0.0407 (18)	0.0356 (15)	0.0143 (15)	0.0119 (13)	0.0154 (15)
O1	0.0832 (19)	0.0469 (17)	0.0662 (16)	0.0112 (15)	−0.0242 (14)	0.0142 (14)
O2	0.0497 (14)	0.0766 (17)	0.0425 (13)	0.0311 (14)	0.0086 (11)	0.0236 (13)
O3	0.0593 (16)	0.103 (2)	0.0558 (16)	0.0393 (16)	0.0228 (13)	0.0376 (16)
O4	0.0846 (18)	0.0442 (16)	0.0443 (13)	0.0112 (14)	−0.0006 (12)	0.0191 (13)
O5	0.0720 (17)	0.0575 (16)	0.0401 (14)	0.0163 (14)	−0.0021 (12)	0.0145 (13)
O6	0.0815 (18)	0.0495 (16)	0.0497 (15)	0.0011 (15)	−0.0116 (13)	0.0156 (13)
O7	0.0578 (15)	0.0422 (15)	0.0531 (14)	0.0132 (13)	−0.0004 (11)	0.0169 (12)
O8	0.0612 (16)	0.0607 (17)	0.0576 (15)	0.0106 (14)	−0.0133 (13)	0.0194 (14)
O9	0.0481 (14)	0.0782 (18)	0.0484 (14)	0.0202 (13)	0.0150 (11)	0.0341 (14)
O10	0.0479 (16)	0.112 (2)	0.0629 (16)	0.0313 (16)	0.0180 (13)	0.0387 (16)
C1	0.046 (2)	0.039 (2)	0.038 (2)	0.0150 (19)	0.0042 (16)	0.0126 (18)
C2	0.060 (2)	0.049 (3)	0.051 (2)	0.020 (2)	−0.0070 (19)	0.018 (2)
C3	0.063 (3)	0.048 (3)	0.055 (2)	0.009 (2)	−0.0164 (19)	0.015 (2)
C4	0.065 (3)	0.037 (2)	0.044 (2)	0.011 (2)	−0.0002 (19)	0.0128 (19)
C5	0.065 (3)	0.048 (3)	0.059 (2)	0.016 (2)	−0.009 (2)	0.024 (2)
C6	0.052 (2)	0.047 (2)	0.045 (2)	0.012 (2)	−0.0086 (17)	0.016 (2)
C7	0.051 (2)	0.038 (2)	0.047 (2)	0.0131 (18)	0.0041 (17)	0.0186 (18)
C8	0.041 (2)	0.035 (2)	0.038 (2)	0.0122 (17)	0.0005 (17)	0.0118 (17)
C9	0.042 (2)	0.046 (2)	0.039 (2)	0.0194 (18)	0.0025 (17)	0.0162 (18)
C10	0.045 (2)	0.052 (2)	0.046 (2)	0.017 (2)	0.0045 (19)	0.016 (2)
C11	0.049 (2)	0.058 (3)	0.058 (2)	0.022 (2)	0.005 (2)	0.016 (2)
C12	0.048 (2)	0.055 (3)	0.070 (3)	0.022 (2)	−0.004 (2)	0.023 (2)
C13	0.061 (2)	0.053 (2)	0.048 (2)	0.022 (2)	0.0008 (19)	0.021 (2)
C14	0.085 (3)	0.135 (4)	0.086 (3)	0.054 (3)	0.050 (3)	0.055 (3)
C15	0.048 (2)	0.045 (2)	0.046 (2)	0.0168 (19)	0.0068 (17)	0.0145 (18)
C16	0.033 (2)	0.044 (2)	0.046 (2)	0.0137 (18)	0.0050 (16)	0.016 (2)
C17	0.036 (2)	0.036 (2)	0.054 (2)	0.0103 (18)	0.0055 (18)	0.018 (2)
C18	0.042 (2)	0.041 (2)	0.044 (2)	0.0126 (19)	−0.0013 (17)	0.009 (2)
C19	0.051 (2)	0.050 (3)	0.056 (2)	0.014 (2)	0.0011 (19)	0.009 (2)
C20	0.057 (2)	0.047 (2)	0.069 (3)	0.003 (2)	−0.001 (2)	0.022 (2)
C21	0.044 (2)	0.058 (3)	0.062 (2)	0.012 (2)	0.0053 (19)	0.032 (2)
C22	0.067 (3)	0.086 (3)	0.040 (2)	0.023 (2)	−0.0016 (19)	0.010 (2)
C23	0.038 (2)	0.040 (2)	0.036 (2)	0.0139 (18)	0.0068 (16)	0.0162 (18)
C24	0.050 (2)	0.044 (2)	0.040 (2)	0.0126 (19)	0.0059 (17)	0.0203 (19)

