Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-Benzoyl-*N*′-(1,10-phenanthrolin-5-yl)-thiourea dichloromethane hemisolvate monohydrate

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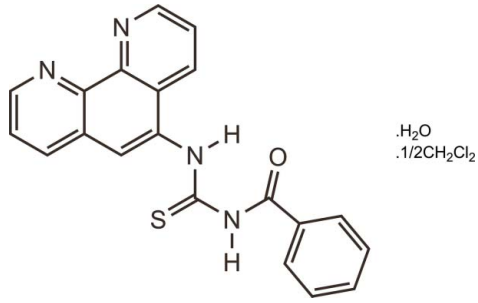
Received 1 March 2011; accepted 8 May 2011

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; some non-H atoms missing; R factor = 0.049; wR factor = 0.139; data-to-parameter ratio = 13.3.

The title compound, $C_{20}H_{14}N_4OS\cdot0.5CH_2Cl_2\cdot H_2O$, contains 1,10-phenanthroline and benzoyl fragments that adopt *cisoid* and *transoid* conformations respectively, with respect to the S atom. In the crystal, molecules are linked by intermolecular O—H···O, O—H···N, N—H···O and C—H···O hydrogen bonds, forming chains along [011]. Weak C—H···π and slipped π–π stacking interactions [centroid–centroid distances = 3.715 (3), 3.684 (3) and 3.574 (2) Å] are also observed. In addition to an ordered water molecule of solvation, there is a disordered dichloromethane solvent molecule which was difficult to model correctly. The contributions to the electron density for this molecule was removed using the *SQUEEZE* procedure in *PLATON* [Spek (2009). *Acta Cryst.* D**65**, 148–155].

Related literature

For related structures, see: Al-abbasi & Kassim (2011); Hassan *et al.* (2008); Yamin & Hassan (2004); Yamin & Yusof (2003); Yunus *et al.* (2008). For standard bond lengths, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{20}H_{14}N_4OS\cdot0.5CH_2Cl_2\cdot H_2O$
M_r = 418.89
Triclinic, $P\overline{1}$
a = 9.385 (5) Å
b = 10.863 (5) Å
c = 10.927 (5) Å
α = 112.949 (5)°
β = 103.984 (5)°
γ = 96.641 (5)°
V = 967.6 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.33 mm^{-1}
T = 298 K
0.47 × 0.19 × 0.14 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.916, T_{max} = 0.974
4400 measured reflections
3254 independent reflections
2450 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.139
S = 1.08
3254 reflections
244 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

*Cg*2 and *Cg*3 are the centroids of the N4,C9–C12,C20 and C1–C6 rings, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*A*···O1	0.86	1.96	2.636 (3)	135
N2—H2*A*···O2*W*	0.86	2.07	2.910 (3)	165
O2*W*—H1*W*···N3^{i}	0.85	2.08	2.889 (3)	160
O2*W*—H2*W*···O1^{ii}	0.85	2.46	3.187 (3)	144
C9—H9···O1^{iii}	0.93	2.56	3.140 (4)	121
C2—H2···*Cg*2^{iv}	0.93	2.99	3.806 (4)	147
C13—H13···*Cg*3^{ii}	0.93	2.88	3.796 (3)	168

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2009).

The authors thank Universiti Kebangsaan Malaysia for providing facilities and the Ministry of Science, Technology

and Innovation for the research fund No. UKM-MGI-NBD0021–2007.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2403).

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supplementary materials

Acta Cryst. (2011). E**67**, o1397-o1398 [**doi:10.1107/S160053681101734X**]

N-Benzoyl-*N*'-(1,10-phenanthrolin-5-yl)thiourea dichloromethane hemisolvate monohydrate

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Comment

The title molecule maintains the *cis-trans* configuration with respect to the positions of the 1,10-phenanthrolin-5-amine and benzoyl groups, respectively, relative to the S atom across the C–N bonds (Fig 1). The bond lengths and angles in the molecules are in normal ranges (Allen *et al.*, 1987).

