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Structure of 8-Amino-3- β -D-ribofuranosyl-1,2,4-triazolo[4,3-*b*]pyridazine, an Analog of Formycin A

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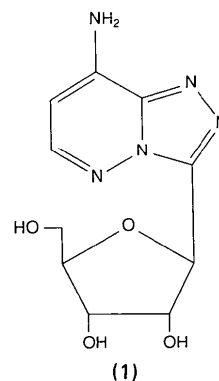
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Abstract. $C_{10}H_{13}N_5O_4$, $M_r = 267.25$, orthorhombic, $P2_12_12_1$, $a = 6.7976$ (5), $b = 7.4170$ (8), $c = 22.1104$ (17) Å, $V = 1114.75$ (16) Å³, $Z = 4$, $D_x = 1.592$ g cm⁻³, $Cu K\alpha$ ($\lambda = 1.54178$ Å), $\mu = 10.236$ cm⁻¹, $F(000) = 560$, $T = 295$ K, $R = 0.0273$ for 2217 reflections ($F \geq 4\sigma_F$). The sugar conformation and puckering parameters are 1T (C_1 -*exo*- C_2 -*endo*), $P = 141.2^\circ$ and $\tau_m = 42.4^\circ$. The side chain is *gt*, $\varphi_{OO} = 57.70$ (14) $^\circ$ and $\varphi_{CO} = 176.06$ (11) $^\circ$. The glycosidic bond length is 1.491 (2) Å; the glycosidic torsion angle, $O4'-C1'-C3-N2$, is 71.3 (2) $^\circ$ corresponding to an *anti* conformation. The triazolo-pyridazine moiety is planar [r.m.s. deviation: 0.005 (2) Å]. The dihedral angle between the pyridazine and triazole planes is 0.51 (5) $^\circ$. Bond lengths [C—C, 1.506 (2)–1.541 (2) Å; C—O, 1.406 (2)–1.453 (2) Å] and angles in the ribose moiety are normal; those in the aglycon are consistent with a non-aromatic system containing four double bonds [three C=N, 1.313 (2)–1.320 (2) Å, and one C=C 1.375 (2) Å]. The three hydroxyl H atoms and one H atom of the amino group are involved in the intermolecular hydrogen bonding [1.78 (3) Å $\leq d(H \cdots A) \leq 2.17$ (3) Å; 159 (2) $^\circ \leq \angle(D-H \cdots A) \leq 168$ (3) $^\circ$].

Experimental. Synthesis of the title compound (1) has been reported recently (Kang, Larson, Robins & Revankar, 1989). The sample crystal was a colorless, transparent, flattened prism grown slowly from water. Table 1 summarizes data collection and refinement. All non-H atom positions were obtained with *SHELXS86* (Sheldrick, 1986). All H atoms were located in a difference map as peaks of density 0.54–0.84 e Å⁻³ at $R = 0.056$. Positional parameters

of all atoms, anisotropic thermal parameters for non-H atoms and isotropic thermal parameters for H atoms were refined with *SHELX76* (Sheldrick, 1976). Scattering factors and anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974) except those of H which were taken from Stewart, Davidson & Simpson (1965). Data were reduced with *SDP-Plus* (Frenz, 1985); least-squares-planes program from Cordes (1983); figures were drawn with *ORTEPII* (Johnson, 1976). The atomic coordinates are listed in Table 2; bond lengths, bond angles and selected torsion angles are listed in Table 3.† The atom labeling and molecular conformation are shown in Fig. 1. The hydrogen bonding is tabulated in Table 4 and illustrated in a packing diagram in Fig. 2.



† Tables of anisotropic thermal parameters, bond lengths and angles involving H atoms, torsion angles, least-squares planes and structure factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51694 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. Crystallographic summary for (1)

