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3,3′-Diphenyl-1,1′-[2,2′-oxybis(2,1-phenylene)]diurea *N*,*N*-dimethylformamide disolvate

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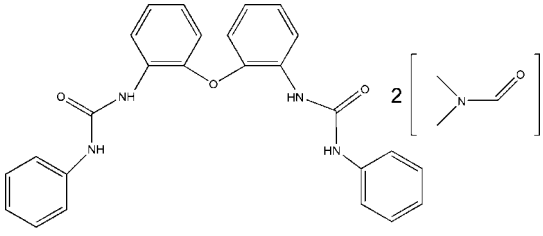
Key indicators: single-crystal X-ray study; T = 180 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.051; wR factor = 0.140; data-to-parameter ratio = 9.5.

In the structure of the title compound, $C_{26}H_{22}N_4O_3\cdot 2C_3H_7NO$, one of the DMF solvent molecules is disordered over two sets of positions in a 0.5:0.5 ratio. In the 1,1′-[2,2′-oxybis(2,1-phenylene)]bis(3-phenylurea) molecule, the two diphenylurea segments are linked *via* an ether O atom and are inclined at an angle of 53.80 (4)° to one another. In the crystal structure, classical N—H···O hydrogen bonds link each molecule to two DMF solvent molecules and these aggregates form columns down *a* through C—H···π interactions. Additional C—H···O interactions link the main molecule and the solvent molecules, forming columns of independent zigzag chains along *b*.

Related literature

For information on anion binding agents, see: Gunnlaugsson *et al.* (2004); Kim & Kim (2005). To our knowledge, no structures of oxybis(phenylene)phenylurea derivatives have been reported previously. However, for structures of *N*,*N*′-diphenylthiourea, see: Dannecker *et al.* (1979); Galkin *et al.* (2006). For structures of oxybis(aminobenzene) derivatives, see: Bensemann *et al.* (2003); Ashton *et al.* (1996).



Experimental

Crystal data

$C_{26}H_{22}N_4O_3\cdot 2C_3H_7NO$
M_r = 584.67
Monoclinic, $P2_1$
a = 11.1035 (2) Å
b = 8.1564 (2) Å
c = 16.8881 (3) Å
β = 103.429 (1)°
V = 1487.65 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 180 (2) K
0.46 × 0.35 × 0.23 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.905, T_{max} = 0.979
16291 measured reflections
3643 independent reflections
3337 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.140
S = 1.04
3643 reflections
385 parameters
25 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.79 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

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

*Cg*1 is the centroid of the C1–C6 ring.

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O4	0.88	1.99	2.847 (4)	163
N2—H2N···O4	0.88	2.17	2.977 (4)	153
N3—H3N···O5^{i}	0.88	2.03	2.809 (8)	147
N3—H3N···O5′^{i}	0.88	2.14	2.935 (7)	150
N4—H4N···O5′^{i}	0.88	1.92	2.765 (8)	161
N4—H4N···O5^{i}	0.88	2.40	3.101 (8)	137
C24—H24···O3^{ii}	0.95	2.68	3.573 (5)	157
C32—H32*B*···O3^{iii}	0.98	2.67	3.455 (11)	138
C31—H31*A*···*Cg*1	0.98	2.53	3.440 (4)	154

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

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*, *enCIFer* (Allen *et al.*, 2004) and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2678).

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3,3′-Diphenyl-1,1′-[2,2′-oxybis(2,1-phenylene)]diurea *N*,*N*-dimethylformamide disolvate

Yiannis C. Charalambides, Stephen C. Moratti and Jim Simpson

S1. Comment

While a large amount of work has been done on organic-metal cation complexes, there is much less known about analogous organic-anion complexes. We are interested in looking at diureas as potential anion binding agents. Ureas and thioureas have previously shown some promise in this area, and bind to anions such as fluoride and chloride through the hydrogen atoms attached to the urea N atoms (Gunnlaugsson *et al.*, 2004; Kim & Kim, 2005).

The title diurea compound, (I), was synthesized by the reaction of phenyl isocyanate and an aromatic diamine. The crystal structure shows that the bis-2,2-oxyphenyl motif used here may not be optimal for anion binding, as the urea units are splayed away from each other, though some of this may be due to binding to the solvent in the solid state. Initial NMR studies showed some affinity for both fluoride and chloride anions in chloroform.

The asymmetric unit of (I) comprises a 1,1′-(2,2′-oxybis(2,1-phenylene))bis(3-phenylurea) molecule and two molecules of dimethylformamide solvate, one of which is disordered equally over two positions (Fig. 1). The two diphenylurea segments of the molecule are linked *via* the ether O2 atom to form a V-shaped molecule with the arms inclined at an angle of 53.80 (4)° to one another. One 1,3-diphenylurea segment of the molecule, incorporating atoms C1···O2, is reasonably planar with an r.m.s. deviation from the plane through all 17 atoms of 0.029 Å. Also, the C1···C6 ring makes a dihedral angle of only 2.8 (2)° with the C8···C13 ring plane. In contrast, the correponding deviation from the plane through the 17 atoms that make up the second O2···C26 diphenylurea unit is 0.241Å with the C41···C19 and C21···C26 rings inclined at 24.08 (18)°. This variation is undoubtedly due to crystal packing effects.

