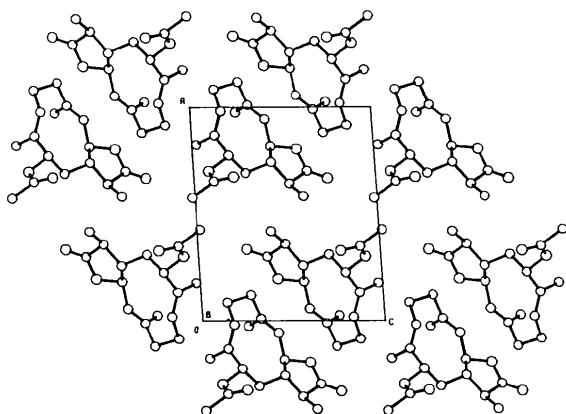


Table 3. *Torsion angles* (°)

Sign convention as defined by Klyne & Prelog (1960).

C(10)–C(1)–C(2)–C(3)	–100.3 (6)	O(2)–C(6)–C(7)–C(11)	–33.7 (4)	O(3)–C(9)–C(10)–C(1)	127.7 (4)
C(2)–C(1)–C(10)–C(9)	164.6 (5)	C(5)–C(6)–O(2)–C(12)	147.5 (4)	O(3)–C(9)–C(10)–C(15)	–57.0 (5)
C(2)–C(1)–C(10)–C(15)	–10.3 (7)	C(7)–C(6)–O(2)–C(12)	25.3 (5)	C(8)–C(9)–O(3)–C(16)	89.7 (5)
C(1)–C(2)–C(3)–C(4)	50.5 (5)	C(6)–C(7)–C(8)–C(9)	–82.2 (5)	C(10)–C(9)–O(3)–C(16)	–147.4 (4)
C(2)–C(3)–C(4)–C(5)	–87.5 (5)	C(11)–C(7)–C(8)–C(9)	161.5 (4)	C(6)–O(2)–C(12)–O(1)	175.1 (4)
C(2)–C(3)–C(4)–C(14)	85.2 (5)	C(6)–C(7)–C(11)–C(12)	30.2 (5)	C(6)–O(2)–C(12)–C(11)	–5.7 (6)
C(3)–C(4)–C(5)–C(6)	157.2 (5)	C(6)–C(7)–C(11)–C(13)	154.0 (4)	O(2)–C(12)–C(11)–C(7)	–16.3 (6)
C(14)–C(4)–C(5)–C(6)	–14.9 (7)	C(8)–C(7)–C(11)–C(12)	156.1 (4)	O(2)–C(12)–C(11)–C(13)	–141.9 (5)
C(4)–C(5)–C(6)–C(7)	–130.9 (5)	C(8)–C(7)–C(11)–C(13)	–80.0 (5)	O(1)–C(12)–C(11)–C(7)	162.8 (6)
C(4)–C(5)–C(6)–O(2)	113.5 (5)	C(7)–C(8)–C(9)–C(10)	75.2 (5)	O(1)–C(12)–C(11)–C(13)	37.3 (8)
C(5)–C(6)–C(7)–C(8)	86.0 (5)	C(7)–C(8)–C(9)–O(3)	–167.3 (4)	C(9)–O(3)–C(16)–O(4)	2.9 (7)
C(5)–C(6)–C(7)–C(11)	–152.9 (4)	C(8)–C(9)–C(10)–C(1)	–114.8 (5)	C(9)–O(3)–C(16)–C(17)	–177.6 (4)
O(2)–C(6)–C(7)–C(8)	–154.7 (4)	C(8)–C(9)–C(10)–C(15)	60.5 (5)		

Fig. 2. Packing diagram projected down *b*; H atoms are omitted for clarity.

C(10) 127.0, C(6)–C(7)–C(8) 117.8, C(3)–C(4)–C(5)–C(6) 157.2°. This is probably due to ring strain, there being two *trans* double bonds in the ten-membered ring. Similar angles are observed in her-

bolide B (Hull & Kennard, 1978), in which there is a C(1)–C(10) epoxide ring.

There are no non-bonded contacts between non-H atoms <3.4 Å.

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References

- HULL, S. E. & KENNARD, O. (1978). *Cryst. Struct. Commun.* **7**, 85–90.
 KLYNE, W. & PRELOG, V. (1960). *Experientia*, **16**, 521–523.
 SEGAL, R., SOKOLOFF, S., HARAN, B., ZAITSCHEK, D. V. & LICHTENBERG, D. (1977). *Phytochemistry*, **16**, 1237–1241.

