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2-*n*-Butyl-3-[2′-(1*H*-tetrazol-5-yl)-biphenyl-4-ylmethyl]-1-azonia-3-aza-spiro[4.4]non-1-en-4-one bromide sesquihydrate

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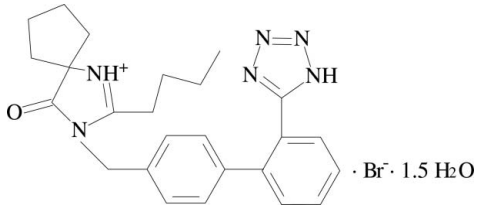
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.049; wR factor = 0.150; data-to-parameter ratio = 13.8.

The crystal structure of the title compound, irbesartan bromide sesquihydrate, $C_{25}H_{29}N_6O^+\cdot Br^-\cdot 1.5H_2O$, has networks of hydrogen bonds which link Br^-, solvent water molecules and irbesartan cations (which adopt tautomeric form *A*) into infinite chains.

Related literature

For related literature, see: Bartolucci *et al.* (2007); Goldmann & Stoltefuss (1991). Tautomeric form *A* is defined by Bernhart *et al.* (1997).



Experimental

Crystal data

$C_{25}H_{29}N_6O^+\cdot Br^-\cdot 1.5H_2O$
M_r = 1072.95
Monoclinic, $P2_1/c$
a = 12.482 (3) Å
b = 25.285 (5) Å
c = 8.6938 (17) Å
β = 105.78 (3)°
V = 2640.4 (9) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.59 mm^{-1}
T = 293 (2) K
0.21 × 0.13 × 0.13 mm

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.727, T_{max} = 0.826
21153 measured reflections
4907 independent reflections
3402 reflections with $I > 2\sigma(I)$
R_{int} = 0.056

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.150
S = 1.04
4907 reflections
355 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.81 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N6—H6A···Br1	0.93 (4)	2.31 (4)	3.241 (3)	174 (3)
O1W—HW1A···Br1^{i}	0.73 (6)	2.59 (7)	3.309 (3)	168 (7)
O1W—HW1B···N3^{ii}	0.87 (6)	2.04 (6)	2.886 (4)	163 (6)
O2W—HW2A···O1	0.87 (2)	2.52 (7)	3.068 (6)	122 (7)
O2W—HW2B···Br1^{iii}	0.88 (2)	2.58 (3)	3.456 (5)	169 (8)
N1—H1A···O1W	0.64 (4)	2.10 (4)	2.735 (5)	171 (5)

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

Data collection: *RAPID-AUTO* (Rigaku/MSC, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); 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: RN2028).

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supplementary materials

Acta Cryst. (2007). E**63**, o4933 [**doi:10.1107/S1600536807061156**]

2-*n*-Butyl-3-[2'-(1*H*-tetrazol-5-yl)biphenyl-4-ylmethyl]-1-azonia-3-azaspiro[4.4]non-1-en-4-one bromide sesquihydrate

L. Wang, L.-N. Zhou, Y. Bao and J.-K. Wang

Comment

2-*n*-Butyl-3-((2'-(2*H*-tetrazol-5-yl)biphenyl-4-yl)methyl)-1,3-diazaspiro(4.4)no n-1-en-4-one is an antihypertensive agent that inhibits the renin-angiotensin system by selectively blocking the AT1 subtype of receptors. The title compound, 2-*n*-Butyl-3-((2'-(2*H*-tetrazol-5-yl)biphenyl-4-yl)methyl)-1,3- diazaspiro(4.4)non-1-en-4-one hydrobromide 1.5-hydrate, is an acidic salt hydrate of irbesartan.

In this paper, the single-crystal of the title compound was obtained and the crystal structure was determined. As shown in Fig. 2, the crystal packing is stabilized by a sets of hydrogen bonds between Br^{-}, O1W of the solvent water and N6 in the irbesartan cation. There are also hydrogen bonds between O1W and N1 of the irbesartan cation.

The crystal structure reported is very similar to $C_{25}H_{29}N_6O^{+}$. Cl^{-}.-1.69H_2O (Bartolucci *et al.*, 2007) which is the Cl rather than Br salt.

