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1-{[2-(Bromomethyl)-2-(4-methoxyphenyl)-1,3-dioxolan-4-yl]methyl}benzo[*d*]imidazole

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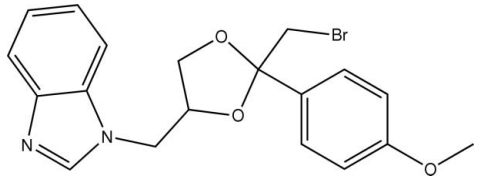
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.042; wR factor = 0.104; data-to-parameter ratio = 15.9.

The title compound, $C_{19}H_{19}BrN_2O_3$, forms an intramolecular C—H···O hydrogen bond which results in the formation of a five-membered ring. In the crystal structure, intermolecular C—H···O hydrogen bonds link the molecules into infinite chains along the b axis. The packing is further stabilized by C—H···π and π-π interactions.

Related literature

For related literature, see: Allen *et al.* (1987); Chai (1985).



Experimental

Crystal data

$C_{19}H_{19}BrN_2O_3$
M_r = 403.26
Monoclinic, $P2_1/c$
a = 12.683 (4) Å
b = 10.967 (4) Å
c = 26.530 (8) Å
β = 99.677 (7)°
V = 3638 (2) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 2.28 mm^{-1}
T = 293 (2) K
0.41 × 0.21 × 0.14 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan *SADABS* (Sheldrick, 1996)
T_{min} = 0.455, T_{max} = 0.741
19950 measured reflections
7191 independent reflections
4269 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.104
S = 0.99
7191 reflections
451 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C37—H37A···Cg	0.97	2.65	3.515	148
C15—H15A···O1^{i}	0.93	2.56	3.362 (5)	144
C20—H20A···O6	0.93	2.44	2.788 (4)	102

Symmetry code: (i) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$. Cg is the centroid of the C1–C6 benzene ring.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RN2025).

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.
Bruker (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Chai, Y. J. (1985). *Pesticides*, **5**, 38–44.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-Ray Systems, Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

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supplementary materials

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1-{[2-(Bromomethyl)-2-(4-methoxyphenyl)-1,3-dioxolan-4-yl]methyl}benzo[*d*]imidazole

C.-F. Ding, S. Bi, H.-S. Ning and Y.-Q. Yu

Comment

Benzimidazole compounds containing 1,3-dioxolane have the ability to prevent plant diseases (Chai, 1985). In the present study, a novel benzimidazole compound, (I), containing 1,3-dioxolane, has been synthesized from cheap 3-chloropropane-1,2-diol instead of expensive pentane-1,2-diol.

There are two molecules, A and B, in the asymmetric unit (Fig. 1). The bond lengths and angles in A and B are within normal ranges (Allen *et al.*, 1987). The benzimidazole units, C11—C17/N1/N2 and C30—C36/N3/N4, are essentially planar with dihedral angles of 0.77 (2)° and 0.90 (2)° in A and B, respectively. Intramolecular C20—H20A···O6 interactions (Table 2) result in the formation of a five-numbered ring.

As can be seen from the packing diagram (Fig. 2), intermolecular C15—H15A···O1^{i} hydrogen bonds (Table 1) link the molecules, forming infinite chains along the *b* axis (Fig. 2). The packing is further stabilized by C—H···π interactions (Table 1),where *Cg* denotes the centroid of C1—C6 benzene ring. The distance of 3.523 Å between the centroids of the rings C30/C31/C36/N3/N4 related by the symmetry operation (2 − *x*, −*y*, 1 − *z*) suggests a π-π interaction.

Experimental

Bromine (1.92 g, 24 mmol) was added dropwise to a solution of 4-methoxyacetophenone (3.00 g, 20 mmol) in anhydrous diethyl ether (50 ml) in an ice-bath. The reaction proceeded for 6 h to give w-bromine-4-methoxyacetophenone. Anhydrous 3-chloro-1,2-propanediol (3.33 g, 30 mmol), 1-butanol (10 ml) and toluene-*p*-sulfonic acid(0.1 g, 0.2 mmol) were then added. The reaction mixture was refluxed for 7 h and then cooled to room temperature. The organic layer was washed with saturated sodium bicarbonate solution and dried over anhydrous sodium sulfate. A white solid was obtained by recrystallization from methanol. A solution of this compound (7.70 g, 24 mmol) in dimethylformamide (15 ml) was added to a solution of benzimidazole (2.81 g, 24 mmol) and dried KI (0.5 g, 3 mmol) in dimethylformamide (50 ml). The mixture was heated to reflux for 5 h and then the solution was washed with water and extracted with chloroform. Single crystals of (I) were obtained by slow evaporation of a solution of the product in petroleum ether-ethyl acetate (3:1 *v*/*v*) at room temperature over a period of 3 d.

Refinement

All H atoms were located in difference Fourier map. Water H atoms were refined with O1W-H1W1 and O1W-H2W1 distance restrains of 0.85±0.01 Å. The remaining H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å, and with U_{iso}(H) = 1.2 U_{eq}(C).

