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tert-Butyl *N*-(4-hydroxybenzyl)-*N*-[4-(prop-2-ynyloxy)benzyl]carbamate

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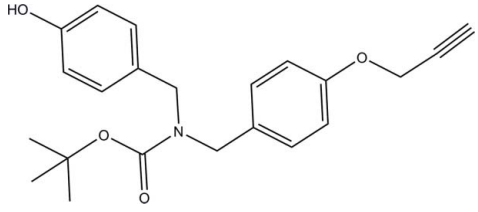
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.046; wR factor = 0.149; data-to-parameter ratio = 14.0.

In the crystal structure of the title compound, $C_{22}H_{25}NO_4$, intermolecular O—H···O hydrogen bonds involving the hydroxy group of the 4-(amimomethyl)phenol fragment and the C=O group connect the molecules into infinite chains along the c axis. Two C atoms of the propyne group are disordered over two sites with occupancy factors of 0.53 (2) and 0.47 (2).

Related literature

For applications of the title compound, see: Späth & König (2010); Juríček *et al.* (2011). For the synthesis of the title compound, see: Kim *et al.* (2004). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{22}H_{25}NO_4$
M_r = 367.43
Monoclinic, $P2_1/c$
a = 18.6904 (8) Å
b = 6.2611 (4) Å
c = 17.3567 (7) Å
β = 96.791 (1)°
V = 2016.87 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 296 K
0.41 × 0.37 × 0.29 mm

Data collection

Rigaku R-AXIS RAPID/ZJUG diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.957, T_{max} = 0.976
15741 measured reflections
3750 independent reflections
2099 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.149
S = 1.00
3750 reflections
268 parameters
4 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O4^{i}	0.82	1.94	2.745 (2)	167

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KJ2185).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc., Perkin Trans. 2*, pp. S1–19.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Juríček, M., Kouwer, P. H. J. & Rowan, A. E. (2011). *Chem. Commun.* **47**, 8740–8749.
Kim, M. K., Kleckley, T. S., Wiemer, A. J., Holstein, S. A., Hohl, R. J. & Wiemer, D. F. (2004). *J. Org. Chem.* **69**, 8186–8193.
Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2002). *CrystalStructure*. Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Späth, A. & König, B. (2010). *Beilstein J. Org. Chem.* **6**, 1–111.

supplementary materials

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tert-Butyl *N*-(4-hydroxybenzyl)-*N*-[4-(prop-2-ynyloxy)benzyl]carbamate

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Comment

The amino group is one of the most important functional groups in molecules of biological relevance, of which histamine and dopamine are two representative examples. In the synthesis of amino-contaning compounds, the boc group is commonly used to protect the amino group when performing parallel chemical transformations (Späth *et al.* , 2010). The acetylene group, due to the presence of the carbon-carbon triple bond, is an ideal functional group for further postmodification by numerous synthetic transformations (Juríček *et al.* , 2011). In our exploration of structure-activity relationships of amino-contaning compounds, we recently obtained a crystal of an intermediate, which contains both a boc-protecting amino group and an acetylene group. We report its crystal structure here.

The molecular structure of the title compound is shown in Fig. 1. The dihedral angle of the rings C1—C6 and C9—C14 is 11.7 (3)°. The bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The C18—N1 distance is 1.348 (3) Å, which is in the range of a typical double C=N bond. Atom O4 has a partial anionic character, as is shown by the lengthening of the C=O bond [1.226 (3) Å] compared to what is normally found for carbonyl groups. This atom acts as a hydrogen-bond acceptor for an intermolecular O—H···O hydrogen bond (Table 1). The hydrogen bonds are forming one-dimensional infinite chains along the *c* axis (Fig. 2).

Experimental

The title compound was synthesized according to the method proposed by Kim *et al.* (2004). 4-(Amimomethyl)phenol (0.01 mol,1.23 g) and 4-(prop-2-gnyloxy)benzoldehyde were heated in anhydrous methanol for 2 h, then $NaBH_4$ (0.1 mol,0.38 g) was added to the solution. The resulting solution was stirred for 30 minutes, then Boc_2O (0.01 mol,2.18 g) was dropped into the solution. Colourless block-shaped single crystals suitable for X-ray structure determination were obtained by slow evaporation of the solution in a mixture of PE:EA(1:1,v:v). Yield: 51.7%.