The phenyl and phenanthroline rings are twisted with relative to the central thiourea fragment dihedral angles of 29.46 (12)° and 74.06 (8)°, respectively. The phenyl and phenantroline rings are almost perpendicular to each other with dihedral angle of 83.15 (10)°.

An intramolecular N1—H1A···O1 (Fig. 1 and Table 2) stabilize the conformation and forms a pseudo six-membered ring (N1/H1A/O1/C7/N2/C8). The crystal packing is stabilized by fours intermolecular O2W—H2W···O1, O2W—H1W···N3, N2—H2A···O2W and C9—H9···O1 hydrogen bonds,(Table 1), which link the molecules into an infinite one-dimensional chain along [0 1 1] direction (Fig 2). There also exist weak C—H···π and slippest π-π stacking interactions which result in a three dimensionnal network.

Experimental

The reaction scheme involved a reaction of benzoyl chloride (8.6 mmol) with ammonium thiocyanate (8.6 mmol) in acetone. The product, benzoyl isothiocyanate (7.7 mmol) was reacted with 1,10-phenanthrolin-5-amine (7.7 mmol) to give the title compound with a 70% yield. A slow evaporation dichloromethane solution of the product gave a colourless crystals suitable for X-ray diffraction.

Refinement

All H atoms attached to C and N atoms were fixed geometrically and treated as riding with C—H = 0.93 Å and N—H = 0.86 Å with U_{iso}(H) = 1.2U_{eq}(C or N). H atoms of water molecule were located in difference Fourier maps and included in the subsequent refinement using restraints (O—H= 0.85 (1)Å and H···H= 1.39 (2) Å) with U_{iso}(H) = 1.5U_{eq}(O). In the last cycles of refinement, they were treated as riding on their parent O atom.

Some residual electron density were difficult to model and therefore, the SQUEEZE function of *PLATON* (Spek, 2009) was used to eliminate the contribution of the electron density in the solvent region from the intensity data, and the solvent-free model was employed for the final refinement. There is one cavity of 122.9 Å^3 per unit cell. *PLATON* estimated that the cavity contains 38.4 electrons which may correspond to a solvent molecule of dichloromethane as suggested by chemical analyses.

This dichloromethane solvent has been however included in the formula.

Figures

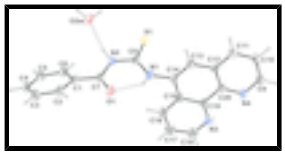

Fig. 1. The molecular structure of (I) with the atom labeling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii. Hydrogen bonds are shown as dashed lines.

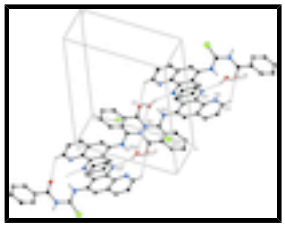

Fig. 2. A partial packing view of (I) showing the O—H···O, O—H···N, N—H···O and C—H···O Hydrogen bonds whiwh are shown as dashed lines. H atoms attached to C atoms not involved in hydrogen bondings have been omitted for clarity.

N-Benzoyl-*N*'-(1,10-phenanthrolin-5-yl)thiourea dichloromethane hemisolvate monohydrate

Crystal data

$C_{20}H_{14}N_4OS·0.5CH_2Cl_2·H_2O$	$Z = 2$
$M_r = 418.89$	$F(000) = 434$
Triclinic, $P\overline{1}$	$D_x = 1.438$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 9.385$ (5) Å	Cell parameters from 4905 reflections
$b = 10.863$ (5) Å	$\theta = 2.1$–$28.4°$
$c = 10.927$ (5) Å	$\mu = 0.33$ mm^{-1}
$\alpha = 112.949$ (5)°	$T = 298$ K
$\beta = 103.984$ (5)°	Block, colourless
$\gamma = 96.641$ (5)°	0.47 × 0.19 × 0.14 mm
$V = 967.6$ (8) Å^3	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3254 independent reflections
Radiation source: fine-focus sealed tube	2450 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.014$
ω scans	$\theta_{max} = 25.0°$, $\theta_{min} = 2.1°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)	$h = -11 \rightarrow 10$
$T_{min} = 0.916$, $T_{max} = 0.974$	$k = -7 \rightarrow 12$
4400 measured reflections	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map

