Chemistry of Heterocyclic Compounds **2019**, *55*(10), 968–977

Synthesis and X-ray structural studies of 5-methyl-6-nitro-7-oxo-4,7-dihydro-1,2,4-triazolo-[1,5-*a*]pyrimidine L-arginine and piperidine salts

Pavel A. Slepukhin[1,2], Egor K. Voinkov[1], Eugeny N. Ulomskiy[1,2], Konstantin V. Savateev[1,2], Dmitry S. Kopchuk[1,2], Grigory V. Zyryanov[1,2]*, Victor V. Fedotov[1], Valery N. Charushin[1,2], Oleg N. Chupakhin[1,2], Vladimir L. Rusinov[1,2]

[1] *Ural Federal University named after the first President of Russia B. N. Yeltsin, 19 Mira St., Yekaterinburg 620002, Russia; e-mail: gvzyryanov@gmail.com*

[2] *Postovsky Institute of Organic Synthesis, Ural Branch of the Russian Academy of Sciences, 22 S. Kovalevskoi / 20 Akademicheskaya St., Yekaterinburg 620990, Russia; e-mail: chupakhin@ios.uran.ru*

SUPPLEMENTARY INFORMATION

Supplement 1A. Results of the structural studies of compound 1

Table 1A. Crystal data and structure **1** refinement

Empirical formula	$C_{12}H_{21}N_9O_6$
Molecular mass	387.38
Temperature/K	295(2)
Syngony	monoclinic
Space group	$P2_1$
a/Å	5.3831(4)
b/Å	18.4228(11)
c/Å	8.7708(7)
α/deg	90.00
β/deg	96.447(7)
γ/deg	90.00
Volume/Å^3	864.32(10)
Z	2
ρ_{calc}, mg/mm^3	1.488
μ/mm^{-1}	0.121
F(000)	408.0
Crystal size/mm	0.25 × 0.2 × 0.15
Scanning angles 2Θ, deg	5.18 > 2Θ > 62.08°
Index range	$-6 \le h \le 7$, $-26 \le k \le 12$, $-11 \le l \le 12$
Reflections total	4428
Independent reflections	3046 [R_{int} = 0.0252]
Data/limitations/parameters	3046/17/286
Goodness-of-fit on F^2	1.006
Final indeces *R* [*I*>=2σ (*I*)]	R_1 = 0.0500, wR_2 = 0.1185
Final index *R* [all raflections]	R_1 = 0.0646, wR_2 = 0.1323
$\Delta\rho_{\bar{e}}$ ē Å^{-3}	0.34/–0.20
Flack parameter	–0.3(15)

Table 2A. Fractional atomic coordinates (×10^4) and equivalent isotropic displacement parameters (Å^2×10^3). Ueq is defined as 1/3 of the trace of the orthogonalized UIJ tensor

Atom	***x***	***y***	***z***	**U(eq)**
O1	2379(4)	–2847.2(12)	647(3)	40.8(5)
N6	–3468(4)	–1736.5(15)	–1758(3)	34.0(6)
C9	609(5)	–2552.5(16)	–113(3)	29.3(6)
N3	–1286(5)	–3634.2(15)	–1364(3)	37.3(6)

O5	1352(4)	444.3(14)	–2020(3)	50.0(7)
O6	1211(5)	1394.8(15)	–3545(4)	65.6(9)
C10	227(5)	912.5(17)	–2862(4)	33.3(6)
O3	–3260(5)	–3978.0(15)	–1475(4)	64.4(8)
O2	747(5)	–3936.9(13)	–1403(3)	49.0(6)
N1	246(4)	–1803.1(13)	3(3)	28.2(5)
C5	–1715(5)	–1422.4(16)	–747(3)	29.3(6)
N2	1778(5)	–1340.2(14)	898(3)	35.4(6)
C3	611(6)	–712.7(19)	625(4)	38.7(7)
N9	–1903(6)	–1070.7(18)	–5878(4)	50.5(8)
N5	–3567(4)	705.0(16)	–1576(3)	29.8(5)
O4	2020(4)	–1044.3(15)	–3169(3)	48.3(6)
N4	–1524(5)	–722.7(14)	–344(3)	36.2(6)
C8	–1341(5)	–2860.2(17)	–1158(3)	31.0(6)
N7	–382(6)	–2202(2)	–5477(4)	63.5(10)
C7	–3266(5)	–2445.3(18)	–1955(4)	32.7(7)
C15	–2053(6)	–1771(2)	–6238(4)	41.6(7)
N8	–3691(5)	–2038(2)	–7298(4)	53.5(8)
C14	–3503(7)	–494(2)	–6535(4)	43.8(8)
C12	–3520(6)	188.3(19)	–4082(4)	29.7(6)
C11	–2669(5)	842.3(18)	–3081(3)	30.2(6)
C1	–5102(6)	–2778(2)	–3176(4)	49.8(9)
C13	–2794(7)	209(2)	–5712(4)	37.0(7)
C2	–3350(60)	–180(14)	–4880(30)	48(8)
C4	-3290(70)	611(16)	–4700(20)	49(8)