Refinement

Two C atoms of the propyne group are disordered over two sites. The occupancy factors refined to 0.53 (2) and 0.47 (2). H atoms were positioned geometrically and refined as riding groups, with O—H = 0.82 and C—H = 0.93 Å for aromatic H, 0.96 for methyl H, 0.97 for methylene H and constrained to ride on their parent atoms, with Uiso(H) = x U_{eq}(C), where x = 1.2 for aromatic and methylene H, x = 1.0 for H atoms bonded to the disorded C atoms of the propyne group and x = 1.5 for methyl H.

Figures

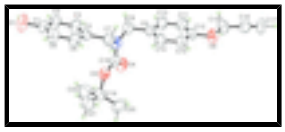

Fig. 1. The molecular structure of title compound. Displacement ellipsoids are drawn at the 40% probability level. Only the major disorder component is shown.

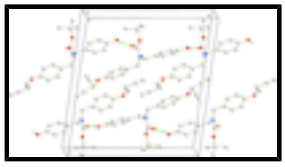

Fig. 2. Crystal packing of the title compound, viewed down the *b* axis, showing the O—H···O the hydrogen bonds as green dashed lines. H atoms not involved in hydrogen bonding have been omitted. Both disorder compounds of the propyne group are shown.

tert-Butyl *N*-(4-hydroxybenzyl)-*N*-[4-(prop-2-ynyloxy)benzyl]carbamate

Crystal data

$C_{22}H_{25}NO_4$	$F(000) = 784$
$M_r = 367.43$	$D_x = 1.210$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 9726 reflections
$a = 18.6904$ (8) Å	$\theta = 3.0$–$27.4°$
$b = 6.2611$ (4) Å	$\mu = 0.08$ mm^{-1}
$c = 17.3567$ (7) Å	$T = 296$ K
$\beta = 96.791$ (1)°	Chunk, colorless
$V = 2016.87$ (18) Å^3	0.41 × 0.37 × 0.29 mm
$Z = 4$	

Data collection

Rigaku R-AXIS RAPID/ZJUG diffractometer	3750 independent reflections
Radiation source: rolling anode	2099 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.038$
Detector resolution: 10.00 pixels mm^{-1}	$\theta_{max} = 25.5°$, $\theta_{min} = 3.0°$
ω scans	$h = -22\rightarrow22$
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	$k = -7\rightarrow7$
$T_{min} = 0.957$, $T_{max} = 0.976$	$l = -21\rightarrow19$
15741 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.046$	H-atom parameters constrained
$wR(F^2) = 0.149$	$w = 1/[\sigma^2(F_o^2) + (0.0635P)^2 + 0.6687P]$

	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} < 0.001$
3750 reflections	$\Delta\rho_{max} = 0.26$ e Å^{-3}
268 parameters	$\Delta\rho_{min} = -0.19$ e Å^{-3}
4 restraints	Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0155 (18)

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 $F^2 > \sigma(F^2)$ 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}	Occ. (<1)
O1	0.17241 (12)	0.2936 (3)	0.76205 (9)	0.0818 (6)	
H1	0.1833	0.3985	0.7892	0.123*	
O2	0.43006 (9)	0.6806 (3)	0.13104 (10)	0.0726 (5)	
O3	0.13748 (8)	0.5836 (3)	0.40395 (8)	0.0592 (5)	
O4	0.19445 (10)	0.8841 (3)	0.36950 (9)	0.0695 (5)	
N1	0.25604 (11)	0.6051 (3)	0.42845 (10)	0.0605 (5)	
C1	0.19319 (13)	0.3250 (4)	0.68966 (12)	0.0564 (6)	
C2	0.22502 (14)	0.5106 (4)	0.66891 (13)	0.0643 (7)	
H2	0.2334	0.6214	0.7046	0.077*	
C3	0.24466 (15)	0.5329 (4)	0.59494 (13)	0.0664 (7)	
H3	0.2663	0.6593	0.5816	0.080*	
C4	0.23302 (11)	0.3722 (4)	0.54024 (12)	0.0513 (6)	
C5	0.20066 (14)	0.1883 (4)	0.56236 (13)	0.0648 (7)	
H5	0.1919	0.0776	0.5268	0.078*	
C6	0.18094 (15)	0.1640 (4)	0.63598 (14)	0.0712 (7)	
H6	0.1592	0.0378	0.6493	0.085*	
C7	0.25534 (14)	0.3881 (4)	0.45966 (13)	0.0633 (7)	
H7A	0.3032	0.3276	0.4604	0.076*	
H7B	0.2227	0.3019	0.4248	0.076*	
C8	0.32381 (13)	0.7155 (5)	0.42417 (13)	0.0712 (8)	
H8A	0.3601	0.6535	0.4622	0.085*	
H8B	0.3180	0.8639	0.4382	0.085*	
C9	0.35077 (12)	0.7072 (4)	0.34535 (12)	0.0584 (6)	
C10	0.39496 (15)	0.8655 (5)	0.32395 (15)	0.0776 (8)	

