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6-Amino-4-(3-nitrophenyl)-2-oxo-1,2-dihydropyridine-3,5-dicarbonitrile ethanol solvate

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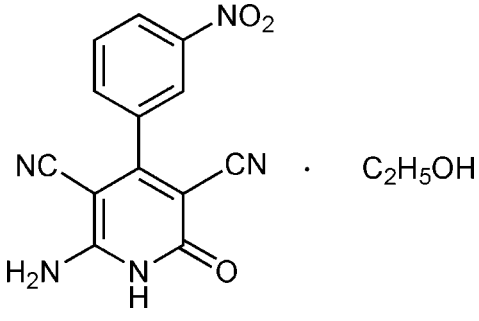
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; disorder in main residue; R factor = 0.058; wR factor = 0.124; data-to-parameter ratio = 12.2.

The title compound, $C_{13}H_7N_5O_3\cdot C_2H_6O$, was synthesized by the reaction of 2-cyanoacetamide, malononitrile and 3-nitrobenzaldehyde in water. The asymmetric unit contains two crystallographically independent 6-amino-4-(3-nitrophenyl)-2-oxo-1,2-dihydropyridine-3,5-dicarbonitrile molecules of similar conformation and two ethanol solvent molecules. The nitro group of one molecule is disordered over two positions with site-occupation factors of 0.688 (17) and 0.312 (17). The dihedral angles between the pyridine and benzene rings are 52.06 (11) and 65.12 (13)° in the two molecules. The crystal packing is stabilized by N—H···O, N—H···N, O—H···O and C—H···O intra- and intermolecular hydrogen bonds.

Related literature

For general background, see: Boeckman *et al.* (1987); Comins *et al.* (1994); Cox & O'Hagan (1991); Deshang *et al.* (1988); Kozikowski *et al.* (1996); Li *et al.* (2000); Nagarajan *et al.* (2003).



Experimental

Crystal data

$C_{13}H_7N_5O_3\cdot C_2H_6O$
M_r = 327.30
Monoclinic, $P2_1/n$
a = 13.079 (3) Å
b = 14.764 (4) Å
c = 16.693 (4) Å
β = 90.923 (4)°
V = 3223.0 (14) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 (2) K
0.45 × 0.36 × 0.31 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.950, T_{max} = 0.962
16780 measured reflections
5690 independent reflections
1952 reflections with $I > 2\sigma(I)$
R_{int} = 0.089

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.124
S = 1.01
5690 reflections
465 parameters
160 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N9—H9A···O8	0.86	1.94	2.785 (5)	167
C22—H22···O5	0.93	2.42	2.802 (19)	104
N1—H1···O4^{i}	0.86	1.98	2.826 (4)	170
N4—H4A···O7^{ii}	0.86	1.97	2.816 (4)	169
N4—H4B···N8^{ii}	0.86	2.17	3.009 (5)	164
C13—H13···O3^{ii}	0.93	2.32	3.228 (6)	167
C25—H25···O3^{ii}	0.93	2.59	3.464 (7)	158
N6—H6···O1^{iii}	0.86	1.98	2.830 (4)	171
O8—H8···O1^{iii}	0.82	2.19	2.824 (4)	135
N9—H9B···N3^{iv}	0.86	2.22	3.047 (5)	163
O7—H7···O4^{v}	0.82	2.01	2.753 (4)	150

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); 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, 1999); 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: RZ2140).

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supplementary materials

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6-Amino-4-(3-nitrophenyl)-2-oxo-1,2-dihydropyridine-3,5-dicarbonitrile ethanol solvate

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Comment

The synthesis of 2-pyridone derivatives is an area of growing interest because compounds with these scaffolds exhibited significant pharmacological properties such as antibacterial (Li *et al.*, 2000), antifungal (Cox & O'Hagan, 1991) and anti-tumor activity (Nagarajan *et al.*, 2003). These compounds have been used also as cardiotonic (Deshang *et al.*, 1988) and psychotherapeutic agents (Kozikowski *et al.*, 1996) and potential HIC-1 specific transcriptase inhibitors (Boeckman *et al.*, 1987). Moreover it is well know that the 2-pyridone ring system is a valuable building block in the symthesis of natural products (Comins *et al.*, 1994). For these reasons, the synthesis of new compounds containing 2-pyridone derivatives is strongly desired. In this paper we report the crystal structure of the title compound.