Table 3A. Anisotropic bias parameters ($Å^2 \times 10^3$). The exponent of the anisotropic bias factor has the form: $-2\pi^2[h^2a^{*2}U_{11}+...+2hka\times b\times U_{12}]$

Atom	**U_{11}**	**U_{22}**	**U_{33}**	**U_{23}**	**U_{13}**	**U_{12}**
O1	40.7(11)	24.4(11)	52.0(12)	1.1(10)	–18.8(10)	3.5(9)
N6	26.2(11)	29.8(13)	43.4(13)	2.8(12)	–7.6(10)	4.3(10)
C9	30.4(13)	22.7(13)	33.3(14)	0.9(12)	–3.1(11)	0.7(11)
N3	38.3(13)	24.3(12)	45.4(14)	1.7(12)	–11.5(11)	–0.5(11)
O5	19.9(9)	58.0(17)	70.3(16)	16.4(14)	–2.4(10)	0.7(10)
O6	43.1(13)	37.6(15)	118(3)	27.5(17)	16.6(14)	–6.0(11)
C10	24.7(12)	28.4(15)	47.0(16)	–7.4(14)	5.8(11)	–2.4(11)
O3	46.6(13)	34.3(14)	106(2)	3.4(15)	–21.0(14)	–13.2(12)
O2	49.6(12)	31.0(13)	65.1(15)	–5.8(12)	0.4(11)	8.5(11)

N1	28(1)	22.0(12)	32.6(11)	0.7(10)	–5.6(9)	–0.7(9)
C5	28.8(12)	23.7(14)	34.6(13)	6.2(13)	–0.2(11)	2.9(11)
N2	40.4(13)	22.3(13)	40.6(13)	–2.8(11)	–7.6(10)	–1.7(11)
C3	45.7(16)	24.4(15)	44.4(16)	–1.8(15)	–1.9(14)	–0.5(13)
N9	46.3(15)	43.9(17)	56.7(17)	–15.1(16)	–14.0(13)	9.8(14)
N5	18.4(10)	27.6(13)	42.1(14)	–4.5(13)	–2.1(9)	1.2(10)
O4	37.2(11)	46.5(15)	57.0(15)	–4.7(13)	–13.1(10)	1.2(11)
N4	38.2(12)	23.7(13)	45.6(14)	2.0(12)	–0.9(11)	3.3(11)
C8	29.8(13)	24.8(14)	36.5(14)	2.8(13)	–5.0(11)	1.0(11)
N7	53.3(17)	56(2)	73(2)	–8.5(19)	–26.9(15)	10.3(16)
C7	27.8(13)	29.9(16)	38.5(15)	3.5(13)	–4.0(11)	–0.8(12)
C15	42.9(16)	37.3(18)	44.0(16)	–4.9(16)	2.6(13)	10.1(14)
N8	42.3(14)	56(2)	58.2(17)	–11.8(18)	–12.4(13)	5.5(15)
C14	52.5(18)	40.6(19)	37.0(15)	–1.5(16)	–1.1(14)	11.9(16)
C12	27.6(14)	25.3(16)	35.9(16)	–3.9(15)	2.2(12)	–3.5(13)
C11	22.6(11)	28.8(15)	38.4(14)	1.7(13)	–0.1(10)	3.4(11)
C1	41.3(16)	40(2)	62(2)	–1.3(18)	–21.7(15)	–4.1(16)
C13	46.4(18)	28.2(17)	36.4(17)	1.2(16)	4.2(15)	2.7(15)
C2	43(11)	55(12)	45(11)	3(9)	8(9)	6(9)
C4	42(11)	46(12)	58(12)	2(9)	3(9)	3(9)