In the crystal, classical N—H···O hydrogen bonds link each molecule to two DMF solvates (Table 1) and these aggregates form columns down *a* through an additional C31—H31A···*Cg*1 interaction, where *Cg*1 is the centroid of the C1···C6 ring. Then, C—H···O interactions further link the molecule and solvates into zigzag chains along *b*. The combination of these interactions stacks the chains into independent but interleaving columns, down *a*, as shown in Fig. 2.

S2. Experimental

To a solution of bis(2-aminophenyl)ether (1.00 g, 5.00 mmol) in dry distilled CH_2Cl_2 (30 ml) was added phenyl isocyanate (1.20 g, 10.00 mmol) and the reaction stirred under nitrogen at room temperature for 24 h. Following evaporation of the solvent a beige crystalline solid was obtained. Recrystallization from dimethylformamide layered with hexane afforded (I) (1.92 g, 88%) as colourless blocks: mp 388–390 K; ν_{max}(MeCN) 258 nm; Found: C, 70.61%; H, 5.08%; N, 12.60%; *M*+ 438.16958 (EI). $C_{26}H_{22}N_4O_3$ requires C, 71.22%; H, 5.06%; N, 12.75; *M*+ 438.16919]; ν_{max}/cm^{-1} 3337 (NH), 1656 (NHCONH), 1599 (aromatic); δH (400 MHz, DMSO-d^6) 9.26 (2*H*, s, (NH)2), 8.60 (2*H*, s, (NH)2) 8.30 (2*H*, d, J 8.1, ArH), 7.42 (4*H*, d, J 8.4, ArH), 7.25 (4*H*, t, J 7.9, ArH), 7.10 (2*H*, t, J 7.5, ArH), 6.94 (4*H*, t, J 7.4, ArH), 6.76 (2*H*, d, J 8.1, ArH); δC (125 MHz, CDCl3) 154.6, 147.7, 138.1, 129.0, 128.9, 125.1, 124.3, 124.1, 123.7, 120.7, 117.9.

S3. Refinement

In the absence of significant anomalous scattering effects, 2780 Friedel pairs were averaged for the refinement. One of the DMF solvate molecules is disordered over two sites and, in the final refinement cycles, the occupancy factors of the two disorder components were each fixed at 0.5. Common, isotropic temperature factors were applied to the non-H atoms of these components. In the final difference map, two peaks > 0.7 e Å^{-3} in the vicinity of the O5 and O5′ atoms of the two components of the disordered dimethylformamide solvate were apparent.

All the H atoms were positioned geometrically (C—H = 0.95–0.98 Å, N—H = 0.88 Å) and refined as riding model with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C, N).

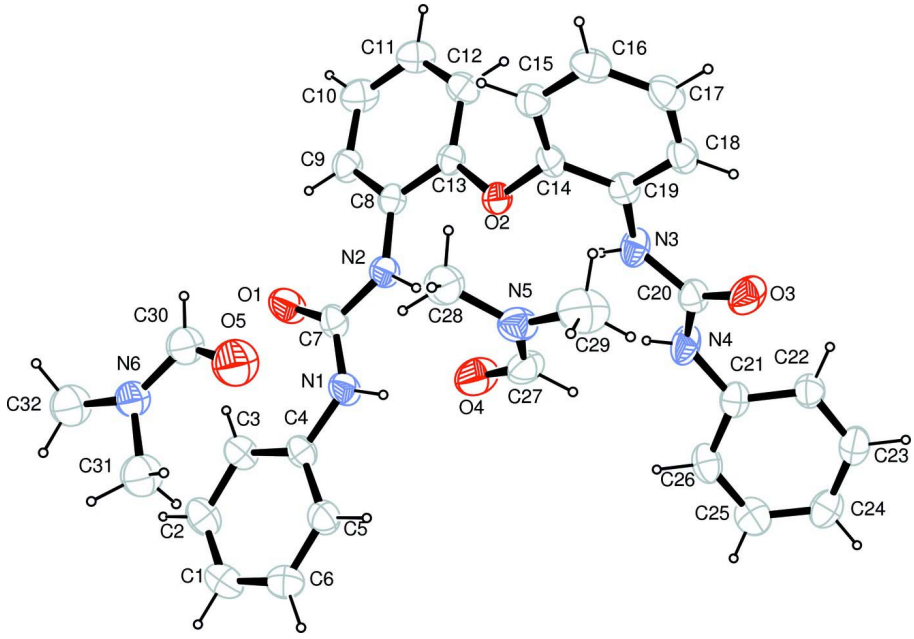

Figure 1

The structure of (I) showing displacement ellipsoids drawn at the 50% probability level. For clarity, only one of the disorder components of the N6 dimethylformamide solvate molecule is shown.