Figures

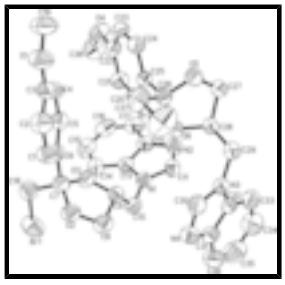

Fig. 1. The structure of the compound (I) showing 50% probability displacement ellipsoids and the atom numbering scheme.

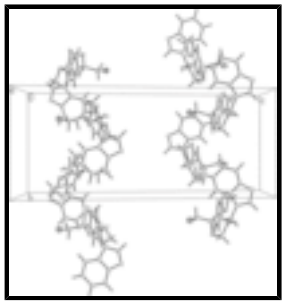

Fig. 2. Packing diagram of (I) showing the intermolecular hydrogen bonds (dished lines), viewed down the *a* axis,

1-{[2-(Bromomethyl)-2-(4-methoxyphenyl)-1,3-dioxolan-4- yl]methyl}benzo[*d*]imidazole

Crystal data

$C_{19}H_{19}BrN_2O_3$	F_{000} = 1648
M_r = 403.26	D_x = 1.473 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 12.683 (4) Å	Cell parameters from 3621 reflections
b = 10.967 (4) Å	θ = 2.5–20.6º
c = 26.530 (8) Å	μ = 2.28 mm^{-1}
β = 99.677 (7)º	T = 293 (2) K
V = 3638 (2) Å^3	Column, colourless
Z = 8	0.41 × 0.21 × 0.14 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer	7191 independent reflections
Radiation source: fine-focus sealed tube	4269 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.036
Detector resolution: 8.33 pixels mm^{-1}	θ_{max} = 26.1º
T = 293(2) K	θ_{min} = 1.6º
ω scans	h = −15→9
Absorption correction: multi-scan SADABS (Sheldrick, 1996)	k = −13→13
T_{min} = 0.455, T_{max} = 0.741	l = −31→32
19950 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
$R[F^2 > 2\sigma(F^2)] = 0.042$	H-atom parameters constrained
$wR(F^2) = 0.104$	$w = 1/[\sigma^2(F_o^2) + (0.0485P)^2 + 0.1464P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.99$	$(\Delta/\sigma)_{max} = 0.001$
7191 reflections	$\Delta\rho_{max} = 0.53$ e Å^{-3}
451 parameters	$\Delta\rho_{min} = -0.32$ 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 $F^2 > 2$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}
Br1	0.78726 (3)	0.28198 (4)	0.167855 (14)	0.07979 (15)
Br2	0.57291 (3)	0.11064 (3)	0.456477 (18)	0.09666 (18)
O3	0.76388 (15)	0.33379 (15)	0.28135 (7)	0.0483 (5)
O6	0.75670 (15)	0.31159 (16)	0.44731 (6)	0.0475 (5)
O5	0.65429 (16)	0.36202 (18)	0.50601 (7)	0.0541 (5)
N1	0.95774 (17)	0.42740 (19)	0.34591 (8)	0.0441 (6)
C7	0.6796 (2)	0.2610 (2)	0.25541 (11)	0.0467 (7)
O2	0.71432 (16)	0.13815 (16)	0.26158 (8)	0.0545 (5)
O1	0.29443 (17)	0.3243 (2)	0.33276 (9)	0.0697 (6)
O4	0.58098 (17)	0.83370 (18)	0.37021 (9)	0.0681 (6)
N3	0.9258 (2)	0.1434 (2)	0.47892 (9)	0.0519 (6)
C6	0.5772 (2)	0.2786 (2)	0.27646 (10)	0.0437 (7)
C11	0.9950 (2)	0.4562 (3)	0.39540 (11)	0.0523 (8)
H11A	1.0158	0.3975	0.4204	0.063*
C22	0.6035 (2)	0.7177 (3)	0.38762 (11)	0.0495 (7)
C20	0.7035 (2)	0.5328 (3)	0.39791 (11)	0.0491 (7)
H20A	0.7605	0.4865	0.3907	0.059*
C10	0.9432 (2)	0.3058 (2)	0.32409 (11)	0.0498 (7)