Table 4A. Bond lengths

Atom	**Atom**	Length/**Å**	**Atom**	**Atom**	Length /**Å**
O1	C9	1.226(3)	C3	N4	1.351(4)
N6	C5	1.351(4)	N9	C15	1.329(5)
N6	C7	1.323(4)	N9	C14	1.445(5)
C9	N1	1.400(4)	N5	C11	1.478(4)
C9	C8	1.431(4)	C8	C7	1.408(4)
N3	O3	1.232(3)	N7	C15	1.323(5)
N3	O2	1.232(3)	C7	C1	1.505(4)
N3	C8	1.438(4)	C15	N8	1.303(4)
O5	C10	1.247(4)	C14	C13	1.511(5)
O6	C10	1.225(4)	C14	C2	1.560(17)
C10	C11	1.554(4)	C12	C11	1.530(5)
N1	C5	1.372(3)	C12	C13	1.524(4)
N1	N2	1.370(3)	C11	C4	1.485(19)
C5	N4	1.337(4)	C2	C4	1.467(19)
N2	C3	1.325(4)			

Table 5A. Bond angles

Atom	Atom	Atom	Angle/deg	Atom	Atom	Atom	Angle/deg
C7	N6	C5	116.5(2)	C7	C8	C9	123.3(3)
O1	C9	N1	120.2(3)	C7	C8	N3	120.0(3)
O1	C9	C8	130.0(3)	N6	C7	C8	122.5(3)
N1	C9	C8	109.8(2)	N6	C7	C1	116.0(3)
O3	N3	O2	121.8(3)	C8	C7	C1	121.3(3)
O3	N3	C8	119.4(3)	N7	C15	N9	116.3(3)
O2	N3	C8	118.9(2)	N8	C15	N9	123.6(3)
O5	C10	C11	115.4(3)	N8	C15	N7	120.1(4)
O6	C10	O5	125.7(3)	N9	C14	C13	109.4(3)
O6	C10	C11	118.9(3)	N9	C14	C2	86.2(9)
C5	N1	C9	125.1(2)	C13	C14	C2	41.6(12)
N2	N1	C9	125.1(2)	C13	C12	C11	115.3(3)
N2	N1	C5	109.7(2)	N5	C11	C10	109.1(2)
N6	C5	N1	122.6(3)	N5	C11	C12	105.7(3)
N4	C5	N6	127.8(3)	N5	C11	C4	138.1(14)
N4	C5	N1	109.5(2)	C12	C11	C10	111.6(2)
C3	N2	N1	101.4(2)	C4	C11	C10	105.0(15)
N2	C3	N4	117.0(3)	C4	C11	C12	37.3(12)
C15	N9	C14	126.9(3)	C14	C13	C12	110.5(3)
C5	N4	C3	102.3(2)	C4	C2	C14	117.7(19)
C9	C8	N3	116.7(2)	C2	C4	C11	112.6(19)

Table 6A. Torsion angles

A	B	C	D	Angles/deg
O1	C9	N1	C5	–177.4(3)
O1	C9	N1	N2	1.7(5)
O1	C9	C8	N3	0.7(5)
O1	C9	C8	C7	–180.0(3)
N6	C5	N4	C3	–177.0(3)
C9	N1	C5	N6	-3.6(5)
C9	N1	C5	N4	178.2(3)
C9	N1	N2	C3	–178.9(3)
C9	C8	C7	N6	–2.3(5)
C9	C8	C7	C1	172.8(3)
N3	C8	C7	N6	177.0(3)
N3	C8	C7	C1	–7.9(5)

O5	C10	C11	N5	45.1(4)
O5	C10	C11	C12	–71.3(4)
O5	C10	C11	C4	–110.1(13)
O6	C10	C11	N5	–137.1(3)
O6	C10	C11	C12	106.5(4)
O6	C10	C11	C4	67.7(13)
C10	C11	C4	C2	90(3)
O3	N3	C8	C9	140.2(3)
O3	N3	C8	C7	–39.1(4)
O2	N3	C8	C9	–38.8(4)
O2	N3	C8	C7	141.9(3)
N1	C9	C8	N3	–177.9(3)
N1	C9	C8	C7	1.4(4)
N1	C5	N4	C3	1.1(3)
N1	N2	C3	N4	0.4(4)
C5	N6	C7	C8	0.2(4)
C5	N6	C7	C1	–175.2(3)
C5	N1	N2	C3	0.3(3)
N2	N1	C5	N6	177.2(3)
N2	N1	C5	N4	–1.0(3)
N2	C3	N4	C5	–1.0(4)
N9	C14	C13	C12	–69.9(4)
N9	C14	C2	C4	144(3)
N5	C11	C4	C2	–53(4)
C8	C9	N1	C5	1.3(4)
C8	C9	N1	N2	–179.6(3)
C7	N6	C5	N1	2.6(4)
C7	N6	C5	N4	–179.5(3)
C15	N9	C14	C13	175.8(4)
C15	N9	C14	C2	140.7(14)
C14	N9	C15	N7	–179.8(4)
C14	N9	C15	N8	1.7(6)
C14	C2	C4	C11	–169(2)
C12	C11	C4	C2	–15.7(14)
C11	C12	C13	C14	174.3(3)
C13	C14	C2	C4	19(2)
C13	C12	C11	C10	–61.4(4)
C13	C12	C11	N5	–180.0(3)
C13	C12	C11	C4	25(2)
C2	C14	C13	C12	–10.0(16)