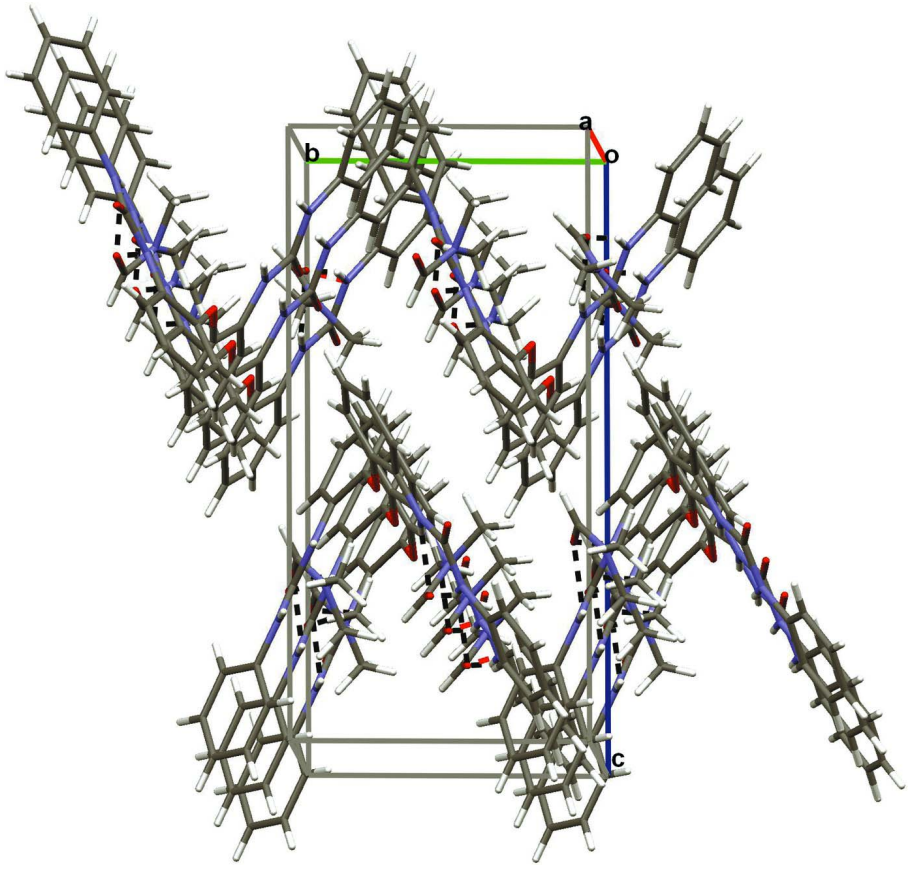

Figure 2

Crystal packing for (I) with hydrogen bonds drawn as dashed lines and the second disorder component of the N6 dimethylformamide solvate molecule omitted.

3,3′-Diphenyl-1,1′-[2,2′-Oxybis(2,1-phenylene)]diurea *N*,*N*-dimethylformamide disolvate

Crystal data

$C_{26}H_{22}N_4O_3\cdot 2(C_3H_7NO)$
$M_r = 584.67$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 11.1035\ (2)$ Å
$b = 8.1564\ (2)$ Å
$c = 16.8881\ (3)$ Å
$\beta = 103.429\ (1)°$
$V = 1487.65\ (5)$ Å^3
$Z = 2$
$F(000) = 620$
$D_x = 1.305$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 11759 reflections
$\theta = 1.0–27.5°$
$\mu = 0.09$ mm^{-1}
$T = 180$ K
Block, colourless
0.46 × 0.35 × 0.23 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Thin–slice ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{min} = 0.905$, $T_{max} = 0.979$
16291 measured reflections
3643 independent reflections
3337 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.5°$

$h = -14 \rightarrow 14$
$k = -9 \rightarrow 10$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.140$
$S = 1.04$
3643 reflections
385 parameters
25 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0791P)^2 + 0.5832P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.79$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.036 (6)

Special details

Experimental. One of the DMF solvate molecules is disordered over two sites: common, isotropic temperature factors were applied to the non-H atoms of this moiety.