(A) Data collection (295 K) ^{a,b}	
Mode	ω -2 θ scan
Scan range (°)	0.80 + 0.15 tan θ
Background	Scan 0.25 times scan range before and after scan
Scan rate (° min ⁻¹)	1.5–16.5
Exposure time (h)	46.7
Stability correction range on I	0.999–1.000
2 θ range (°)	3.0–152.0
Range in <i>hkl</i> , min.	0, -9, -27
max.	8, 9, 27
Total reflections, measd, unique	5026, 2327
R_{int}	0.0171
Crystal dimensions (mm)	0.24 × 0.23 × 0.11
Crystal volume (mm ³)	0.00574
Crystal faces	{001}; {011}; {102}; {10 $\bar{2}$ }; cut \sim {100}
Transmission-factor range	0.748–0.900
(B) Structure refinement ^c	
Reflections used, m ($F \geq 4\sigma_F$)	2217
No. of variables, n	225
Extinction parameter	8.2 (16) × 10 ⁻⁷
Goodness of fit, S	1.544
R , wR	0.0273, 0.0396
R for all data	0.0293
Max. ave. Δ/σ	0.0038, 0.0006
Max., min. density in ΔF map (e Å ⁻³)	0.38, -0.21

(a) Unit-cell parameters were obtained by least-squares refinement of the setting angles of 25 reflections with $57.2^\circ < 2\theta < 59.1^\circ$.

(b) Enraf–Nonius CAD-4 diffractometer with a graphite monochromator was used. Data reduction was accomplished with the *SDP-Plus* software (Frenz, 1985). Crystal and instrument stability were monitored by re-measurement of 3 check reflections (2, 1, 12; $\bar{1}, \bar{3}, \bar{10}$; $\bar{2}, \bar{4}, \bar{2}$) every hour. A linear fit of the intensities of these reflections was used to correct the data.

(c) Function minimized was $\sum w(F_o - F_c)^2$, where $w^{-1} = (\sigma_F^2 + 0.0004F^2)$. $\sigma_F = F_o/2I$; $\sigma_I = [N_{pk} + N_{bg1} + N_{bg2}]^{1/2}$.

Table 2. Positional and isotropic thermal parameters for atoms in (1)

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}^*/U (Å ²)
N1	0.7650 (2)	0.84696 (13)	0.58591 (5)	0.0329 (3)
N2	0.7706 (2)	0.70709 (15)	0.62707 (5)	0.0327 (3)
C3	0.7739 (2)	0.5533 (2)	0.59754 (5)	0.0282 (3)
N4	0.7700 (2)	0.58723 (14)	0.53704 (4)	0.0273 (3)
N5	0.7697 (2)	0.4626 (2)	0.49115 (5)	0.0351 (3)
C6	0.7655 (2)	0.5403 (2)	0.43782 (6)	0.0392 (4)
C7	0.7605 (2)	0.7267 (2)	0.42591 (6)	0.0384 (4)
C8	0.7623 (2)	0.8503 (2)	0.47225 (6)	0.0324 (3)
C9	0.7653 (2)	0.7718 (2)	0.53179 (5)	0.0281 (3)
N10	0.7613 (2)	1.0300 (2)	0.46606 (6)	0.0446 (4)
C1'	0.7912 (2)	0.3705 (2)	0.62509 (5)	0.0283 (3)
C2'	0.9445 (2)	0.3583 (2)	0.67531 (6)	0.0286 (3)
C3'	0.8821 (2)	0.1849 (2)	0.70788 (5)	0.0279 (3)
C4'	0.6568 (2)	0.1887 (2)	0.70011 (5)	0.0287 (3)
C5'	0.5556 (2)	0.2339 (2)	0.75878 (6)	0.0331 (4)
O2'	1.1373 (2)	0.3587 (2)	0.65228 (5)	0.0460 (3)
O3'	0.9729 (2)	0.02870 (14)	0.68458 (6)	0.0413 (3)
O4'	0.61202 (14)	0.3264 (2)	0.65542 (4)	0.0364 (3)
O5'	0.3489 (2)	0.2487 (2)	0.74958 (4)	0.0414 (3)
H6	0.778 (3)	0.465 (3)	0.4068 (9)	0.045 (5)
H7	0.754 (3)	0.769 (3)	0.3846 (10)	0.058 (5)
H10A	0.748 (3)	1.085 (3)	0.4285 (8)	0.048 (5)
H10B	0.765 (4)	1.096 (3)	0.5048 (11)	0.063 (6)
H1'	0.818 (2)	0.285 (2)	0.5927 (7)	0.030 (4)
H2'	0.919 (2)	0.457 (2)	0.7029 (7)	0.030 (4)
H3'	0.915 (3)	0.190 (3)	0.7509 (9)	0.039 (5)
H4'	0.618 (3)	0.071 (2)	0.6851 (8)	0.033 (4)
H5'A	0.615 (4)	0.351 (3)	0.7804 (10)	0.066 (6)
H5'B	0.576 (3)	0.129 (3)	0.7848 (8)	0.043 (5)
HO2'	1.225 (4)	0.315 (4)	0.6824 (12)	0.079 (8)
HO3'	0.915 (5)	0.000 (4)	0.6555 (12)	0.090 (10)
HO5'	0.276 (5)	0.246 (5)	0.7901 (13)	0.095 (9)

* For non-hydrogen atoms, $U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* A_{ij}$, where A_{ij} is the dot product of the *i*th and *j*th direct-space unit-cell vectors.