H10	0.4067	0.9780	0.3581	0.093*	
C11	0.42283 (15)	0.8647 (5)	0.25335 (15)	0.0762 (8)	
H11	0.4527	0.9746	0.2404	0.091*	
C12	0.40561 (12)	0.6991 (4)	0.20300 (13)	0.0579 (6)	
C13	0.36058 (13)	0.5388 (4)	0.22214 (14)	0.0649 (7)	
H13	0.3485	0.4271	0.1877	0.078*	
C14	0.33342 (13)	0.5442 (4)	0.29254 (14)	0.0652 (7)	
H14	0.3027	0.4359	0.3049	0.078*	
C15	0.48249 (14)	0.8306 (4)	0.11323 (15)	0.0756 (8)	
H15A	0.5194	0.8459	0.1571	0.091*	
H15	0.4601	0.9688	0.1024	0.091*	0.532 (4)
H15B	0.5046	0.7474	0.0752	0.091*	0.468 (4)
C16A	0.5155 (2)	0.7544 (7)	0.0443 (2)	0.0661 (14)	0.532 (4)
C17A	0.5407 (3)	0.6902 (8)	−0.0069 (2)	0.0760 (17)	0.532 (4)
H17A	0.5615	0.6372	−0.0491	0.091*	0.532 (4)
C16B	0.4509 (3)	1.0432 (7)	0.0868 (3)	0.0689 (17)	0.468 (4)
C17B	0.4307 (4)	1.2108 (7)	0.0727 (4)	0.086 (2)	0.468 (4)
H17B	0.4141	1.3484	0.0611	0.103*	0.468 (4)
C18	0.19532 (13)	0.7046 (4)	0.39779 (12)	0.0566 (6)	
C19	0.06391 (13)	0.6547 (4)	0.37645 (14)	0.0623 (6)	
C20	0.05670 (17)	0.6953 (5)	0.28996 (16)	0.0932 (10)	
H20A	0.0806	0.8265	0.2800	0.140*	
H20B	0.0066	0.7054	0.2703	0.140*	
H20C	0.0783	0.5797	0.2647	0.140*	
C21	0.01977 (15)	0.4612 (5)	0.39359 (16)	0.0809 (8)	
H21A	0.0320	0.3431	0.3624	0.121*	
H21B	−0.0306	0.4932	0.3818	0.121*	
H21C	0.0299	0.4245	0.4475	0.121*	
C22	0.04566 (18)	0.8454 (5)	0.4239 (2)	0.1049 (11)	
H22A	0.0587	0.8160	0.4780	0.157*	
H22B	−0.0051	0.8734	0.4144	0.157*	
H22C	0.0718	0.9680	0.4093	0.157*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.1286 (16)	0.0695 (13)	0.0529 (10)	−0.0072 (12)	0.0346 (10)	0.0001 (9)
O2	0.0827 (12)	0.0748 (13)	0.0651 (11)	−0.0114 (10)	0.0292 (9)	−0.0031 (9)
O3	0.0653 (10)	0.0561 (10)	0.0566 (9)	0.0006 (8)	0.0088 (7)	0.0070 (8)
O4	0.0927 (13)	0.0557 (11)	0.0608 (10)	−0.0063 (9)	0.0113 (9)	0.0095 (8)
N1	0.0651 (12)	0.0708 (14)	0.0463 (10)	0.0014 (11)	0.0101 (9)	0.0085 (10)
C1	0.0717 (15)	0.0532 (15)	0.0454 (12)	0.0012 (12)	0.0109 (11)	0.0046 (11)
C2	0.0915 (18)	0.0562 (16)	0.0455 (13)	−0.0079 (14)	0.0090 (12)	−0.0060 (11)
C3	0.0921 (18)	0.0566 (16)	0.0522 (14)	−0.0155 (13)	0.0155 (13)	−0.0014 (12)
C4	0.0559 (13)	0.0566 (15)	0.0412 (11)	0.0052 (11)	0.0046 (9)	0.0014 (10)
C5	0.0825 (17)	0.0591 (16)	0.0529 (14)	−0.0081 (13)	0.0089 (12)	−0.0107 (12)
C6	0.102 (2)	0.0533 (16)	0.0612 (15)	−0.0136 (14)	0.0206 (14)	−0.0054 (12)
C7	0.0765 (16)	0.0657 (17)	0.0485 (13)	0.0155 (13)	0.0105 (11)	0.0065 (11)