H10A	0.9895	0.2497	0.3458	0.060*
H10B	0.9653	0.3062	0.2908	0.060*
C26	0.6549 (2)	0.3585 (2)	0.45304 (10)	0.0469 (7)
C18	0.6593 (3)	0.2961 (3)	0.19921 (12)	0.0620 (8)
H18A	0.6336	0.3795	0.1959	0.074*
H18B	0.6038	0.2440	0.1810	0.074*
C23	0.5348 (2)	0.6716 (3)	0.41814 (11)	0.0531 (8)
H23A	0.4775	0.7180	0.4250	0.064*
C5	0.5499 (2)	0.3928 (2)	0.29164 (10)	0.0483 (7)
H5A	0.5961	0.4578	0.2896	0.058*
C25	0.6379 (2)	0.4852 (2)	0.42916 (10)	0.0424 (7)
C1	0.5066 (2)	0.1839 (3)	0.27949 (11)	0.0555 (8)
H1A	0.5228	0.1063	0.2689	0.067*
N2	0.9992 (2)	0.5726 (2)	0.40511 (9)	0.0585 (7)
C14	0.8955 (2)	0.5644 (3)	0.26991 (11)	0.0544 (8)
H14A	0.8794	0.5037	0.2453	0.065*
C4	0.4555 (2)	0.4132 (2)	0.30981 (10)	0.0496 (7)
H4A	0.4382	0.4912	0.3195	0.060*
C13	0.9350 (2)	0.5368 (2)	0.32053 (10)	0.0428 (7)
C28	0.8287 (2)	0.3282 (2)	0.49474 (10)	0.0445 (7)
H28A	0.8888	0.3801	0.4895	0.053*
C21	0.6867 (2)	0.6487 (3)	0.37683 (11)	0.0518 (7)
H21A	0.7317	0.6791	0.3555	0.062*
N4	0.9507 (3)	0.0307 (2)	0.41143 (10)	0.0699 (8)
C32	1.0312 (3)	0.1611 (3)	0.47307 (11)	0.0518 (8)
C9	0.8295 (2)	0.2592 (2)	0.31804 (11)	0.0461 (7)
H9A	0.8037	0.2596	0.3508	0.055*
C24	0.5520 (2)	0.5564 (3)	0.43830 (10)	0.0531 (8)
H24A	0.5052	0.5252	0.4585	0.064*
C12	0.9611 (2)	0.6265 (3)	0.35794 (11)	0.0488 (7)
C17	0.9458 (3)	0.7485 (3)	0.34484 (15)	0.0641 (9)
H17A	0.9623	0.8099	0.3691	0.077*
C2	0.4127 (3)	0.2027 (3)	0.29789 (12)	0.0613 (9)
H2B	0.3664	0.1377	0.2998	0.074*
C33	1.1130 (3)	0.2305 (3)	0.50087 (13)	0.0649 (9)
H33A	1.1023	0.2780	0.5286	0.078*
C30	0.8832 (3)	0.0654 (3)	0.44097 (12)	0.0656 (9)
H30A	0.8125	0.0392	0.4364	0.079*
C27	0.7613 (2)	0.3925 (3)	0.52859 (11)	0.0530 (8)
H27A	0.7777	0.3631	0.5635	0.064*
H27B	0.7725	0.4800	0.5284	0.064*
C8	0.8164 (3)	0.1342 (2)	0.29330 (12)	0.0569 (8)
H8A	0.8722	0.1190	0.2732	0.068*
H8B	0.8189	0.0708	0.3190	0.068*
C29	0.8691 (2)	0.2051 (3)	0.51457 (11)	0.0539 (8)
H29A	0.9165	0.2153	0.5470	0.065*
H29B	0.8091	0.1553	0.5204	0.065*
C3	0.3870 (2)	0.3165 (3)	0.31343 (11)	0.0502 (7)
C15	0.8816 (2)	0.6849 (3)	0.25791 (13)	0.0649 (9)