Table 7A. Hydrogen atom coordinates (Å×10^4) and equivalent isotropic displacement parameters (Å^2×10^3)

Atom	*x*	*y*	*z*	U(eq)
H9	–729	–947	–5180	61
H4C	1712	–648	–2730	72
H4D	3422	–1209	–2776	72
H7A	–375	–2658	–5681	76
H7B	698	–2025	–4778	76
H8A	–3687	–2494	–7503	64
H8B	–4777	–1757	–7792	64
H14A	–5235	–610	–6436	53
H14B	–3320	–445	–7618	53
H14C	–5160	–652	–6951	53
H14D	–2745	–189	–7256	53
H12A	–2820	–247	–3583	36
H12B	–5325	152	–4136	36
H1A	–6457	–2992	–2710	75
H1B	–4279	–3146	–3711	75
H1C	–5742	–2409	–3888	75
H13A	–3639	610	–6268	44
H13B	–1007	285	–5681	44
H3	1110(90)	–260(30)	1190(50)	79(15)
H5A	–3290(70)	1040(20)	–960(40)	42(11)
H5B	–5170(70)	630(20)	–1770(40)	40(9)
H5C	–2860(70)	350(20)	–1170(40)	40(10)
H11	–3260(60)	1280(20)	–3450(30)	30(8)
H2A	–4777	–362	–4407	57
H2B	–1862	–376	–4293	57
H4A	–4914	808	–5089	59
H4B	–2066	811	–5315	59

Supplement 1B. Results of the structural studies of compound 2

Table 1B. Crystal data and structure **2** refinement

Empirical formula	$C_{11}H_{16}N_6O_3$
Molecular mass	280.30
Temperature/K	295(2)
Syngony	triclinic
Space group	*P*-1

a/Å	9.0670(4)
b/Å	12.3598(7)
c/Å	13.2585(9)
α/deg	107.251(6)
β/deg	90.631(5)
γ/deg	105.390(5)
Volume/Å^3	1361.61(14)
Z	4
ρ_{calc}, mg/mm^3	1.367
μ/mm^{-1}	0.103
F(000)	592.0
Crystal size/mm	0.25 × 0.2 × 0.15
Scanning angles, 2Θ, deg	5.04 < 2Θ < 61.76°
Index range	–12 ≤ h ≤ 12, –17 ≤ k ≤ 15, –15 ≤ l ≤ 17
Reflections total	12860
Independent reflections	7416[R_{int} = 0.0157]
Data/limitations/parameters	7416/0/387
Goodness-of-fit on F^2	1.009
Final indeces *R* [*I*>=2σ (*I*)]	R_1 = 0.0512, wR_2 = 0.1495
Final infeces *R* [all reflections]	R_1 = 0.0818, wR_2 = 0.1804
$\Delta\rho_{\bar{e}}$ ēÅ^{-3}	0.18/–0.23

Table 2B. Fractional atomic coordinates (×10^4) and equivalent isotropic displacement parameters (Å^2×10^3). Ueq is defined as 1/3 of the trace of the orthogonalized UIJ tensor