$R[F^2 > 2\sigma(F^2)] = 0.049$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.139$	H-atom parameters constrained
$S = 1.08$	$w = 1/[\sigma^2(F_o^2) + (0.0792P)^2 + 0.0787P]$ where $P = (F_o^2 + 2F_c^2)/3$
3254 reflections	$(\Delta/\sigma)_{max} < 0.001$
244 parameters	$\Delta\rho_{max} = 0.28$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.19$ e Å^{-3}

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}
C8	0.1675 (2)	0.7038 (2)	0.1383 (2)	0.0355 (5)
C7	0.1658 (3)	0.4881 (2)	0.1675 (2)	0.0392 (6)
C14	0.0884 (3)	0.8891 (2)	0.3010 (2)	0.0376 (5)
C13	−0.0219 (3)	0.9315 (2)	0.2377 (2)	0.0419 (6)
H13	−0.0864	0.8715	0.1488	0.050*
C12	−0.0432 (3)	1.0675 (2)	0.3038 (2)	0.0384 (5)
C20	0.0499 (3)	1.1569 (2)	0.4405 (2)	0.0359 (5)
C19	0.1718 (3)	1.1125 (2)	0.5081 (2)	0.0366 (5)
C15	0.1916 (3)	0.9784 (2)	0.4377 (2)	0.0386 (5)
C1	0.2387 (3)	0.3698 (2)	0.1377 (2)	0.0374 (5)
C6	0.3666 (3)	0.3676 (3)	0.0951 (3)	0.0444 (6)
H6	0.4046	0.4380	0.0751	0.053*
C5	0.4371 (3)	0.2607 (3)	0.0825 (3)	0.0528 (7)
H5	0.5231	0.2593	0.0544	0.063*
C4	0.3802 (3)	0.1559 (3)	0.1116 (3)	0.0521 (7)
H4	0.4284	0.0843	0.1034	0.063*
C3	0.2526 (3)	0.1569 (3)	0.1525 (3)	0.0556 (7)
H3	0.2136	0.0853	0.1704	0.067*
C2	0.1833 (3)	0.2638 (2)	0.1669 (3)	0.0472 (6)
H2	0.0983	0.2653	0.1965	0.057*
C11	−0.1558 (3)	1.1162 (3)	0.2395 (3)	0.0490 (6)
H11	−0.2178	1.0612	0.1482	0.059*
C10	−0.1746 (3)	1.2442 (3)	0.3108 (3)	0.0533 (7)
H10	−0.2479	1.2787	0.2689	0.064*