Experimental

The title compound was prepared as follows:2-*n*-Butyl-4-spirocyclopentane −1-[(2'-(triphenyl-methyltetrazol-5-yl)biphenyl-4-yl)methyl]-2-imidazolin-5-one (1.96 g) was dissolved in 10 ml me thanol and 10 ml THF. After the reaction medium had been cooled to 298 K, 4*M* hydrochloric acid (1.5 ml) was added and the mixture stirred for 3 h at RT and 1 h at 303 K. After evaporation of the solvents, the residue is taken up in water and the pH is brought to 12 by addition of 10*M* sodium hydroxide solution. The aqueous phase is extracted with ether, toluene and ether, acidified to pH 2 by 1*M* hydrochloric acid, extracted with ethyl acetate and the extract is dried and evaporated. Then the product powder is dispersed or dissolved in ethanol and mixed with HBr solution, after the addition of water crystals of good quality appeared after a few days.

Refinement

H atoms were placed in calculated positions and constrained to ride on their parent atoms with distances C–H =0.93–0.98 Å, N–H =0.90Å and U_{iso}(H) = 1.2U_{eq}(C).

Figures

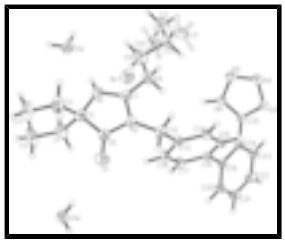

Fig. 1. An *ORTEP* plot of the title compound (I). Displacement ellipsoids are drawn at the 30% probability level.

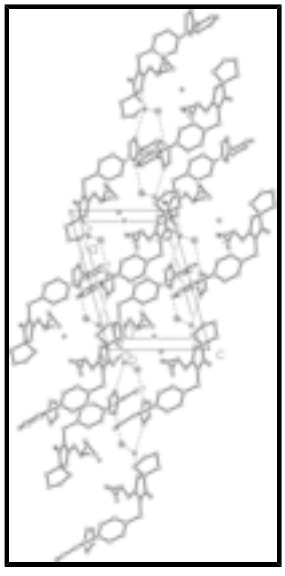

Fig. 2. The packing diagram of (I).

2-*n*-Butyl-3-[2'-(1*H*-tetrazol-5-yl)biphenyl-4-ylmethyl]- 1-azonia-3-azaspiro[4.4]non-1-en-4-one bromide sesquihydrate

Crystal data

$C_{25}H_{29}N_6O^+\cdot Br^-\cdot 1.5H_2O$	F_{000} = 1116
M_r = 1072.95	D_x = 1.350 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 12.482 (3) Å	Cell parameters from 14089 reflections
b = 25.285 (5) Å	θ = 3.0–27.5º
c = 8.6938 (17) Å	μ = 1.59 mm^{-1}
β = 105.78 (3)º	T = 293 (2) K
V = 2640.4 (9) Å^3	Plate, colorless
Z = 2	0.21 × 0.13 × 0.13 mm

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer	4907 independent reflections
Radiation source: rotating anode	3402 reflections with $I > 2\sigma(I)$

Monochromator: graphite	R_{int} = 0.056
T = 293(2) K	θ_{max} = 25.5º
oscillation scans	θ_{min} = 3.0º
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	h = −14→15
T_{min} = 0.727, T_{max} = 0.826	k = −30→30
21153 measured reflections	l = −10→9

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.049	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.150	$w = 1/[\sigma^2(F_o^2) + (0.0885P)^2 + 0.3523P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max}$ = 0.001
4907 reflections	$\Delta\rho_{max}$ = 0.81 e Å^{-3}
355 parameters	$\Delta\rho_{min}$ = −0.53 e Å^{-3}
3 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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}	Occ. (<1)
N1	0.0224 (3)	0.10652 (14)	0.6152 (4)	0.0506 (9)	
N2	0.1841 (2)	0.14371 (11)	0.6523 (3)	0.0418 (6)	
N3	0.6216 (2)	0.03416 (11)	1.3916 (3)	0.0445 (7)	
N4	0.5717 (3)	−0.01012 (12)	1.3141 (4)	0.0530 (8)	
N5	0.4646 (3)	−0.00437 (11)	1.2715 (3)	0.0507 (7)	
N6	0.4445 (2)	0.04420 (10)	1.3203 (3)	0.0408 (6)	
H6A	0.371 (3)	0.0550 (14)	1.305 (4)	0.042 (9)*	
O1	0.1343 (3)	0.22304 (12)	0.5236 (4)	0.0809 (9)	
O1W	−0.1488 (3)	0.03602 (13)	0.5792 (5)	0.0806 (11)	

