Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,5-Bis(4-methylphenyl)-7-phenyl-6,7-dihydropyrazolo[1,5-*a*]pyrimidine-3,6-dicarbaldehyde: hydrogen-bonded sheets built from N—H···O and C—H···O hydrogen bonds

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.047
wR factor = 0.133
Data-to-parameter ratio = 17.3

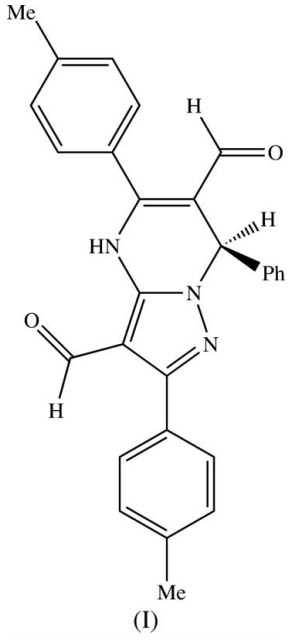
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 29 September 2006
Accepted 29 September 2006

The molecules of the title compound, $C_{28}H_{23}N_3O_2$, are linked into complex sheets by a combination of one N—H···O and two C—H···O hydrogen bonds.

Comment

As part of a continuing search for biologically active molecules containing fused pyrazole systems, we have now prepared 2,5-bis(4-methylphenyl)-7-phenyl-6,7-dihydropyrazolo[1,5-*a*]pyrimidine-3,6-dicarbaldehyde, (I), of potential value as a precursor for modified or fused polycyclic pyrazolo[1,5-*a*]pyrimidine systems, and we report its structure here.



(I)

Within the molecule of (I), atom C7 is a stereogenic centre, and in the selected reference molecule this atom has the (*R*)-configuration; however, the space group accommodates equal numbers of (*R*) and (S) enantiomers. For the atom sequence C3*A*—N4—C5—C6—C7—N7*A*, the ring-puckering parameters (Cremer & Pople, 1975) are θ = 70.2 (5)° and φ = 169.4 (5)°, indicating a ring conformation intermediate between boat [θ = 90.0°, φ = (60*k*)°, where *k* is zero or an integer)] and screw-boat [θ = 67.5° and φ = (60*k* + 30)°]. While the aryl rings are all significantly twisted away from the mean plane of the heterocyclic core, as shown by the key torsion angles (Table 1), the two carboxaldehyde groups are nearly coplanar with the heterocycle. The short intramolecular N—H···O contact to O3 (Table 2) may contribute to this.

The molecules of (I) are linked into complex sheets by one N—H···O hydrogen bond and two C—H···O hydrogen bonds (Table 2), and the sheet formation is easily analysed in terms of two one-dimensional sub-structures. Atoms N4 and C73 in the molecule at (x, y, z) act as hydrogen-bond donors, respectively, to atoms O3 and O6 in the molecules at ($2-x$, $1-y$, $1-z$) and ($-x$, $1-y$, $-z$), respectively. Propagation by inversion of these two interactions then generates a chain of centrosymmetric rings running parallel to the [201] direction with $R_2^2(16)$ (Bernstein *et al.*, 1995) rings centred at ($2n$, $\frac{1}{2}$, n) (n = zero or an integer), and $R_2^2(12)$ rings centred at ($2n+1$, $\frac{1}{2}$, $n+\frac{1}{2}$) (n = zero or an integer) (Fig. 2). In addition, atom C74 in the molecule at (x, y, z) acts as a hydrogen-bond donor to atom O3 in the molecule ($-1+x$, y, $-1+z$), so generating by translation a $C(11)$ chain running parallel to the [101] direction (Fig. 3). The combination of the [101] and [201] chains generates a sheet parallel to (010) (Fig. 4).