C25	0.056 (2)	0.048 (2)	0.035 (2)	0.014 (2)	−0.0028 (17)	0.013 (2)
C26	0.041 (2)	0.041 (2)	0.048 (2)	0.0066 (19)	−0.0029 (18)	0.013 (2)
C27	0.049 (2)	0.044 (2)	0.045 (2)	0.0121 (19)	0.0071 (17)	0.0237 (19)
C28	0.050 (2)	0.045 (2)	0.0343 (19)	0.011 (2)	0.0024 (17)	0.0140 (19)
C29	0.045 (2)	0.047 (2)	0.039 (2)	0.0167 (19)	0.0099 (17)	0.0123 (18)
C30	0.046 (2)	0.042 (2)	0.0344 (19)	0.0163 (19)	0.0110 (16)	0.0131 (18)
C31	0.055 (2)	0.033 (2)	0.039 (2)	0.0121 (19)	0.0170 (17)	0.0134 (18)
C32	0.050 (2)	0.042 (2)	0.036 (2)	0.009 (2)	0.0028 (18)	0.0050 (19)
C33	0.056 (2)	0.046 (3)	0.047 (2)	0.004 (2)	0.0112 (19)	0.005 (2)
C34	0.061 (3)	0.045 (2)	0.059 (2)	0.019 (2)	0.018 (2)	0.015 (2)
C35	0.050 (2)	0.048 (2)	0.052 (2)	0.022 (2)	0.0125 (18)	0.018 (2)
C36	0.074 (3)	0.090 (3)	0.076 (3)	−0.005 (3)	−0.028 (2)	0.031 (3)
C37	0.053 (2)	0.040 (2)	0.044 (2)	0.0095 (19)	0.0027 (17)	0.0134 (18)
C38	0.049 (2)	0.036 (2)	0.037 (2)	0.0080 (18)	0.0118 (17)	0.0137 (18)
C39	0.047 (2)	0.042 (2)	0.0300 (19)	0.0022 (18)	0.0081 (17)	0.0125 (17)
C40	0.041 (2)	0.061 (3)	0.041 (2)	0.006 (2)	0.0055 (18)	0.018 (2)
C41	0.056 (3)	0.072 (3)	0.044 (2)	0.007 (2)	0.0194 (19)	0.018 (2)
C42	0.075 (3)	0.064 (3)	0.048 (2)	0.004 (2)	0.016 (2)	0.028 (2)
C43	0.066 (3)	0.045 (2)	0.048 (2)	0.012 (2)	0.0113 (19)	0.018 (2)
C44	0.069 (3)	0.157 (5)	0.115 (3)	0.055 (3)	0.050 (3)	0.073 (3)

Geometric parameters (Å, °)