HW1A	−0.149 (6)	0.009 (3)	0.610 (8)	0.121*	
HW1B	−0.215 (5)	0.042 (2)	0.516 (7)	0.121*	
O2W	0.0459 (4)	0.3369 (2)	0.4848 (6)	0.0544 (13)	0.50
HW2A	0.056 (8)	0.313 (2)	0.559 (7)	0.082*	0.50
HW2B	0.074 (7)	0.360 (2)	0.563 (7)	0.082*	0.50
C1	−0.0819 (4)	0.19128 (18)	0.6060 (5)	0.0668 (11)	
H1B	−0.1215	0.1697	0.6649	0.080*	
H1C	−0.0394	0.2179	0.6773	0.080*	
C2	−0.1620 (4)	0.2167 (2)	0.4636 (6)	0.0894 (16)	
H2A	−0.2324	0.2247	0.4857	0.107*	
H2B	−0.1312	0.2491	0.4334	0.107*	
C3	−0.1763 (4)	0.1762 (3)	0.3367 (6)	0.1002 (19)	
H3B	−0.2026	0.1923	0.2320	0.120*	
H3C	−0.2299	0.1497	0.3480	0.120*	
C4	−0.0651 (4)	0.1516 (2)	0.3560 (5)	0.0735 (13)	
H4B	−0.0244	0.1700	0.2918	0.088*	
H4C	−0.0727	0.1147	0.3240	0.088*	
C5	−0.0047 (3)	0.15675 (14)	0.5355 (4)	0.0464 (8)	
C6	0.1091 (3)	0.18060 (14)	0.5643 (4)	0.0486 (9)	
C7	0.1278 (3)	0.10035 (13)	0.6782 (4)	0.0445 (8)	
C8	0.1858 (3)	0.05499 (18)	0.7725 (6)	0.0751 (13)	
H8A	0.2271	0.0369	0.7084	0.090*	
H8B	0.2400	0.0690	0.8656	0.090*	
C9	0.1196 (5)	0.0156 (2)	0.8278 (6)	0.0840 (15)	
H9A	0.0651	0.0004	0.7372	0.101*	
H9B	0.0804	0.0321	0.8973	0.101*	
C10	0.1988 (9)	−0.0287 (4)	0.9208 (13)	0.071 (3)	0.636 (14)
H10A	0.2286	−0.0486	0.8465	0.085*	0.636 (14)
H10B	0.2608	−0.0125	0.9988	0.085*	0.636 (14)
C10'	0.1449 (16)	−0.0210 (7)	0.944 (2)	0.071 (5)	0.364 (14)
H10C	0.0833	−0.0454	0.9336	0.085*	0.364 (14)
H10D	0.1600	−0.0041	1.0484	0.085*	0.364 (14)
C11	0.1412 (8)	−0.0645 (4)	1.0018 (10)	0.094 (4)	0.636 (14)
H11A	0.1921	−0.0910	1.0576	0.141*	0.636 (14)
H11B	0.0807	−0.0811	0.9247	0.141*	0.636 (14)
H11C	0.1126	−0.0449	1.0768	0.141*	0.636 (14)
C11'	0.2466 (16)	−0.0499 (6)	0.9276 (16)	0.069 (5)	0.364 (14)
H11D	0.2684	−0.0759	1.0107	0.103*	0.364 (14)
H11E	0.3062	−0.0251	0.9360	0.103*	0.364 (14)
H11F	0.2299	−0.0670	0.8253	0.103*	0.364 (14)
C12	0.3046 (3)	0.14967 (16)	0.6926 (4)	0.0481 (9)	
H12A	0.3223	0.1807	0.6385	0.058*	
H12B	0.3361	0.1191	0.6532	0.058*	
C13	0.3579 (3)	0.15515 (13)	0.8688 (4)	0.0401 (7)	
C14	0.4569 (3)	0.12912 (13)	0.9378 (4)	0.0407 (7)	
H14A	0.4902	0.1088	0.8744	0.049*	
C15	0.5066 (3)	0.13312 (12)	1.0994 (4)	0.0389 (7)	
H15A	0.5744	0.1164	1.1433	0.047*	
C16	0.4569 (3)	0.16183 (12)	1.1981 (4)	0.0379 (7)	

