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N-(3-Oxo-2-phenyl-2,3-dihydro-1*H*-pyrrolo[1,2-*c*]imidazol-1-yl)-*N*′-phenyl-*N*-(2,4,6-trimethylphenyl)urea ethyl acetate solvate

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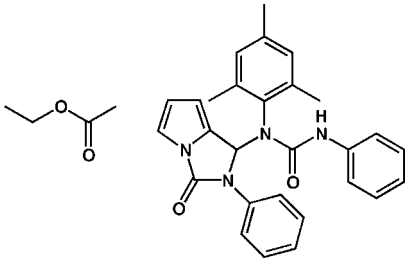
Key indicators: single-crystal X-ray study; T = 203 K; mean σ(C–C) = 0.003 Å; R factor = 0.042; wR factor = 0.115; data-to-parameter ratio = 13.1.

The title compound, $C_{28}H_{26}N_4O_2\cdot C_4H_8O_2$, was prepared from (2,4,6-trimethylphenyl)(1*H*-pyrrol-2-ylmethylene)amine and phenyl isocyanate in the presence of catalytic amounts of $[Pd(PPh_3)_4]$. Recrystallization of the crude product from a pentane–ethyl acetate mixture (10:1) yielded the title compound as the ethyl acetate solvate. The pyrrolo[1,2-*c*]imidazole core is essentially planar. In addition, a new stereogenic center at the ring C atom of the biheterocyclic group attached to the urea N atom has been formed during the reaction sequence. The crystal structure is influenced by various N—H···O and C—H···O hydrogen bonds.

Related literature

For related literature, see: Mishriky *et al.* (1998); Imhof (2007); Desiraju & Steiner (1999).



Experimental

Crystal data

$C_{28}H_{26}N_4O_2\cdot C_4H_8O_2$
M_r = 538.63
Monoclinic, $P2_1/c$
a = 16.2289 (11) Å
b = 11.0873 (7) Å
c = 16.8630 (8) Å
β = 111.907 (4)°
V = 2815.1 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 203 (2) K
0.4 × 0.3 × 0.3 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
8455 measured reflections
4780 independent reflections
3265 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.115
S = 0.83
4780 reflections
366 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···O1^{i}	0.86	2.11	2.899 (2)	151
C24—H24···O1^{i}	0.93	2.64	3.181 (2)	118
C18—H18···O3^{ii}	0.93	2.67	3.556 (2)	160
C3—H3···O4^{iii}	0.93	2.62	3.467 (2)	151
C27—H27···O4^{iv}	0.93	2.65	3.496 (2)	151

Symmetry codes: (i) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (ii) $x, -y+\frac{3}{2}, z-\frac{1}{2}$; (iii) $-x, -y+1, -z$; (iv) $x, y-1, z$.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1990); software used to prepare material for publication: *XP*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2325).

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  doi:10.1107/S1600536807060928

supplementary materials

Acta Cryst. (2007). E**63**, o4910 [**doi:10.1107/S1600536807060928**]

N-(3-Oxo-2-phenyl-2,3-dihydro-1*H*-pyrrolo[1,2-*c*]imidazol-1-yl)-*N*'-phenyl-*N*-(2,4,6-trimethylphenyl)urea ethyl acetate solvate

W. Imhof

Comment

Derivatives of the title compound have only been described once and have been synthesized due to their molluscicidal activity (Mishriky *et al.*, 1998). To the best of our knowledge this report represents the first structural investigation of a 3-Oxo-2-phenyl-2,3-dihydro-1*H*- pyrrolo[1,2-*c*]imidazole derivative.

The title compound (Scheme 1) is produced by the reaction of one equivalent of the imine with two equivalents of phenyl-isocyanate. Corresponding to the observations of Mishriky *et al.* this reaction most probably proceeds in two steps. First, one isocyanate moiety inserts into the N—H bond of the pyrrole ring forming 2-(2',4',6'-trimethyl-phenyl)iminomethyl-pyrrole-1-carboxylic acid phenylamide. This class of heterocyclic compounds has already been described as intermediates in the synthesis of 1*H*-pyrrolo[1,2-*c*]imidazoles (Mishriky *et al.*, 1998) and have in one case also been structurally characterized (Imhof, 2007). The bicyclic ring system of the title compound is then produced *via* the nucleophilic attack of the amide nitrogen towards the imine carbon atom with the concomitant transfer of the amide hydrogen atom towards the former imine nitrogen. Subsequent insertion of the second isocyanate moiety into the newly formed amide N—H function at the former imine nitrogen atom leads to the formation of the title compound.