In the asymmetric units there are two title molecules of similar geometry and two ethanol molecules (Fig. 1). One nitro group is disordered over two positions, with refined site occupation factors of 0.688 (17) and 0.312 (17) for the major and minor components, respectively. The dihedral angle between the C1/C2/C3/C4/C5/N1 plane and the C8—C13 benzene ring is 52.06 (11) °, while the corresponding angle between the C14/C15/C16/C17/C18/N6 plane and the C21—C26 benzene ring is 65.12 (13)°. The crystal packing is stabilized by an extended network of N—H···O, N—H···N, O—H···O and C—H···O intra- and intermolecular hydrogen bonds (Table 1, Fig. 2).

Experimental

The title Compound was prepared by the reaction of 2-cyanoacetamide (2 mmol), malononitrile (2 mmol) and 3-nitrobenzaldehyde (2 mmol) in water (2 ml). Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of a 95% aqueous ethanol solution (yield 48%; m.p. >573 K). IR (cm^{-1}): 3546, 3338, 3189, 2214, 1670, 1646, 1529; ^1H NMR (DMSO-d_6): 7.87 (2*H*, brs, NH_2), 7.88–8.01 (4*H*, m, ArH), 11.85 (1*H*, s, NH).

Refinement

All H atoms were positioned geometrically and treated as riding, with N—H = 0.86 Å, O—H = 0.82 Å and C—H = 0.93–0.97 Å, and with U_{iso}(H) = 1.2 U_{eq}(C, N) or 1.5 U_{eq}(C, O) for methyl and hydroxy groups.

Figures

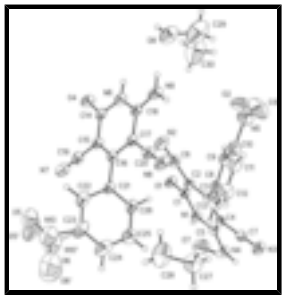

Fig. 1. The molecular structure of the title compound, showing 30% probability displacement ellipsoids.

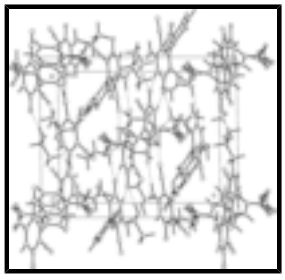

Fig. 2. Packing diagram of the title compound viewed along the *b* axis. Dashed lines indicate hydrogen bonds.

6-Amino-4-(3-nitrophenyl)-2-oxo-1,2-dihydropyridine-3,5-dicarbonitrile ethanol solvate

Crystal data

$C_{13}H_7N_5O_3 \cdot C_2H_6O$	F_{000} = 1360
M_r = 327.30	D_x = 1.349 Mg m^{-3}
Monoclinic, $P2_1/n$	Melting point: >573 K
Hall symbol: -P 2yn	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 13.079 (3) Å	Cell parameters from 1628 reflections
b = 14.764 (4) Å	θ = 2.4–19,693º
c = 16.693 (4) Å	μ = 0.10 mm^{-1}
β = 90.923 (4)º	T = 298 (2) K
V = 3223.0 (14) Å^3	Block, yellow
Z = 8	0.45 × 0.36 × 0.31 mm

Data collection

Bruker SMART CCD area-detector diffractometer	5690 independent reflections
Radiation source: fine-focus sealed tube	1952 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.089
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.0º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −15→15
T_{min} = 0.950, T_{max} = 0.962	k = −15→17
16780 measured reflections	l = −19→16

