(1987); as model enzymatic reaction intermediates: Colucci & Gandour (1988). Structure of *rel*-(2*S*,6*S*)-2-(6-hydroxy-4,4,6-trimethyl)morpholinomethanesulfonate: Colucci, Fronczek, Gandour & Watkins (1988).

References

COLUCCI, W. J., FRONCZEK, F. R., GANDOUR, R. D. & WATKINS, S. F. (1988). *Acta Cryst.* C**44**, 1845–1847.

COLUCCI, W. J. & GANDOUR, R. D. (1988). *Bioorg. Chem.* **16**, 307–334.

COLUCCI, W. J., GANDOUR, R. D., FRONCZEK, F. R., BRADY, P. S. & BRADY, L. J. (1987). *J. Am. Chem. Soc.* **109**, 7915–7916.

CROMER, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

FRENZ, B. A. & OKAYA, Y. (1980). *Enraf–Nonius Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.

GANDOUR, R. D., COLUCCI, W. J. & FRONCZEK, F. R. (1985). *Bioorg. Chem.* **13**, 197–208.

GANDOUR, R. D., COLUCCI, W. J., STELLY, T. C., BRADY, P. S. & BRADY, L. J. (1986). *Biochem. Biophys. Res. Commun.* **138**, 735–741.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

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An Aminocyclohexanecarboxylic Acid

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Abstract. 8-Amino-1,4-dioxaspiro[4.5]decane-8-carboxylic acid, $C_9H_{15}NO_4$, $M_r = 201{\cdot}2$, orthorhombic, $Pna2_1$, $a = 27{\cdot}234\ (4)$, $b = 5{\cdot}5786\ (10)$, $c = 6{\cdot}4522\ (9)$ Å, $V = 980{\cdot}3\ (5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}363$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 1{\cdot}00$ cm^{-1}, $F(000) = 432$, $T = 297$ K, $R = 0{\cdot}036$ for 1469 data having $F_o^2 > 0$. The six-membered ring adopts a near ideal chair conformation with the carboxylate as an axial substituent. The ammonio group is nearly antiperiplanar to the carboxylate. All three NH bonds are involved in intermolecular hydrogen bonding with neighboring carboxylates.

Experimental. The title compound was prepared according to Britten & Lockwood (1974). Colorless, crystals, dec. 573 K, suitable for single-crystal X-ray diffraction were crystallized from methanol/water at room temperature. All standard spectroscopic measurements support the X-ray structure determination.

Intensity data were obtained from an irregular fragment of dimensions 0·28 × 0·40 × 0·48 mm mounted in a random orientation on an Enraf–Nonius CAD-4 diffractometer. Cell dimensions were determined at 297 K by a least-squares fit to setting angles of 25 reflections having $22 > 2\theta > 19°$. Two octants of data having $2 < 2\theta < 60°$, $-38 \le h \le 38$, $0 \le k \le 9$, $0 \le l \le 7$ were measured using graphite-monochromated Mo $K\alpha$ radiation. The ω–2θ scans were made at speeds ranging from 0·45 to 4·0° min^{-1} to measure all significant data with approximately equal precision. Three standard reflections (800, 020, 002) exhibited no decline in intensity during data collection. Data included corrections for background, Lorentz, and polarization. Absorption was negligible.

The space group was determined by systematic absences $0kl$ with $k+l$ odd, $h0l$ with h odd, and successful refinement of the noncentrosymmetric model. The structure was solved by direct methods and refined by full-matrix least squares based upon F, with weights $w = 4F_o^2[\sigma^2(I) + (0{\cdot}02F_o^2)^2]^{-1}$ using the Enraf–Nonius *SDP* (Frenz, 1985), scattering factors of Cromer &

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters*

$B_{eq} = \frac{8}{3}\pi^2(U_{11} + U_{22} + U_{33})$.

	x	y	z	B_{eq}(Å^2)
O1	0·55512 (4)	−0·1689 (2)	1·0000	3·06 (2)
O2	0·53117 (4)	−0·2371 (2)	0·6789 (2)	3·27 (2)
O3	0·67193 (4)	0·4357 (2)	0·6062 (2)	3·35 (2)
O4	0·71427 (4)	0·0842 (2)	0·6064 (3)	3·66 (2)
N	0·51540 (4)	0·2913 (2)	0·7889 (2)	2·05 (2)
C1	0·54913 (4)	−0·1085 (2)	0·8170 (3)	2·11 (2)
C2	0·56257 (4)	0·1553 (2)	0·7592 (2)	1·86 (2)
C3	0·57845 (5)	0·1833 (2)	0·5333 (2)	2·21 (2)
C4	0·62917 (5)	0·0736 (3)	0·4974 (3)	2·65 (3)
C5	0·66668 (5)	0·1850 (3)	0·6428 (3)	2·62 (2)
C6	0·65149 (5)	0·1516 (3)	0·8668 (3)	2·83 (3)
C7	0·60097 (5)	0·2609 (3)	0·9060 (3)	2·39 (2)
C8	0·70990 (6)	0·4610 (4)	0·4552 (3)	3·97 (4)
C9	0·74349 (8)	0·2556 (4)	0·5033 (4)	5·32 (4)