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Structure of 3-(7-Adeninyl)propionic Acid Lactam

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Abstract. C₈H₇N₅O, FW = 189, orthorhombic, *Pbca*, *a* = 14.006 (2), *b* = 6.751 (1), *c* = 16.442 (2) Å, *D_m* (floatation) = 1.61₆, *D_x* = 1.616 Mg m^{–3}, *Z* = 8, *R* = 0.053 for 1499 reflexions (Mo *K*α). As compared with the adenine derivatives alkylated at N(9), the N(7)–C(8) bond is lengthened, the C(8)–N(9) bond is

shortened, the C(5)–N(7)–C(8) and C(5)–C(4)–N(9) angles are expanded, and the C(4)–N(9)–C(8) and C(4)–C(5)–N(7) angles are contracted.

Introduction. In the preparation of 3-(9-adeninyl)propionic acid (I), we have found that the reaction

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reported by Kondo, Miyata & Takemoto (1971) gives a by-product which is deposited as hexagonal plates after (I) has crystallized. An elemental analysis indicated it to be the anhydride of (I). To determine the structure of the by-product, an X-ray analysis has been performed.

A crystal $0.2 \times 0.2 \times 0.6$ mm was used for data collection on a Rigaku computer-controlled four-circle diffractometer, with graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0.71069$ Å). The unit-cell dimensions were determined with 30 high-angle reflexions. Intensities were measured in the ω - 2θ scan mode with a scan width of 1.00° (in ω) plus $\alpha_1 - \alpha_2$ divergence, and a scan speed of $4^\circ (2\theta) \text{ min}^{-1}$. Five reference reflexions showed no significant intensity variations throughout the data collection. Of 1787 independent reflexions in the range $2 \leq 2\theta \leq 55^\circ$, 410 weak reflexions below background were considered zero reflexions. The data were corrected for Lorentz and polarization factors but not for absorption effects.

The structure was solved by the direct method and its parameters were refined by a block-diagonal least-squares technique. All the H atoms, found on a difference map, were included. The quantity minimized was $\sum w(|F_o| - |F_c|)^2$, with $w = 1/(\sigma_p^2 + qF_o^2)$; σ_p is from counting statistics and q (3.66×10^{-4}) is derived from the variation of the monitored reflexions. The zero reflexions were included in the least-squares calculation by assuming $F_o = F_{\text{lim}}$ and $w = w(F_{\text{lim}})$, where F_{lim} is 1.91, an observational threshold value; those for which $|F_c| < F_{\text{lim}}$ were omitted. The final R was 0.066 ($R = 0.053$ for $F_o > 3/\sqrt{w}$); the maximum shift of parameters in the last cycle was 0.02σ for C, N and O, and 0.23σ for H atoms. Atomic scattering factors used were

Table 1. Atomic coordinates ($\times 10^5$, for H $\times 10^4$) with their estimated standard deviations in parentheses

	x	y	z
N(1)	21952 (12)	13687 (31)	55104 (10)
C(2)	16101 (17)	15925 (39)	61566 (13)
N(3)	6884 (13)	20697 (31)	61667 (11)
C(4)	3305 (15)	23185 (33)	54140 (13)
C(5)	8714 (15)	21016 (31)	47077 (12)
C(6)	18300 (14)	16322 (32)	47694 (13)
N(7)	2602 (12)	24935 (29)	40704 (11)
C(8)	-5960 (15)	29060 (35)	44132 (14)
N(9)	-5961 (12)	28305 (29)	52186 (11)
N(6)	24603 (13)	14727 (29)	41231 (10)
C(11)	5092 (16)	24289 (37)	32061 (13)
C(12)	12363 (16)	7848 (36)	30526 (13)
C(13)	22545 (14)	11893 (33)	33143 (13)
O(13)	29106 (11)	11995 (29)	28220 (9)
H(2)	1903 (15)	1350 (34)	6707 (13)
H(6)	3096 (16)	1562 (36)	4302 (13)
H(8)	-1167 (15)	3212 (32)	4069 (13)
H(11A)	761 (15)	3792 (35)	3012 (12)
H(11B)	-69 (14)	2109 (33)	2924 (11)
H(12A)	1286 (15)	551 (33)	2468 (13)
H(12B)	1020 (15)	-417 (33)	3316 (13)

taken from *International Tables for X-ray Crystallography* (1974). Atomic parameters are listed in Table 1.*

Discussion. As shown in Fig. 1, the 2-carboxyethyl group is attached, contrary to expectation, to N(7) of the adenine, and a lactam is formed with the amino group N(6).