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.058$	H-atom parameters constrained
$wR(F^2) = 0.124$	$w = 1/[\sigma^2(F_o^2) + (0.0257P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} < 0.001$
5690 reflections	$\Delta\rho_{max} = 0.38$ e Å^{-3}
465 parameters	$\Delta\rho_{min} = -0.33$ e Å^{-3}
160 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	0.5413 (2)	−0.0007 (2)	0.88884 (17)	0.0437 (9)	
H1	0.5749	−0.0469	0.9064	0.052*	
N2	0.6368 (3)	0.2644 (3)	0.7473 (2)	0.0869 (14)	
N3	0.1889 (3)	0.0708 (2)	0.8869 (2)	0.0649 (11)	
N4	0.3967 (2)	−0.0744 (2)	0.92758 (17)	0.0535 (10)	
H4A	0.4351	−0.1178	0.9448	0.064*	
H4B	0.3315	−0.0780	0.9321	0.064*	
N5	0.3662 (4)	0.4712 (3)	0.8369 (3)	0.0937 (16)	
N6	0.6783 (2)	0.4244 (2)	0.59015 (17)	0.0485 (9)	
H6	0.7123	0.4700	0.6084	0.058*	
N7	0.7718 (3)	0.1339 (3)	0.4923 (2)	0.0779 (13)	
N8	0.3234 (3)	0.3745 (2)	0.5484 (2)	0.0711 (12)	
N9	0.5327 (2)	0.5071 (2)	0.61243 (18)	0.0612 (10)	
H9A	0.5706	0.5513	0.6288	0.073*	
H9B	0.4673	0.5127	0.6117	0.073*	
N10	0.447 (3)	0.099 (3)	0.305 (2)	0.107 (9)	0.312 (17)
N10'	0.4599 (12)	0.0567 (11)	0.3306 (8)	0.098 (4)	0.688 (17)

O1	0.69115 (19)	0.06659 (18)	0.85852 (15)	0.0582 (8)	
O2	0.4461 (4)	0.4791 (3)	0.8736 (3)	0.1390 (18)	
O3	0.3055 (3)	0.5318 (3)	0.8276 (2)	0.1389 (18)	
O4	0.82853 (19)	0.35442 (18)	0.56654 (15)	0.0615 (9)	
O5	0.4957 (16)	0.1407 (14)	0.2640 (11)	0.097 (6)	0.312 (17)
O6	0.3707 (16)	0.0454 (18)	0.2843 (11)	0.128 (7)	0.312 (17)
O5'	0.5210 (8)	0.0869 (12)	0.2864 (7)	0.136 (4)	0.688 (17)
O6'	0.4135 (8)	−0.0099 (8)	0.3139 (6)	0.137 (4)	0.688 (17)
O7	0.0021 (2)	0.2717 (2)	0.5108 (2)	0.0806 (10)	
H7	−0.0559	0.2762	0.5283	0.121*	
O8	0.6269 (2)	0.6604 (3)	0.6769 (2)	0.1075 (13)	
H8	0.6889	0.6636	0.6709	0.161*	
C1	0.5962 (3)	0.0702 (3)	0.8570 (2)	0.0447 (11)	
C2	0.5381 (3)	0.1436 (3)	0.8234 (2)	0.0431 (11)	
C3	0.4334 (3)	0.1448 (3)	0.8294 (2)	0.0408 (10)	
C4	0.3825 (3)	0.0726 (3)	0.8654 (2)	0.0400 (10)	
C5	0.4381 (3)	−0.0024 (3)	0.8943 (2)	0.0406 (11)	
C6	0.5925 (3)	0.2113 (3)	0.7813 (3)	0.0572 (13)	
C7	0.2748 (3)	0.0719 (3)	0.8769 (2)	0.0459 (11)	
C8	0.3733 (3)	0.2238 (3)	0.8012 (2)	0.0466 (11)	
C9	0.3988 (3)	0.3087 (3)	0.8279 (2)	0.0544 (12)	
H9	0.4558	0.3170	0.8612	0.065*	
C10	0.3397 (4)	0.3813 (3)	0.8054 (3)	0.0634 (14)	
C11	0.2560 (4)	0.3732 (4)	0.7573 (3)	0.0808 (17)	
H11	0.2168	0.4236	0.7438	0.097*	
C12	0.2302 (4)	0.2888 (4)	0.7289 (3)	0.0791 (16)	
H12	0.1734	0.2818	0.6953	0.095*	
C13	0.2886 (3)	0.2144 (3)	0.7503 (2)	0.0628 (13)	
H13	0.2711	0.1575	0.7305	0.075*	
C14	0.7335 (3)	0.3502 (3)	0.5651 (2)	0.0453 (11)	
C15	0.6741 (3)	0.2752 (3)	0.5383 (2)	0.0445 (11)	
C16	0.5687 (3)	0.2784 (3)	0.5358 (2)	0.0424 (11)	
C17	0.5190 (3)	0.3557 (3)	0.5605 (2)	0.0430 (11)	
C18	0.5750 (3)	0.4312 (3)	0.5884 (2)	0.0449 (11)	
C19	0.7282 (3)	0.1969 (3)	0.5126 (3)	0.0547 (13)	
C20	0.4105 (3)	0.3644 (3)	0.5539 (2)	0.0507 (12)	
C21	0.5069 (3)	0.1995 (3)	0.5085 (3)	0.0462 (11)	
C22	0.5108 (3)	0.1706 (3)	0.4295 (3)	0.0638 (14)	
H22	0.5532	0.1988	0.3929	0.077*	
C23	0.4500 (4)	0.0988 (4)	0.4074 (3)	0.0753 (16)	
C24	0.3867 (4)	0.0549 (3)	0.4580 (4)	0.0872 (19)	
H24	0.3459	0.0073	0.4398	0.105*	
C25	0.3839 (3)	0.0819 (3)	0.5366 (4)	0.0821 (16)	
H25	0.3425	0.0523	0.5730	0.098*	
C26	0.4444 (3)	0.1545 (3)	0.5604 (3)	0.0621 (13)	
H26	0.4426	0.1733	0.6136	0.074*	
C27	0.0420 (4)	0.1856 (4)	0.5300 (3)	0.0975 (18)	
H27A	−0.0073	0.1394	0.5149	0.117*	
H27B	0.0540	0.1816	0.5874	0.117*	