C9	−0.0820 (3)	1.3217 (3)	0.4472 (3)	0.0506 (7)
H9	−0.0988	1.4074	0.4964	0.061*
C16	0.3117 (3)	0.9399 (3)	0.5063 (3)	0.0470 (6)
H16	0.3276	0.8523	0.4638	0.056*
C17	0.4052 (3)	1.0305 (3)	0.6353 (3)	0.0562 (7)
H17	0.4865	1.0067	0.6814	0.067*
C18	0.3767 (3)	1.1599 (3)	0.6968 (3)	0.0550 (7)
H18	0.4411	1.2211	0.7852	0.066*
N1	0.1038 (2)	0.75069 (19)	0.2384 (2)	0.0462 (5)
H1A	0.0695	0.6940	0.2674	0.055*
N2	0.1897 (2)	0.57097 (18)	0.1030 (2)	0.0384 (5)
H2A	0.2221	0.5371	0.0325	0.046*
N3	0.2644 (2)	1.2012 (2)	0.6378 (2)	0.0475 (5)
N4	0.0287 (2)	1.2829 (2)	0.5128 (2)	0.0462 (5)
O1	0.0911 (2)	0.51100 (18)	0.2479 (2)	0.0574 (5)
S1	0.22322 (8)	0.79160 (7)	0.05756 (7)	0.0509 (2)
O2W	0.2509 (2)	0.46575 (19)	−0.1633 (2)	0.0658 (6)
H1W	0.2519	0.3976	−0.2352	0.099*
H2W	0.1823	0.5024	−0.1909	0.099*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C8	0.0397 (12)	0.0285 (11)	0.0331 (12)	0.0102 (9)	0.0112 (10)	0.0075 (9)
C7	0.0467 (14)	0.0288 (12)	0.0386 (13)	0.0088 (10)	0.0173 (11)	0.0089 (10)
C14	0.0492 (14)	0.0276 (11)	0.0418 (13)	0.0106 (10)	0.0272 (11)	0.0128 (10)
C13	0.0523 (15)	0.0341 (12)	0.0350 (13)	0.0085 (11)	0.0179 (11)	0.0087 (10)
C12	0.0489 (14)	0.0342 (12)	0.0376 (13)	0.0121 (10)	0.0224 (11)	0.0151 (11)
C20	0.0448 (13)	0.0286 (11)	0.0373 (13)	0.0088 (10)	0.0204 (10)	0.0129 (10)
C19	0.0430 (13)	0.0314 (11)	0.0378 (13)	0.0075 (10)	0.0205 (10)	0.0131 (10)
C15	0.0480 (14)	0.0357 (12)	0.0430 (13)	0.0132 (10)	0.0272 (11)	0.0195 (11)
C1	0.0448 (13)	0.0295 (11)	0.0347 (12)	0.0089 (10)	0.0132 (10)	0.0101 (10)
C6	0.0443 (14)	0.0456 (14)	0.0506 (15)	0.0134 (11)	0.0194 (12)	0.0247 (12)
C5	0.0499 (15)	0.0639 (17)	0.0500 (15)	0.0254 (13)	0.0205 (12)	0.0239 (14)
C4	0.0623 (17)	0.0432 (14)	0.0516 (16)	0.0266 (12)	0.0155 (13)	0.0187 (12)
C3	0.0677 (18)	0.0408 (14)	0.0679 (18)	0.0191 (13)	0.0272 (15)	0.0279 (14)
C2	0.0545 (15)	0.0352 (13)	0.0558 (16)	0.0126 (11)	0.0265 (13)	0.0176 (12)
C11	0.0552 (16)	0.0505 (15)	0.0403 (14)	0.0159 (12)	0.0124 (12)	0.0194 (12)
C10	0.0619 (17)	0.0511 (16)	0.0582 (17)	0.0276 (13)	0.0226 (14)	0.0289 (14)
C9	0.0629 (17)	0.0386 (13)	0.0566 (17)	0.0225 (12)	0.0263 (14)	0.0197 (13)
C16	0.0527 (15)	0.0452 (14)	0.0556 (16)	0.0205 (12)	0.0286 (13)	0.0250 (13)
C17	0.0478 (15)	0.0695 (19)	0.0607 (18)	0.0216 (14)	0.0183 (13)	0.0345 (16)
C18	0.0511 (16)	0.0588 (17)	0.0458 (15)	0.0110 (13)	0.0117 (12)	0.0160 (13)
N1	0.0699 (14)	0.0278 (10)	0.0503 (12)	0.0157 (9)	0.0354 (11)	0.0155 (9)
N2	0.0488 (12)	0.0315 (10)	0.0372 (11)	0.0155 (8)	0.0204 (9)	0.0112 (9)
N3	0.0485 (12)	0.0442 (12)	0.0432 (12)	0.0098 (10)	0.0151 (10)	0.0122 (10)
N4	0.0589 (13)	0.0325 (10)	0.0487 (12)	0.0184 (9)	0.0243 (10)	0.0125 (9)
O1	0.0855 (13)	0.0415 (10)	0.0681 (13)	0.0283 (9)	0.0510 (11)	0.0274 (9)