Experimental

A mixture of 5-amino-3-(4-methylphenyl-1*H*-pyrazole (1.9 mmol) and 1-(4-methylphenyl)-3-phenylpropenone (1.9 mmol) in dimethylformamide (1 ml) was heated under reflux for 20 min, to afford the intermediate 2,5-di-(4-methylphenyl)-7-phenyl-6,7-dihydropyrazolo[1,5-*a*]pyrimidine. The reaction mixture was cooled to ambient temperature and the intermediate was collected by filtration, washed with ethanol and dried, and then purified by chromatography on alumina using chloroform as the eluent. Phosphoryl chloride (2.1 mmol) was then added dropwise to a suspension of the pyrazolopyrimidine intermediate (1.0 mmol) in dimethylformamide (2 ml) while cooling within an ice–water bath. When the addition had been completed the reaction mixture was stirred vigorously for 0.5 h at ambient temperature. The resulting solid product (I) was collected by filtration, dried and recrystallized from dimethylformamide to give yellow crystals suitable for single-crystal X-ray diffraction: yield 65%, m.p. 544 K; MS (m/z, %): 433 (92, M^+), 356 (100), 91 (44), 65 (25).

Crystal data

$C_{28}H_{23}N_3O_2$
M_r = 433.49
Monoclinic, $P2_1/c$
a = 10.4688 (3) Å
b = 22.3018 (7) Å
c = 11.0754 (4) Å
β = 118.723 (2)°
V = 2267.63 (13) Å^3
Z = 4
D_x = 1.270 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 120 (2) K
Lath, yellow
0.44 × 0.40 × 0.19 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.968, T_{max} = 0.986
24100 measured reflections
5194 independent reflections
3286 reflections with $I > 2\sigma(I)$
R_{int} = 0.049
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.133
S = 0.93
5194 reflections
300 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0711P)^2 + 0.5212P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.032
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

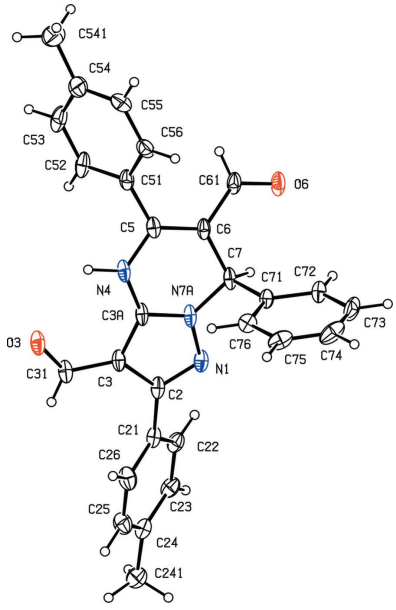


Figure 1
The molecular structure of the (R) enantiomer of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

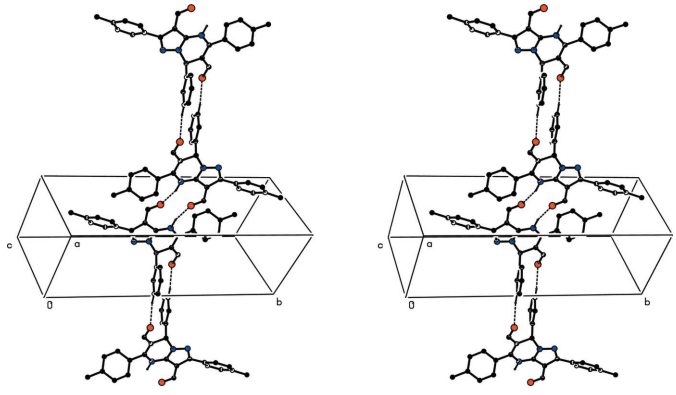


Figure 2
A stereoscopic view of part of the crystal structure of (I) showing the formation of a hydrogen-bonded chain of rings along [201]. Hydrogen bonds are shown as dashed lines, and for the sake of clarity the H atoms not involved in the motif shown have been omitted.