Table 3. Bond lengths (Å) and bond angles (°) in (1)

	1	2	3	1–2	1–2–3
N2	N1	C9		1.3805 (15)	106.26 (10)
C3	N2	N1		1.315 (2)	108.97 (10)
N4	C3	C1'		1.361 (2)	124.83 (11)
N4	C3	N2			109.10 (10)
C1'	C3	N2		1.491 (2)	125.98 (11)
N5	N4	C9		1.3726 (14)	127.47 (10)
N5	N4	C3			127.00 (10)
C9	N4	C3		1.374 (2)	105.53 (10)
C6	N5	N4		1.313 (2)	111.62 (11)
C7	C6	N5		1.408 (2)	126.85 (12)
C8	C7	C6		1.375 (2)	121.00 (12)
C9	C8	N10		1.440 (2)	119.72 (11)
C9	C8	C7			114.33 (12)
N10	C8	C7		1.340 (2)	125.95 (12)
N1	C9	N4		1.320 (2)	110.14 (10)
N1	C9	C8			131.15 (11)
N4	C9	C8			118.72 (11)
C2'	C1'	O4'		1.526 (2)	103.12 (9)
C2'	C1'	C3			113.93 (10)
O4'	C1'	C3		1.428 (2)	109.45 (10)
C3'	C2'	O2'		1.533 (2)	115.47 (11)
C3'	C2'	C1'			101.70 (10)
O2'	C2'	C1'		1.406 (2)	111.90 (10)
C4'	C3'	O3'		1.541 (2)	114.15 (10)
C4'	C3'	C2'			101.97 (10)
O3'	C3'	C2'		1.410 (2)	113.33 (10)
C5'	C4'	O4'		1.506 (2)	109.47 (10)
C5'	C4'	C3'			111.21 (10)
O4'	C4'	C3'		1.453 (2)	107.27 (10)
O5'	C5'	C4'		1.424 (2)	110.17 (11)
C1'	O4'	C4'			107.56 (9)
Selected torsion angles (°)					
χ	N2	C3	C1'	O4'	71.3 (2)
θ_0	C1'	C2'	C3'	C4'	-33.04 (11)
θ_1	C2'	C3'	C4'	O4'	12.51 (12)
θ_2	C3'	C4'	O4'	C1'	14.82 (12)
θ_3	C4'	O4'	C1'	C2'	-36.29 (12)
θ_4	O4'	C1'	C2'	C3'	43.25 (12)
φ_{00}	O4'	C4'	C5'	O5'	57.70 (14)
φ_{00}	C3'	C4'	C5'	O5'	176.06 (11)

Table 4. Hydrogen bonding in (1)

<i>D</i> –H... <i>A</i>	Symmetry of <i>A</i> relative to <i>D</i>	<i>d</i> (<i>D</i> ... <i>A</i>) (Å)	<i>d</i> (H... <i>A</i>) (Å)	\angle (<i>D</i> –H... <i>A</i>) (°)
N10 H10A O2'	$x-0.5, 1.5-y, 1.0-z$	2.871 (2)	1.98 (2)	159. (2)
O2' HO2' O5'	$1.0+x, y, z$	2.714 (2)	1.78 (3)	168. (3)
O3' HO3' N1	$x, y-1.0, z$	2.929 (2)	2.17 (3)	164. (3)
O5' HO5' N2	$1.0-x, y-0.5, 1.5-z$	2.863 (1)	1.88 (3)	159. (3)

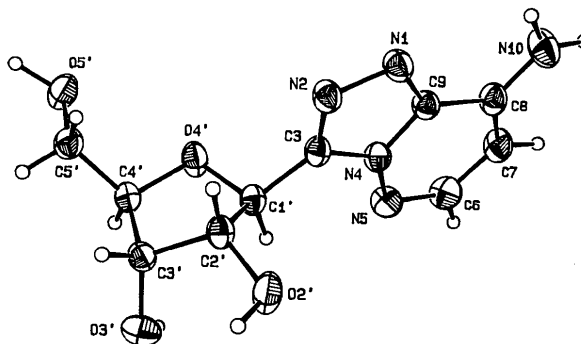


Fig. 1. Perspective drawing of (1) showing labeling of non-hydrogen atoms. Double bonds in the heterocyclic ring are N2=C3, N5=C6, N1=C9 and C7=C8. Hydrogens are included as open circles. Thermal ellipsoids are drawn at the 50% probability level.