The molecular structure of the title compound is shown in Figure 1. A s it is expected the 1,2-dihydro-pyrrolo[1,2-*c*]imidazol-3-one core (C1, C2, C3, C4, C5, C6, N1, N2, O1) is essentially planar. During the reaction a new stereogenic center at C5 is produced. Nevertheless, due to the non-chirality of all substrates and solvents of course the racemate is formed. The observed space group is $P2_1/c$, therefore exhibiting both enantiomeric forms of the title compound. Figure 1 represents the *R*-enantiomer. Bond lengths and angles in the heterocyclic compound show expected values (*cf.* Supplementary Material).

The title compound crystallizes as ethyl acetate solvate. The crystal structure is determined by N—H···O and C—H···O hydrogen bonds between two neighboring molecules of the title compound with O1 acting as the acceptor of a bifurcated hydrogen bond, therefore prducing infinite chains (Figure 2). In addition, weaker C—H···O hydrogen bonds of the title compound towards the solvent molecules with the carbonyl oxygen atom of ethyl acetate also act as the acceptor of a bifurcated hydrogen bond to produce four membered cyclic systems consisting of two molecules of the title compound and two molecules of ethyl acetate (Figure 3). The center of this cyclic arrangement is also a crystallographic center of inversion. The cyclic aggregates are further connected by another C—H···O interaction of the ester oxygen atom towards an aromatic C—H group of a mesityl substituent ending up in the formation of infinte chains of the cyclic arrangements shown in Figure 3. The lengths of these C—H···O hydrogen bonds are in the typical range compared to other structural investigations (Desiraju & Steiner, 1999).

Experimental

215 mg (1.01 mmol) Mesityl-(1*H*-pyrrol-2-ylmethylene)-amine and 180 mg (1.515 mmol) phenylisocyanate were refluxed in 20 ml of THF together with 46 mg (0.04 mmol) [$Pd(PPh_3)_4$] and 3 mg glacial acetic acid for 2 hrs. Evaporation of the solvent yielded an orange oil. Column chromatography on silica yielded 40 mg (12%) of 2-mesityliminomethyl-pyrrole-1-carboxylic acid phenylamidethe using a mixture of pentane and ethyl acetate (8:1) as the eluent. With a ratio of 4:1 the title compound was obtained (108 mg, 24%). Colorless crystals of the title compound were produced from a pentane solution at 253 K.

Refinement

Hydrogen atoms were calculated in idealized positions and refined with distances of 0.86 Å (N4—H4), 0.98 Å (C5—H5), 0.96 Å (CH_3) and 0.93 Å (aromatic CH). All hydrogen atoms were refined using a riding model with U_{iso}(H) = 1.5 times U_{iso}(C, N).

Figures

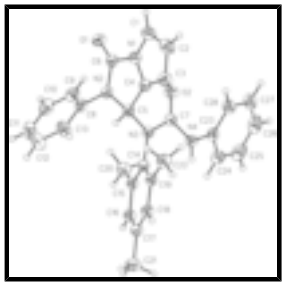

Fig. 1. The molecular structure of the title compoud showing the labelling scheme. Displacement ellipsoids are presented at the 40% probalitiy level.

Fig. 2. Infinite chains of the title compound along the *c* axis.

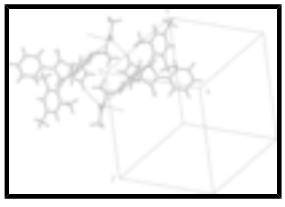

Fig. 3. Infinite chains of cyclic hydrogen bonded aggregates.