C28	0.1378 (4)	0.1693 (4)	0.4885 (3)	0.140 (2)
H28A	0.1244	0.1647	0.4320	0.210*
H28B	0.1679	0.1139	0.5076	0.210*
H28C	0.1841	0.2186	0.4988	0.210*
C29	0.6040 (6)	0.6753 (5)	0.7591 (5)	0.180 (4)
H29A	0.5308	0.6793	0.7670	0.216*
H29B	0.6364	0.7300	0.7792	0.216*
C30	0.6487 (6)	0.5913 (5)	0.7994 (5)	0.210 (4)
H30A	0.6229	0.5381	0.7729	0.315*
H30B	0.6295	0.5902	0.8546	0.315*
H30C	0.7219	0.5927	0.7960	0.315*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.035 (2)	0.043 (2)	0.053 (2)	0.0012 (18)	−0.0031 (17)	0.0028 (17)
N2	0.075 (3)	0.077 (4)	0.109 (4)	−0.005 (3)	0.027 (3)	0.023 (3)
N3	0.038 (2)	0.068 (3)	0.088 (3)	0.005 (2)	0.008 (2)	0.008 (2)
N4	0.0299 (19)	0.057 (3)	0.074 (3)	0.0025 (19)	−0.0017 (18)	0.007 (2)
N5	0.120 (5)	0.046 (4)	0.116 (4)	0.016 (4)	0.017 (4)	0.005 (3)
N6	0.032 (2)	0.050 (3)	0.063 (2)	−0.0055 (19)	−0.0007 (18)	−0.0132 (19)
N7	0.061 (3)	0.071 (3)	0.101 (3)	0.006 (2)	0.012 (2)	−0.017 (3)
N8	0.036 (2)	0.072 (3)	0.105 (3)	0.001 (2)	0.008 (2)	−0.022 (2)
N9	0.035 (2)	0.058 (3)	0.092 (3)	0.0023 (19)	0.0032 (19)	−0.019 (2)
N10	0.105 (12)	0.112 (12)	0.105 (12)	−0.017 (9)	0.001 (9)	−0.032 (9)
N10'	0.083 (7)	0.116 (11)	0.095 (9)	−0.039 (7)	0.000 (7)	−0.048 (7)
O1	0.0329 (17)	0.062 (2)	0.080 (2)	−0.0014 (15)	0.0019 (15)	0.0115 (15)
O2	0.133 (4)	0.062 (3)	0.221 (5)	−0.007 (3)	−0.013 (4)	−0.019 (3)
O3	0.213 (5)	0.066 (3)	0.137 (4)	0.069 (3)	0.002 (3)	0.007 (2)
O4	0.0309 (17)	0.060 (2)	0.094 (2)	−0.0044 (15)	−0.0003 (16)	−0.0134 (16)
O5	0.100 (9)	0.111 (9)	0.081 (8)	−0.026 (7)	0.011 (7)	−0.023 (7)
O6	0.124 (9)	0.142 (10)	0.119 (9)	−0.041 (8)	−0.006 (7)	−0.048 (7)
O5'	0.121 (6)	0.177 (8)	0.111 (6)	−0.032 (6)	0.028 (5)	−0.068 (6)
O6'	0.152 (6)	0.122 (7)	0.137 (6)	−0.020 (5)	−0.022 (5)	−0.061 (5)
O7	0.056 (2)	0.070 (2)	0.116 (3)	−0.0024 (19)	0.009 (2)	−0.014 (2)
O8	0.067 (2)	0.105 (3)	0.151 (4)	−0.005 (2)	0.003 (2)	−0.043 (3)
C1	0.033 (3)	0.053 (3)	0.048 (3)	−0.006 (3)	0.005 (2)	−0.006 (2)
C2	0.039 (3)	0.040 (3)	0.050 (3)	0.004 (2)	0.003 (2)	0.003 (2)
C3	0.041 (3)	0.038 (3)	0.043 (3)	0.001 (2)	0.002 (2)	−0.006 (2)
C4	0.030 (2)	0.040 (3)	0.050 (3)	0.001 (2)	−0.001 (2)	−0.004 (2)
C5	0.032 (3)	0.044 (3)	0.047 (3)	−0.007 (2)	0.004 (2)	−0.005 (2)
C6	0.041 (3)	0.051 (4)	0.080 (4)	0.004 (2)	0.011 (3)	0.006 (3)
C7	0.042 (3)	0.041 (3)	0.055 (3)	0.004 (2)	0.002 (2)	0.000 (2)
C8	0.043 (3)	0.043 (3)	0.054 (3)	0.002 (2)	0.004 (2)	0.001 (2)
C9	0.045 (3)	0.049 (3)	0.069 (3)	0.007 (3)	0.006 (2)	0.003 (3)
C10	0.064 (4)	0.051 (4)	0.076 (4)	0.011 (3)	0.012 (3)	0.007 (3)
C11	0.074 (4)	0.075 (5)	0.094 (4)	0.028 (4)	0.018 (3)	0.034 (3)
C12	0.061 (3)	0.088 (5)	0.088 (4)	0.009 (4)	−0.017 (3)	0.026 (4)