H15A	0.8555	0.7070	0.2243	0.078*
C37	0.5676 (3)	0.2741 (3)	0.42735 (13)	0.0655 (9)
H37A	0.5729	0.2677	0.3914	0.079*
H37B	0.4987	0.3101	0.4297	0.079*
C31	1.0450 (3)	0.0900 (3)	0.43109 (12)	0.0600 (9)
C16	0.9053 (3)	0.7754 (3)	0.29473 (16)	0.0705 (10)
H16A	0.8935	0.8565	0.2852	0.085*
C36	1.1447 (4)	0.0868 (3)	0.41568 (14)	0.0819 (11)
H36A	1.1557	0.0400	0.3878	0.098*
C19	0.2627 (3)	0.4400 (3)	0.34839 (14)	0.0859 (11)
H19A	0.1964	0.4324	0.3611	0.129*
H19B	0.2534	0.4948	0.3198	0.129*
H19C	0.3167	0.4714	0.3749	0.129*
C34	1.2100 (3)	0.2246 (4)	0.48492 (17)	0.0868 (11)
H34A	1.2672	0.2685	0.5026	0.104*
C38	0.6542 (3)	0.8897 (3)	0.34226 (14)	0.0837 (11)
H38A	0.6295	0.9701	0.3320	0.125*
H38B	0.6593	0.8418	0.3125	0.125*
H38C	0.7234	0.8948	0.3634	0.125*
C35	1.2257 (4)	0.1541 (4)	0.44256 (19)	0.0967 (13)
H35A	1.2926	0.1532	0.4327	0.116*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0835 (3)	0.0932 (3)	0.0707 (3)	−0.0159 (2)	0.0360 (2)	−0.00961 (19)
Br2	0.0870 (3)	0.0610 (2)	0.1373 (4)	−0.0144 (2)	0.0053 (3)	0.0236 (2)
O3	0.0438 (12)	0.0378 (10)	0.0612 (13)	−0.0029 (9)	0.0027 (10)	−0.0005 (9)
O6	0.0492 (12)	0.0533 (11)	0.0394 (11)	0.0115 (9)	0.0054 (9)	−0.0002 (9)
O5	0.0516 (13)	0.0695 (13)	0.0438 (12)	0.0080 (10)	0.0158 (10)	0.0097 (10)
N1	0.0421 (14)	0.0436 (13)	0.0450 (14)	−0.0011 (11)	0.0027 (11)	−0.0011 (11)
C7	0.0458 (18)	0.0403 (15)	0.0527 (19)	−0.0047 (13)	0.0048 (14)	−0.0043 (13)
O2	0.0483 (13)	0.0421 (11)	0.0715 (14)	−0.0002 (9)	0.0053 (11)	−0.0120 (10)
O1	0.0551 (14)	0.0748 (15)	0.0847 (16)	−0.0046 (12)	0.0280 (12)	−0.0170 (12)
O4	0.0647 (15)	0.0530 (12)	0.0869 (16)	0.0094 (11)	0.0140 (12)	0.0155 (12)
N3	0.0592 (18)	0.0472 (14)	0.0467 (15)	0.0145 (13)	0.0012 (13)	−0.0014 (12)
C6	0.0439 (17)	0.0407 (15)	0.0441 (17)	−0.0025 (14)	0.0006 (13)	−0.0030 (13)
C11	0.0461 (19)	0.062 (2)	0.0464 (19)	−0.0042 (15)	0.0020 (14)	0.0052 (15)
C22	0.0470 (19)	0.0467 (17)	0.0524 (19)	0.0010 (15)	0.0015 (15)	0.0027 (14)
C20	0.0443 (18)	0.0499 (17)	0.0563 (19)	0.0037 (14)	0.0176 (14)	−0.0023 (14)
C10	0.0475 (19)	0.0474 (16)	0.0536 (18)	0.0049 (14)	0.0061 (14)	0.0001 (14)
C26	0.0456 (19)	0.0514 (17)	0.0443 (18)	0.0040 (14)	0.0091 (14)	0.0056 (13)
C18	0.056 (2)	0.072 (2)	0.059 (2)	−0.0019 (17)	0.0093 (16)	−0.0008 (16)
C23	0.0484 (19)	0.0570 (19)	0.0555 (19)	0.0168 (15)	0.0139 (15)	0.0053 (15)
C5	0.0512 (19)	0.0421 (16)	0.0514 (18)	−0.0026 (14)	0.0078 (14)	0.0025 (13)
C25	0.0377 (16)	0.0492 (16)	0.0398 (16)	0.0025 (13)	0.0050 (13)	−0.0011 (13)
C1	0.054 (2)	0.0441 (17)	0.069 (2)	−0.0031 (15)	0.0117 (16)	−0.0111 (15)
N2	0.0551 (17)	0.0638 (17)	0.0552 (16)	−0.0059 (14)	0.0051 (13)	−0.0098 (14)

C14	0.055 (2)	0.0559 (19)	0.052 (2)	−0.0095 (15)	0.0081 (15)	0.0048 (15)
C4	0.058 (2)	0.0401 (15)	0.0506 (18)	0.0059 (15)	0.0082 (15)	−0.0033 (13)
C13	0.0356 (16)	0.0451 (16)	0.0486 (18)	−0.0033 (13)	0.0093 (13)	0.0038 (14)
C28	0.0470 (18)	0.0471 (16)	0.0385 (16)	0.0028 (14)	0.0041 (13)	−0.0030 (13)
C21	0.0473 (19)	0.0535 (18)	0.0576 (19)	−0.0047 (15)	0.0176 (15)	0.0075 (15)
N4	0.093 (2)	0.0568 (16)	0.0565 (18)	0.0188 (17)	0.0036 (17)	−0.0117 (13)
C32	0.067 (2)	0.0425 (16)	0.0447 (18)	0.0194 (16)	0.0062 (16)	0.0063 (14)
C9	0.0510 (19)	0.0432 (15)	0.0437 (17)	0.0011 (14)	0.0067 (14)	0.0024 (13)
C24	0.0443 (19)	0.069 (2)	0.0486 (18)	0.0007 (16)	0.0157 (14)	0.0067 (15)
C12	0.0402 (18)	0.0482 (18)	0.059 (2)	−0.0062 (13)	0.0105 (15)	−0.0053 (15)
C17	0.052 (2)	0.054 (2)	0.089 (3)	−0.0063 (16)	0.0194 (19)	−0.0116 (18)
C2	0.055 (2)	0.0499 (18)	0.080 (2)	−0.0143 (16)	0.0147 (17)	−0.0096 (16)
C33	0.069 (3)	0.061 (2)	0.062 (2)	0.0053 (19)	0.0019 (19)	0.0013 (17)
C30	0.080 (3)	0.0473 (17)	0.061 (2)	0.0081 (18)	−0.0121 (19)	−0.0066 (16)
C27	0.058 (2)	0.0592 (18)	0.0424 (17)	0.0102 (16)	0.0096 (15)	−0.0020 (14)
C8	0.059 (2)	0.0398 (16)	0.070 (2)	−0.0016 (15)	0.0057 (17)	0.0015 (14)
C29	0.060 (2)	0.0574 (18)	0.0435 (17)	0.0101 (16)	0.0060 (15)	0.0000 (14)
C3	0.0467 (19)	0.0559 (19)	0.0478 (18)	−0.0010 (15)	0.0069 (14)	−0.0065 (14)
C15	0.053 (2)	0.067 (2)	0.075 (2)	−0.0106 (17)	0.0108 (17)	0.0244 (19)
C37	0.054 (2)	0.0583 (19)	0.080 (2)	−0.0057 (16)	−0.0011 (18)	0.0133 (17)
C31	0.076 (3)	0.0487 (18)	0.054 (2)	0.0194 (18)	0.0079 (19)	0.0075 (16)
C16	0.059 (2)	0.0464 (19)	0.108 (3)	−0.0020 (17)	0.020 (2)	0.017 (2)
C36	0.103 (3)	0.078 (3)	0.070 (3)	0.028 (3)	0.028 (2)	0.003 (2)
C19	0.071 (3)	0.088 (3)	0.106 (3)	0.013 (2)	0.036 (2)	−0.022 (2)
C34	0.077 (3)	0.090 (3)	0.093 (3)	−0.007 (2)	0.012 (2)	0.004 (2)
C38	0.093 (3)	0.062 (2)	0.098 (3)	−0.006 (2)	0.021 (2)	0.025 (2)
C35	0.076 (3)	0.109 (3)	0.111 (4)	0.018 (3)	0.033 (3)	0.017 (3)