C17	0.3577 (3)	0.18849 (13)	1.1274 (4)	0.0444 (8)
H17A	0.3234	0.2084	1.1906	0.053*
C18	0.3103 (3)	0.18558 (14)	0.9653 (4)	0.0466 (8)
H18A	0.2453	0.2043	0.9197	0.056*
C19	0.5100 (3)	0.16517 (12)	1.3729 (3)	0.0357 (7)
C20	0.5238 (3)	0.21398 (13)	1.4513 (4)	0.0461 (8)
H20A	0.4935	0.2441	1.3947	0.055*
C21	0.5811 (3)	0.21855 (14)	1.6101 (4)	0.0502 (9)
H21A	0.5884	0.2514	1.6599	0.060*
C22	0.6275 (3)	0.17448 (15)	1.6950 (4)	0.0526 (9)
H22A	0.6679	0.1777	1.8016	0.063*
C23	0.6142 (3)	0.12572 (14)	1.6229 (4)	0.0455 (8)
H23A	0.6449	0.0959	1.6813	0.055*
C24	0.5553 (3)	0.12060 (12)	1.4634 (4)	0.0368 (7)
C25	0.5414 (3)	0.06719 (12)	1.3938 (3)	0.0358 (7)
H1A	−0.014 (4)	0.0882 (16)	0.613 (5)	0.047 (14)*
Br1	0.19252 (3)	0.083154 (16)	1.29277 (5)	0.06089 (18)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0354 (18)	0.0413 (19)	0.072 (2)	−0.0086 (15)	0.0101 (15)	−0.0011 (15)
N2	0.0329 (14)	0.0439 (16)	0.0464 (15)	−0.0026 (12)	0.0070 (11)	−0.0012 (12)
N3	0.0402 (16)	0.0370 (15)	0.0542 (16)	0.0048 (12)	0.0096 (13)	−0.0015 (12)
N4	0.056 (2)	0.0385 (17)	0.0635 (18)	0.0048 (14)	0.0142 (15)	−0.0057 (14)
N5	0.059 (2)	0.0331 (16)	0.0590 (18)	−0.0020 (13)	0.0133 (15)	−0.0055 (13)
N6	0.0365 (16)	0.0325 (15)	0.0534 (16)	−0.0006 (12)	0.0126 (13)	−0.0033 (12)
O1	0.067 (2)	0.0533 (18)	0.117 (2)	−0.0077 (15)	0.0161 (18)	0.0279 (17)
O1W	0.0474 (17)	0.071 (2)	0.107 (3)	−0.0213 (16)	−0.0074 (16)	0.0237 (18)
O2W	0.047 (3)	0.055 (3)	0.050 (3)	0.009 (2)	−0.007 (2)	−0.014 (2)
C1	0.060 (3)	0.069 (3)	0.074 (3)	0.015 (2)	0.023 (2)	0.002 (2)
C2	0.065 (3)	0.106 (4)	0.105 (4)	0.040 (3)	0.036 (3)	0.040 (3)
C3	0.058 (3)	0.142 (5)	0.085 (3)	0.018 (3)	−0.007 (3)	0.018 (4)
C4	0.052 (2)	0.099 (4)	0.059 (2)	0.009 (2)	−0.0013 (19)	−0.004 (2)
C5	0.0358 (18)	0.047 (2)	0.0525 (19)	0.0030 (15)	0.0061 (15)	0.0030 (15)
C6	0.042 (2)	0.046 (2)	0.056 (2)	−0.0014 (16)	0.0097 (16)	0.0057 (16)
C7	0.041 (2)	0.0366 (18)	0.055 (2)	−0.0014 (15)	0.0122 (16)	−0.0017 (15)
C8	0.052 (3)	0.049 (3)	0.119 (4)	0.0085 (19)	0.015 (3)	0.024 (2)
C9	0.098 (4)	0.075 (3)	0.085 (3)	0.022 (3)	0.035 (3)	0.026 (3)
C10	0.067 (7)	0.046 (6)	0.092 (7)	0.019 (5)	0.009 (5)	0.012 (5)
C10'	0.081 (12)	0.062 (10)	0.077 (9)	0.028 (9)	0.037 (9)	0.010 (7)
C11	0.119 (8)	0.081 (7)	0.077 (5)	−0.005 (5)	0.018 (5)	0.021 (5)
C11'	0.091 (13)	0.047 (9)	0.060 (8)	0.008 (7)	0.009 (8)	−0.004 (6)
C12	0.0321 (18)	0.063 (2)	0.0475 (19)	−0.0043 (16)	0.0084 (14)	−0.0026 (16)
C13	0.0348 (17)	0.0400 (18)	0.0456 (17)	−0.0040 (14)	0.0109 (14)	−0.0030 (14)
C14	0.0377 (18)	0.0417 (19)	0.0459 (18)	−0.0005 (14)	0.0169 (14)	−0.0043 (14)
C15	0.0321 (17)	0.0364 (17)	0.0492 (18)	0.0038 (13)	0.0126 (14)	0.0009 (14)
C16	0.0356 (17)	0.0295 (16)	0.0493 (17)	−0.0013 (13)	0.0130 (14)	−0.0006 (13)