Atom	***x***	***y***	***z***	**U(eq)**
O1	2284.0(15)	–705.7(10)	5501.8(9)	56.9(3)
O1A	–2686.7(14)	–802.5(10)	9108.6(9)	57.6(3)
O2	3993.2(19)	–1787.0(13)	4122.7(12)	83.8(5)
O2A	–1026.7(19)	–1972.3(15)	9893.4(13)	85.4(4)
O3	3602(2)	–3594.8(12)	4032.3(13)	93.6(5)
O3A	–1703(2)	–3790.5(14)	8969.4(18)	117.3(7)
N1	2345.0(13)	–1162.1(10)	7030.3(10)	39.9(3)
N1A	–2589.7(13)	–1239.5(10)	7328.4(9)	40.5(3)
N2	1706.5(14)	–302.2(10)	7604.1(10)	44.8(3)
N2A	–3189.1(14)	–361.6(10)	7225.5(10)	44.1(3)
N3	3637.9(16)	–2585.1(12)	4520.1(12)	57.1(4)
N3A	–1475.8(19)	–2751.4(15)	9052.1(16)	67.1(4)
N4	2263.0(15)	–1364.3(11)	8614.2(11)	50.3(3)
N4A	–2614.4(16)	–1409.2(11)	5637.5(11)	52.3(3)
N5	–1871.0(16)	–1576.1(11)	3536.0(11)	48.1(3)

N5A	3058.3(16)	–1525.8(12)	10639.9(12)	48.2(3)
N6	3292.0(15)	–2691.8(11)	7285.8(12)	51.4(3)
N6A	–1634.1(15)	–2761.9(11)	6241.3(12)	54.2(3)
C1	4248(2)	–3968.1(15)	5904.3(19)	69.2(5)
C1A	–716(2)	–4055.6(16)	6943(2)	77.0(6)
C3	1692.1(18)	–482.4(13)	8533.8(13)	47.2(3)
C3A	–3173.9(18)	–519.4(13)	6202.2(12)	47.8(4)
C5	2670.3(16)	–1789.6(12)	7647.3(12)	42.3(3)
C5A	–2239.3(17)	–1855.1(12)	6371.9(12)	43.9(3)
C7	3561.7(17)	–2952.2(12)	6268.5(14)	48.8(4)
C7A	–1416.0(17)	–3049.6(13)	7112.6(15)	52.2(4)
C8	3265.4(16)	–2311.9(12)	5604.8(12)	44.9(3)
C8A	–1763.4(18)	–2432.5(13)	8115.3(14)	49.4(4)
C9	2622.4(16)	–1336.7(12)	5970.5(12)	42.0(3)
C9A	–2353.0(17)	–1435.4(12)	8294.3(12)	43.7(3)
C10	–3044.8(19)	–2436.5(14)	2693.2(14)	54.9(4)
C10A	1998.4(19)	–2432.0(15)	11009.8(15)	56.7(4)
C11	–3130(2)	–3665.8(15)	2677.5(19)	69.3(5)
C11A	2011(2)	–3640.4(15)	10358.7(18)	70.3(5)
C12	–1600(2)	–3916.6(16)	2533.2(19)	76.1(6)
C12A	3604(2)	–3782.3(16)	10362.2(19)	72.7(6)
C13	–408(2)	–3013.7(18)	3374.7(19)	75.1(6)
C13A	4677(2)	–2841.1(19)	10010.6(19)	76.0(6)
C14	–332(2)	–1778.0(16)	3411.9(16)	64.8(5)
C14A	4647(2)	–1624.9(16)	10652.5(17)	63.4(5)

Table 3B. Anisotropic bias parameters ($Å^2 \times 10^3$). The exponent of the anisotropic bias factor has the form: $-2\pi^2[h^2a^{*2}U_{11}+...+2hka\times b\times U_{12}]$

Атом	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	87.1(8)	55.7(6)	41.1(6)	21.7(5)	12.0(6)	34.1(6)
O1A	84.6(8)	60.2(7)	36.9(6)	17.1(5)	9.5(5)	32.6(6)
O2	122.2(13)	89.8(10)	61.7(9)	33.9(8)	40.0(9)	53.4(9)
O2A	107.1(12)	106.5(12)	61.4(10)	36.9(9)	–2.7(8)	49.7(9)
O3	119.8(12)	61.0(8)	77.6(11)	–9.4(7)	25.4(9)	22.6(8)
O3A	166.8(18)	77.6(11)	136.7(17)	68.5(11)	9.0(14)	43.2(11)
N1	48.1(6)	36.7(6)	37.9(6)	13.2(5)	4.5(5)	14.8(5)
N1A	49.8(7)	37.7(6)	35.4(6)	11.1(5)	5.4(5)	14.9(5)
N2	56.5(7)	42.3(6)	38.2(7)	11.6(5)	5.7(5)	19.4(5)
N2A	57.4(7)	41.8(6)	37.2(7)	14.3(5)	4.7(5)	18.0(5)
N3	58.5(8)	54.6(8)	51.0(8)	3.8(7)	8.3(7)	18.2(6)
N3A	71.2(10)	67.7(10)	80.9(12)	42.8(9)	8.3(9)	27.9(8)
N4	59.2(8)	56.1(7)	43.9(8)	24.9(6)	7.7(6)	19.3(6)