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)
N1	0.0475 (2)	0.8656 (4)	0.19863 (16)	0.0384 (6)	
H1N	0.1190	0.8925	0.1882	0.046*	
O1	−0.0703 (2)	0.9052 (4)	0.29150 (15)	0.0546 (7)	
C1	−0.1737 (3)	0.5733 (5)	0.0162 (2)	0.0487 (8)	
H1	−0.2225	0.5077	−0.0257	0.058*	
N2	0.1272 (2)	1.0072 (3)	0.31451 (15)	0.0343 (6)	
H2N	0.1937	1.0089	0.2946	0.041*	
O2	0.33284 (17)	1.1674 (3)	0.37323 (11)	0.0365 (5)	
C2	−0.2174 (3)	0.6157 (4)	0.0830 (2)	0.0436 (8)	
H2	−0.2966	0.5780	0.0872	0.052*	
O3	0.56816 (19)	1.4761 (4)	0.21808 (13)	0.0506 (7)	
N3	0.4261 (2)	1.3857 (4)	0.28714 (16)	0.0381 (6)	
H3N	0.3461	1.3781	0.2841	0.046*	
C3	−0.1478 (3)	0.7131 (4)	0.14500 (19)	0.0385 (7)	
H3	−0.1798	0.7421	0.1906	0.046*	
N4	0.3619 (2)	1.4974 (4)	0.16115 (17)	0.0446 (7)	
H4N	0.2880	1.4767	0.1694	0.054*	
C4	−0.0314 (3)	0.7675 (4)	0.13956 (17)	0.0327 (6)	
C5	0.0135 (3)	0.7227 (4)	0.07149 (19)	0.0409 (7)	

H5	0.0933	0.7583	0.0674	0.049*
C6	−0.0570 (3)	0.6275 (5)	0.0104 (2)	0.0491 (8)
H6	−0.0260	0.5988	−0.0356	0.059*
C7	0.0261 (3)	0.9232 (4)	0.26955 (18)	0.0348 (6)
C8	0.1356 (2)	1.0902 (4)	0.38836 (16)	0.0305 (6)
C9	0.0432 (3)	1.0957 (4)	0.43132 (18)	0.0367 (6)
H9	−0.0329	1.0401	0.4108	0.044*
C10	0.0610 (3)	1.1817 (5)	0.50385 (19)	0.0435 (8)
H10	−0.0032	1.1842	0.5325	0.052*
C11	0.1701 (3)	1.2635 (5)	0.53523 (18)	0.0418 (7)
H11	0.1814	1.3211	0.5853	0.050*
C12	0.2634 (3)	1.2609 (4)	0.49290 (18)	0.0359 (6)
H12	0.3386	1.3183	0.5134	0.043*
C13	0.2462 (2)	1.1743 (4)	0.42062 (16)	0.0297 (5)
C14	0.4548 (2)	1.2147 (4)	0.40545 (17)	0.0312 (6)
C15	0.5259 (3)	1.1477 (4)	0.47623 (18)	0.0375 (6)
H15	0.4908	1.0705	0.5065	0.045*
C16	0.6488 (3)	1.1939 (5)	0.50276 (19)	0.0417 (7)
H16	0.6979	1.1500	0.5518	0.050*
C17	0.6997 (3)	1.3043 (5)	0.45735 (19)	0.0407 (7)
H17	0.7840	1.3358	0.4757	0.049*
C18	0.6295 (3)	1.3696 (4)	0.38541 (18)	0.0374 (6)
H18	0.6658	1.4443	0.3546	0.045*
C19	0.5055 (3)	1.3254 (4)	0.35856 (17)	0.0319 (6)
C20	0.4606 (3)	1.4552 (4)	0.22158 (18)	0.0345 (6)
C21	0.3665 (3)	1.5712 (4)	0.08639 (19)	0.0346 (6)
C22	0.4558 (3)	1.6869 (4)	0.08045 (19)	0.0391 (7)
H22	0.5172	1.7173	0.1273	0.047*
C23	0.4548 (3)	1.7576 (5)	0.0059 (2)	0.0449 (7)
H23	0.5162	1.8364	0.0021	0.054*
C24	0.3659 (3)	1.7156 (5)	−0.0634 (2)	0.0448 (8)
H24	0.3658	1.7652	−0.1143	0.054*
C25	0.2777 (3)	1.6003 (5)	−0.0568 (2)	0.0468 (8)
H25	0.2164	1.5703	−0.1038	0.056*
C26	0.2771 (3)	1.5281 (4)	0.0171 (2)	0.0417 (7)
H26	0.2157	1.4491	0.0207	0.050*
O4	0.2870 (2)	0.9862 (5)	0.19570 (14)	0.0576 (8)
N5	0.4865 (2)	0.9358 (4)	0.25860 (15)	0.0422 (6)
C27	0.3979 (3)	1.0063 (5)	0.20292 (18)	0.0436 (8)
H27	0.4227	1.0786	0.1655	0.065*
C28	0.4566 (4)	0.8214 (5)	0.3171 (2)	0.0520 (9)
H28A	0.3726	0.7790	0.2963	0.078*
H28B	0.4613	0.8778	0.3689	0.078*
H28C	0.5157	0.7303	0.3253	0.078*
C29	0.6164 (3)	0.9735 (7)	0.2665 (3)	0.0654 (12)
H29A	0.6244	1.0593	0.2274	0.098*
H29B	0.6601	0.8747	0.2556	0.098*
H29C	0.6524	1.0121	0.3219	0.098*