C17	0.0400 (19)	0.0425 (19)	0.0518 (19)	0.0051 (15)	0.0143 (15)	−0.0056 (15)
C18	0.0331 (17)	0.049 (2)	0.055 (2)	0.0071 (15)	0.0059 (15)	−0.0008 (16)
C19	0.0354 (17)	0.0331 (17)	0.0424 (17)	−0.0008 (13)	0.0167 (14)	−0.0044 (13)
C20	0.049 (2)	0.0365 (19)	0.057 (2)	0.0043 (15)	0.0219 (16)	−0.0009 (15)
C21	0.062 (2)	0.041 (2)	0.051 (2)	−0.0072 (17)	0.0224 (17)	−0.0145 (16)
C22	0.064 (2)	0.052 (2)	0.0434 (19)	−0.0108 (19)	0.0172 (17)	−0.0103 (16)
C23	0.049 (2)	0.044 (2)	0.0416 (18)	−0.0025 (16)	0.0085 (15)	0.0005 (15)
C24	0.0313 (16)	0.0330 (17)	0.0481 (18)	−0.0043 (13)	0.0139 (13)	−0.0052 (14)
C25	0.0357 (17)	0.0326 (16)	0.0381 (16)	0.0005 (13)	0.0086 (13)	0.0000 (13)
Br1	0.0465 (3)	0.0590 (3)	0.0773 (3)	0.00469 (18)	0.0172 (2)	0.00558 (19)

Geometric parameters (Å, °)

N1—C7	1.290 (5)	C9—H9B	0.9700
N1—C5	1.442 (5)	C10—C11	1.452 (15)
N1—H1A	0.64 (4)	C10—H10A	0.9700
N2—C7	1.354 (4)	C10—H10B	0.9700
N2—C6	1.394 (4)	C10'—C11'	1.51 (2)
N2—C12	1.456 (4)	C10'—H10C	0.9700
N3—C25	1.308 (4)	C10'—H10D	0.9700
N3—N4	1.366 (4)	C11—H11A	0.9600
N4—N5	1.295 (4)	C11—H11B	0.9600
N5—N6	1.345 (4)	C11—H11C	0.9600
N6—C25	1.337 (4)	C11'—H11D	0.9600
N6—H6A	0.93 (4)	C11'—H11E	0.9600
O1—C6	1.199 (4)	C11'—H11F	0.9600
O1W—HW1A	0.73 (6)	C12—C13	1.501 (4)
O1W—HW1B	0.87 (6)	C12—H12A	0.9700
O2W—HW2A	0.87 (2)	C12—H12B	0.9700
O2W—HW2B	0.88 (2)	C13—C14	1.385 (5)
C1—C2	1.508 (6)	C13—C18	1.386 (5)
C1—C5	1.546 (5)	C14—C15	1.377 (5)
C1—H1B	0.9700	C14—H14A	0.9300
C1—H1C	0.9700	C15—C16	1.392 (4)
C2—C3	1.481 (8)	C15—H15A	0.9300
C2—H2A	0.9700	C16—C17	1.397 (4)
C2—H2B	0.9700	C16—C19	1.486 (4)
C3—C4	1.487 (7)	C17—C18	1.374 (5)
C3—H3B	0.9700	C17—H17A	0.9300
C3—H3C	0.9700	C18—H18A	0.9300
C4—C5	1.543 (5)	C19—C20	1.397 (4)
C4—H4B	0.9700	C19—C24	1.403 (4)
C4—H4C	0.9700	C20—C21	1.377 (5)
C5—C6	1.500 (5)	C20—H20A	0.9300
C7—C8	1.480 (5)	C21—C22	1.375 (5)
C8—C9	1.456 (7)	C21—H21A	0.9300
C8—H8A	0.9700	C22—C23	1.373 (5)
C8—H8B	0.9700	C22—H22A	0.9300
C9—C10'	1.345 (17)	C23—C24	1.387 (4)