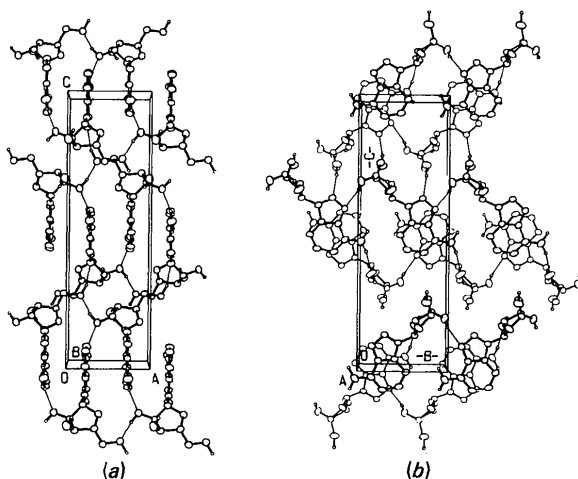


Fig. 2. Unit-cell packing diagrams with hydrogen bonds indicated by thin lines. There are no interbase hydrogen bonds. Hydrogens attached to carbon atoms have been omitted for clarity. (a) Projection along the *b* axis illustrating the base stacking along the twofold screw axis parallel to the *a* axis. The bases are essentially perpendicular to the *a* axis and stacked about 3.4 Å apart. (b) Projection along the *a* axis showing the flip-flop overlap of bases in the stacks and the alignment of the amino bond with the N4–C9 bond.

Related literature. The title compound is related to formycin A first isolated by Hori, Ito, Takita, Koyama, Takeuchi & Umezawa (1964). Structures of formycin and derivatives include formycin monohydrate (Prusiner, Brennan & Sundaralingam, 1973), 2-methylformycin (Abola, Sims, Abraham, Lewis & Townsend, 1974) and formycin 5'-phosphate (Giranda, Berman & Schramm, 1988). Comparison of (1) with the structure of the unsubstituted 1,2,4-triazolo[4,3-*b*]pyridazine

reported by Golic, Leban, Stanovnik & Tisler (1978) indicates a slight bond elongation occurring at bonds involving the substitution sites, C3 and C8. The conformational parameters follow the convention of Altona & Sundaralingam (1972). The title compound was found to lack significant biological activity (Kang, Larson, Robins & Revankar, 1989).

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1-Phenyl-*N*²-(1-phenylmethyl-3-isoquinoly)acetamidinium Trifluoromethanesulfonate

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Abstract. $C_{24}H_{22}N_3^+ \cdot CF_3O_3S^-$, $M_r = 501.5$, monoclinic, $P2_1/c$, $a = 7.022$ (1), $b = 18.294$ (3), $c = 18.617$ (4) Å, $\beta = 91.33$ (2)°, $V = 2390.9$ Å³, $Z = 4$, $D_x = 1.392$ Mg m⁻³, $F(000) = 1040$, $\lambda(Mo K\alpha) = 0.71069$ Å, $\mu = 0.148$ mm⁻¹, $T = 293$ K, $R = 0.041$ for 2260 unique reflexions [$F \geq 3\sigma(F)$]. Coplanar acetamidinium and isoquinoline moieties [C–C–N–C = –179.3 (1)°] are able to interact through their π

systems. The protonated =NH group takes part in two hydrogen bonds: one is a weak intramolecular interaction with the isoquinoline nitrogen [=NH...N = 1.98 (3), N...N = 2.658 (5) Å, N–H...N = 135 (2)°] and the other is to an oxygen O(33) of the anion [=NH...O(33) = 2.02 (3) Å, N...O(33) = 2.886 (5) Å, N–H...O(33) = 171 (2)°]. The second acetamidinium nitrogen also forms an H bond to