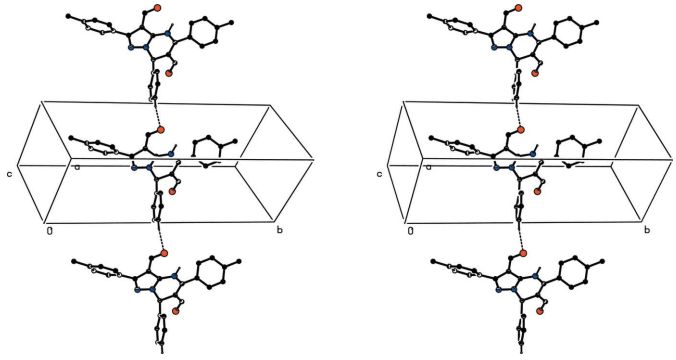


Figure 3
A stereoscopic view of part of the crystal structure of (I) showing the formation of a hydrogen-bonded chain along [101]. Hydrogen bonds are shown as dashed lines, and for the sake of clarity the H atoms not involved in the motif shown have been omitted.

Table 1
Selected torsion angles (°).

N1—C2—C21—C22	−39.4 (2)	C3*A*—C3—C31—O3	−8.3 (3)
N4—C5—C51—C52	−54.56 (19)	C5—C6—C61—O6	−176.16 (15)
N7*A*—C7—C71—C72	137.35 (15)	C7—C6—C61—O6	−0.3 (2)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4···O3	0.98	2.36	2.933 (2)	117
N4—H4···O3^{i}	0.98	2.02	2.968 (2)	163
C73—H73···O6^{ii}	0.95	2.37	3.294 (3)	165
C74—H74···O3^{iii}	0.95	2.44	3.376 (2)	170

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

All H atoms were located in difference maps and then treated as riding atoms with distances C—H 0.95 Å (aromatic and aldehydic), 0.98 Å (methyl) or 1.00 Å (aliphatic CH), and N—H 0.98 Å, and with $U_{iso}(H) = kU_{eq}(C,N)$, where k = 1.5 for the methyl groups and 1.2 for all other H atoms.

Data collection: *COLLECT* (Hooft, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

X-ray data were collected at the EPSRC X-ray Crystallographic Service, University of Southampton, England. JC thanks the Consejería de Innovación, Ciencia y Empresa (Junta de Andalucía, Spain) and the Universidad de Jaén for financial support. JT thanks COLCIENCIAS and UNIVALLE (Universidad del Valle, Colombia) for financial support.

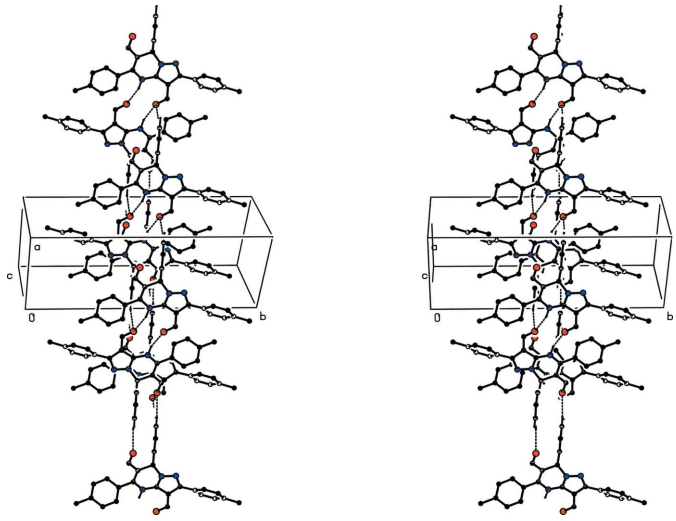


Figure 4
A stereoscopic view of part of the crystal structure of (I) showing the formation of a hydrogen-bonded sheet parallel to (010). Hydrogen bonds are shown as dashed lines, and for the sake of clarity the H atoms not involved in the motif shown have been omitted.