S1	0.0700 (5)	0.0451 (4)	0.0598 (4)	0.0279 (3)	0.0388 (4)	0.0303 (3)
O2W	0.0810 (14)	0.0525 (11)	0.0523 (11)	0.0243 (10)	0.0246 (10)	0.0067 (9)

Geometric parameters (Å, °)

C8—N1	1.329 (3)	C5—H5	0.9300
C8—N2	1.395 (3)	C4—C3	1.375 (4)
C8—S1	1.651 (2)	C4—H4	0.9300
C7—O1	1.222 (3)	C3—C2	1.371 (4)
C7—N2	1.372 (3)	C3—H3	0.9300
C7—C1	1.486 (3)	C2—H2	0.9300
C14—C13	1.331 (3)	C11—C10	1.360 (4)
C14—N1	1.431 (3)	C11—H11	0.9300
C14—C15	1.437 (3)	C10—C9	1.383 (4)
C13—C12	1.432 (3)	C10—H10	0.9300
C13—H13	0.9300	C9—N4	1.322 (3)
C12—C11	1.397 (4)	C9—H9	0.9300
C12—C20	1.407 (3)	C16—C17	1.358 (4)
C20—N4	1.354 (3)	C16—H16	0.9300
C20—C19	1.444 (3)	C17—C18	1.391 (4)
C19—N3	1.355 (3)	C17—H17	0.9300
C19—C15	1.416 (3)	C18—N3	1.317 (3)
C15—C16	1.398 (3)	C18—H18	0.9300
C1—C6	1.388 (3)	N1—H1A	0.8600
C1—C2	1.388 (3)	N2—H2A	0.8600
C6—C5	1.379 (4)	O2W—H1W	0.8472
C6—H6	0.9300	O2W—H2W	0.8513
C5—C4	1.381 (4)		
N1—C8—N2	116.0 (2)	C5—C4—H4	119.8
N1—C8—S1	124.83 (17)	C2—C3—C4	119.8 (3)
N2—C8—S1	119.18 (17)	C2—C3—H3	120.1
O1—C7—N2	122.2 (2)	C4—C3—H3	120.1
O1—C7—C1	121.1 (2)	C3—C2—C1	120.7 (2)
N2—C7—C1	116.7 (2)	C3—C2—H2	119.7
C13—C14—N1	121.2 (2)	C1—C2—H2	119.7
C13—C14—C15	121.4 (2)	C10—C11—C12	119.8 (2)
N1—C14—C15	117.4 (2)	C10—C11—H11	120.1
C14—C13—C12	121.3 (2)	C12—C11—H11	120.1
C14—C13—H13	119.3	C11—C10—C9	118.3 (2)
C12—C13—H13	119.3	C11—C10—H10	120.8
C11—C12—C20	117.5 (2)	C9—C10—H10	120.8
C11—C12—C13	122.8 (2)	N4—C9—C10	124.8 (2)
C20—C12—C13	119.7 (2)	N4—C9—H9	117.6
N4—C20—C12	122.7 (2)	C10—C9—H9	117.6
N4—C20—C19	118.2 (2)	C17—C16—C15	119.9 (2)
C12—C20—C19	119.1 (2)	C17—C16—H16	120.0
N3—C19—C15	121.9 (2)	C15—C16—H16	120.0
N3—C19—C20	118.7 (2)	C16—C17—C18	118.5 (3)
C15—C19—C20	119.4 (2)	C16—C17—H17	120.7