C9—C10	1.565 (10)	C23—H23A	0.9300
C9—H9A	0.9700	C24—C25	1.471 (4)
C7—N1—C5	112.9 (3)	C9—C10—H10A	109.3
C7—N1—H1A	123 (4)	C11—C10—H10B	109.3
C5—N1—H1A	124 (4)	C9—C10—H10B	109.3
C7—N2—C6	109.4 (3)	H10A—C10—H10B	107.9
C7—N2—C12	126.4 (3)	C9—C10'—C11'	106.9 (12)
C6—N2—C12	124.0 (3)	C9—C10'—H10C	110.3
C25—N3—N4	106.4 (3)	C11'—C10'—H10C	110.3
N5—N4—N3	110.2 (3)	C9—C10'—H10D	110.3
N4—N5—N6	106.2 (3)	C11'—C10'—H10D	110.3
C25—N6—N5	109.0 (3)	H10C—C10'—H10D	108.6
C25—N6—H6A	132 (2)	C10—C11—H11A	109.5
N5—N6—H6A	119 (2)	C10—C11—H11B	109.5
HW1A—O1W—HW1B	107 (6)	H11A—C11—H11B	109.5
HW2A—O2W—HW2B	87 (2)	C10—C11—H11C	109.5
C2—C1—C5	105.1 (3)	H11A—C11—H11C	109.5
C2—C1—H1B	110.7	H11B—C11—H11C	109.5
C5—C1—H1B	110.7	C10'—C11'—H11D	109.5
C2—C1—H1C	110.7	C10'—C11'—H11E	109.5
C5—C1—H1C	110.7	H11D—C11'—H11E	109.5
H1B—C1—H1C	108.8	C10'—C11'—H11F	109.5
C3—C2—C1	103.6 (4)	H11D—C11'—H11F	109.5
C3—C2—H2A	111.0	H11E—C11'—H11F	109.5
C1—C2—H2A	111.0	N2—C12—C13	113.4 (3)
C3—C2—H2B	111.0	N2—C12—H12A	108.9
C1—C2—H2B	111.0	C13—C12—H12A	108.9
H2A—C2—H2B	109.0	N2—C12—H12B	108.9
C2—C3—C4	106.6 (4)	C13—C12—H12B	108.9
C2—C3—H3B	110.4	H12A—C12—H12B	107.7
C4—C3—H3B	110.4	C14—C13—C18	118.8 (3)
C2—C3—H3C	110.4	C14—C13—C12	119.6 (3)
C4—C3—H3C	110.4	C18—C13—C12	121.7 (3)
H3B—C3—H3C	108.6	C15—C14—C13	120.5 (3)
C3—C4—C5	105.3 (4)	C15—C14—H14A	119.7
C3—C4—H4B	110.7	C13—C14—H14A	119.7
C5—C4—H4B	110.7	C14—C15—C16	121.0 (3)
C3—C4—H4C	110.7	C14—C15—H15A	119.5
C5—C4—H4C	110.7	C16—C15—H15A	119.5
H4B—C4—H4C	108.8	C15—C16—C17	118.1 (3)
N1—C5—C6	100.7 (3)	C15—C16—C19	120.7 (3)
N1—C5—C4	113.4 (3)	C17—C16—C19	121.2 (3)
C6—C5—C4	112.5 (3)	C18—C17—C16	120.6 (3)
N1—C5—C1	113.4 (3)	C18—C17—H17A	119.7
C6—C5—C1	111.8 (3)	C16—C17—H17A	119.7
C4—C5—C1	105.2 (3)	C17—C18—C13	121.0 (3)
O1—C6—N2	124.8 (3)	C17—C18—H18A	119.5
O1—C6—C5	128.6 (3)	C13—C18—H18A	119.5
N2—C6—C5	106.7 (3)	C20—C19—C24	117.3 (3)