O5	0.1857 (6)	0.5004 (9)	0.2790 (4)	0.0671 (11)*	0.50
N6	−0.0062 (7)	0.3841 (11)	0.2555 (5)	0.0401 (9)*	0.50
C30	0.0876 (6)	0.4795 (10)	0.3014 (4)	0.0455 (11)*	0.50
H30	0.0770	0.5292	0.3502	0.055*	0.50
C31	0.0181 (8)	0.3207 (12)	0.1803 (5)	0.0566 (10)*	0.50
H31A	−0.0117	0.3992	0.1362	0.085*	0.50
H31B	−0.0251	0.2159	0.1668	0.085*	0.50
H31C	0.1073	0.3041	0.1871	0.085*	0.50
C32	−0.1324 (8)	0.3298 (12)	0.2555 (5)	0.0566 (10)*	0.50
H32A	−0.1576	0.2436	0.2146	0.085*	0.50
H32B	−0.1894	0.4229	0.2424	0.085*	0.50
H32C	−0.1343	0.2869	0.3094	0.085*	0.50
O5′	0.1596 (5)	0.3843 (9)	0.2144 (4)	0.0671 (11)*	0.50
N6′	−0.0096 (7)	0.4027 (12)	0.2653 (5)	0.0401 (9)*	0.50
C30′	0.0532 (7)	0.3524 (10)	0.2138 (5)	0.0455 (11)*	0.50
H30′	0.0108	0.2831	0.1712	0.055*	0.50
C31′	0.0366 (7)	0.5061 (11)	0.3332 (5)	0.0566 (10)*	0.50
H31D	0.1145	0.5561	0.3278	0.085*	0.50
H31E	0.0511	0.4412	0.3834	0.085*	0.50
H31F	−0.0240	0.5924	0.3353	0.085*	0.50
C32′	−0.1265 (8)	0.3719 (13)	0.2756 (5)	0.0566 (10)*	0.50
H32D	−0.1433	0.2540	0.2702	0.085*	0.50
H32E	−0.1872	0.4313	0.2342	0.085*	0.50
H32F	−0.1323	0.4086	0.3299	0.085*	0.50

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0290 (11)	0.0458 (15)	0.0429 (13)	−0.0094 (11)	0.0133 (10)	−0.0104 (12)
O1	0.0420 (12)	0.0728 (19)	0.0552 (13)	−0.0216 (13)	0.0239 (11)	−0.0253 (13)
C1	0.0487 (18)	0.0443 (19)	0.0481 (18)	−0.0029 (16)	0.0009 (15)	−0.0107 (16)
N2	0.0282 (11)	0.0417 (14)	0.0357 (12)	−0.0061 (11)	0.0129 (9)	−0.0054 (11)
O2	0.0290 (9)	0.0486 (13)	0.0329 (9)	−0.0108 (10)	0.0093 (7)	−0.0012 (10)
C2	0.0309 (14)	0.0406 (18)	0.0566 (19)	−0.0002 (13)	0.0050 (13)	−0.0065 (15)
O3	0.0309 (10)	0.080 (2)	0.0430 (11)	−0.0025 (12)	0.0138 (9)	0.0070 (13)
N3	0.0253 (11)	0.0438 (15)	0.0477 (14)	0.0001 (11)	0.0136 (10)	0.0085 (13)
C3	0.0318 (13)	0.0387 (17)	0.0445 (15)	0.0010 (12)	0.0082 (12)	−0.0058 (13)
N4	0.0274 (11)	0.0579 (19)	0.0509 (15)	−0.0042 (13)	0.0138 (10)	0.0129 (14)
C4	0.0293 (13)	0.0287 (14)	0.0388 (14)	0.0008 (11)	0.0056 (11)	−0.0028 (12)
C5	0.0438 (16)	0.0362 (17)	0.0448 (16)	−0.0041 (14)	0.0149 (13)	−0.0051 (13)
C6	0.059 (2)	0.048 (2)	0.0433 (17)	−0.0045 (17)	0.0180 (15)	−0.0097 (15)
C7	0.0295 (13)	0.0336 (15)	0.0424 (15)	−0.0048 (12)	0.0104 (11)	−0.0027 (12)
C8	0.0305 (13)	0.0286 (13)	0.0329 (13)	0.0006 (11)	0.0081 (10)	0.0029 (11)
C9	0.0294 (13)	0.0417 (16)	0.0404 (15)	−0.0019 (13)	0.0111 (11)	0.0019 (13)
C10	0.0358 (14)	0.056 (2)	0.0409 (15)	0.0076 (15)	0.0140 (12)	−0.0029 (16)
C11	0.0383 (15)	0.0491 (19)	0.0377 (14)	0.0090 (14)	0.0084 (12)	−0.0040 (14)
C12	0.0320 (14)	0.0356 (15)	0.0384 (14)	0.0013 (13)	0.0046 (11)	−0.0037 (13)
C13	0.0285 (12)	0.0290 (13)	0.0322 (12)	0.0021 (11)	0.0085 (10)	0.0041 (11)