N1—C1	1.438 (3)	C15—H15B	0.9700
N1—C7	1.478 (3)	C16—C21	1.388 (4)
N1—C15	1.487 (3)	C16—C17	1.390 (4)
N2—C23	1.446 (3)	C17—C18	1.408 (4)
N2—C37	1.457 (3)	C18—C19	1.363 (4)
N2—C29	1.488 (3)	C19—C20	1.388 (4)
O1—C4	1.371 (3)	C19—H19	0.9300
O1—H1	0.8200	C20—C21	1.383 (4)
O2—C9	1.383 (3)	C20—H20	0.9300
O2—H2	0.8200	C21—H21	0.9300
O3—C10	1.362 (3)	C22—H22A	0.9600
O3—C14	1.414 (3)	C22—H22B	0.9600
O4—C17	1.368 (3)	C22—H22C	0.9600
O4—H4	0.8200	C23—C28	1.378 (4)
O5—C18	1.366 (3)	C23—C24	1.396 (3)
O5—C22	1.430 (3)	C24—C25	1.395 (4)
O6—C26	1.377 (3)	C24—H24	0.9300
O6—H6	0.8200	C25—C26	1.371 (4)
O7—C31	1.378 (3)	C25—H25	0.9300
O7—H7	0.8200	C26—C27	1.381 (3)
O8—C32	1.362 (3)	C27—C28	1.382 (4)
O8—C36	1.408 (3)	C27—H27	0.9300
O9—C39	1.361 (3)	C28—H28	0.9300
O9—H9	0.8200	C29—C30	1.513 (3)
O10—C40	1.375 (3)	C29—H29A	0.9700
O10—C44	1.421 (3)	C29—H29B	0.9700

C1—C2	1.381 (3)	C30—C31	1.379 (4)
C1—C6	1.383 (4)	C30—C35	1.390 (4)
C2—C3	1.396 (4)	C31—C32	1.403 (4)
C2—H2A	0.9300	C32—C33	1.373 (4)
C3—C4	1.367 (4)	C33—C34	1.390 (4)
C3—H3	0.9300	C33—H33	0.9300
C4—C5	1.381 (4)	C34—C35	1.373 (4)
C5—C6	1.374 (4)	C34—H34	0.9300
C5—H5	0.9300	C35—H35	0.9300
C6—H6A	0.9300	C36—H36A	0.9600
C7—C8	1.518 (4)	C36—H36B	0.9600
C7—H7A	0.9700	C36—H36C	0.9600
C7—H7B	0.9700	C37—C38	1.508 (4)
C8—C9	1.368 (4)	C37—H37A	0.9700
C8—C13	1.397 (3)	C37—H37B	0.9700
C9—C10	1.403 (4)	C38—C43	1.384 (4)
C10—C11	1.379 (4)	C38—C39	1.385 (4)
C11—C12	1.381 (4)	C39—C40	1.400 (4)
C11—H11	0.9300	C40—C41	1.370 (4)
C12—C13	1.376 (4)	C41—C42	1.393 (4)
C12—H12	0.9300	C41—H41	0.9300
C13—H13	0.9300	C42—C43	1.377 (4)
C14—H14A	0.9600	C42—H42	0.9300
C14—H14B	0.9600	C43—H43	0.9300
C14—H14C	0.9600	C44—H44A	0.9600
C15—C16	1.507 (4)	C44—H44B	0.9600
C15—H15A	0.9700	C44—H44C	0.9600
C1—N1—C7	117.0 (2)	C20—C21—H21	119.5
C1—N1—C15	114.4 (2)	C16—C21—H21	119.5
C7—N1—C15	110.1 (2)	O5—C22—H22A	109.5
C23—N2—C37	116.8 (2)	O5—C22—H22B	109.5
C23—N2—C29	114.3 (2)	H22A—C22—H22B	109.5
C37—N2—C29	111.6 (2)	O5—C22—H22C	109.5
C4—O1—H1	109.5	H22A—C22—H22C	109.5
C9—O2—H2	109.5	H22B—C22—H22C	109.5
C10—O3—C14	118.7 (3)	C28—C23—C24	118.3 (3)
C17—O4—H4	109.5	C28—C23—N2	118.9 (3)
C18—O5—C22	116.9 (3)	C24—C23—N2	122.6 (3)
C26—O6—H6	109.5	C25—C24—C23	120.1 (3)
C31—O7—H7	109.5	C25—C24—H24	119.9
C32—O8—C36	117.8 (3)	C23—C24—H24	119.9
C39—O9—H9	109.5	C26—C25—C24	120.1 (3)
C40—O10—C44	118.3 (3)	C26—C25—H25	120.0
C2—C1—C6	118.1 (3)	C24—C25—H25	120.0
C2—C1—N1	124.5 (3)	C25—C26—O6	123.1 (3)
C6—C1—N1	117.3 (3)	C25—C26—C27	120.3 (3)
C1—C2—C3	120.9 (3)	O6—C26—C27	116.6 (3)
C1—C2—H2A	119.5	C26—C27—C28	119.3 (3)
C3—C2—H2A	119.5	C26—C27—H27	120.3