Geometric parameters (Å, °)

Br1—C18	1.950 (3)	C14—C13	1.385 (4)
Br2—C37	1.949 (3)	C14—H14A	0.9300
O3—C7	1.416 (3)	C4—C3	1.385 (4)
O3—C9	1.427 (3)	C4—H4A	0.9300
O6—C26	1.421 (3)	C13—C12	1.397 (4)
O6—C28	1.437 (3)	C28—C29	1.506 (4)
O5—C26	1.407 (3)	C28—C27	1.514 (4)
O5—C27	1.428 (3)	C28—H28A	0.9800
N1—C11	1.356 (3)	C21—H21A	0.9300
N1—C13	1.383 (3)	N4—C30	1.311 (4)
N1—C10	1.454 (3)	N4—C31	1.385 (4)
C7—O2	1.418 (3)	C32—C33	1.394 (4)
C7—C6	1.510 (4)	C32—C31	1.395 (4)
C7—C18	1.519 (4)	C9—C8	1.518 (4)
O2—C8	1.422 (3)	C9—H9A	0.9800
O1—C3	1.360 (4)	C24—H24A	0.9300
O1—C19	1.414 (4)	C12—C17	1.388 (4)
O4—C22	1.367 (3)	C17—C16	1.374 (5)
O4—C38	1.422 (4)	C17—H17A	0.9300

N3—C30	1.361 (4)	C2—C3	1.371 (4)
N3—C32	1.385 (4)	C2—H2B	0.9300
N3—C29	1.449 (4)	C33—C34	1.368 (5)
C6—C5	1.378 (4)	C33—H33A	0.9300
C6—C1	1.382 (4)	C30—H30A	0.9300
C11—N2	1.301 (4)	C27—H27A	0.9700
C11—H11A	0.9300	C27—H27B	0.9700
C22—C21	1.366 (4)	C8—H8A	0.9700
C22—C23	1.382 (4)	C8—H8B	0.9700
C20—C25	1.373 (4)	C29—H29A	0.9700
C20—C21	1.391 (4)	C29—H29B	0.9700
C20—H20A	0.9300	C15—C16	1.390 (5)
C10—C9	1.512 (4)	C15—H15A	0.9300
C10—H10A	0.9700	C37—H37A	0.9700
C10—H10B	0.9700	C37—H37B	0.9700
C26—C37	1.515 (4)	C31—C36	1.392 (5)
C26—C25	1.528 (4)	C16—H16A	0.9300
C18—H18A	0.9700	C36—C35	1.365 (5)
C18—H18B	0.9700	C36—H36A	0.9300
C23—C24	1.375 (4)	C19—H19A	0.9600
C23—H23A	0.9300	C19—H19B	0.9600
C5—C4	1.382 (4)	C19—H19C	0.9600
C5—H5A	0.9300	C34—C35	1.406 (6)
C25—C24	1.394 (4)	C34—H34A	0.9300
C1—C2	1.376 (4)	C38—H38A	0.9600
C1—H1A	0.9300	C38—H38B	0.9600
N2—C12	1.395 (4)	C38—H38C	0.9600
C14—C15	1.364 (4)	C35—H35A	0.9300
C7—O3—C9	108.34 (19)	C33—C32—C31	123.1 (3)
C26—O6—C28	108.4 (2)	O3—C9—C10	108.4 (2)
C26—O5—C27	105.1 (2)	O3—C9—C8	102.4 (2)
C11—N1—C13	106.2 (2)	C10—C9—C8	112.6 (2)
C11—N1—C10	126.9 (2)	O3—C9—H9A	111.1
C13—N1—C10	126.9 (2)	C10—C9—H9A	111.1
O3—C7—O2	106.6 (2)	C8—C9—H9A	111.1
O3—C7—C6	112.0 (2)	C23—C24—C25	121.6 (3)
O2—C7—C6	110.4 (2)	C23—C24—H24A	119.2
O3—C7—C18	108.9 (2)	C25—C24—H24A	119.2
O2—C7—C18	110.6 (2)	C17—C12—N2	130.2 (3)
C6—C7—C18	108.4 (2)	C17—C12—C13	119.6 (3)
C7—O2—C8	109.38 (19)	N2—C12—C13	110.1 (2)
C3—O1—C19	118.2 (2)	C16—C17—C12	117.5 (3)
C22—O4—C38	117.4 (2)	C16—C17—H17A	121.2
C30—N3—C32	106.0 (3)	C12—C17—H17A	121.2
C30—N3—C29	126.6 (3)	C3—C2—C1	120.5 (3)
C32—N3—C29	127.0 (3)	C3—C2—H2B	119.8
C5—C6—C1	118.0 (3)	C1—C2—H2B	119.8
C5—C6—C7	119.9 (2)	C34—C33—C32	116.0 (3)
C1—C6—C7	122.1 (2)	C34—C33—H33A	122.0