C14	0.0255 (12)	0.0305 (14)	0.0383 (13)	−0.0043 (10)	0.0088 (10)	−0.0060 (11)
C15	0.0385 (14)	0.0375 (16)	0.0372 (14)	−0.0016 (13)	0.0098 (11)	−0.0032 (13)
C16	0.0361 (15)	0.0473 (19)	0.0386 (14)	0.0044 (14)	0.0025 (12)	−0.0050 (14)
C17	0.0265 (13)	0.0485 (19)	0.0465 (16)	−0.0034 (13)	0.0069 (11)	−0.0128 (15)
C18	0.0318 (14)	0.0386 (16)	0.0443 (15)	−0.0065 (13)	0.0139 (12)	−0.0073 (14)
C19	0.0302 (13)	0.0308 (14)	0.0369 (13)	0.0012 (11)	0.0120 (11)	−0.0042 (12)
C20	0.0311 (13)	0.0339 (15)	0.0419 (15)	−0.0003 (12)	0.0153 (11)	−0.0039 (13)
C21	0.0298 (13)	0.0320 (15)	0.0445 (15)	0.0031 (11)	0.0137 (11)	0.0029 (13)
C22	0.0374 (14)	0.0390 (17)	0.0427 (15)	−0.0049 (13)	0.0130 (12)	−0.0020 (14)
C23	0.0437 (17)	0.0413 (18)	0.0542 (18)	−0.0043 (15)	0.0204 (14)	0.0034 (16)
C24	0.0440 (16)	0.0453 (19)	0.0475 (17)	0.0057 (14)	0.0155 (14)	0.0110 (15)
C25	0.0440 (17)	0.0413 (18)	0.0506 (18)	0.0042 (15)	0.0015 (14)	0.0056 (15)
C26	0.0308 (14)	0.0353 (16)	0.0566 (18)	−0.0011 (13)	0.0057 (13)	0.0050 (14)
O4	0.0337 (11)	0.097 (2)	0.0431 (12)	−0.0073 (14)	0.0105 (9)	−0.0005 (15)
N5	0.0361 (13)	0.0534 (18)	0.0373 (12)	0.0007 (13)	0.0085 (10)	−0.0056 (13)
C27	0.0353 (15)	0.065 (2)	0.0325 (14)	−0.0025 (15)	0.0118 (12)	0.0009 (15)
C28	0.071 (2)	0.0381 (18)	0.0484 (18)	0.0005 (17)	0.0160 (17)	−0.0008 (16)
C29	0.0347 (16)	0.098 (4)	0.061 (2)	0.007 (2)	0.0054 (15)	−0.007 (3)

Geometric parameters (Å, º)

N1—C7	1.358 (4)	C18—H18	0.9500
N1—C4	1.413 (4)	C21—C22	1.389 (4)
N1—H1N	0.8800	C21—C26	1.393 (5)
O1—C7	1.220 (4)	C22—C23	1.382 (5)
C1—C2	1.372 (5)	C22—H22	0.9500
C1—C6	1.393 (5)	C23—C24	1.387 (5)
C1—H1	0.9500	C23—H23	0.9500
N2—C7	1.383 (4)	C24—C25	1.381 (5)
N2—C8	1.403 (4)	C24—H24	0.9500
N2—H2N	0.8800	C25—C26	1.382 (5)
O2—C13	1.388 (3)	C25—H25	0.9500
O2—C14	1.392 (3)	C26—H26	0.9500
C2—C3	1.396 (4)	O4—C27	1.220 (4)
C2—H2	0.9500	N5—C27	1.324 (4)
O3—C20	1.221 (3)	N5—C29	1.450 (4)
N3—C20	1.375 (4)	N5—C28	1.452 (5)
N3—C19	1.407 (4)	C27—H27	0.9500
N3—H3N	0.8800	C28—H28A	0.9800
C3—C4	1.390 (4)	C28—H28B	0.9800
C3—H3	0.9500	C28—H28C	0.9800
N4—C20	1.357 (4)	C29—H29A	0.9800
N4—C21	1.410 (4)	C29—H29B	0.9800
N4—H4N	0.8800	C29—H29C	0.9800
C4—C5	1.403 (4)	O5—C30	1.245 (8)
C5—C6	1.380 (5)	N6—C30	1.383 (10)
C5—H5	0.9500	N6—C31	1.454 (11)
C6—H6	0.9500	N6—C32	1.469 (9)