C4—C3—C2	119.8 (3)	C28—C27—H27	120.3
C4—C3—H3	120.1	C23—C28—C27	121.7 (3)
C2—C3—H3	120.1	C23—C28—H28	119.1
C3—C4—O1	123.7 (3)	C27—C28—H28	119.1
C3—C4—C5	119.8 (3)	N2—C29—C30	110.5 (2)
O1—C4—C5	116.4 (3)	N2—C29—H29A	109.5
C6—C5—C4	120.0 (3)	C30—C29—H29A	109.5
C6—C5—H5	120.0	N2—C29—H29B	109.5
C4—C5—H5	120.0	C30—C29—H29B	109.5
C5—C6—C1	121.3 (3)	H29A—C29—H29B	108.1
C5—C6—H6A	119.3	C31—C30—C35	117.7 (3)
C1—C6—H6A	119.3	C31—C30—C29	119.9 (3)
N1—C7—C8	113.4 (3)	C35—C30—C29	122.3 (3)
N1—C7—H7A	108.9	O7—C31—C30	118.7 (3)
C8—C7—H7A	108.9	O7—C31—C32	119.7 (3)
N1—C7—H7B	108.9	C30—C31—C32	121.5 (3)
C8—C7—H7B	108.9	O8—C32—C33	127.7 (3)
H7A—C7—H7B	107.7	O8—C32—C31	113.5 (3)
C9—C8—C13	119.4 (3)	C33—C32—C31	118.9 (3)
C9—C8—C7	121.3 (3)	C32—C33—C34	120.7 (3)
C13—C8—C7	118.9 (3)	C32—C33—H33	119.6
C8—C9—O2	123.1 (3)	C34—C33—H33	119.6
C8—C9—C10	121.0 (3)	C35—C34—C33	119.0 (3)
O2—C9—C10	115.8 (3)	C35—C34—H34	120.5
O3—C10—C11	126.2 (3)	C33—C34—H34	120.5
O3—C10—C9	114.6 (3)	C34—C35—C30	122.1 (3)
C11—C10—C9	119.2 (3)	C34—C35—H35	118.9
C10—C11—C12	119.6 (3)	C30—C35—H35	118.9
C10—C11—H11	120.2	O8—C36—H36A	109.5
C12—C11—H11	120.2	O8—C36—H36B	109.5
C13—C12—C11	121.2 (3)	H36A—C36—H36B	109.5
C13—C12—H12	119.4	O8—C36—H36C	109.5
C11—C12—H12	119.4	H36A—C36—H36C	109.5
C12—C13—C8	119.5 (3)	H36B—C36—H36C	109.5
C12—C13—H13	120.2	N2—C37—C38	115.6 (3)
C8—C13—H13	120.2	N2—C37—H37A	108.4
O3—C14—H14A	109.5	C38—C37—H37A	108.4
O3—C14—H14B	109.5	N2—C37—H37B	108.4
H14A—C14—H14B	109.5	C38—C37—H37B	108.4
O3—C14—H14C	109.5	H37A—C37—H37B	107.4
H14A—C14—H14C	109.5	C43—C38—C39	119.4 (3)
H14B—C14—H14C	109.5	C43—C38—C37	119.8 (3)
N1—C15—C16	111.2 (2)	C39—C38—C37	120.4 (3)
N1—C15—H15A	109.4	O9—C39—C38	124.1 (3)
C16—C15—H15A	109.4	O9—C39—C40	116.0 (3)
N1—C15—H15B	109.4	C38—C39—C40	120.0 (3)
C16—C15—H15B	109.4	C41—C40—O10	125.7 (3)
H15A—C15—H15B	108.0	C41—C40—C39	120.4 (3)
C21—C16—C17	118.4 (3)	O10—C40—C39	114.0 (3)