N4A	63.8(8)	56.1(8)	37.1(7)	12.6(6)	6.4(6)	18.9(6)
N5	69.7(9)	44.1(7)	35.9(7)	11.5(5)	10.1(6)	25.8(6)
N5A	64.4(8)	44.2(7)	42.0(8)	16.3(6)	4.3(6)	22.0(6)
N6	54.0(7)	46.2(7)	63.4(9)	25.9(6)	8.6(6)	19.8(6)
N6A	54.6(7)	47.4(7)	55.8(9)	4.6(6)	6.4(6)	19.5(6)
C1	72.8(11)	47.8(9)	94.3(15)	21.0(9)	20.9(11)	29.9(8)
C1A	70.4(11)	50.5(10)	109.4(18)	14.4(10)	–0.7(11)	28.4(8)
C3	56.1(9)	50.2(8)	38.3(8)	16.6(6)	6.3(7)	16.8(7)
C3A	57.6(9)	48.0(8)	39.1(8)	15.8(6)	5.3(7)	14.7(7)
C5	44.3(7)	43.5(7)	44.1(8)	21.6(6)	3.5(6)	11.4(6)
C5A	48.1(8)	41.6(7)	38.6(8)	7.7(6)	6.0(6)	12.2(6)
C7	43.6(7)	36.0(7)	65.4(11)	14.5(7)	8.2(7)	10.3(6)
C7A	41.5(8)	40.5(8)	70.5(12)	11.8(7)	–0.9(7)	10.9(6)
C8	45.9(7)	39.2(7)	46.5(9)	9.6(6)	6.6(6)	11.1(6)
C8A	49.1(8)	45.4(8)	57.1(10)	21.7(7)	0.8(7)	12.6(6)
C9	47.9(7)	40.2(7)	37.8(8)	13.1(6)	5.3(6)	11.3(6)
C9A	49.4(8)	42.5(7)	41.9(8)	17.4(6)	3.8(6)	12.6(6)
C10	56.0(9)	61.7(10)	51.9(10)	16.9(7)	1.9(7)	25.4(7)
C10A	52.7(9)	68.8(10)	57.5(10)	29.3(8)	12.6(8)	20.9(8)
C11	66.9(11)	49.9(9)	84.1(14)	13.2(9)	2(1)	13.7(8)
C11A	68.1(11)	50.1(10)	86.6(15)	23.5(9)	–5.2(10)	4.5(8)
C12	93.3(14)	55.6(10)	83.3(15)	9.3(10)	13.2(12)	41.2(10)
C12A	88.1(13)	51.5(10)	85.2(15)	19.0(9)	–0.8(11)	33.5(9)
C13	69.6(12)	91.3(14)	81.0(15)	26.9(11)	7.4(10)	50.2(11)
C13A	66.6(11)	101.1(15)	79.7(15)	34.1(12)	21(1)	48.2(11)
C14	52.6(9)	72.6(12)	60.4(11)	12.8(9)	–0.5(8)	12.6(8)
C14A	52.3(9)	64.2(10)	76.9(13)	32.7(9)	7.6(9)	9.4(8)

Table 4B. Bond lengths

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O1	C9	1.2243(17)	N5	C14	1.484(2)
O1A	C9A	1.2252(18)	N5A	C10A	1.478(2)
O2	N3	1.2238(19)	N5A	C14A	1.478(2)
O2A	N3A	1.217(2)	N6	C5	1.3466(19)
O3	N3	1.2149(18)	N6	C7	1.332(2)
O3A	N3A	1.216(2)	N6A	C5A	1.3419(19)
N1	N2	1.3784(16)	N6A	C7A	1.333(2)
N1	C5	1.3603(18)	C1	C7	1.503(2)
N1	C9	1.3932(19)	C1A	C7A	1.502(2)
N1A	N2A	1.3750(17)	C7	C8	1.412(2)
N1A	C5A	1.3614(18)	C7A	C8A	1.408(2)
N1A	C9A	1.3964(19)	C8	C9	1.435(2)