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supporting information

Acta Cryst. (2006). E**62**, o4930–o4932 [https://doi.org/10.1107/S1600536806040177]

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$c = 11.0754$ (4) Å
$\beta = 118.723$ (2)°
$V = 2267.63$ (13) Å^3
$Z = 4$
$F(000) = 912$
$D_x = 1.270$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5194 reflections
$\theta = 2.4$–27.5°
$\mu = 0.08$ mm^{-1}
$T = 120$ K
Lath, yellow
0.44 × 0.40 × 0.19 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Radiation source: Bruker-Nonius FR591 rotating anode
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
$T_{min} = 0.968$, $T_{max} = 0.986$
24100 measured reflections
5194 independent reflections
3286 reflections with $I > 2\sigma(I)$
$R_{int} = 0.049$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.4°$
$h = -12\rightarrow13$
$k = -28\rightarrow28$
$l = -14\rightarrow14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.133$
$S = 0.93$
5194 reflections
300 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0711P)^2 + 0.5212P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.032$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.53507 (14)	0.36610 (7)	0.37300 (13)	0.0362 (3)
C2	0.66984 (16)	0.34501 (8)	0.42271 (15)	0.0338 (4)
C21	0.69963 (16)	0.28096 (8)	0.45871 (16)	0.0346 (4)
C22	0.62975 (18)	0.25175 (8)	0.52193 (17)	0.0383 (4)
C23	0.66112 (19)	0.19257 (8)	0.56254 (18)	0.0406 (4)
C24	0.76215 (18)	0.16014 (8)	0.54152 (17)	0.0391 (4)
C241	0.7985 (2)	0.09603 (9)	0.5878 (2)	0.0509 (5)
C25	0.8283 (2)	0.18923 (9)	0.4751 (2)	0.0473 (5)
C26	0.79841 (19)	0.24850 (9)	0.43499 (19)	0.0454 (5)
C3	0.77137 (16)	0.39198 (7)	0.44303 (15)	0.0330 (4)
C31	0.92724 (17)	0.39201 (8)	0.50462 (17)	0.0385 (4)
O3	0.99610 (12)	0.43759 (6)	0.51326 (13)	0.0459 (3)
C3A	0.68660 (16)	0.44389 (8)	0.40379 (15)	0.0330 (4)
N4	0.72056 (13)	0.50336 (6)	0.40806 (13)	0.0345 (3)
C5	0.61577 (16)	0.54631 (8)	0.38226 (15)	0.0318 (4)
C51	0.67015 (17)	0.60881 (8)	0.41815 (16)	0.0345 (4)
C52	0.77948 (18)	0.62163 (9)	0.55105 (18)	0.0460 (5)
C53	0.8253 (2)	0.67996 (11)	0.5900 (2)	0.0556 (6)
C54	0.7645 (2)	0.72720 (10)	0.4981 (2)	0.0552 (5)
C541	0.8098 (3)	0.79111 (11)	0.5435 (3)	0.0853 (9)
C55	0.6590 (2)	0.71403 (9)	0.3644 (2)	0.0534 (5)
C56	0.61142 (18)	0.65559 (8)	0.32453 (18)	0.0413 (4)
C6	0.47288 (16)	0.53011 (8)	0.33359 (15)	0.0326 (4)
C61	0.36371 (17)	0.57250 (9)	0.32273 (17)	0.0441 (5)
O6	0.23758 (12)	0.55809 (7)	0.28624 (14)	0.0579 (4)
C7	0.42139 (15)	0.46566 (8)	0.29714 (15)	0.0334 (4)
C71	0.33917 (16)	0.45440 (7)	0.14263 (15)	0.0306 (4)
C72	0.18835 (18)	0.45125 (8)	0.07359 (18)	0.0414 (4)
C73	0.1146 (2)	0.44338 (9)	−0.0689 (2)	0.0547 (6)
C74	0.1921 (3)	0.43808 (9)	−0.13992 (19)	0.0584 (6)
C75	0.3409 (2)	0.44043 (8)	−0.07132 (19)	0.0480 (5)
C76	0.41463 (19)	0.44856 (7)	0.06949 (17)	0.0362 (4)
N7A	0.54869 (13)	0.42700 (6)	0.36316 (13)	0.0347 (3)
H22	0.5599	0.2727	0.5373	0.046*
H23	0.6125	0.1736	0.6058	0.049*
H24A	0.7203	0.0698	0.5233	0.076*
H24B	0.8086	0.0916	0.6800	0.076*
H24C	0.8903	0.0849	0.5901	0.076*
H25	0.8957	0.1679	0.4569	0.057*
H26	0.8461	0.2672	0.3906	0.054*
H31	0.9786	0.3555	0.5398	0.046*
H4	0.8219	0.5152	0.4423	0.041*
H52	0.8230	0.5900	0.6155	0.055*
H53	0.8996	0.6879	0.6813	0.067*
H54A	0.7436	0.8089	0.5729	0.128*