As compared with the adenine derivatives alkylated at N(9) (Voet & Rich, 1970; Kistenmacher & Rossi, 1977; Ohki, Takenaka, Shimanouchi & Sasada, 1977*a,b,c*), the N(7)-C(8) bond is lengthened, the C(8)-N(9) bond is shortened, the C(5)-N(7)-C(8) and C(5)-C(4)-N(9) angles are expanded, and the C(4)-N(9)-C(8) and C(4)-C(5)-N(7) angles are contracted. A similar trend is observed in the purine structure (Watson, Sweet & Marsh, 1965), in which the H atom is bonded to N(7).

The purine ring of the present compound is planar within 0.012 Å but the amino N(6) deviates -0.072 Å from the ring plane ($-0.2397X + 0.9701Y + 0.0390Z = 0.255$, where X, Y and Z are in Å along the crystal axes). This large deviation is due to the formation of the lactam ring.

The torsion angles of the seven-membered lactam ring are C(5)-C(6)-N(6)-C(13) -19.8 (4), C(6)-N(6)-C(13)-C(12) -6.8 (3), N(6)-C(13)-C(12)-C(11) 63.6 (3), C(13)-C(12)-C(11)-N(7) -75.5 (2), and C(12)-C(11)-N(7)-C(5) 34.7 (3) $^\circ$. The adenine part of this ring causes the C(11)-C(12)-C(13) and C(6)-N(6)-C(13) angles to expand. The amide group takes a *cis* conformation, the torsion angle

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34249 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

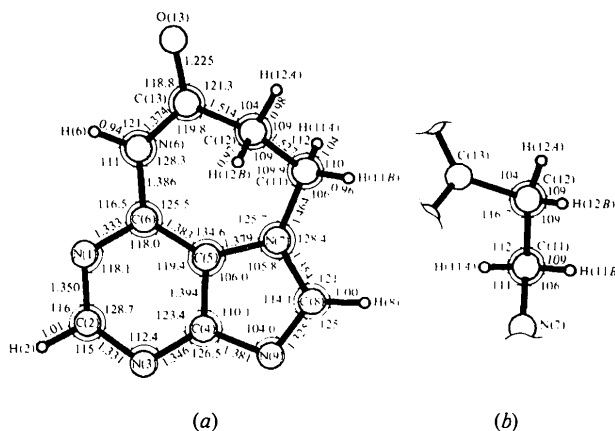


Fig. 1. 3-(7-Adeniny)propionic acid lactam, showing (a) the atom-numbering scheme, bond distances (Å) and angles ($^\circ$), and (b) the bond angles which are omitted from (a) for clarity. E.s.d.'s are 0.003 Å and 0.2° for C, N and O; those involving H are 0.02 Å and $0.1-0.2^\circ$.

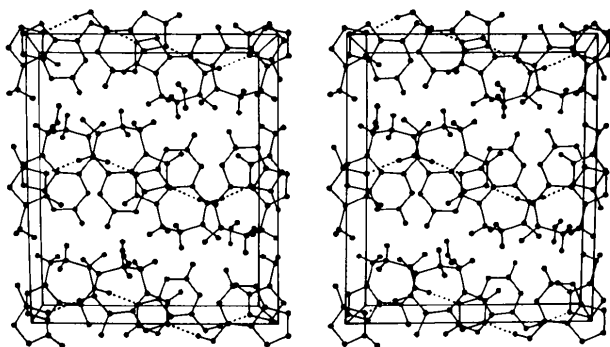


Fig. 2. Stereoscopic diagram showing the molecular packing viewed along the *b* axis. For the origin at the upper rear left-hand corner, *a* is to the right, *c* is down and *b* is out of the paper.