2-(3-Oxo-2-phenyl-2,3-dihydro-1*H*-pyrrolo[1,2-*c*]imidazol-1-yl)- *N*-phenyl-2-(2,4,6-trimethylphenyl)acetamide ethyl acetate solvate

Crystal data

$C_{28}H_{26}N_4O_2 \cdot C_4H_8O_2$	F_{000} = 1144
M_r = 538.63	D_x = 1.271 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation

	λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 8455 reflections
a = 16.2289 (11) Å	θ = 2.3–25.0º
b = 11.0873 (7) Å	μ = 0.09 mm^{-1}
c = 16.8630 (8) Å	T = 203 (2) K
β = 111.907 (4)º	Quader, light yellow
V = 2815.1 (3) $Å^3$	0.4 × 0.3 × 0.3 mm
Z = 4	

Data collection

Nonius KappaCCD diffractometer	3265 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.026
Monochromator: graphite	θ_{max} = 25.0º
T = 203(2) K	θ_{min} = 2.3º
ω–scan, phi–scan	h = −19→19
Absorption correction: none	k = −12→13
8455 measured reflections	l = −18→18
4780 independent 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
$wR(F^2)$ = 0.115	$w = 1/[\sigma^2(F_o^2) + (0.0883P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.83	$(\Delta/\sigma)_{max}$ = 0.025
4780 reflections	$\Delta\rho_{max}$ = 0.17 e $Å^{-3}$
366 parameters	$\Delta\rho_{min}$ = −0.20 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 $F^2 > 2\text{sigma}(F^2)$ 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}
O1	0.26955 (9)	0.30094 (11)	0.32304 (7)	0.0502 (3)
N1	0.14653 (10)	0.29652 (13)	0.19593 (9)	0.0392 (4)
C1	0.07187 (14)	0.25677 (17)	0.20841 (13)	0.0521 (5)
H1	0.0688	0.2283	0.2591	0.078*
O2	0.23868 (8)	0.13806 (10)	0.11232 (7)	0.0432 (3)
N2	0.27078 (10)	0.37447 (12)	0.19455 (8)	0.0355 (3)
C2	0.00374 (14)	0.26760 (18)	0.13176 (14)	0.0553 (5)
H2	−0.0551	0.2471	0.1207	0.083*
N3	0.23943 (9)	0.31621 (12)	0.04532 (8)	0.0324 (3)
C3	0.03661 (12)	0.31543 (17)	0.07091 (12)	0.0461 (5)
H3	0.0040	0.3320	0.0137	0.069*
N4	0.26166 (9)	0.13536 (12)	−0.01321 (8)	0.0356 (3)
H4	0.2563	0.1775	−0.0577	0.053*
C4	0.12517 (12)	0.33192 (15)	0.11321 (10)	0.0368 (4)
C5	0.20816 (11)	0.38032 (15)	0.10422 (10)	0.0335 (4)
H5	0.1985	0.4651	0.0868	0.050*
C6	0.23449 (13)	0.32051 (15)	0.24712 (11)	0.0392 (4)
C7	0.24625 (11)	0.19124 (15)	0.05201 (10)	0.0336 (4)
C8	0.35420 (12)	0.43446 (15)	0.22519 (10)	0.0367 (4)
C9	0.42748 (13)	0.38264 (17)	0.28751 (10)	0.0447 (5)
H9	0.4230	0.3067	0.3090	0.067*
C10	0.50732 (13)	0.4440 (2)	0.31774 (12)	0.0531 (5)
H10	0.5561	0.4095	0.3603	0.080*
C11	0.51548 (14)	0.5558 (2)	0.28541 (13)	0.0571 (5)
H11	0.5695	0.5965	0.3057	0.086*
C12	0.44309 (15)	0.60616 (19)	0.22320 (14)	0.0614 (6)
H12	0.4483	0.6813	0.2010	0.092*
C13	0.36229 (13)	0.54674 (17)	0.19292 (12)	0.0508 (5)
H13	0.3135	0.5822	0.1509	0.076*
C14	0.26130 (11)	0.38359 (14)	−0.01744 (9)	0.0321 (4)
C15	0.34965 (11)	0.38869 (14)	−0.01265 (10)	0.0346 (4)
C16	0.36662 (12)	0.45074 (15)	−0.07668 (10)	0.0378 (4)
H16	0.4246	0.4529	−0.0749	0.057*
C17	0.30080 (12)	0.50961 (15)	−0.14316 (10)	0.0382 (4)
C18	0.21474 (12)	0.50428 (15)	−0.14451 (10)	0.0389 (4)
H18	0.1699	0.5446	−0.1877	0.058*
C19	0.19304 (11)	0.44124 (15)	−0.08405 (10)	0.0345 (4)
C20	0.42454 (12)	0.32755 (17)	0.05762 (11)	0.0440 (4)
H20A	0.4187	0.2417	0.0503	0.066*
H20B	0.4224	0.3491	0.1120	0.066*
H20C	0.4802	0.3529	0.0555	0.066*
C21	0.32114 (14)	0.57668 (18)	−0.21132 (11)	0.0511 (5)
H21A	0.3843	0.5802	−0.1962	0.077*
H21B	0.2977	0.6571	−0.2163	0.077*
H21C	0.2944	0.5355	−0.2649	0.077*