C8	0.0694 (16)	0.093 (2)	0.0519 (13)	−0.0119 (15)	0.0077 (12)	−0.0025 (13)
C9	0.0558 (13)	0.0720 (17)	0.0475 (12)	−0.0059 (12)	0.0061 (10)	0.0016 (12)
C10	0.0908 (19)	0.087 (2)	0.0562 (15)	−0.0329 (17)	0.0127 (14)	−0.0139 (14)
C11	0.0831 (18)	0.084 (2)	0.0631 (16)	−0.0333 (16)	0.0160 (13)	−0.0054 (14)
C12	0.0586 (13)	0.0661 (16)	0.0506 (13)	0.0006 (12)	0.0131 (11)	0.0036 (12)
C13	0.0726 (16)	0.0621 (16)	0.0632 (15)	−0.0091 (13)	0.0215 (12)	−0.0066 (12)
C14	0.0676 (15)	0.0668 (17)	0.0636 (15)	−0.0105 (13)	0.0179 (12)	0.0011 (13)
C15	0.0773 (18)	0.078 (2)	0.0759 (17)	−0.0071 (15)	0.0281 (14)	0.0091 (15)
C16A	0.074 (3)	0.065 (3)	0.059 (3)	−0.008 (3)	0.006 (2)	0.015 (2)
C17A	0.099 (4)	0.082 (4)	0.052 (3)	0.004 (3)	0.032 (3)	0.003 (3)
C16B	0.078 (4)	0.074 (4)	0.058 (3)	−0.014 (3)	0.022 (3)	−0.009 (3)
C17B	0.124 (6)	0.056 (4)	0.083 (4)	−0.005 (4)	0.030 (4)	−0.002 (3)
C18	0.0739 (16)	0.0567 (16)	0.0407 (12)	−0.0044 (13)	0.0126 (11)	−0.0007 (11)
C19	0.0630 (14)	0.0604 (16)	0.0641 (14)	0.0043 (12)	0.0096 (12)	−0.0031 (12)
C20	0.100 (2)	0.100 (2)	0.0728 (17)	−0.0091 (19)	−0.0149 (16)	0.0252 (17)
C21	0.0818 (18)	0.080 (2)	0.0820 (19)	−0.0154 (16)	0.0134 (15)	0.0000 (15)
C22	0.091 (2)	0.078 (2)	0.150 (3)	0.0079 (17)	0.034 (2)	−0.037 (2)

Geometric parameters (Å, °)

O1—C1	1.372 (3)	C10—H10	0.9300
O1—H1	0.8200	C11—C12	1.369 (3)
O2—C12	1.385 (3)	C11—H11	0.9300
O2—C15	1.417 (3)	C12—C13	1.376 (3)
O3—C18	1.335 (3)	C13—C14	1.378 (3)
O3—C19	1.470 (3)	C13—H13	0.9300
O4—C18	1.226 (3)	C14—H14	0.9300
N1—C18	1.348 (3)	C15—C16A	1.488 (3)
N1—C8	1.452 (3)	C15—C16B	1.506 (3)
N1—C7	1.464 (3)	C15—H15A	0.9700
C1—C2	1.373 (3)	C15—H15	0.9700
C1—C6	1.374 (3)	C15—H15B	0.9700
C2—C3	1.383 (3)	C16A—C17A	1.128 (3)
C2—H2	0.9300	C16A—H15B	0.5978
C3—C4	1.383 (3)	C17A—H17A	0.9300
C3—H3	0.9300	C16B—C17B	1.132 (3)
C4—C5	1.376 (3)	C17B—H17B	0.9300
C4—C7	1.509 (3)	C19—C22	1.512 (4)
C5—C6	1.379 (3)	C19—C20	1.513 (4)
C5—H5	0.9300	C19—C21	1.515 (4)
C6—H6	0.9300	C20—H20A	0.9600
C7—H7A	0.9700	C20—H20B	0.9600
C7—H7B	0.9700	C20—H20C	0.9600
C8—C9	1.514 (3)	C21—H21A	0.9600
C8—H8A	0.9700	C21—H21B	0.9600
C8—H8B	0.9700	C21—H21C	0.9600
C9—C10	1.369 (3)	C22—H22A	0.9600
C9—C14	1.384 (3)	C22—H22B	0.9600
C10—C11	1.387 (3)	C22—H22C	0.9600