Table 2. Hydrogen-bond geometry

N(6)⋯N(9 ⁱ)	2.967 (3) Å	H(6)⋯N(9 ⁱ)	2.035 (23) Å
C(6)—N(6)⋯N(9 ⁱ)	107.0 (1)°	H(6)—N(6)⋯N(9 ⁱ)	6.4 (14)°
C(13)—N(6)⋯N(9 ⁱ)	124.6 (1)	N(6)⋯N(9 ⁱ)⋯H(6)	2.9 (6)
N(6)⋯N(9 ⁱ)—C(4 ⁱ)	144.8 (1)	H(6)⋯N(9 ⁱ)—C(4 ⁱ)	143.7 (7)
N(6)⋯N(9 ⁱ)—C(8 ⁱ)	111.0 (1)	H(6)⋯N(9 ⁱ)—C(8 ⁱ)	112.3 (7)
N(6)—H(6)⋯N(9 ⁱ)	170.6 (20)		

Symmetry code: superscript: none *x, y, z*; (i) $-\frac{1}{2} + x, \frac{1}{2} - y, 1 - z$.

of C(6)—N(6)—C(13)—O(13) being 175.8 (2)°. Although this conformation is similar to that of ϵ -caprolactam (Winkler & Dunitz, 1975), shortening of C(13)—O(13) and lengthening of C(13)—N(6) are observed.

The crystal structure is shown in Fig. 2. Hydrogen-bond distances and angles are given in Table 2. The N(6)—H(6) group is the donor for a hydrogen bond to N(9) of the neighbouring molecule related by 2₁

symmetry along the *a* axis. The carbonyl oxygen, O(13), of the amide group does not participate in hydrogen bonding.

Parallel adenine moieties approach each other across inversion centres. The spacings are 3.323 and 3.226 Å. For the latter spacing, the shortest contact (3.258 Å) is between C(4) and C(8).

Figs. 1 and 2 were drawn by *TSD:XTAL*, which is a computer-graphics interactive modelling program for the Nova 3 computer (Takenaka & Sasada, 1978). The present work was supported by a Grant-in-Aid for Scientific Research from the Ministry of Education and by the Kawakami Foundation, to which the authors' thanks are due.

References

- International Tables for X-ray Crystallography* (1974). Vol. IV, pp. 71–147. Birmingham: Kynoch Press.
- KISTENMACHER, T. J. & ROSSI, M. (1977). *Acta Cryst.* **B33**, 253–256.
- KONDO, K., MIYATA, M. & TAKEMOTO, K. (1971). *Bull. Chem. Soc. Jpn.* **44**, 2554–2555.
- OHKI, M., TAKENAKA, A., SHIMANOUCI, H. & SASADA, Y. (1977a). *Acta Cryst.* **B33**, 2954–2956.
- OHKI, M., TAKENAKA, A., SHIMANOUCI, H. & SASADA, Y. (1977b). *Acta Cryst.* **B33**, 2956–2958.
- OHKI, M., TAKENAKA, A., SHIMANOUCI, H. & SASADA, Y. (1977c). *Bull. Chem. Soc. Jpn.* **50**, 2573–2578.
- TAKENAKA, A. & SASADA, Y. (1978). Annual Meeting Crystallogr. Soc. Jpn, Hiroshima. Abstracts, p. 32.
- VOET, D. & RICH, A. (1970). *Prog. Nucleic Acid Res. Mol. Biol.* **10**, 183–265.
- WATSON, D. G., SWEET, R. M. & MARSH, R. E. (1965). *Acta Cryst.* **19**, 573–580.
- WINKLER, F. K. & DUNITZ, J. D. (1975). *Acta Cryst.* **B31**, 268–269.

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5,6-Dimethyl-1-(α -D-ribofuranosyl)benzimidazole

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Abstract. C₁₄H₁₈N₂O₄, *M_r* = 278.31, monoclinic *P*2₁, *a* = 10.849 (3), *b* = 5.460 (3), *c* = 11.717 (6) Å, β = 103.94 (1)°, *Z* = 2, Cu *K* α , λ = 1.54178 Å. Final *R* = 0.067 for 1241 diffractometer data. The molecule is in the *syn* conformation. The ribose moiety has the C(3')-*endo*, ³*T*₂ conformation. The staggered conformation about C(4')—C(5') is *trans*. The bases form a stacked

column along *b* with an interplanar base separation of 5.460 Å. The molecules are connected by O(2')—H(O2')⋯N(9) [2.701 (6) Å] hydrogen bonds.

Introduction. C₁₄H₁₈N₂O₄ (ribazole) is of biological interest since its nucleotide counterpart occurs as a fragment of vitamin B₁₂. The aim of this structure deter-