C22	0.09814 (12)	0.43324 (18)	−0.09162 (11)	0.0445 (4)
H22A	0.0898	0.4823	−0.0483	0.067*
H22B	0.0839	0.3509	−0.0844	0.067*
H22C	0.0600	0.4614	−0.1470	0.067*
C23	0.28586 (11)	0.01230 (15)	−0.01252 (10)	0.0356 (4)
C24	0.34908 (12)	−0.01721 (16)	−0.04676 (10)	0.0424 (4)
H24	0.3751	0.0432	−0.0679	0.064*
C25	0.37323 (15)	−0.13587 (18)	−0.04937 (12)	0.0558 (5)
H25	0.4149	−0.1552	−0.0731	0.084*
C26	0.33646 (15)	−0.22613 (18)	−0.01726 (13)	0.0597 (6)
H26	0.3539	−0.3059	−0.0181	0.090*
C27	0.27349 (15)	−0.19684 (17)	0.01613 (12)	0.0537 (5)
H27	0.2477	−0.2576	0.0372	0.081*
C28	0.24790 (13)	−0.07770 (15)	0.01877 (11)	0.0438 (4)
H28	0.2054	−0.0589	0.0415	0.066*
O3	0.08918 (9)	0.79550 (12)	0.18966 (8)	0.0534 (4)
O4	0.14584 (10)	0.66986 (14)	0.11965 (9)	0.0656 (4)
C29	0.15269 (16)	0.6166 (2)	0.25872 (13)	0.0648 (6)
H29A	0.1092	0.5538	0.2481	0.097*
H29B	0.1533	0.6643	0.3064	0.097*
H29C	0.2103	0.5815	0.2714	0.097*
C30	0.13003 (12)	0.69448 (18)	0.18177 (12)	0.0477 (5)
C31	0.06762 (15)	0.88484 (19)	0.12199 (13)	0.0580 (5)
H31A	0.0655	0.8472	0.0693	0.087*
H31B	0.1128	0.9472	0.1372	0.087*
C32	−0.01968 (17)	0.9382 (2)	0.10986 (19)	0.0841 (8)
H32A	−0.0651	0.8781	0.0877	0.126*
H32B	−0.0315	1.0042	0.0703	0.126*
H32C	−0.0192	0.9671	0.1637	0.126*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0721 (10)	0.0464 (8)	0.0334 (7)	−0.0057 (7)	0.0210 (6)	0.0013 (5)
N1	0.0473 (10)	0.0367 (8)	0.0400 (8)	−0.0032 (7)	0.0236 (7)	−0.0015 (6)
C1	0.0641 (14)	0.0455 (11)	0.0614 (13)	−0.0056 (10)	0.0403 (12)	−0.0001 (9)
O2	0.0604 (9)	0.0351 (7)	0.0410 (6)	0.0059 (6)	0.0270 (6)	0.0056 (5)
N2	0.0429 (9)	0.0327 (8)	0.0302 (7)	−0.0008 (7)	0.0131 (6)	−0.0002 (6)
C2	0.0476 (13)	0.0550 (13)	0.0712 (14)	−0.0049 (10)	0.0313 (11)	0.0003 (10)
N3	0.0393 (8)	0.0284 (8)	0.0324 (7)	0.0038 (6)	0.0168 (6)	0.0010 (5)
C3	0.0416 (12)	0.0457 (11)	0.0528 (11)	0.0004 (9)	0.0198 (9)	−0.0018 (8)
N4	0.0451 (9)	0.0308 (8)	0.0343 (7)	0.0044 (6)	0.0187 (6)	0.0030 (6)
C4	0.0437 (11)	0.0316 (9)	0.0398 (9)	0.0018 (8)	0.0209 (8)	−0.0022 (7)
C5	0.0371 (10)	0.0311 (9)	0.0328 (8)	0.0028 (7)	0.0138 (7)	−0.0003 (6)
C6	0.0541 (12)	0.0301 (10)	0.0371 (10)	0.0000 (8)	0.0212 (9)	−0.0022 (7)
C7	0.0341 (10)	0.0337 (10)	0.0330 (9)	0.0028 (7)	0.0124 (7)	0.0009 (7)
C8	0.0430 (11)	0.0335 (10)	0.0348 (8)	0.0009 (8)	0.0156 (8)	−0.0046 (7)
C9	0.0499 (12)	0.0443 (11)	0.0388 (9)	0.0088 (9)	0.0153 (9)	−0.0005 (8)