N2	C3	1.316(2)	C8A	C9A	1.428(2)
N2A	C3A	1.313(2)	C10	C11	1.495(2)
N3	C8	1.442(2)	C10A	C11A	1.490(3)
N3A	C8A	1.451(2)	C11	C12	1.502(3)
N4	C3	1.355(2)	C11A	C12A	1.500(3)
N4	C5	1.330(2)	C12	C13	1.501(3)
N4A	C3A	1.358(2)	C12A	C13A	1.500(3)
N4A	C5A	1.333(2)	C13	C14	1.496(3)
N5	C10	1.477(2)	C13A	C14A	1.500(3)

Table 5B. Valence angles

Atom	**Atom**	**Atom**	**Angle/deg**	**Atom**	**Atom**	**Atom**	**Angle/deg**
N2	N1	C9	123.77(12)	N6	C7	C1	113.69(16)
C5	N1	N2	110.45(12)	N6	C7	C8	122.95(14)
C5	N1	C9	125.77(12)	C8	C7	C1	123.33(16)
N2A	N1A	C9A	123.68(11)	N6A	C7A	C1A	114.41(17)
C5A	N1A	N2A	110.53(12)	N6A	C7A	C8A	122.64(14)
C5A	N1A	C9A	125.78(13)	C8A	C7A	C1A	122.91(18)
C3	N2	N1	101.06(12)	C7	C8	N3	121.40(13)
C3A	N2A	N1A	101.33(12)	C7	C8	C9	122.49(14)
O2	N3	C8	118.75(13)	C9	C8	N3	116.08(14)
O3	N3	O2	122.06(16)	C7A	C8A	N3A	121.17(15)
O3	N3	C8	119.19(16)	C7A	C8A	C9A	123.30(15)
O2A	N3A	C8A	118.95(15)	C9A	C8A	N3A	115.48(15)
O3A	N3A	O2A	122.35(19)	O1	C9	N1	119.51(13)
O3A	N3A	C8A	118.70(18)	O1	C9	C8	130.61(14)
C5	N4	C3	103.34(13)	N1	C9	C8	109.86(13)
C5A	N4A	C3A	103.28(13)	O1A	C9A	N1A	119.65(13)
C10	N5	C14	112.31(12)	O1A	C9A	C8A	131.06(15)
C10A	N5A	C14A	112.22(13)	N1A	C9A	C8A	109.26(13)
C7	N6	C5	115.84(13)	N5	C10	C11	110.12(14)
C7A	N6A	C5A	115.74(14)	N5A	C10A	C11A	110.81(15)
N2	C3	N4	116.41(14)	C10	C11	C12	111.78(16)
N2A	C3A	N4A	116.29(15)	C10A	C11A	C12A	111.48(15)
N4	C5	N1	108.74(13)	C13	C12	C11	109.94(15)
N4	C5	N6	128.21(13)	C13A	C12A	C11A	110.30(15)
N6	C5	N1	123.05(14)	C14	C13	C12	112.32(17)
N4A	C5A	N1A	108.58(13)	C14A	C13A	C12A	112.21(17)
N4A	C5A	N6A	128.23(14)	N5	C14	C13	110.45(15)
N6A	C5A	N1A	123.18(14)	N5A	C14A	C13A	110.48(15)

Table 6B. Torsion angles

A	**B**	**C**	**D**	Angle/**deg**
O2	N3	C8	C7	–149.14(16)
O2	N3	C8	C9	29.1(2)
O2A	N3A	C8A	C7A	142.87(18)
O2A	N3A	C8A	C9A	–34.7(2)
O3	N3	C8	C7	30.3(2)
O3	N3	C8	C9	–151.44(16)
O3A	N3A	C8A	C7A	–37.6(3)
O3A	N3A	C8A	C9A	144.84(18)
N1	N2	C3	N4	0.34(17)
N1A	N2A	C3A	N4A	–0.37(18)
N2	N1	C5	N4	–0.04(16)
N2	N1	C5	N6	179.46(13)
N2	N1	C9	O1	–0.3(2)
N2	N1	C9	C8	–178.93(12)
N2A	N1A	C5A	N4A	–0.77(16)
N2A	N1A	C5A	N6A	179.92(13)
N2A	N1A	C9A	O1A	–0.1(2)
N2A	N1A	C9A	C8A	178.23(12)
N3	C8	C9	O1	2.6(2)
N3	C8	C9	N1	–179.00(12)
N3A	C8A	C9A	O1A	–1.3(2)
N3A	C8A	C9A	N1A	–179.34(13)
N5	C10	C11	C12	–56.7(2)
N5A	C10A	C11A	C12A	56.2(2)
N6	C7	C8	N3	177.10(13)
N6	C7	C8	C9	–1.1(2)
N6A	C7A	C8A	N3A	–178.45(14)
N6A	C7A	C8A	C9A	–1.1(2)
C1	C7	C8	N3	–1.2(2)
C1	C7	C8	C9	–179.40(15)
C1A	C7A	C8A	N3A	–0.8(2)
C1A	C7A	C8A	C9A	176.56(15)
C3	N4	C5	N1	0.23(16)
C3	N4	C5	N6	–179.23(15)
C3A	N4A	C5A	N1A	0.50(16)
C3A	N4A	C5A	N6A	179.76(15)
C5	N1	N2	C3	–0.17(15)
C5	N1	C9	O1	–179.57(13)
C5	N1	C9	C8	1.85(19)
C5	N4	C3	N2	–0.37(18)
C5	N6	C7	C1	–179.72(13)
C5	N6	C7	C8	1.8(2)