C13	0.056 (3)	0.067 (4)	0.064 (3)	0.001 (3)	−0.004 (3)	0.002 (3)
C14	0.034 (3)	0.053 (3)	0.049 (3)	0.001 (2)	0.004 (2)	−0.008 (2)
C15	0.034 (3)	0.047 (3)	0.053 (3)	0.000 (2)	0.003 (2)	−0.010 (2)
C16	0.034 (3)	0.047 (3)	0.046 (3)	−0.005 (2)	0.001 (2)	−0.006 (2)
C17	0.027 (2)	0.052 (3)	0.050 (3)	−0.004 (2)	0.002 (2)	−0.008 (2)
C18	0.029 (3)	0.049 (3)	0.056 (3)	0.002 (2)	0.003 (2)	−0.006 (2)
C19	0.038 (3)	0.063 (4)	0.063 (3)	−0.010 (3)	0.006 (2)	−0.006 (3)
C20	0.047 (3)	0.043 (3)	0.062 (3)	−0.003 (3)	0.005 (3)	−0.012 (2)
C21	0.036 (3)	0.049 (3)	0.053 (3)	−0.002 (2)	0.004 (2)	−0.010 (3)
C22	0.041 (3)	0.080 (4)	0.071 (4)	−0.018 (3)	0.006 (2)	−0.023 (3)
C23	0.051 (3)	0.089 (4)	0.087 (4)	−0.010 (3)	0.008 (3)	−0.050 (4)
C24	0.053 (4)	0.068 (4)	0.142 (6)	−0.016 (3)	0.014 (4)	−0.037 (4)
C25	0.057 (3)	0.067 (4)	0.122 (5)	−0.015 (3)	0.016 (3)	−0.004 (4)
C26	0.049 (3)	0.070 (4)	0.067 (3)	−0.009 (3)	0.016 (3)	−0.007 (3)
C27	0.100 (5)	0.090 (5)	0.102 (5)	0.010 (4)	−0.007 (4)	0.009 (3)
C28	0.079 (4)	0.175 (7)	0.166 (6)	0.048 (4)	0.017 (4)	0.008 (5)
C29	0.155 (7)	0.210 (10)	0.177 (8)	0.007 (7)	0.016 (6)	−0.073 (7)
C30	0.175 (7)	0.281 (8)	0.175 (7)	−0.054 (6)	0.038 (5)	−0.045 (6)