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Waber (1974), anomalous-dispersion coefficients of Cromer (1974), and 1469 data having $F_o^2 > 0$. Non-H atoms were refined anisotropically; the H atoms were located by ΔF syntheses and were refined isotropically. Final $R = 0{\cdot}036$ ($R = 0{\cdot}043$ for all 1544 data), $wR = 0{\cdot}036$, $S = 1{\cdot}652$ for 187 variables. The largest shift was $0{\cdot}12\sigma$ in the final cycle, maximum residual density 0·18, minimum −0·17 e Å^{-3}, extinction coefficient $g = 6{\cdot}5\ (18) \times 10^{-7}$, where the correction factor $(1 + gIc)^{-1}$ was applied to F_c. The fractional coordinates of the title compound are given in Table 1. Fig. 1 is a line drawing and Fig. 2 is a perspective drawing showing the atom numbering. Selected distances,

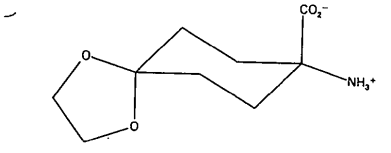

Fig. 1. 8-Amino-1,4-dioxaspiro[4.5]decane-8-carboxylic acid.

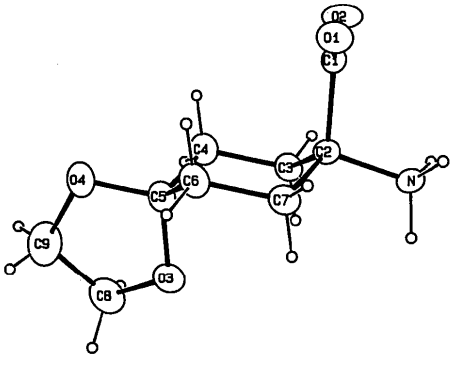

Fig. 2. *ORTEP* drawing (Johnson, 1965) of the molecule, representing heavy atoms as 40% probability ellipsoids and H atoms as circles of arbitrary radius.

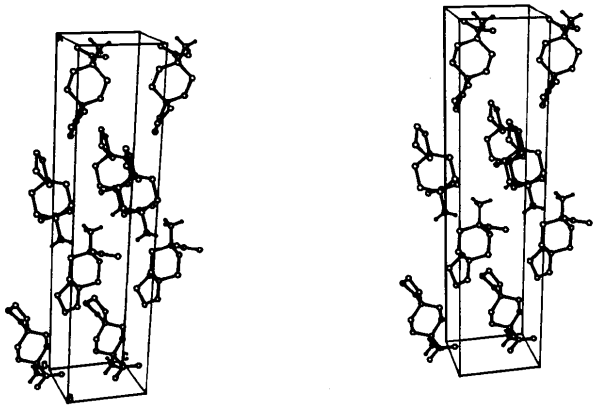

Fig. 3. *PLUTO*78 drawing (Motherwell & Clegg, 1978) illustrating the molecular packing and hydrogen bonding using circles of arbitrary radius for the heavy atoms and only the NH H atoms.

Table 2. *Bond distances* (Å), *angles* (°), *and selected torsion angles* (°)

O1	C1	1·239 (2)	O2	C1	1·244 (2)	O3	C5	1·426 (2)
O3	C8	1·428 (2)	O4	C5	1·432 (2)	O4	C9	1·411 (2)
N	C2	1·505 (1)	C1	C2	1·562 (2)	C2	C3	1·528 (2)
C2	C7	1·529 (2)	C3	C4	1·528 (2)	C4	C5	1·520 (2)
C5	C6	1·514 (2)	C6	C7	1·526 (2)	C8	C9	1·499 (3)

C5	O3	C8	106·4 (1)	C5	O4	C9	108·8 (1)	O1	C1	O2	125·2 (1)
O1	C1	C2	117·0 (1)	O2	C1	C2	117·7 (1)	N	C2	C1	104·16 (9)
N	C2	C3	108·2 (1)	N	C2	C7	108·1 (1)	C1	C2	C3	113·0 (1)
C1	C2	C7	112·0 (1)	C3	C2	C7	110·98 (9)	C2	C3	C4	111·0 (1)
C3	C4	C5	110·5 (1)	O3	C5	O4	105·5 (1)	O3	C5	C4	111·5 (1)
O3	C5	C6	107·8 (1)	O4	C5	C4	110·3 (1)	O4	C5	C6	110·8 (1)
C4	C5	C6	110·8 (1)	C5	C6	C7	110·8 (1)	C2	C7	C6	111·1 (1)
O3	C8	C9	103·0 (1)	O4	C9	C8	105·8 (1)				