N1—C7—N2	110.3 (3)	C20—C19—C16	120.7 (3)
N1—C7—C8	128.2 (4)	C24—C19—C16	121.9 (3)
N2—C7—C8	121.4 (3)	C21—C20—C19	121.6 (3)
C9—C8—C7	118.4 (4)	C21—C20—H20A	119.2
C9—C8—H8A	107.7	C19—C20—H20A	119.2
C7—C8—H8A	107.7	C22—C21—C20	119.9 (3)
C9—C8—H8B	107.7	C22—C21—H21A	120.0
C7—C8—H8B	107.7	C20—C21—H21A	120.0
H8A—C8—H8B	107.1	C23—C22—C21	120.1 (3)
C10'—C9—C8	133.0 (10)	C23—C22—H22A	119.9
C10'—C9—C10	29.9 (7)	C21—C22—H22A	119.9
C8—C9—C10	108.9 (6)	C22—C23—C24	120.4 (3)
C10'—C9—H9A	107.9	C22—C23—H23A	119.8
C8—C9—H9A	109.9	C24—C23—H23A	119.8
C10—C9—H9A	109.9	C23—C24—C19	120.6 (3)
C10'—C9—H9B	83.1	C23—C24—C25	117.9 (3)
C8—C9—H9B	109.9	C19—C24—C25	121.5 (3)
C10—C9—H9B	109.9	N3—C25—N6	108.2 (3)
H9A—C9—H9B	108.3	N3—C25—C24	125.9 (3)
C11—C10—C9	111.7 (9)	N6—C25—C24	125.9 (3)
C11—C10—H10A	109.3		
C25—N3—N4—N5	−0.4 (4)	C10—C9—C10'—C11'	−5.0 (11)
N3—N4—N5—N6	0.6 (4)	C7—N2—C12—C13	−72.3 (4)
N4—N5—N6—C25	−0.5 (4)	C6—N2—C12—C13	115.0 (4)
C5—C1—C2—C3	33.1 (5)	N2—C12—C13—C14	138.8 (3)
C1—C2—C3—C4	−38.6 (6)	N2—C12—C13—C18	−40.9 (5)
C2—C3—C4—C5	28.4 (6)	C18—C13—C14—C15	0.5 (5)
C7—N1—C5—C6	−0.2 (4)	C12—C13—C14—C15	−179.3 (3)
C7—N1—C5—C4	120.3 (4)	C13—C14—C15—C16	2.2 (5)
C7—N1—C5—C1	−119.7 (4)	C14—C15—C16—C17	−2.9 (5)
C3—C4—C5—N1	117.3 (4)	C14—C15—C16—C19	178.9 (3)
C3—C4—C5—C6	−129.1 (4)	C15—C16—C17—C18	1.0 (5)
C3—C4—C5—C1	−7.2 (5)	C19—C16—C17—C18	179.2 (3)
C2—C1—C5—N1	−140.4 (4)	C16—C17—C18—C13	1.7 (5)
C2—C1—C5—C6	106.5 (4)	C14—C13—C18—C17	−2.4 (5)
C2—C1—C5—C4	−15.9 (5)	C12—C13—C18—C17	177.4 (3)
C7—N2—C6—O1	179.7 (4)	C15—C16—C19—C20	130.9 (3)
C12—N2—C6—O1	−6.6 (6)	C17—C16—C19—C20	−47.3 (5)
C7—N2—C6—C5	0.4 (4)	C15—C16—C19—C24	−44.4 (5)
C12—N2—C6—C5	174.2 (3)	C17—C16—C19—C24	137.5 (3)
N1—C5—C6—O1	−179.4 (4)	C24—C19—C20—C21	1.1 (5)
C4—C5—C6—O1	59.5 (6)	C16—C19—C20—C21	−174.4 (3)
C1—C5—C6—O1	−58.6 (5)	C19—C20—C21—C22	0.8 (6)
N1—C5—C6—N2	−0.2 (4)	C20—C21—C22—C23	−1.7 (6)
C4—C5—C6—N2	−121.3 (3)	C21—C22—C23—C24	0.9 (6)
C1—C5—C6—N2	120.6 (3)	C22—C23—C24—C19	1.0 (5)
C5—N1—C7—N2	0.4 (4)	C22—C23—C24—C25	−178.4 (3)
C5—N1—C7—C8	178.1 (4)	C20—C19—C24—C23	−1.9 (5)
C6—N2—C7—N1	−0.5 (4)	C16—C19—C24—C23	173.5 (3)