Geometric parameters (Å, °)

N1—C5	1.355 (4)	C8—C13	1.392 (5)
N1—C1	1.380 (4)	C9—C10	1.370 (5)
N1—H1	0.8600	C9—H9	0.9300
N2—C6	1.132 (5)	C10—C11	1.352 (6)
N3—C7	1.139 (4)	C11—C12	1.373 (6)
N4—C5	1.320 (4)	C11—H11	0.9300
N4—H4A	0.8600	C12—C13	1.382 (6)
N4—H4B	0.8600	C12—H12	0.9300
N5—O3	1.205 (5)	C13—H13	0.9300
N5—O2	1.210 (5)	C14—C15	1.421 (5)
N5—C10	1.466 (6)	C15—C16	1.380 (5)
N6—C18	1.354 (4)	C15—C19	1.426 (5)
N6—C14	1.380 (4)	C16—C17	1.379 (5)
N6—H6	0.8600	C16—C21	1.485 (5)
N7—C19	1.145 (5)	C17—C18	1.409 (5)
N8—C20	1.151 (4)	C17—C20	1.427 (5)
N9—C18	1.316 (4)	C21—C26	1.374 (5)
N9—H9A	0.8600	C21—C22	1.386 (5)
N9—H9B	0.8600	C22—C23	1.373 (5)
N10—O5	1.13 (4)	C22—H22	0.9300
N10—O6	1.32 (4)	C23—C24	1.357 (6)
N10—C23	1.70 (4)	C24—C25	1.373 (6)
N10'—O5'	1.183 (13)	C24—H24	0.9300
N10'—O6'	1.186 (11)	C25—C26	1.386 (5)
N10'—C23	1.433 (13)	C25—H25	0.9300
O1—C1	1.242 (4)	C26—H26	0.9300
O4—C14	1.245 (4)	C27—C28	1.460 (6)
O7—C27	1.410 (5)	C27—H27A	0.9700

O7—H7	0.8200	C27—H27B	0.9700
O8—C29	1.427 (7)	C28—H28A	0.9600
O8—H8	0.8200	C28—H28B	0.9600
C1—C2	1.433 (5)	C28—H28C	0.9600
C2—C3	1.374 (5)	C29—C30	1.523 (8)
C2—C6	1.420 (5)	C29—H29A	0.9700
C3—C4	1.396 (5)	C29—H29B	0.9700
C3—C8	1.480 (5)	C30—H30A	0.9600
C4—C5	1.406 (5)	C30—H30B	0.9600
C4—C7	1.425 (5)	C30—H30C	0.9600
C8—C9	1.370 (5)		
C5—N1—C1	124.4 (4)	N6—C14—C15	115.3 (4)
C5—N1—H1	117.8	C16—C15—C14	121.6 (4)
C1—N1—H1	117.8	C16—C15—C19	121.3 (4)
C5—N4—H4A	120.0	C14—C15—C19	117.1 (4)
C5—N4—H4B	120.0	C17—C16—C15	119.7 (4)
H4A—N4—H4B	120.0	C17—C16—C21	118.9 (4)
O3—N5—O2	123.7 (6)	C15—C16—C21	121.4 (4)
O3—N5—C10	118.4 (6)	C16—C17—C18	120.5 (4)
O2—N5—C10	117.8 (6)	C16—C17—C20	121.6 (4)
C18—N6—C14	125.3 (4)	C18—C17—C20	117.7 (4)
C18—N6—H6	117.3	N9—C18—N6	118.7 (4)
C14—N6—H6	117.3	N9—C18—C17	123.7 (4)
C18—N9—H9A	120.0	N6—C18—C17	117.5 (4)
C18—N9—H9B	120.0	N7—C19—C15	179.7 (6)
H9A—N9—H9B	120.0	N8—C20—C17	177.8 (5)
O5—N10—O6	127 (3)	C26—C21—C22	118.8 (4)
O5—N10—C23	128 (3)	C26—C21—C16	120.8 (4)
O6—N10—C23	106 (3)	C22—C21—C16	120.3 (4)
O5'—N10'—O6'	121.0 (13)	C23—C22—C21	117.6 (4)
O5'—N10'—C23	117.8 (11)	C23—C22—H22	121.2
O6'—N10'—C23	121.1 (12)	C21—C22—H22	121.2
C27—O7—H7	109.5	C24—C23—C22	123.9 (5)
C29—O8—H8	109.5	C24—C23—N10'	114.5 (8)
O1—C1—N1	119.1 (4)	C22—C23—N10'	121.0 (7)
O1—C1—C2	124.3 (4)	C24—C23—N10	128.3 (15)
N1—C1—C2	116.6 (4)	C22—C23—N10	105.7 (14)
C3—C2—C6	122.4 (4)	C23—C24—C25	118.8 (5)
C3—C2—C1	120.3 (4)	C23—C24—H24	120.6
C6—C2—C1	117.3 (4)	C25—C24—H24	120.6
C2—C3—C4	120.3 (4)	C24—C25—C26	118.4 (5)
C2—C3—C8	120.8 (4)	C24—C25—H25	120.8
C4—C3—C8	118.9 (4)	C26—C25—H25	120.8
C3—C4—C5	120.1 (4)	C21—C26—C25	122.4 (5)
C3—C4—C7	122.9 (4)	C21—C26—H26	118.8
C5—C4—C7	117.0 (4)	C25—C26—H26	118.8
N4—C5—N1	117.3 (4)	O7—C27—C28	111.0 (5)
N4—C5—C4	124.5 (4)	O7—C27—H27A	109.4
N1—C5—C4	118.2 (4)	C28—C27—H27A	109.4