H54B	0.8058	0.8143	0.4666	0.128*
H54C	0.9095	0.7916	0.6205	0.128*
H55	0.6184	0.7455	0.2991	0.064*
H56	0.5384	0.6476	0.2328	0.050*
H61	0.3909	0.6133	0.3448	0.053*
H7	0.3551	0.4562	0.3361	0.040*
H72	0.1355	0.4544	0.1230	0.050*
H73	0.0112	0.4417	−0.1170	0.066*
H74	0.1417	0.4328	−0.2370	0.070*
H75	0.3937	0.4364	−0.1207	0.058*
H76	0.5180	0.4502	0.1166	0.043*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0254 (7)	0.0515 (9)	0.0286 (7)	−0.0079 (6)	0.0104 (6)	0.0061 (6)
C2	0.0226 (8)	0.0536 (11)	0.0231 (8)	−0.0070 (7)	0.0095 (6)	0.0039 (7)
C21	0.0254 (8)	0.0486 (10)	0.0264 (8)	−0.0096 (7)	0.0097 (7)	0.0006 (7)
C22	0.0364 (9)	0.0457 (10)	0.0365 (9)	−0.0123 (8)	0.0205 (8)	−0.0069 (8)
C23	0.0459 (10)	0.0435 (11)	0.0392 (10)	−0.0158 (8)	0.0259 (8)	−0.0057 (8)
C24	0.0339 (9)	0.0446 (10)	0.0343 (9)	−0.0107 (8)	0.0129 (7)	−0.0041 (8)
C241	0.0515 (12)	0.0476 (11)	0.0540 (12)	−0.0085 (9)	0.0257 (10)	−0.0029 (9)
C25	0.0376 (10)	0.0554 (12)	0.0552 (12)	0.0003 (9)	0.0274 (9)	0.0041 (9)
C26	0.0347 (9)	0.0610 (12)	0.0476 (11)	−0.0022 (9)	0.0256 (8)	0.0104 (9)
C3	0.0212 (8)	0.0494 (10)	0.0240 (8)	−0.0034 (7)	0.0074 (6)	0.0095 (7)
C31	0.0231 (8)	0.0522 (11)	0.0346 (9)	−0.0027 (8)	0.0093 (7)	0.0134 (8)
O3	0.0226 (6)	0.0565 (8)	0.0512 (8)	−0.0064 (6)	0.0119 (5)	0.0154 (6)
C3A	0.0181 (7)	0.0530 (11)	0.0243 (8)	−0.0051 (7)	0.0075 (6)	0.0112 (7)
N4	0.0177 (6)	0.0493 (9)	0.0328 (7)	−0.0020 (6)	0.0091 (5)	0.0146 (6)
C5	0.0226 (8)	0.0540 (10)	0.0188 (7)	−0.0005 (7)	0.0099 (6)	0.0049 (7)
C51	0.0230 (8)	0.0539 (11)	0.0300 (8)	−0.0039 (7)	0.0154 (7)	−0.0027 (7)
C52	0.0281 (9)	0.0731 (13)	0.0355 (9)	−0.0181 (9)	0.0141 (7)	−0.0008 (9)
C53	0.0359 (10)	0.0846 (16)	0.0494 (12)	−0.0257 (10)	0.0229 (9)	−0.0189 (11)
C54	0.0372 (11)	0.0628 (13)	0.0767 (15)	−0.0076 (10)	0.0363 (11)	−0.0195 (12)
C541	0.0608 (15)	0.0679 (16)	0.132 (2)	−0.0137 (12)	0.0498 (16)	−0.0378 (16)
C55	0.0447 (11)	0.0497 (12)	0.0695 (14)	0.0128 (9)	0.0303 (11)	−0.0033 (10)
C56	0.0333 (9)	0.0503 (11)	0.0401 (10)	0.0098 (8)	0.0175 (8)	−0.0033 (8)
C6	0.0199 (7)	0.0580 (11)	0.0186 (7)	−0.0039 (7)	0.0082 (6)	−0.0038 (7)
C61	0.0236 (9)	0.0764 (13)	0.0317 (9)	−0.0066 (8)	0.0128 (7)	−0.0250 (9)
O6	0.0224 (6)	0.0986 (11)	0.0515 (8)	−0.0084 (6)	0.0168 (6)	−0.0384 (7)
C7	0.0172 (7)	0.0595 (11)	0.0224 (8)	−0.0025 (7)	0.0086 (6)	0.0049 (7)
C71	0.0240 (8)	0.0404 (9)	0.0228 (7)	−0.0031 (7)	0.0075 (6)	0.0030 (6)
C72	0.0261 (8)	0.0526 (11)	0.0350 (9)	−0.0050 (8)	0.0064 (7)	0.0002 (8)
C73	0.0407 (11)	0.0534 (12)	0.0376 (10)	−0.0118 (9)	−0.0073 (8)	0.0006 (9)
C74	0.0884 (17)	0.0463 (12)	0.0254 (9)	−0.0212 (11)	0.0153 (10)	−0.0033 (8)
C75	0.0760 (15)	0.0396 (10)	0.0345 (10)	−0.0096 (9)	0.0314 (10)	−0.0045 (8)
C76	0.0402 (10)	0.0397 (10)	0.0323 (9)	−0.0019 (8)	0.0202 (8)	0.0023 (7)
N7A	0.0175 (7)	0.0522 (9)	0.0296 (7)	−0.0044 (6)	0.0075 (5)	0.0107 (6)