C1—O1—H1	109.5	C14—C13—H13	120.1
C12—O2—C15	116.86 (19)	C13—C14—C9	121.6 (2)
C18—O3—C19	122.50 (19)	C13—C14—H14	119.2
C18—N1—C8	117.3 (2)	C9—C14—H14	119.2
C18—N1—C7	122.1 (2)	O2—C15—C16A	109.0 (3)
C8—N1—C7	120.4 (2)	O2—C15—C16B	113.3 (3)
C2—C1—O1	122.7 (2)	C16A—C15—C16B	102.9 (3)
C2—C1—C6	119.1 (2)	O2—C15—H15A	109.9
O1—C1—C6	118.3 (2)	C16A—C15—H15A	109.9
C1—C2—C3	120.0 (2)	C16B—C15—H15A	111.6
C1—C2—H2	120.0	O2—C15—H15	109.9
C3—C2—H2	120.0	C16A—C15—H15	109.9
C4—C3—C2	121.8 (2)	H15A—C15—H15	108.3
C4—C3—H3	119.1	O2—C15—H15B	98.9
C2—C3—H3	119.1	C16B—C15—H15B	116.8
C5—C4—C3	117.1 (2)	H15A—C15—H15B	105.5
C5—C4—C7	119.5 (2)	H15—C15—H15B	123.8
C3—C4—C7	123.4 (2)	C17A—C16A—C15	177.7 (5)
C4—C5—C6	121.7 (2)	C17A—C16A—H15B	154.1
C4—C5—H5	119.2	C16A—C17A—H17A	180.0
C6—C5—H5	119.2	C17B—C16B—C15	173.8 (6)
C1—C6—C5	120.4 (2)	C16B—C17B—H17B	180.0
C1—C6—H6	119.8	O4—C18—O3	125.5 (2)
C5—C6—H6	119.8	O4—C18—N1	123.5 (2)
N1—C7—C4	114.8 (2)	O3—C18—N1	111.0 (2)
N1—C7—H7A	108.6	O3—C19—C22	109.0 (2)
C4—C7—H7A	108.6	O3—C19—C20	110.1 (2)
N1—C7—H7B	108.6	C22—C19—C20	114.1 (3)
C4—C7—H7B	108.6	O3—C19—C21	101.8 (2)
H7A—C7—H7B	107.5	C22—C19—C21	111.2 (2)
N1—C8—C9	114.55 (19)	C20—C19—C21	110.0 (2)
N1—C8—H8A	108.6	C19—C20—H20A	109.5
C9—C8—H8A	108.6	C19—C20—H20B	109.5
N1—C8—H8B	108.6	H20A—C20—H20B	109.5
C9—C8—H8B	108.6	C19—C20—H20C	109.5
H8A—C8—H8B	107.6	H20A—C20—H20C	109.5
C10—C9—C14	117.1 (2)	H20B—C20—H20C	109.5
C10—C9—C8	119.8 (2)	C19—C21—H21A	109.5
C14—C9—C8	123.1 (2)	C19—C21—H21B	109.5
C9—C10—C11	122.6 (2)	H21A—C21—H21B	109.5
C9—C10—H10	118.7	C19—C21—H21C	109.5
C11—C10—H10	118.7	H21A—C21—H21C	109.5
C12—C11—C10	118.8 (2)	H21B—C21—H21C	109.5
C12—C11—H11	120.6	C19—C22—H22A	109.5
C10—C11—H11	120.6	C19—C22—H22B	109.5
C11—C12—C13	120.2 (2)	H22A—C22—H22B	109.5
C11—C12—O2	124.1 (2)	C19—C22—H22C	109.5
C13—C12—O2	115.7 (2)	H22A—C22—H22C	109.5
C12—C13—C14	119.7 (2)	H22B—C22—H22C	109.5