C9	O3	C5	O4	29·6 (2)	C8	O3	C5	C4	−90·2 (2)
C5	O3	C8	C9	−32·7 (2)	C9	O4	C5	O3	−13·9 (2)
C9	O4	C5	C4	106·6 (2)	O1	C1	C2	N	−91·1 (1)
O1	C1	C2	C3	151·7 (1)	O2	C1	C2	N	85·5 (1)
O2	C1	C2	C3	−31·6 (2)	N	C2	C3	C4	173·5 (1)
C7	C2	C3	C4	55·1 (2)	O3	C8	C9	O4	23·7 (2)

angles, and torsion angles are presented in Table 2.* The crystal lattice is stabilized by intermolecular hydrogen bonds formed by all three NH bonds to neighboring carboxylates, Fig. 3.

Related literature. For amino-acid structures showing the ammonio group nearly antiperiplanar to the carboxylate and extensive intermolecular hydrogen bonding see Chacko & Zand (1973) and references cited.

The structural features of the dioxolane ring of 1,4-dioxaspiro[4.5]dec-8-yl *p*-toluenesulfonate (Bocelli, Grenier-Loustalot & Iratcabal, 1982) are similar to those in the title molecule. A bis(dioxolane) derivative of 1,4-cyclohexanedione reported by Chadwick, Dunitz & Schweizer (1977) is between an envelope and a twist structure.

We thank the donors of the Petroleum Research Fund, administered by the American Chemical Society, for support of this research.

* Lists of H-atom coordinates and isotropic thermal parameters, anisotropic thermal parameters, structure-factor amplitudes, bond distances and angles involving H, intermolecular hydrogen-bond distances and angles, and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51721 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

BOCELLI, G., GRENIER-LOUSTALOT, M.-F. & IRATCABAL, P. (1982). *Acta Cryst.* B**38**, 2068–2070.

BRITTEN, A. & LOCKWOOD, G. (1974). *J. Chem. Soc. Perkin Trans.* 1, pp. 1824–1827.

CHACKO, K. K. & ZAND, R. (1973). *Acta Cryst.* B**29**, 2681–2686.

CHADWICK, D. J., DUNITZ, J. D. & SCHWEIZER, W. B. (1977). *Acta Cryst.* B**33**, 1643–1645.

CROMER, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
FRENZ, B. A. (1985). *Enraf–Nonius Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.
JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO78*. A program for plotting molecular and crystal structures. Univ. of Cambridge, England.

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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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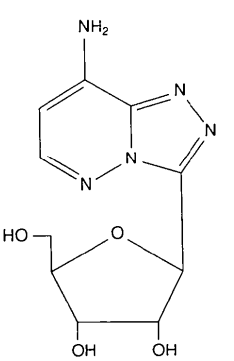
Abstract. $C_{10}H_{13}N_5O_4$, $M_r = 267{\cdot}25$, orthorhombic, $P2_12_12_1$, $a = 6{\cdot}7976\ (5)$, $b = 7{\cdot}4170\ (8)$, $c = 22{\cdot}1104\ (17)$ Å, $V = 1114{\cdot}75\ (16)$ Å^3, $Z = 4$, $D_x = 1{\cdot}592$ g cm^{-3}, Cu $K\alpha$ ($\lambda = 1{\cdot}54178$ Å), $\mu = 10{\cdot}236$ cm^{-1}, $F(000) = 560$, $T = 295$ K, $R = 0{\cdot}0273$ for 2217 reflections ($F \geq 4\sigma_F$). The sugar conformation and puckering parameters are ${}^2_1T$ ($C_{1'}$-*exo*–$C_{2'}$-*endo*), $P = 141{\cdot}2°$ and $\tau_m = 42{\cdot}4°$. The side chain is *gt*, $\varphi_{OO} = 57{\cdot}70\ (14)°$ and $\varphi_{CO} = 176{\cdot}06\ (11)°$. The glycosidic bond length is 1·491 (2) Å; the glycosidic torsion angle, O4′–C1′–C3–N2, is 71·3 (2)° corresponding to an *anti* conformation. The triazolopyridazine moiety is planar [r.m.s. deviation: 0·005 (2) Å]. The dihedral angle between the pyridazine and triazole planes is 0·51 (5)°. 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 [$1{\cdot}78\ (3)$ Å $\leq d(H\cdots A) \leq 2{\cdot}17\ (3)$ Å; $159\ (2)° \leq \angle(D{-}H\cdots A) \leq 168\ (3)°$].

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 *SHELXS*86 (Sheldrick, 1986). All H atoms were located in a difference map as peaks of density 0·54–0·84 e Å^{-3} at $R = 0{\cdot}056$. Positional parameters of all atoms, anisotropic thermal parameters for non-H atoms and isotropic thermal parameters for H atoms were refined with *SHELX*76 (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 *ORTEP*II (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.



(1)

† 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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