C21—C16—C15	122.8 (3)	C40—C41—C42	119.3 (3)
C17—C16—C15	118.8 (3)	C40—C41—H41	120.3
O4—C17—C16	118.8 (3)	C42—C41—H41	120.3
O4—C17—C18	120.4 (3)	C43—C42—C41	120.5 (3)
C16—C17—C18	120.8 (3)	C43—C42—H42	119.8
C19—C18—O5	127.5 (3)	C41—C42—H42	119.8
C19—C18—C17	119.2 (3)	C42—C43—C38	120.5 (3)
O5—C18—C17	113.2 (3)	C42—C43—H43	119.8
C18—C19—C20	120.9 (3)	C38—C43—H43	119.8
C18—C19—H19	119.6	O10—C44—H44A	109.5
C20—C19—H19	119.6	O10—C44—H44B	109.5
C21—C20—C19	119.6 (3)	H44A—C44—H44B	109.5
C21—C20—H20	120.2	O10—C44—H44C	109.5
C19—C20—H20	120.2	H44A—C44—H44C	109.5
C20—C21—C16	121.0 (3)	H44B—C44—H44C	109.5
C7—N1—C1—C2	8.2 (4)	C37—N2—C23—C28	−169.1 (3)
C15—N1—C1—C2	−122.7 (3)	C29—N2—C23—C28	57.9 (3)
C7—N1—C1—C6	−169.5 (3)	C37—N2—C23—C24	6.7 (4)
C15—N1—C1—C6	59.6 (4)	C29—N2—C23—C24	−126.3 (3)
C6—C1—C2—C3	0.2 (5)	C28—C23—C24—C25	1.6 (4)
N1—C1—C2—C3	−177.5 (3)	N2—C23—C24—C25	−174.3 (3)
C1—C2—C3—C4	−1.5 (5)	C23—C24—C25—C26	0.2 (5)
C2—C3—C4—O1	−178.8 (3)	C24—C25—C26—O6	175.0 (3)
C2—C3—C4—C5	2.4 (6)	C24—C25—C26—C27	−2.8 (5)
C3—C4—C5—C6	−2.0 (5)	C25—C26—C27—C28	3.6 (5)
O1—C4—C5—C6	179.1 (3)	O6—C26—C27—C28	−174.3 (3)
C4—C5—C6—C1	0.7 (5)	C24—C23—C28—C27	−0.8 (5)
C2—C1—C6—C5	0.2 (5)	N2—C23—C28—C27	175.2 (3)
N1—C1—C6—C5	178.1 (3)	C26—C27—C28—C23	−1.8 (5)
C1—N1—C7—C8	74.4 (3)	C23—N2—C29—C30	−158.7 (2)
C15—N1—C7—C8	−152.7 (2)	C37—N2—C29—C30	66.0 (3)
N1—C7—C8—C9	38.0 (4)	N2—C29—C30—C31	66.8 (4)
N1—C7—C8—C13	−149.7 (3)	N2—C29—C30—C35	−109.3 (3)
C13—C8—C9—O2	−178.2 (3)	C35—C30—C31—O7	−179.0 (3)
C7—C8—C9—O2	−5.9 (5)	C29—C30—C31—O7	4.7 (4)
C13—C8—C9—C10	−0.3 (5)	C35—C30—C31—C32	−0.2 (5)
C7—C8—C9—C10	172.0 (3)	C29—C30—C31—C32	−176.5 (3)
C14—O3—C10—C11	−3.4 (5)	C36—O8—C32—C33	−1.7 (5)
C14—O3—C10—C9	176.7 (3)	C36—O8—C32—C31	178.0 (3)
C8—C9—C10—O3	−179.7 (3)	O7—C31—C32—O8	0.7 (4)
O2—C9—C10—O3	−1.7 (4)	C30—C31—C32—O8	−178.0 (3)
C8—C9—C10—C11	0.4 (5)	O7—C31—C32—C33	−179.6 (3)
O2—C9—C10—C11	178.3 (3)	C30—C31—C32—C33	1.7 (5)
O3—C10—C11—C12	−179.7 (3)	O8—C32—C33—C34	177.8 (3)
C9—C10—C11—C12	0.2 (5)	C31—C32—C33—C34	−1.9 (5)
C10—C11—C12—C13	−0.8 (5)	C32—C33—C34—C35	0.7 (5)
C11—C12—C13—C8	0.8 (5)	C33—C34—C35—C30	0.8 (5)
C9—C8—C13—C12	−0.3 (5)	C31—C30—C35—C34	−1.1 (5)
C7—C8—C13—C12	−172.7 (3)	C29—C30—C35—C34	175.1 (3)