N2—C6—C2	179.0 (6)	O7—C27—H27B	109.4
N3—C7—C4	179.3 (5)	C28—C27—H27B	109.4
C9—C8—C13	118.5 (4)	H27A—C27—H27B	108.0
C9—C8—C3	119.5 (4)	C27—C28—H28A	109.5
C13—C8—C3	122.0 (4)	C27—C28—H28B	109.5
C10—C9—C8	119.5 (4)	H28A—C28—H28B	109.5
C10—C9—H9	120.2	C27—C28—H28C	109.5
C8—C9—H9	120.2	H28A—C28—H28C	109.5
C11—C10—C9	122.8 (5)	H28B—C28—H28C	109.5
C11—C10—N5	118.5 (5)	O8—C29—C30	102.3 (6)
C9—C10—N5	118.7 (5)	O8—C29—H29A	111.3
C10—C11—C12	118.5 (5)	C30—C29—H29A	111.3
C10—C11—H11	120.7	O8—C29—H29B	111.3
C12—C11—H11	120.7	C30—C29—H29B	111.3
C11—C12—C13	120.0 (5)	H29A—C29—H29B	109.2
C11—C12—H12	120.0	C29—C30—H30A	109.5
C13—C12—H12	120.0	C29—C30—H30B	109.5
C12—C13—C8	120.7 (5)	H30A—C30—H30B	109.5
C12—C13—H13	119.7	C29—C30—H30C	109.5
C8—C13—H13	119.7	H30A—C30—H30C	109.5
O4—C14—N6	118.8 (4)	H30B—C30—H30C	109.5
O4—C14—C15	125.9 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N9—H9A···O8	0.86	1.94	2.785 (5)	167
C22—H22···O5	0.93	2.42	2.802 (19)	104
N1—H1···O4^{i}	0.86	1.98	2.826 (4)	170
N4—H4A···O7^{ii}	0.86	1.97	2.816 (4)	169
N4—H4B···N8^{ii}	0.86	2.17	3.009 (5)	164
C13—H13···O3^{ii}	0.93	2.32	3.228 (6)	167
C25—H25···O3^{ii}	0.93	2.59	3.464 (7)	158
N6—H6···O1^{iii}	0.86	1.98	2.830 (4)	171
O8—H8···O1^{iii}	0.82	2.19	2.824 (4)	135
N9—H9B···N3^{iv}	0.86	2.22	3.047 (5)	163
O7—H7···O4^{v}	0.82	2.01	2.753 (4)	150