N2—C11—N1	114.6 (3)	C32—C33—H33A	122.0
N2—C11—H11A	122.7	N4—C30—N3	114.0 (3)
N1—C11—H11A	122.7	N4—C30—H30A	123.0
C21—C22—O4	124.7 (3)	N3—C30—H30A	123.0
C21—C22—C23	120.4 (3)	O5—C27—C28	103.5 (2)
O4—C22—C23	114.9 (3)	O5—C27—H27A	111.1
C25—C20—C21	121.5 (3)	C28—C27—H27A	111.1
C25—C20—H20A	119.2	O5—C27—H27B	111.1
C21—C20—H20A	119.2	C28—C27—H27B	111.1
N1—C10—C9	114.0 (2)	H27A—C27—H27B	109.0
N1—C10—H10A	108.7	O2—C8—C9	104.5 (2)
C9—C10—H10A	108.7	O2—C8—H8A	110.8
N1—C10—H10B	108.7	C9—C8—H8A	110.8
C9—C10—H10B	108.7	O2—C8—H8B	110.8
H10A—C10—H10B	107.6	C9—C8—H8B	110.8
O5—C26—O6	105.9 (2)	H8A—C8—H8B	108.9
O5—C26—C37	109.9 (2)	N3—C29—C28	111.6 (2)
O6—C26—C37	109.8 (2)	N3—C29—H29A	109.3
O5—C26—C25	111.4 (2)	C28—C29—H29A	109.3
O6—C26—C25	110.7 (2)	N3—C29—H29B	109.3
C37—C26—C25	109.1 (2)	C28—C29—H29B	109.3
C7—C18—Br1	112.5 (2)	H29A—C29—H29B	108.0
C7—C18—H18A	109.1	O1—C3—C2	115.8 (3)
Br1—C18—H18A	109.1	O1—C3—C4	124.7 (3)
C7—C18—H18B	109.1	C2—C3—C4	119.5 (3)
Br1—C18—H18B	109.1	C14—C15—C16	121.6 (3)
H18A—C18—H18B	107.8	C14—C15—H15A	119.2
C24—C23—C22	119.3 (3)	C16—C15—H15A	119.2
C24—C23—H23A	120.3	C26—C37—Br2	114.2 (2)
C22—C23—H23A	120.3	C26—C37—H37A	108.7
C6—C5—C4	121.6 (3)	Br2—C37—H37A	108.7
C6—C5—H5A	119.2	C26—C37—H37B	108.7
C4—C5—H5A	119.2	Br2—C37—H37B	108.7
C20—C25—C24	117.6 (3)	H37A—C37—H37B	107.6
C20—C25—C26	122.6 (2)	N4—C31—C36	130.3 (3)
C24—C25—C26	119.8 (3)	N4—C31—C32	110.3 (3)
C2—C1—C6	121.0 (3)	C36—C31—C32	119.4 (4)
C2—C1—H1A	119.5	C17—C16—C15	121.9 (3)
C6—C1—H1A	119.5	C17—C16—H16A	119.0
C11—N2—C12	104.0 (2)	C15—C16—H16A	119.0
C15—C14—C13	116.7 (3)	C35—C36—C31	118.3 (4)
C15—C14—H14A	121.7	C35—C36—H36A	120.9
C13—C14—H14A	121.7	C31—C36—H36A	120.9
C5—C4—C3	119.4 (3)	O1—C19—H19A	109.5
C5—C4—H4A	120.3	O1—C19—H19B	109.5
C3—C4—H4A	120.3	H19A—C19—H19B	109.5
N1—C13—C14	132.3 (3)	O1—C19—H19C	109.5
N1—C13—C12	105.1 (2)	H19A—C19—H19C	109.5
C14—C13—C12	122.6 (3)	H19B—C19—H19C	109.5