C1—N1—C15—C16	−151.0 (3)	C23—N2—C37—C38	69.6 (3)
C7—N1—C15—C16	74.8 (3)	C29—N2—C37—C38	−156.3 (2)
N1—C15—C16—C21	−107.1 (3)	N2—C37—C38—C43	−148.5 (3)
N1—C15—C16—C17	71.1 (3)	N2—C37—C38—C39	39.3 (4)
C21—C16—C17—O4	−178.7 (3)	C43—C38—C39—O9	−177.6 (3)
C15—C16—C17—O4	3.0 (4)	C37—C38—C39—O9	−5.4 (5)
C21—C16—C17—C18	2.3 (5)	C43—C38—C39—C40	1.2 (5)
C15—C16—C17—C18	−176.0 (3)	C37—C38—C39—C40	173.4 (3)
C22—O5—C18—C19	−2.5 (5)	C44—O10—C40—C41	−6.3 (5)
C22—O5—C18—C17	176.1 (3)	C44—O10—C40—C39	173.9 (3)
O4—C17—C18—C19	179.5 (3)	O9—C39—C40—C41	177.0 (3)
C16—C17—C18—C19	−1.5 (5)	C38—C39—C40—C41	−1.9 (5)
O4—C17—C18—O5	0.8 (4)	O9—C39—C40—O10	−3.3 (4)
C16—C17—C18—O5	179.8 (3)	C38—C39—C40—O10	177.9 (3)
O5—C18—C19—C20	178.5 (3)	O10—C40—C41—C42	−178.9 (3)
C17—C18—C19—C20	0.0 (5)	C39—C40—C41—C42	0.9 (5)
C18—C19—C20—C21	0.7 (5)	C40—C41—C42—C43	0.9 (5)
C19—C20—C21—C16	0.1 (5)	C41—C42—C43—C38	−1.6 (5)
C17—C16—C21—C20	−1.6 (5)	C39—C38—C43—C42	0.5 (5)
C15—C16—C21—C20	176.6 (3)	C37—C38—C43—C42	−171.7 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O9^{i}	0.82	2.38	3.185 (3)	168
O1—H1···O10^{i}	0.82	2.35	2.911 (3)	127
O2—H2···N1	0.82	1.97	2.697 (3)	147
O4—H4···O5	0.82	2.19	2.640 (3)	115
O4—H4···O6^{ii}	0.82	2.49	2.941 (3)	116
O6—H6···O2^{iii}	0.82	2.10	2.913 (3)	171
O6—H6···O3^{iii}	0.82	2.50	3.008 (3)	121
O7—H7···O1^{iv}	0.82	2.11	2.835 (3)	148
O7—H7···O8	0.82	2.17	2.629 (3)	115
O9—H9···O7	0.82	2.45	3.023 (3)	128
O9—H9···N2	0.82	2.04	2.753 (3)	146
C5—H5···O7^{ii}	0.93	2.50	3.218 (4)	134