C8—C9	1.389 (4)	C30—H30	0.9500
C8—C13	1.402 (4)	C31—H31A	0.9800
C9—C10	1.385 (5)	C31—H31B	0.9800
C9—H9	0.9500	C31—H31C	0.9800
C10—C11	1.376 (5)	C32—H32A	0.9800
C10—H10	0.9500	C32—H32B	0.9800
C11—C12	1.389 (4)	C32—H32C	0.9800
C11—H11	0.9500	O5′—C30′	1.208 (8)
C12—C13	1.385 (4)	N6′—C30′	1.301 (10)
C12—H12	0.9500	N6′—C32′	1.372 (9)
C14—C15	1.383 (4)	N6′—C31′	1.420 (10)
C14—C19	1.402 (4)	C30′—H30′	0.9500
C15—C16	1.385 (4)	C31′—H31D	0.9800
C15—H15	0.9500	C31′—H31E	0.9800
C16—C17	1.386 (5)	C31′—H31F	0.9800
C16—H16	0.9500	C32′—H32D	0.9800
C17—C18	1.389 (5)	C32′—H32E	0.9800
C17—H17	0.9500	C32′—H32F	0.9800
C18—C19	1.393 (4)		

C7—N1—C4	127.7 (2)	C18—C19—C14	118.6 (3)
C7—N1—H1N	116.1	C18—C19—N3	124.3 (3)
C4—N1—H1N	116.1	C14—C19—N3	117.1 (2)
C2—C1—C6	119.3 (3)	O3—C20—N4	123.8 (3)
C2—C1—H1	120.3	O3—C20—N3	123.7 (3)
C6—C1—H1	120.3	N4—C20—N3	112.5 (2)
C7—N2—C8	127.3 (2)	C22—C21—C26	119.5 (3)
C7—N2—H2N	116.4	C22—C21—N4	122.0 (3)
C8—N2—H2N	116.4	C26—C21—N4	118.5 (3)
C13—O2—C14	120.3 (2)	C23—C22—C21	119.6 (3)
C1—C2—C3	121.3 (3)	C23—C22—H22	120.2
C1—C2—H2	119.3	C21—C22—H22	120.2
C3—C2—H2	119.3	C22—C23—C24	121.3 (3)
C20—N3—C19	126.7 (2)	C22—C23—H23	119.3
C20—N3—H3N	116.6	C24—C23—H23	119.3
C19—N3—H3N	116.6	C25—C24—C23	118.6 (3)
C4—C3—C2	119.6 (3)	C25—C24—H24	120.7
C4—C3—H3	120.2	C23—C24—H24	120.7
C2—C3—H3	120.2	C24—C25—C26	121.1 (3)
C20—N4—C21	126.2 (2)	C24—C25—H25	119.5
C20—N4—H4N	116.9	C26—C25—H25	119.5
C21—N4—H4N	116.9	C25—C26—C21	119.9 (3)
C3—C4—C5	118.9 (3)	C25—C26—H26	120.0
C3—C4—N1	124.6 (3)	C21—C26—H26	120.0
C5—C4—N1	116.5 (3)	C27—N5—C29	122.2 (3)
C6—C5—C4	120.7 (3)	C27—N5—C28	120.8 (3)
C6—C5—H5	119.6	C29—N5—C28	116.9 (3)
C4—C5—H5	119.6	O4—C27—N5	125.4 (3)

C5—C6—C1	120.1 (3)	O4—C27—H27	117.3
C5—C6—H6	120.0	N5—C27—H27	117.3
C1—C6—H6	120.0	N5—C28—H28A	109.5
O1—C7—N1	124.9 (3)	N5—C28—H28B	109.5
O1—C7—N2	123.4 (3)	H28A—C28—H28B	109.5
N1—C7—N2	111.7 (2)	N5—C28—H28C	109.5
C9—C8—C13	117.9 (3)	H28A—C28—H28C	109.5
C9—C8—N2	125.1 (3)	H28B—C28—H28C	109.5
C13—C8—N2	117.0 (2)	N5—C29—H29A	109.5
C10—C9—C8	120.4 (3)	N5—C29—H29B	109.5
C10—C9—H9	119.8	H29A—C29—H29B	109.5
C8—C9—H9	119.8	N5—C29—H29C	109.5
C11—C10—C9	121.2 (3)	H29A—C29—H29C	109.5
C11—C10—H10	119.4	H29B—C29—H29C	109.5
C9—C10—H10	119.4	C30—N6—C31	114.9 (7)
C10—C11—C12	119.3 (3)	C30—N6—C32	139.8 (8)
C10—C11—H11	120.4	C31—N6—C32	105.2 (7)
C12—C11—H11	120.4	O5—C30—N6	120.3 (7)
C13—C12—C11	119.7 (3)	O5—C30—H30	119.8
C13—C12—H12	120.2	N6—C30—H30	119.8
C11—C12—H12	120.2	C30′—N6′—C32′	134.2 (8)
C12—C13—O2	123.9 (2)	C30′—N6′—C31′	125.3 (7)
C12—C13—C8	121.4 (3)	C32′—N6′—C31′	100.4 (7)
O2—C13—C8	114.7 (2)	O5′—C30′—N6′	127.0 (8)
C15—C14—O2	122.5 (3)	O5′—C30′—H30′	116.5
C15—C14—C19	121.3 (3)	N6′—C30′—H30′	116.5
O2—C14—C19	116.1 (2)	N6′—C31′—H31D	109.5
C14—C15—C16	119.6 (3)	N6′—C31′—H31E	109.5
C14—C15—H15	120.2	H31D—C31′—H31E	109.5
C16—C15—H15	120.2	N6′—C31′—H31F	109.5
C15—C16—C17	119.7 (3)	H31D—C31′—H31F	109.5
C15—C16—H16	120.1	H31E—C31′—H31F	109.5
C17—C16—H16	120.1	N6′—C32′—H32D	109.5
C16—C17—C18	121.0 (3)	N6′—C32′—H32E	109.5
C16—C17—H17	119.5	H32D—C32′—H32E	109.5
C18—C17—H17	119.5	N6′—C32′—H32F	109.5
C17—C18—C19	119.8 (3)	H32D—C32′—H32F	109.5
C17—C18—H18	120.1	H32E—C32′—H32F	109.5
C19—C18—H18	120.1		