C10	0.0422 (12)	0.0663 (14)	0.0436 (10)	0.0079 (11)	0.0076 (9)	−0.0084 (9)
C11	0.0431 (13)	0.0571 (14)	0.0667 (13)	−0.0074 (10)	0.0153 (10)	−0.0182 (11)
C12	0.0512 (14)	0.0457 (12)	0.0831 (15)	−0.0077 (10)	0.0201 (12)	0.0027 (11)
C13	0.0446 (12)	0.0411 (11)	0.0599 (12)	−0.0011 (9)	0.0119 (9)	0.0071 (9)
C14	0.0389 (10)	0.0266 (9)	0.0333 (8)	0.0009 (7)	0.0162 (7)	−0.0013 (7)
C15	0.0389 (10)	0.0305 (9)	0.0339 (8)	0.0004 (8)	0.0131 (7)	−0.0025 (7)
C16	0.0389 (10)	0.0358 (10)	0.0427 (9)	−0.0036 (8)	0.0197 (8)	−0.0055 (7)
C17	0.0464 (11)	0.0336 (10)	0.0364 (9)	−0.0026 (8)	0.0174 (8)	−0.0016 (7)
C18	0.0461 (11)	0.0347 (10)	0.0336 (9)	0.0035 (8)	0.0123 (8)	0.0030 (7)
C19	0.0392 (10)	0.0297 (9)	0.0341 (9)	0.0018 (8)	0.0132 (8)	−0.0015 (7)
C20	0.0385 (11)	0.0454 (11)	0.0473 (10)	0.0040 (9)	0.0151 (8)	0.0043 (8)
C21	0.0590 (13)	0.0518 (12)	0.0468 (10)	−0.0017 (10)	0.0247 (9)	0.0076 (9)
C22	0.0408 (11)	0.0492 (11)	0.0418 (9)	0.0063 (9)	0.0136 (8)	0.0072 (8)
C23	0.0385 (10)	0.0322 (10)	0.0331 (8)	0.0029 (8)	0.0100 (7)	−0.0023 (7)
C24	0.0442 (11)	0.0433 (11)	0.0400 (9)	0.0074 (9)	0.0160 (8)	−0.0004 (8)
C25	0.0647 (14)	0.0527 (13)	0.0493 (11)	0.0187 (11)	0.0203 (10)	−0.0055 (9)
C26	0.0779 (16)	0.0367 (12)	0.0545 (12)	0.0147 (11)	0.0131 (11)	−0.0064 (9)
C27	0.0682 (14)	0.0328 (11)	0.0529 (11)	−0.0049 (10)	0.0142 (10)	−0.0003 (9)
C28	0.0479 (12)	0.0360 (10)	0.0472 (10)	−0.0012 (9)	0.0174 (9)	−0.0005 (8)
O3	0.0595 (9)	0.0494 (8)	0.0511 (8)	0.0117 (7)	0.0205 (7)	−0.0004 (6)
O4	0.0703 (11)	0.0714 (10)	0.0585 (9)	0.0184 (8)	0.0280 (8)	−0.0045 (7)
C29	0.0708 (16)	0.0627 (14)	0.0616 (13)	0.0168 (12)	0.0254 (11)	0.0055 (10)
C30	0.0391 (11)	0.0529 (12)	0.0479 (11)	0.0006 (10)	0.0125 (9)	−0.0074 (9)
C31	0.0669 (15)	0.0512 (13)	0.0535 (11)	0.0068 (10)	0.0197 (10)	0.0030 (9)
C32	0.0685 (17)	0.0624 (16)	0.118 (2)	0.0154 (13)	0.0304 (15)	0.0237 (15)