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

Fig. 1

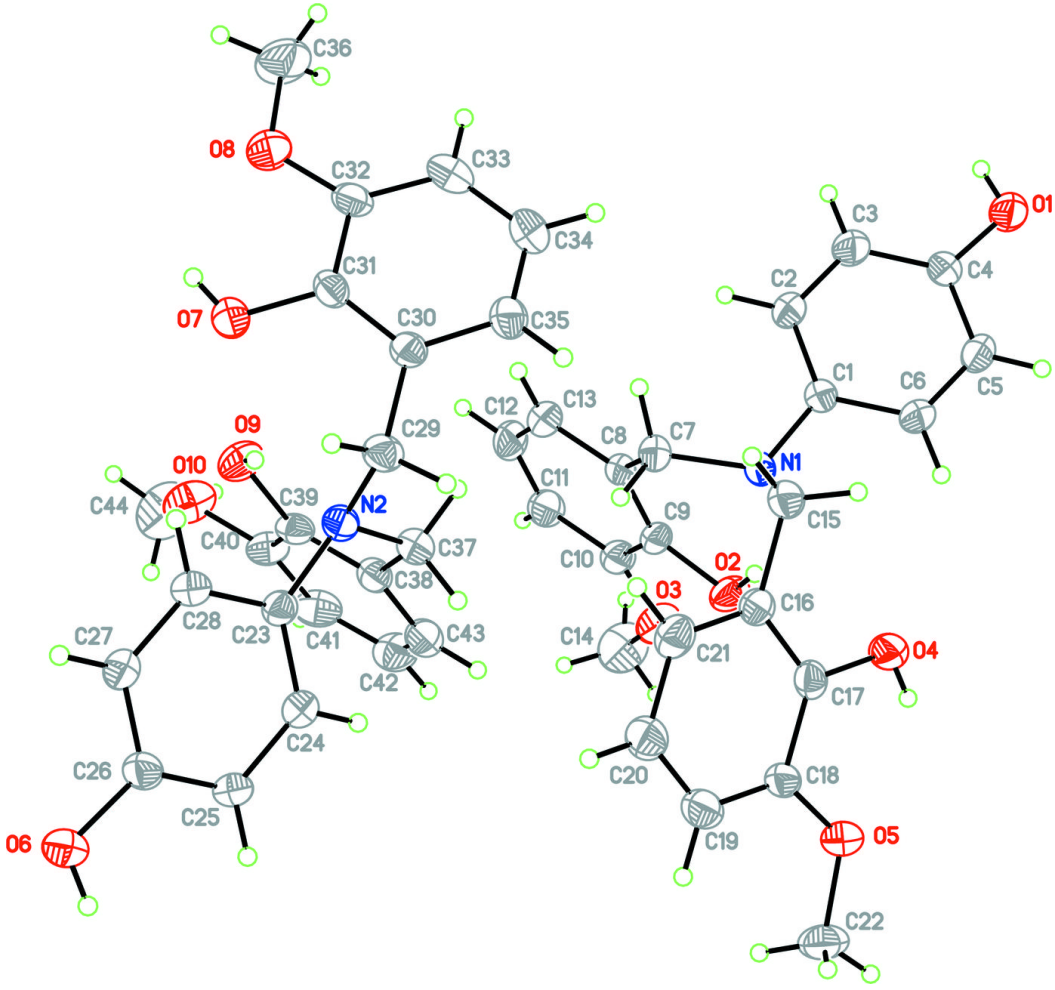
C36
O8
C32
C33
C34
C31
O7
C30
C35
C29
O9
O10
C44
C39
N2
C40
C37
C38
C28
C23
C41
C43
C27
C42
C24
C26
C25
O6
C12
C13
C8
C7
N1
C11
C9
C15
C10
O2
O3
C16
C14
C21
O4
C17
C20
C18
C19
O5
C22
C3
O1
C4
C2
C1
C6
C5