C16—C15—C19	117.6 (2)	C18—C17—H17	120.7
C16—C15—C14	123.4 (2)	N3—C18—C17	124.2 (3)
C19—C15—C14	119.0 (2)	N3—C18—H18	117.9
C6—C1—C2	119.3 (2)	C17—C18—H18	117.9
C6—C1—C7	122.8 (2)	C8—N1—C14	124.39 (19)
C2—C1—C7	117.6 (2)	C8—N1—H1A	117.8
C5—C6—C1	119.9 (2)	C14—N1—H1A	117.8
C5—C6—H6	120.1	C7—N2—C8	127.66 (19)
C1—C6—H6	120.1	C7—N2—H2A	116.2
C6—C5—C4	120.0 (2)	C8—N2—H2A	116.2
C6—C5—H5	120.0	C18—N3—C19	117.9 (2)
C4—C5—H5	120.0	C9—N4—C20	116.8 (2)
C3—C4—C5	120.4 (2)	H1W—O2W—H2W	106.6
C3—C4—H4	119.8		

Hydrogen-bond geometry (Å, °)

Cg2 and Cg3 are the centroids of the N4,C9–C12,C20 and C1–C6 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O1	0.86	1.96	2.636 (3)	135
N2—H2A···O2W	0.86	2.07	2.910 (3)	165
O2W—H1W···N3^{i}	0.85	2.08	2.889 (3)	160
O2W—H2W···O1^{ii}	0.85	2.46	3.187 (3)	144
C9—H9···O1^{iii}	0.93	2.56	3.140 (4)	121
C2—H2···Cg2^{iv}	0.93	2.99	3.806 (4)	147
C13—H13···Cg3^{ii}	0.93	2.88	3.796 (3)	168

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

Table 2

Table 2 π-π stacking interactions (Å,°)

Cg1 is the centroid of the C12—C20 ring.
Cg2 is the centroid of the N4—C20 ring
Cg4 is the centroid of the N3—C19 ring

CgI	CgJ	CgI···CgJ^{a}	α	CgI···P(J)^{b}	CgJ···P(I)^{c}	Slippage
Cg1	Cg2^{v}	3.715 (3)	4.14	3.438	3.387	1.47 (mean value)
Cg1	Cg4^{v}	3.684 (3)	1.64	3.395	3.352	1.48 (mean value)
Cg4	Cg4^{v}	3.574 (2)	0.02	3.359	3.359	1.222

Symmetry codes: (v)-x,2-y,1-z
Notes:
a : Distance between centroids
b : Perpendicular distance of CgI on ring plan J
c : Perpendicular distance of CgJ on ring plan I
α = Dihedral Angle between the ring planes Slippage = vertical displacement between ring centroids.

Fig. 1

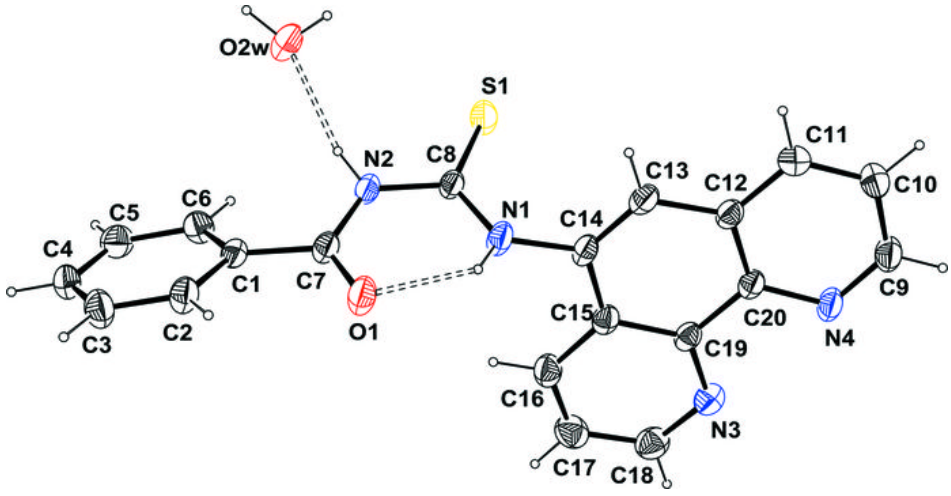
O2w
S1
N2
C8
C11
C13
C12
C10
C5
C6
N1
C14
C4
C1
C7
C9
C15
C20
O1
N4
C3
C2
C19
C16
N3
C17
C18

Fig. 2