Geometric parameters (Å, º)

N1—C2	1.330 (2)	C52—C53	1.382 (3)
N1—N7A	1.375 (2)	C52—H52	0.95
C2—C3	1.431 (2)	C53—C54	1.389 (3)
C2—C21	1.476 (2)	C53—H53	0.95
C21—C26	1.387 (2)	C54—C55	1.388 (3)
C21—C22	1.394 (2)	C54—C541	1.510 (3)
C22—C23	1.382 (2)	C541—H54A	0.98
C22—H22	0.95	C541—H54B	0.98
C23—C24	1.390 (2)	C541—H54C	0.98
C23—H23	0.95	C55—C56	1.389 (3)
C24—C25	1.389 (2)	C55—H55	0.95
C24—C241	1.505 (3)	C56—H56	0.95
C241—H24A	0.98	C6—C61	1.443 (2)
C241—H24B	0.98	C6—C7	1.520 (2)
C241—H24C	0.98	C61—O6	1.2236 (19)
C25—C26	1.382 (3)	C61—H61	0.95
C25—H25	0.95	C7—N7A	1.455 (2)
C26—H26	0.95	C7—C71	1.522 (2)
C3—C3A	1.395 (2)	C7—H7	1.00
C3—C31	1.435 (2)	C71—C76	1.383 (2)
C31—O3	1.223 (2)	C71—C72	1.386 (2)
C31—H31	0.95	C72—C73	1.395 (3)
C3A—N7A	1.3434 (19)	C72—H72	0.95
C3A—N4	1.368 (2)	C73—C74	1.379 (3)
N4—C5	1.379 (2)	C73—H73	0.95
N4—H4	0.98	C74—C75	1.368 (3)
C5—C6	1.372 (2)	C74—H74	0.95
C5—C51	1.486 (2)	C75—C76	1.380 (2)
C51—C56	1.389 (2)	C75—H75	0.95
C51—C52	1.393 (2)	C76—H76	0.95
C2—N1—N7A	104.62 (12)	C52—C53—H53	119.4
N1—C2—C3	111.49 (15)	C54—C53—H53	119.4
N1—C2—C21	119.92 (14)	C55—C54—C53	118.08 (19)
C3—C2—C21	128.43 (14)	C55—C54—C541	121.1 (2)
C26—C21—C22	118.06 (16)	C53—C54—C541	120.8 (2)
C26—C21—C2	122.29 (15)	C54—C541—H54A	109.5
C22—C21—C2	119.63 (15)	C54—C541—H54B	109.5
C23—C22—C21	120.65 (16)	H54A—C541—H54B	109.5
C23—C22—H22	119.7	C54—C541—H54C	109.5
C21—C22—H22	119.7	H54A—C541—H54C	109.5
C22—C23—C24	121.55 (16)	H54B—C541—H54C	109.5
C22—C23—H23	119.2	C54—C55—C56	121.2 (2)
C24—C23—H23	119.2	C54—C55—H55	119.4
C25—C24—C23	117.29 (16)	C56—C55—H55	119.4
C25—C24—C241	120.99 (17)	C51—C56—C55	120.31 (17)