Geometric parameters (Å, °)

O1—C6	1.2109 (19)	C17—C18	1.390 (2)
N1—C4	1.364 (2)	C17—C21	1.505 (2)
N1—C1	1.377 (2)	C18—C19	1.386 (2)
N1—C6	1.391 (2)	C18—H18	0.9300
C1—C2	1.357 (3)	C19—C22	1.500 (2)
C1—H1	0.9300	C20—H20A	0.9600
O2—C7	1.2207 (19)	C20—H20B	0.9600
N2—C6	1.371 (2)	C20—H20C	0.9600
N2—C8	1.421 (2)	C21—H21A	0.9600
N2—C5	1.484 (2)	C21—H21B	0.9600
C2—C3	1.424 (3)	C21—H21C	0.9600
C2—H2	0.9300	C22—H22A	0.9600
N3—C7	1.391 (2)	C22—H22B	0.9600
N3—C14	1.4436 (19)	C22—H22C	0.9600
N3—C5	1.4579 (19)	C23—C28	1.377 (2)
C3—C4	1.357 (3)	C23—C24	1.391 (2)
C3—H3	0.9300	C24—C25	1.378 (3)
N4—C7	1.364 (2)	C24—H24	0.9300
N4—C23	1.419 (2)	C25—C26	1.375 (3)
N4—H4	0.8600	C25—H25	0.9300
C4—C5	1.509 (2)	C26—C27	1.377 (3)

C5—H5	0.9800	C26—H26	0.9300
C8—C9	1.384 (2)	C27—C28	1.390 (3)
C8—C13	1.384 (3)	C27—H27	0.9300
C9—C10	1.381 (3)	C28—H28	0.9300
C9—H9	0.9300	O3—C30	1.333 (2)
C10—C11	1.381 (3)	O3—C31	1.452 (2)
C10—H10	0.9300	O4—C30	1.199 (2)
C11—C12	1.368 (3)	C29—C30	1.486 (3)
C11—H11	0.9300	C29—H29A	0.9600
C12—C13	1.384 (3)	C29—H29B	0.9600
C12—H12	0.9300	C29—H29C	0.9600
C13—H13	0.9300	C31—C32	1.478 (3)
C14—C19	1.403 (2)	C31—H31A	0.9700
C14—C15	1.407 (2)	C31—H31B	0.9700
C15—C16	1.392 (2)	C32—H32A	0.9600
C15—C20	1.505 (2)	C32—H32B	0.9600
C16—C17	1.389 (2)	C32—H32C	0.9600
C16—H16	0.9300		
C4—N1—C1	110.25 (15)	C18—C17—C21	120.92 (16)
C4—N1—C6	112.97 (14)	C19—C18—C17	122.64 (16)
C1—N1—C6	136.39 (15)	C19—C18—H18	118.7
C2—C1—N1	106.06 (16)	C17—C18—H18	118.7
C2—C1—H1	127.0	C18—C19—C14	118.51 (16)
N1—C1—H1	127.0	C18—C19—C22	120.14 (15)
C6—N2—C8	123.34 (13)	C14—C19—C22	121.34 (15)
C6—N2—C5	112.82 (14)	C15—C20—H20A	109.5
C8—N2—C5	123.18 (12)	C15—C20—H20B	109.5
C1—C2—C3	109.16 (18)	H20A—C20—H20B	109.5
C1—C2—H2	125.4	C15—C20—H20C	109.5
C3—C2—H2	125.4	H20A—C20—H20C	109.5
C7—N3—C14	122.75 (12)	H20B—C20—H20C	109.5
C7—N3—C5	117.88 (12)	C17—C21—H21A	109.5
C14—N3—C5	119.36 (12)	C17—C21—H21B	109.5
C4—C3—C2	106.22 (17)	H21A—C21—H21B	109.5
C4—C3—H3	126.9	C17—C21—H21C	109.5
C2—C3—H3	126.9	H21A—C21—H21C	109.5
C7—N4—C23	124.30 (13)	H21B—C21—H21C	109.5
C7—N4—H4	117.9	C19—C22—H22A	109.5
C23—N4—H4	117.9	C19—C22—H22B	109.5
C3—C4—N1	108.30 (15)	H22A—C22—H22B	109.5
C3—C4—C5	143.58 (16)	C19—C22—H22C	109.5
N1—C4—C5	108.02 (14)	H22A—C22—H22C	109.5
N3—C5—N2	113.50 (13)	H22B—C22—H22C	109.5
N3—C5—C4	116.47 (13)	C28—C23—C24	119.55 (16)
N2—C5—C4	100.16 (12)	C28—C23—N4	122.56 (15)
N3—C5—H5	108.8	C24—C23—N4	117.88 (15)
N2—C5—H5	108.8	C25—C24—C23	120.02 (18)
C4—C5—H5	108.8	C25—C24—H24	120.0
O1—C6—N2	128.03 (18)	C23—C24—H24	120.0