Fig. 2

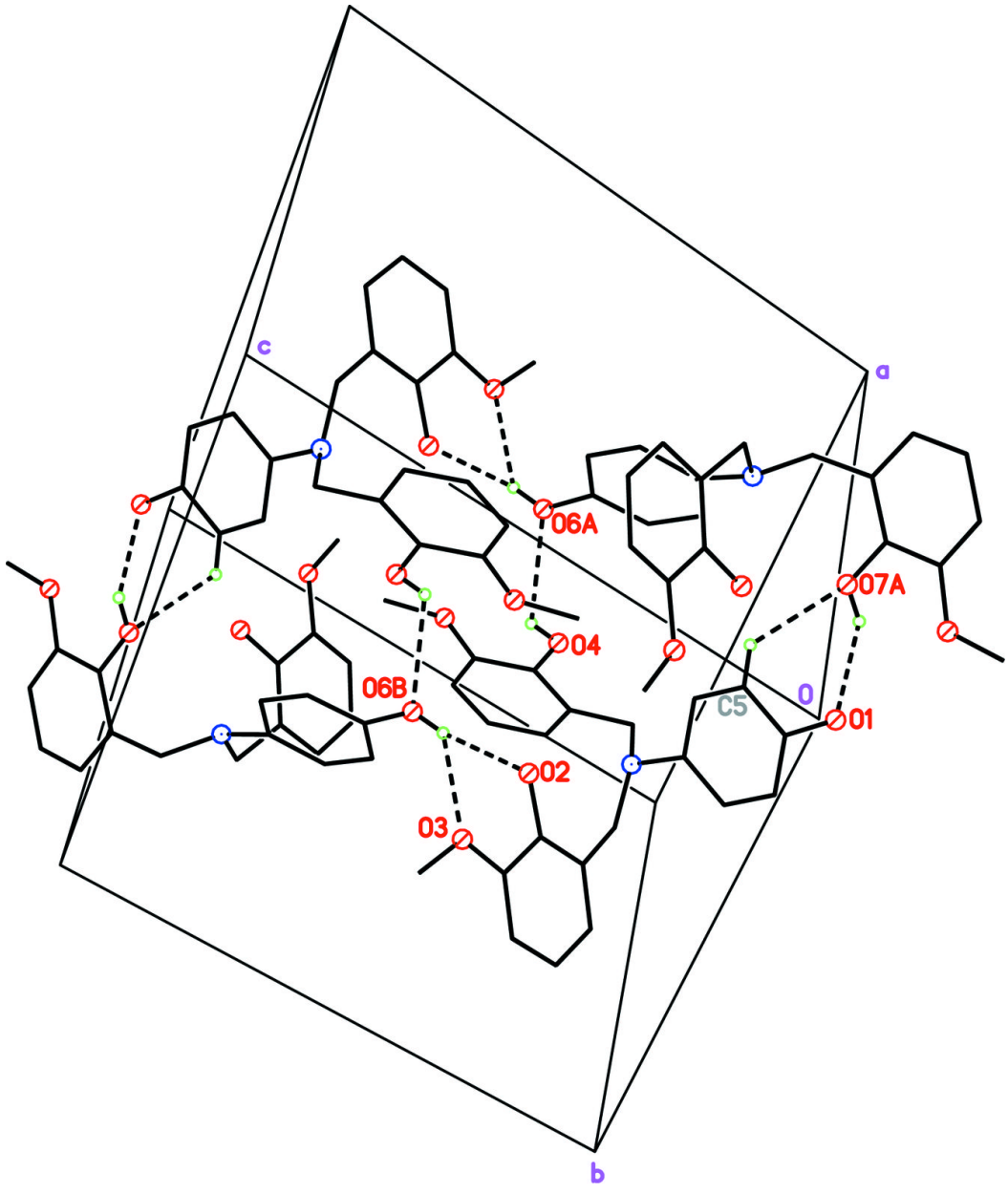

Fig. 3

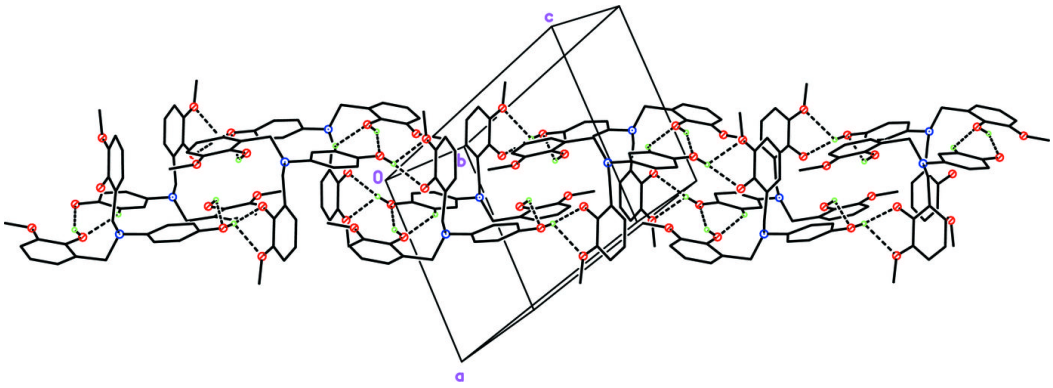

Fig. 4