C23—C24—C241	121.71 (16)	C51—C56—H56	119.8
C24—C241—H24A	109.5	C55—C56—H56	119.8
C24—C241—H24B	109.5	C5—C6—C61	122.03 (16)
H24A—C241—H24B	109.5	C5—C6—C7	122.25 (15)
C24—C241—H24C	109.5	C61—C6—C7	115.58 (14)
H24A—C241—H24C	109.5	O6—C61—C6	122.77 (18)
H24B—C241—H24C	109.5	O6—C61—H61	118.6
C26—C25—C24	121.58 (17)	C6—C61—H61	118.6
C26—C25—H25	119.2	N7A—C7—C6	107.74 (12)
C24—C25—H25	119.2	N7A—C7—C71	110.72 (13)
C25—C26—C21	120.83 (16)	C6—C7—C71	113.04 (13)
C25—C26—H26	119.6	N7A—C7—H7	108.4
C21—C26—H26	119.6	C6—C7—H7	108.4
C3A—C3—C2	104.20 (13)	C71—C7—H7	108.4
C3A—C3—C31	123.76 (15)	C76—C71—C72	119.46 (15)
C2—C3—C31	131.63 (15)	C76—C71—C7	120.04 (14)
O3—C31—C3	122.04 (16)	C72—C71—C7	120.49 (14)
O3—C31—H31	119.0	C71—C72—C73	119.64 (18)
C3—C31—H31	119.0	C71—C72—H72	120.2
N7A—C3A—N4	120.20 (15)	C73—C72—H72	120.2
N7A—C3A—C3	106.98 (14)	C74—C73—C72	119.91 (18)
N4—C3A—C3	132.68 (14)	C74—C73—H73	120.0
C3A—N4—C5	119.91 (13)	C72—C73—H73	120.0
C3A—N4—H4	119.5	C75—C74—C73	120.34 (17)
C5—N4—H4	120.0	C75—C74—H74	119.8
C6—C5—N4	120.46 (15)	C73—C74—H74	119.8
C6—C5—C51	123.86 (15)	C74—C75—C76	120.11 (18)
N4—C5—C51	115.58 (13)	C74—C75—H75	119.9
C56—C51—C52	118.62 (17)	C76—C75—H75	119.9
C56—C51—C5	121.99 (15)	C75—C76—C71	120.53 (17)
C52—C51—C5	119.34 (16)	C75—C76—H76	119.7
C53—C52—C51	120.59 (19)	C71—C76—H76	119.7
C53—C52—H52	119.7	C3A—N7A—N1	112.71 (13)
C51—C52—H52	119.7	C3A—N7A—C7	125.42 (14)
C52—C53—C54	121.15 (18)	N1—N7A—C7	121.34 (12)