C12—N2—C7—N1	−174.1 (3)	C20—C19—C24—C25	177.5 (3)
C6—N2—C7—C8	−178.4 (4)	C16—C19—C24—C25	−7.1 (5)
C12—N2—C7—C8	8.0 (5)	N4—N3—C25—N6	0.1 (4)
N1—C7—C8—C9	−11.7 (7)	N4—N3—C25—C24	−179.2 (3)
N2—C7—C8—C9	165.7 (4)	N5—N6—C25—N3	0.2 (4)
C7—C8—C9—C10'	−160.0 (11)	N5—N6—C25—C24	179.6 (3)
C7—C8—C9—C10	178.8 (6)	C23—C24—C25—N3	−56.0 (5)
C10'—C9—C10—C11	22.4 (18)	C19—C24—C25—N3	124.6 (4)
C8—C9—C10—C11	170.4 (7)	C23—C24—C25—N6	124.7 (4)
C8—C9—C10'—C11'	−48.2 (19)	C19—C24—C25—N6	−54.7 (5)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N6—H6A···Br1	0.93 (4)	2.31 (4)	3.241 (3)	174 (3)
O1W—HW1A···Br1^{i}	0.73 (6)	2.59 (7)	3.309 (3)	168 (7)
O1W—HW1B···N3^{ii}	0.87 (6)	2.04 (6)	2.886 (4)	163 (6)
O2W—HW2A···O1	0.87 (2)	2.52 (7)	3.068 (6)	122 (7)
O2W—HW2B···Br1^{iii}	0.88 (2)	2.58 (3)	3.456 (5)	169 (8)
N1—H1A···O1W	0.64 (4)	2.10 (4)	2.735 (5)	171 (5)

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

Fig. 1

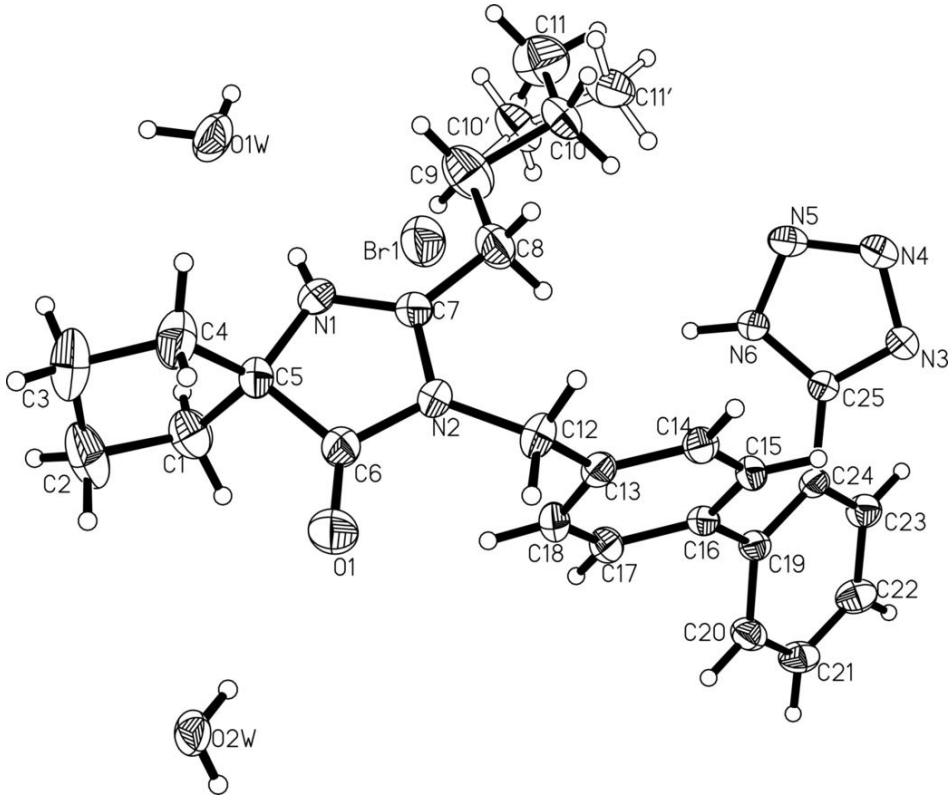
C11
C11'
O1W
C10'
C10
C9
Br1
C8
N5
N4
N1
C7
C4
N6
N3
C5
C25
C3
N2
C12
C14
C15
C1
C6
C24
C2
C13
C23
O1
C18
C16
C19
C17
C22
C20
C21
O2W

Fig. 2