C5A	N1A	N2A	C3A	0.68(15)
C5A	N1A	C9A	O1A	178.39(14)
C5A	N1A	C9A	C8A	–3.3(2)
C5A	N4A	C3A	N2A	–0.07(18)
C5A	N6A	C7A	C1A	–179.13(14)
C5A	N6A	C7A	C8A	–1.3(2)
C7	N6	C5	N1	–0.7(2)
C7	N6	C5	N4	178.65(14)
C7	C8	C9	O1	–179.11(15)
C7	C8	C9	N1	–0.7(2)
C7A	N6A	C5A	N1A	1.3(2)
C7A	N6A	C5A	N4A	–177.91(14)
C7A	C8A	C9A	O1A	–178.80(16)
C7A	C8A	C9A	N1A	3.2(2)
C9	N1	N2	C3	–179.50(13)
C9	N1	C5	N4	179.27(13)
C9	N1	C5	N6	–1.2(2)
C9A	N1A	N2A	C3A	179.34(13)
C9A	N1A	C5A	N4A	–179.40(13)
C9A	N1A	C5A	N6A	1.3(2)
C10	N5	C14	C13	–55.9(2)
C10	C11	C12	C13	55.2(2)
C10A	N5A	C14A	C13A	55.6(2)
C10A	C11A	C12A	C13A	–54.8(2)
C11	C12	C13	C14	–54.2(3)
C11A	C12A	C13A	C14A	54.2(2)
C12	C13	C14	N5	54.5(2)
C12A	C13A	C14A	N5A	–54.5(2)
C14	N5	C10	C11	57.1(2)
C14A	N5A	C10A	C11A	–56.8(2)

Table 7B. Hydrogen atom coordinates (Å×10^4) and equivalent isotropic displacement parameters (Å^2×10^3)

Atom	***x***	***y***	***z***	**U(eq)**
H5A	–1890(20)	–826(18)	3531(17)	76(6)
H5B	–2120(20)	–1609(16)	4249(19)	79(6)
H5AA	2710(20)	–1655(15)	9963(18)	68(6)
H5AB	3030(20)	–821(19)	11015(19)	77(6)
H1A	3476	–4657	5481	104
H1B	4627	–4124	6511	104
H1C	5080	–3773	5489	104
H1AA	–1491	–4753	6950	115

H1AB	87	–3861	7499	115
H1AC	–295	–4196	6270	115
H3	1366(17)	17(14)	9169(14)	50(4)
H3A	–3579(18)	2(14)	5878(14)	54(5)
H10C	–2785	–2342	2011	66
H10D	–4038	–2293	2819	66
H10A	965	–2355	10962	68
H10B	2304	–2310	11748	68
H11C	–3876	–4222	2103	83
H11D	–3479	–3776	3338	83
H11A	1608	–3789	9635	84
H11B	1348	–4218	10638	84
H12C	–1678	–4699	2578	91
H12D	–1300	–3896	1838	91
H12A	3963	–3731	11071	87
H12B	3589	–4554	9889	87
H13C	–644	–3111	4060	90
H13D	589	–3146	3239	90
H13A	5715	–2906	10074	91
H13B	4394	–2964	9269	91
H14C	388	–1225	4002	78
H14D	35	–1643	2763	78
H14A	5058	–1458	11378	76
H14B	5289	–1048	10363	76