C6—C1—C2—C3	0.5 (6)	C19—C14—C15—C16	1.8 (5)
C1—C2—C3—C4	−0.5 (5)	C14—C15—C16—C17	−1.2 (5)
C2—C3—C4—C5	0.0 (5)	C15—C16—C17—C18	−0.1 (5)
C2—C3—C4—N1	−179.6 (3)	C16—C17—C18—C19	0.7 (5)
C7—N1—C4—C3	0.2 (5)	C17—C18—C19—C14	−0.1 (4)
C7—N1—C4—C5	−179.4 (3)	C17—C18—C19—N3	179.3 (3)
C3—C4—C5—C6	0.6 (5)	C15—C14—C19—C18	−1.1 (4)
N1—C4—C5—C6	−179.8 (3)	O2—C14—C19—C18	−176.3 (3)

C4—C5—C6—C1	−0.6 (6)	C15—C14—C19—N3	179.4 (3)
C2—C1—C6—C5	0.0 (6)	O2—C14—C19—N3	4.3 (4)
C4—N1—C7—O1	−4.1 (6)	C20—N3—C19—C18	20.4 (5)
C4—N1—C7—N2	176.4 (3)	C20—N3—C19—C14	−160.2 (3)
C8—N2—C7—O1	−3.1 (5)	C21—N4—C20—O3	−0.4 (6)
C8—N2—C7—N1	176.4 (3)	C21—N4—C20—N3	179.9 (3)
C7—N2—C8—C9	3.0 (5)	C19—N3—C20—O3	−1.3 (5)
C7—N2—C8—C13	−176.7 (3)	C19—N3—C20—N4	178.4 (3)
C13—C8—C9—C10	−0.2 (5)	C20—N4—C21—C22	−36.4 (5)
N2—C8—C9—C10	−179.9 (3)	C20—N4—C21—C26	144.9 (3)
C8—C9—C10—C11	0.0 (5)	C26—C21—C22—C23	−0.1 (5)
C9—C10—C11—C12	0.6 (5)	N4—C21—C22—C23	−178.8 (3)
C10—C11—C12—C13	−1.0 (5)	C21—C22—C23—C24	0.2 (5)
C11—C12—C13—O2	178.6 (3)	C22—C23—C24—C25	−0.2 (5)
C11—C12—C13—C8	0.8 (5)	C23—C24—C25—C26	0.2 (5)
C14—O2—C13—C12	15.8 (4)	C24—C25—C26—C21	−0.1 (5)
C14—O2—C13—C8	−166.3 (3)	C22—C21—C26—C25	0.0 (5)
C9—C8—C13—C12	−0.2 (4)	N4—C21—C26—C25	178.8 (3)
N2—C8—C13—C12	179.5 (3)	C29—N5—C27—O4	−176.1 (4)
C9—C8—C13—O2	−178.2 (3)	C28—N5—C27—O4	1.2 (6)
N2—C8—C13—O2	1.6 (4)	C31—N6—C30—O5	−2.7 (13)
C13—O2—C14—C15	54.7 (4)	C32—N6—C30—O5	−178.0 (11)
C13—O2—C14—C19	−130.2 (3)	C32′—N6′—C30′—O5′	−176.1 (11)
O2—C14—C15—C16	176.6 (3)	C31′—N6′—C30′—O5′	−0.2 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O4	0.88	1.99	2.847 (4)	163
N2—H2*N*···O4	0.88	2.17	2.977 (4)	153
N3—H3*N*···O5^{i}	0.88	2.03	2.809 (8)	147
N3—H3*N*···O5′^{i}	0.88	2.14	2.935 (7)	150
N4—H4*N*···O5′^{i}	0.88	1.92	2.765 (8)	161
N4—H4*N*···O5^{i}	0.88	2.40	3.101 (8)	137
C24—H24···O3^{ii}	0.95	2.68	3.573 (5)	157
C32—H32*B*···O3^{iii}	0.98	2.67	3.455 (11)	138
C31—H31*A*···*Cg*1	0.98	2.53	3.440 (4)	154

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