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

Fig. 1

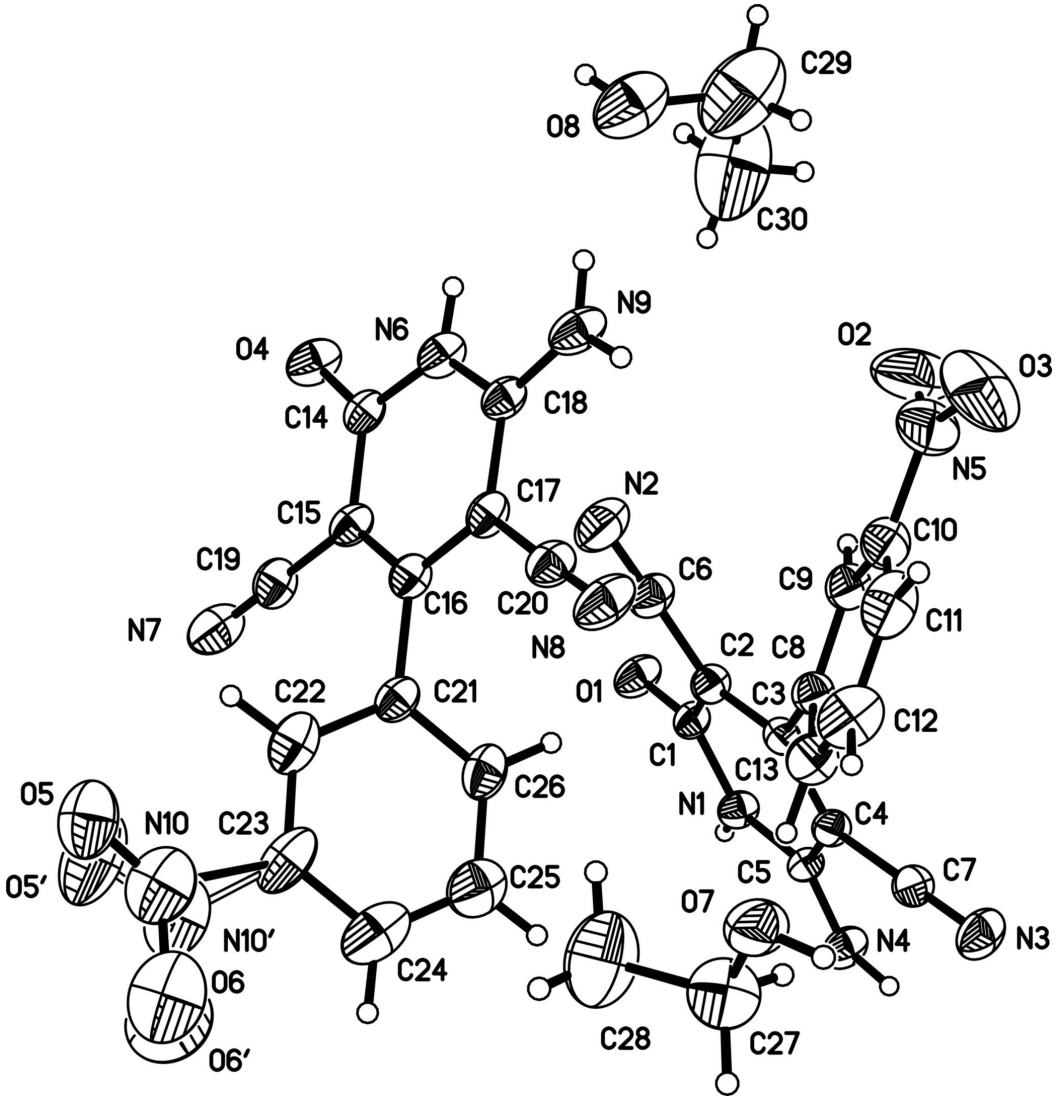
C29
O8
C30
N9
N6
O4
C14
C18
O2
O3
N5
C17
N2
C15
C10
C19
C6
C9
C16
C20
C11
N7
N8
C2
C8
C22
C21
O1
C3
C12
C1
C13
C26
O5
N10
C23
N1
C4
C5
C7
O5′
C25
O7
N10′
N4
N3
C24
O6
C28
C27
O6′

Fig. 2