O1—C6—N1	126.19 (16)	C26—C25—C24	120.78 (19)
N2—C6—N1	105.73 (14)	C26—C25—H25	119.6
O2—C7—N4	123.91 (15)	C24—C25—H25	119.6
O2—C7—N3	121.27 (13)	C25—C26—C27	119.12 (18)
N4—C7—N3	114.82 (13)	C25—C26—H26	120.4
C9—C8—C13	119.40 (17)	C27—C26—H26	120.4
C9—C8—N2	120.92 (16)	C26—C27—C28	120.88 (19)
C13—C8—N2	119.68 (15)	C26—C27—H27	119.6
C8—C9—C10	119.86 (18)	C28—C27—H27	119.6
C8—C9—H9	120.1	C23—C28—C27	119.64 (17)
C10—C9—H9	120.1	C23—C28—H28	120.2
C11—C10—C9	120.75 (19)	C27—C28—H28	120.2
C11—C10—H10	119.6	C30—O3—C31	118.53 (14)
C9—C10—H10	119.6	C30—C29—H29A	109.5
C12—C11—C10	119.18 (19)	C30—C29—H29B	109.5
C12—C11—H11	120.4	H29A—C29—H29B	109.5
C10—C11—H11	120.4	C30—C29—H29C	109.5
C11—C12—C13	120.9 (2)	H29A—C29—H29C	109.5
C11—C12—H12	119.6	H29B—C29—H29C	109.5
C13—C12—H12	119.6	O4—C30—O3	123.69 (18)
C12—C13—C8	119.93 (18)	O4—C30—C29	124.52 (19)
C12—C13—H13	120.0	O3—C30—C29	111.79 (16)
C8—C13—H13	120.0	O3—C31—C32	108.63 (18)
C19—C14—C15	120.72 (14)	O3—C31—H31A	110.0
C19—C14—N3	119.08 (14)	C32—C31—H31A	110.0
C15—C14—N3	120.18 (14)	O3—C31—H31B	110.0
C16—C15—C14	117.89 (15)	C32—C31—H31B	110.0
C16—C15—C20	119.87 (16)	H31A—C31—H31B	108.3
C14—C15—C20	122.22 (14)	C31—C32—H32A	109.5
C17—C16—C15	122.90 (16)	C31—C32—H32B	109.5
C17—C16—H16	118.6	H32A—C32—H32B	109.5
C15—C16—H16	118.6	C31—C32—H32C	109.5
C16—C17—C18	117.31 (15)	H32A—C32—H32C	109.5
C16—C17—C21	121.77 (17)	H32B—C32—H32C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4···O1^{i}	0.86	2.11	2.899 (2)	151
C24—H24···O1^{i}	0.93	2.64	3.181 (2)	118
C18—H18···O3^{ii}	0.93	2.67	3.556 (2)	160
C3—H3···O4^{iii}	0.93	2.62	3.467 (2)	151
C27—H27···O4^{iv}	0.93	2.65	3.496 (2)	151