C12—C13—H13	120.1		
O1—C1—C2—C3	−179.9 (2)	C10—C11—C12—C13	0.9 (4)
C6—C1—C2—C3	−0.4 (4)	C10—C11—C12—O2	−180.0 (2)
C1—C2—C3—C4	0.1 (4)	C15—O2—C12—C11	7.5 (4)
C2—C3—C4—C5	0.2 (4)	C15—O2—C12—C13	−173.4 (2)
C2—C3—C4—C7	−178.4 (2)	C11—C12—C13—C14	−0.6 (4)
C3—C4—C5—C6	−0.4 (4)	O2—C12—C13—C14	−179.8 (2)
C7—C4—C5—C6	178.3 (2)	C12—C13—C14—C9	−0.5 (4)
C2—C1—C6—C5	0.3 (4)	C10—C9—C14—C13	1.3 (4)
O1—C1—C6—C5	179.8 (2)	C8—C9—C14—C13	−178.2 (2)
C4—C5—C6—C1	0.1 (4)	C12—O2—C15—C16A	165.7 (3)
C18—N1—C7—C4	−79.6 (3)	C12—O2—C15—C16B	−80.4 (3)
C8—N1—C7—C4	105.1 (2)	C19—O3—C18—O4	0.1 (3)
C5—C4—C7—N1	151.9 (2)	C19—O3—C18—N1	178.88 (18)
C3—C4—C7—N1	−29.5 (3)	C8—N1—C18—O4	−2.7 (3)
C18—N1—C8—C9	−77.4 (3)	C7—N1—C18—O4	−178.1 (2)
C7—N1—C8—C9	98.1 (3)	C8—N1—C18—O3	178.44 (18)
N1—C8—C9—C10	154.0 (3)	C7—N1—C18—O3	3.0 (3)
N1—C8—C9—C14	−26.5 (4)	C18—O3—C19—C22	−64.6 (3)
C14—C9—C10—C11	−1.1 (4)	C18—O3—C19—C20	61.2 (3)
C8—C9—C10—C11	178.5 (3)	C18—O3—C19—C21	177.9 (2)
C9—C10—C11—C12	−0.1 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O4^{i}	0.82	1.94	2.745 (2)	167

Symmetry codes: (i) *x*, −*y*+3/2, *z*+1/2.

Fig. 1

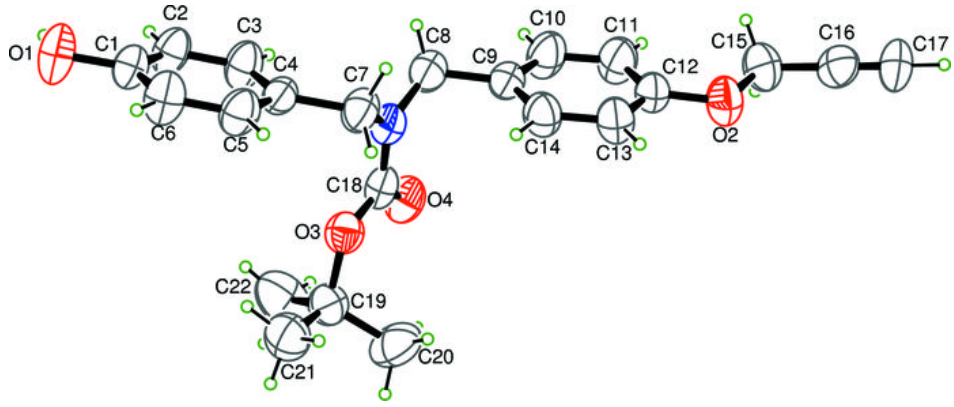
O1
C1
C2
C3
C4
C5
C6
C7
C8
C9
C10
C11
C12
C13
C14
C15
C16
C17
O2
C18
O3
O4
C19
C20
C21
C22

Fig. 2