N7A—N1—C2—C3	−1.08 (17)	C52—C53—C54—C55	−1.7 (3)
N7A—N1—C2—C21	174.63 (13)	C52—C53—C54—C541	176.89 (18)
N1—C2—C21—C26	142.21 (16)	C53—C54—C55—C56	2.1 (3)
C3—C2—C21—C26	−42.9 (2)	C541—C54—C55—C56	−176.41 (18)
N1—C2—C21—C22	−39.4 (2)	C52—C51—C56—C55	−1.5 (2)
N4—C5—C51—C52	−54.56 (19)	C5—C51—C56—C55	176.00 (15)
N7A—C7—C71—C72	137.35 (15)	C54—C55—C56—C51	−0.6 (3)
C3—C2—C21—C22	135.47 (17)	N4—C5—C6—C61	171.58 (14)
C26—C21—C22—C23	1.5 (2)	C51—C5—C6—C61	−4.8 (2)
C2—C21—C22—C23	−176.90 (15)	N4—C5—C6—C7	−4.0 (2)
C21—C22—C23—C24	−0.3 (3)	C51—C5—C6—C7	179.59 (14)
C22—C23—C24—C25	−1.4 (3)	C5—C6—C61—O6	−176.16 (15)

C22—C23—C24—C241	178.92 (16)	C7—C6—C61—O6	−0.3 (2)
C23—C24—C25—C26	1.8 (3)	C5—C6—C7—N7A	18.00 (19)
C241—C24—C25—C26	−178.48 (17)	C61—C6—C7—N7A	−157.89 (13)
C24—C25—C26—C21	−0.6 (3)	C5—C6—C7—C71	−104.66 (16)
C22—C21—C26—C25	−1.1 (3)	C61—C6—C7—C71	79.45 (17)
C2—C21—C26—C25	177.28 (16)	N7A—C7—C71—C76	−44.1 (2)
N1—C2—C3—C3A	1.18 (17)	C6—C7—C71—C76	76.88 (19)
C21—C2—C3—C3A	−174.08 (15)	C6—C7—C71—C72	−101.67 (17)
N1—C2—C3—C31	173.76 (17)	C76—C71—C72—C73	−1.2 (3)
C21—C2—C3—C31	−1.5 (3)	C7—C71—C72—C73	177.35 (16)
C3A—C3—C31—O3	−8.3 (3)	C71—C72—C73—C74	0.8 (3)
C2—C3—C31—O3	−179.62 (17)	C72—C73—C74—C75	0.1 (3)
C2—C3—C3A—N7A	−0.75 (17)	C73—C74—C75—C76	−0.5 (3)
C31—C3—C3A—N7A	−174.09 (15)	C74—C75—C76—C71	0.0 (3)
C2—C3—C3A—N4	174.95 (16)	C72—C71—C76—C75	0.8 (2)
C31—C3—C3A—N4	1.6 (3)	C7—C71—C76—C75	−177.74 (16)
N7A—C3A—N4—C5	5.7 (2)	N4—C3A—N7A—N1	−176.21 (13)
C3—C3A—N4—C5	−169.56 (16)	C3—C3A—N7A—N1	0.14 (18)
C3A—N4—C5—C6	−9.2 (2)	N4—C3A—N7A—C7	12.1 (2)
C3A—N4—C5—C51	167.49 (13)	C3—C3A—N7A—C7	−171.52 (14)
C6—C5—C51—C56	−55.5 (2)	C2—N1—N7A—C3A	0.59 (17)
N4—C5—C51—C56	127.95 (16)	C2—N1—N7A—C7	172.63 (13)
C6—C5—C51—C52	121.96 (18)	C6—C7—N7A—C3A	−22.4 (2)
C56—C51—C52—C53	2.0 (3)	C71—C7—N7A—C3A	101.65 (18)
C5—C51—C52—C53	−175.60 (15)	C6—C7—N7A—N1	166.60 (13)
C51—C52—C53—C54	−0.4 (3)	C71—C7—N7A—N1	−69.33 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4···O3	0.98	2.36	2.933 (2)	117
N4—H4···O3^{i}	0.98	2.02	2.968 (2)	163
C73—H73···O6^{ii}	0.95	2.37	3.294 (3)	165
C74—H74···O3^{iii}	0.95	2.44	3.376 (2)	170

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