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

Fig. 1

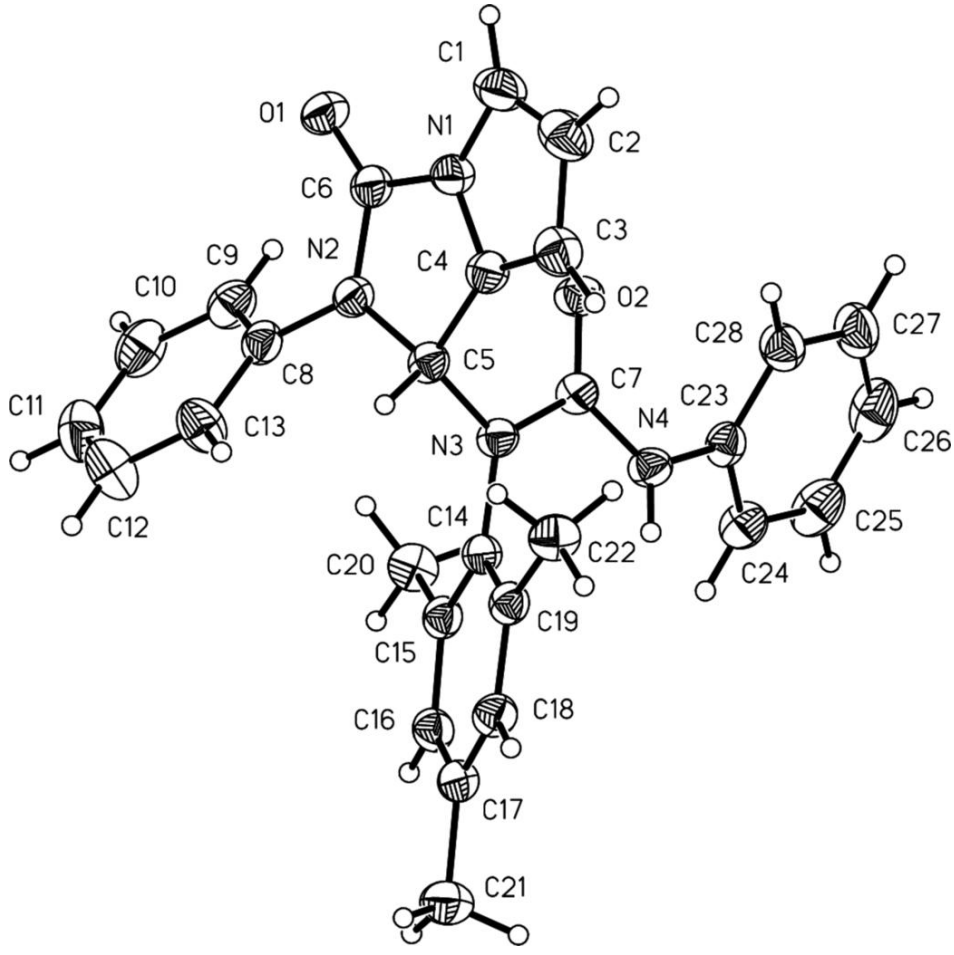
C1
O1
N1
C2
C6
C3
C9
N2
C4
C10
O2
C28
C27
C8
C5
C7
C23
C11
C13
N3
N4
C26
C12
C14
C25
C20
C22
C24
C19
C15
C16
C18
C17
C21

Fig. 2

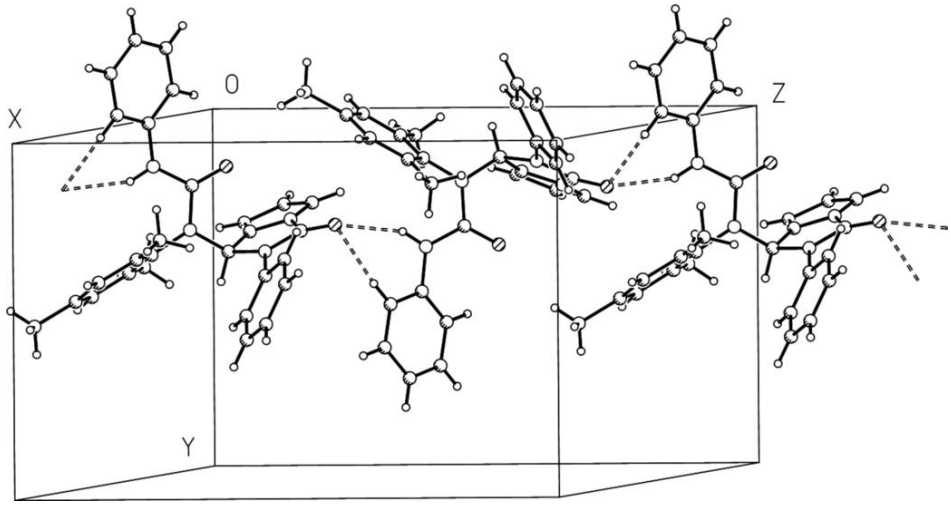

Fig. 3