O6—C28—C29	108.7 (2)	C33—C34—C35	121.9 (4)
O6—C28—C27	103.8 (2)	C33—C34—H34A	119.0
C29—C28—C27	113.8 (2)	C35—C34—H34A	119.0
O6—C28—H28A	110.1	O4—C38—H38A	109.5
C29—C28—H28A	110.1	O4—C38—H38B	109.5
C27—C28—H28A	110.1	H38A—C38—H38B	109.5
C22—C21—C20	119.6 (3)	O4—C38—H38C	109.5
C22—C21—H21A	120.2	H38A—C38—H38C	109.5
C20—C21—H21A	120.2	H38B—C38—H38C	109.5
C30—N4—C31	104.4 (3)	C36—C35—C34	121.3 (4)
N3—C32—C33	131.6 (3)	C36—C35—H35A	119.3
N3—C32—C31	105.3 (3)	C34—C35—H35A	119.3
C9—O3—C7—O2	−21.4 (3)	C30—N3—C32—C31	−0.4 (3)
C9—O3—C7—C6	99.3 (2)	C29—N3—C32—C31	−174.4 (2)
C9—O3—C7—C18	−140.8 (2)	C7—O3—C9—C10	148.8 (2)
O3—C7—O2—C8	3.1 (3)	C7—O3—C9—C8	29.7 (3)
C6—C7—O2—C8	−118.7 (3)	N1—C10—C9—O3	65.3 (3)
C18—C7—O2—C8	121.3 (3)	N1—C10—C9—C8	177.8 (2)
O3—C7—C6—C5	37.9 (3)	C22—C23—C24—C25	0.7 (4)
O2—C7—C6—C5	156.4 (2)	C20—C25—C24—C23	−1.9 (4)
C18—C7—C6—C5	−82.3 (3)	C26—C25—C24—C23	179.0 (3)
O3—C7—C6—C1	−144.5 (3)	C11—N2—C12—C17	178.9 (3)
O2—C7—C6—C1	−25.9 (4)	C11—N2—C12—C13	−0.2 (3)
C18—C7—C6—C1	95.4 (3)	N1—C13—C12—C17	−179.0 (3)
C13—N1—C11—N2	−0.1 (3)	C14—C13—C12—C17	1.1 (4)
C10—N1—C11—N2	−179.3 (3)	N1—C13—C12—N2	0.2 (3)
C38—O4—C22—C21	−5.2 (4)	C14—C13—C12—N2	−179.7 (3)
C38—O4—C22—C23	174.8 (3)	N2—C12—C17—C16	−179.2 (3)
C11—N1—C10—C9	98.5 (3)	C13—C12—C17—C16	−0.2 (4)
C13—N1—C10—C9	−80.5 (3)	C6—C1—C2—C3	0.3 (5)
C27—O5—C26—O6	35.2 (3)	N3—C32—C33—C34	178.4 (3)
C27—O5—C26—C37	153.8 (2)	C31—C32—C33—C34	−0.5 (5)
C27—O5—C26—C25	−85.2 (3)	C31—N4—C30—N3	−0.1 (3)
C28—O6—C26—O5	−20.4 (3)	C32—N3—C30—N4	0.4 (3)
C28—O6—C26—C37	−139.0 (2)	C29—N3—C30—N4	174.3 (3)
C28—O6—C26—C25	100.5 (2)	C26—O5—C27—C28	−35.5 (3)
O3—C7—C18—Br1	56.3 (3)	O6—C28—C27—O5	22.5 (3)
O2—C7—C18—Br1	−60.5 (3)	C29—C28—C27—O5	−95.5 (3)
C6—C7—C18—Br1	178.31 (18)	C7—O2—C8—C9	14.9 (3)
C21—C22—C23—C24	1.1 (4)	O3—C9—C8—O2	−26.8 (3)
O4—C22—C23—C24	−178.8 (3)	C10—C9—C8—O2	−143.0 (2)
C1—C6—C5—C4	0.5 (4)	C30—N3—C29—C28	−91.2 (3)
C7—C6—C5—C4	178.3 (3)	C32—N3—C29—C28	81.6 (3)
C21—C20—C25—C24	1.2 (4)	O6—C28—C29—N3	60.3 (3)
C21—C20—C25—C26	−179.7 (3)	C27—C28—C29—N3	175.4 (2)
O5—C26—C25—C20	129.8 (3)	C19—O1—C3—C2	−178.7 (3)
O6—C26—C25—C20	12.2 (4)	C19—O1—C3—C4	2.9 (4)
C37—C26—C25—C20	−108.7 (3)	C1—C2—C3—O1	−177.6 (3)
O5—C26—C25—C24	−51.2 (3)	C1—C2—C3—C4	0.9 (5)

O6—C26—C25—C24	−168.7 (2)	C5—C4—C3—O1	177.0 (3)
C37—C26—C25—C24	70.3 (3)	C5—C4—C3—C2	−1.4 (4)
C5—C6—C1—C2	−1.0 (4)	C13—C14—C15—C16	−0.3 (5)
C7—C6—C1—C2	−178.7 (3)	O5—C26—C37—Br2	−54.0 (3)
N1—C11—N2—C12	0.2 (3)	O6—C26—C37—Br2	62.2 (3)
C6—C5—C4—C3	0.7 (4)	C25—C26—C37—Br2	−176.4 (2)
C11—N1—C13—C14	179.8 (3)	C30—N4—C31—C36	179.2 (3)
C10—N1—C13—C14	−1.0 (5)	C30—N4—C31—C32	−0.2 (3)
C11—N1—C13—C12	0.0 (3)	N3—C32—C31—N4	0.4 (3)
C10—N1—C13—C12	179.1 (2)	C33—C32—C31—N4	179.5 (3)
C15—C14—C13—N1	179.3 (3)	N3—C32—C31—C36	−179.0 (3)
C15—C14—C13—C12	−0.8 (4)	C33—C32—C31—C36	0.1 (4)
C26—O6—C28—C29	119.8 (2)	C12—C17—C16—C15	−0.9 (5)
C26—O6—C28—C27	−1.6 (3)	C14—C15—C16—C17	1.2 (5)
O4—C22—C21—C20	178.2 (3)	N4—C31—C36—C35	−179.3 (3)
C23—C22—C21—C20	−1.7 (4)	C32—C31—C36—C35	0.0 (5)
C25—C20—C21—C22	0.5 (4)	C32—C33—C34—C35	0.9 (5)
C30—N3—C32—C33	−179.5 (3)	C31—C36—C35—C34	0.3 (6)
C29—N3—C32—C33	6.6 (5)	C33—C34—C35—C36	−0.8 (6)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C37—H37A···Cg	0.97	2.65	3.515	148
C15—H15A···O1^{i}	0.93	2.56	3.362 (5)	144
C20—H20A···O6	0.93	2.44	2.788 (4)	102

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

Fig. 1

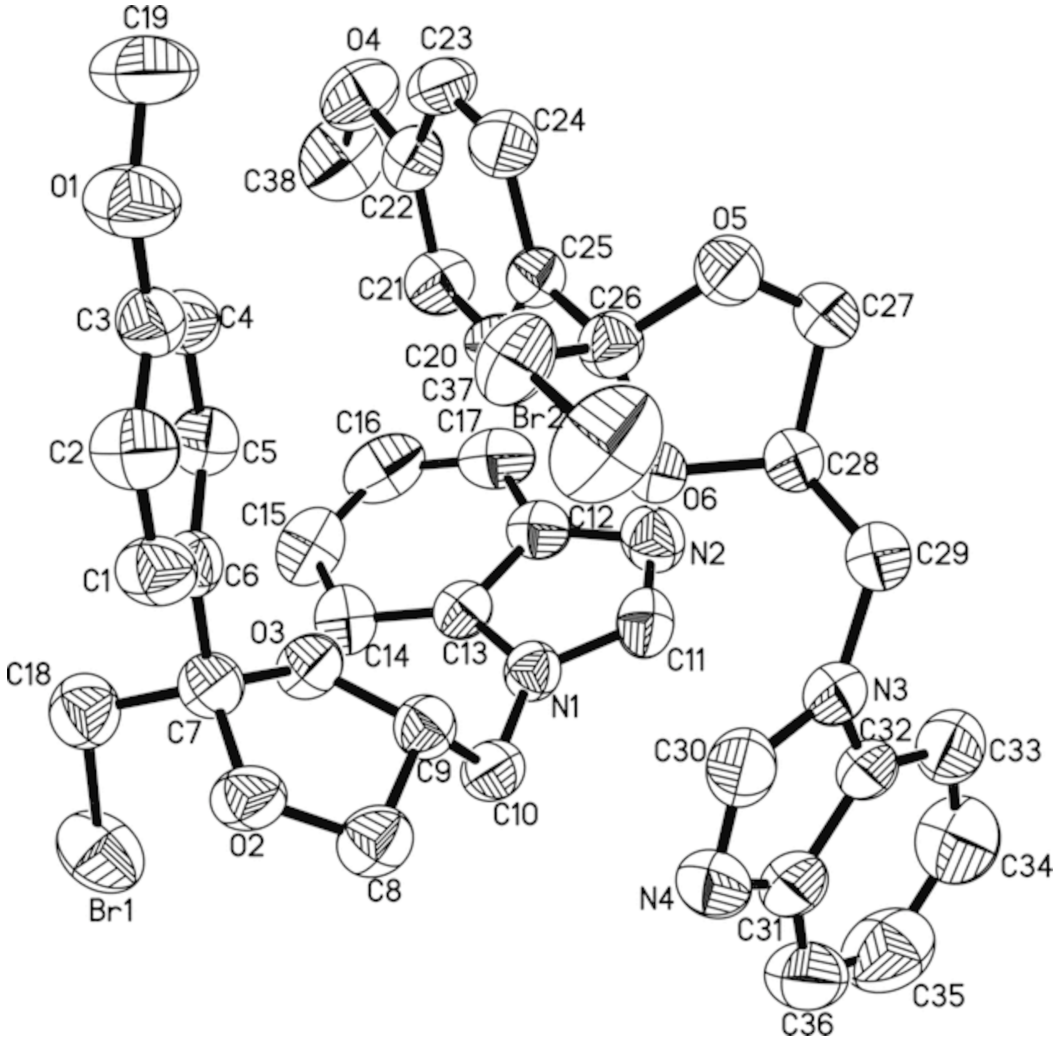
C19
O4
C23
C24
O1
C38
C22
O5
C25
C21
C26
C27
C3
C4
C20
C37
Br2
C16
C17
C2
C5
O6
C28
C15
C12
N2
C29
C1
C6
O3
C14
C13
C11
C18
N3
N1
C7
C32
C33
C30
C9
C10
O2
C34
C8
N4
C31
Br1
C35
C36

Fig. 2