

Fig. 2. Molecular deformation of 3-filicanone (scale divisions in Å).

C(3), and C(23) is 0.18 (2) Å out of the plane. The C(3)—O distance is 1.23 (2) Å, and thus of normal length.

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## Structure of (4*S*)-2,4-Dimethyl-1,2-dihydropyrazino[2,1-*b*]quinazoline-3(4*H*),6-dione

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**Abstract.** C<sub>13</sub>H<sub>13</sub>N<sub>3</sub>O<sub>2</sub>, orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, *a* = 17.443 (5), *b* = 11.650 (4), *c* = 5.784 (1) Å, *Z* = 4, *d<sub>m</sub>* = 1.456, *d<sub>c</sub>* = 1.429 Mg m<sup>-3</sup>, *F*(000) = 512, μ(Cu *K*α) = 0.843 mm<sup>-1</sup>. The *R* index is 0.040 for 1358 significant reflections. The structure is stabilized by C—H...O interactions. The *N*-methylated *cis* peptide group which forms part of a six-membered ring is non-planar. The torsion angle about the peptide bond is -6.1 (4)° and the peptide bond length is 1.337 (3) Å.

**Introduction.** *N*-Methylated peptides occur in cyclic antibiotics where they introduce significant conformational changes. The structure analysis of the present compound has been undertaken with a view to studying (i) the effect of *N*-methylation on the geometry of the amide group and (ii) the packing of the molecules where the amide proton, which can normally participate in hydrogen bonding, is absent. NMR studies (Rajappa & Advani, 1974) have earlier indicated the presence of an axial methyl group in this compound.

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The title compound, a tricyclic quinazolone (Fig. 1), was derived from *cyclo*(-Sar-L-Ala-) (Rajappa & Advani, 1974) and was crystallized from ethyl acetate/hexane solutions. The unit-cell dimensions were initially obtained from photographs and then refined by least-squares calculation based on 25 reflections on a Nonius CAD-4 diffractometer. Intensity data were collected in the ω-2θ scan mode using monochromated Cu *K*α radiation (λ = 1.5418 Å) with a crystal of dimensions 0.6 × 0.2 × 0.1 mm. Of the

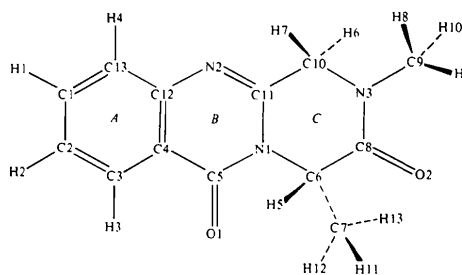


Fig. 1. The numbering of the atoms.

1447 reflections observed up to  $\theta = 75^\circ$ , 1358 were considered significant [ $|F| > 2\sigma(|F|)$ ]. No correction was made for absorption ( $\mu r \leq 0.2$ ).

The structure was solved by direct methods with *MULTAN* (Main, Woolfson & Germain, 1971) and was refined by block-diagonal least squares to an *R* index of 0.084. At this stage all the H atoms were located from a difference map. Refinement including H atoms converged with *R* = 0.040.\* The quantity

\* Lists of structure factors, anisotropic thermal parameters, C...O contacts, and distances and angles involving H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36620 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

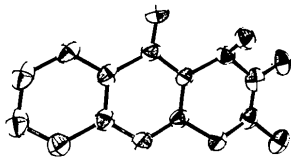


Fig. 2. A diagram of the molecule showing the thermal ellipsoids.

Table 1. Fractional coordinates ( $\times 10^4$ ) and isotropic temperature factors with *e.s.d.*'s in parentheses

For non-H atoms  $U_{eq} = 1/(6\pi^2) \sum_i \sum_j \beta_{ij} a_i \cdot a_j$ .

	x	y	z	$U_{eq}/U$ ( $\text{\AA}^2$ )
O(1)	5344 (1)	4058 (1)	1562 (4)	0.060 (1)
O(2)	5123 (1)	1302 (2)	7863 (3)	0.064 (1)
N(1)	5863 (1)	2311 (1)	2369 (4)	0.039 (1)
N(2)	6910 (1)	1531 (2)	320 (4)	0.044 (1)
N(3)	5999 (1)	475 (2)	5514 (4)	0.048 (1)
C(1)	7409 (2)	3484 (2)	-4425 (5)	0.059 (1)
C(2)	6883 (2)	4371 (2)	-4119 (5)	0.055 (1)
C(3)	6368 (1)	4332 (2)	-2304 (5)	0.050 (1)
C(4)	6380 (1)	3395 (2)	-779 (4)	0.042 (1)
C(5)	5828 (1)	3326 (2)	1105 (5)	0.043 (1)
C(6)	5272 (1)	2164 (2)	4193 (4)	0.045 (1)
C(7)	4495 (2)	1869 (3)	3121 (6)	0.060 (1)
C(8)	5471 (2)	1268 (2)	6019 (4)	0.049 (1)
C(9)	6106 (2)	-524 (3)	7020 (5)	0.062 (2)
C(10)	6331 (2)	372 (2)	3234 (5)	0.049 (1)
C(11)	6387 (1)	1466 (2)	1913 (4)	0.039 (1)
C(12)	6905 (1)	2499 (2)	-1084 (4)	0.041 (1)
C(13)	7425 (2)	2565 (2)	-2936 (5)	0.054 (1)
H(1)	7813 (18)	3491 (23)	-5765 (62)	0.057 (8)
H(2)	6929 (17)	5079 (23)	-5082 (57)	0.054 (8)
H(3)	5999 (17)	4930 (23)	-2114 (54)	0.047 (8)
H(4)	7841 (17)	1952 (24)	-3225 (55)	0.053 (8)
H(5)	5189 (15)	2909 (22)	4895 (53)	0.041 (7)
H(6)	6016 (16)	-165 (21)	2427 (53)	0.039 (7)
H(7)	6883 (21)	94 (25)	3380 (62)	0.066 (9)
H(8)	6691 (20)	-601 (32)	7475 (77)	0.068 (10)
H(9)	5725 (44)	-264 (54)	8356 (13)	0.188 (31)
H(10)	5896 (21)	-1234 (31)	6371 (75)	0.092 (1)
H(11)	4101 (21)	1750 (32)	4334 (76)	0.085 (1)
H(12)	4288 (20)	2479 (26)	1949 (68)	0.079 (1)
H(13)	4491 (21)	1103 (26)	2317 (70)	0.072 (10)

minimized was  $\sum w(|F_o| - |F_c|)^2$  where  $w = 1/(a + b|F_o| + c|F_o|^2)$  (Cruickshank, 1961) with  $a = 1.815$ ,  $b = -0.139$  and  $c = 0.019$ . Final parameters of the non-H and H atoms are listed in Table 1. The thermal vibrational ellipsoids are shown in Fig. 2.

**Discussion.** The bond lengths (Table 2) and bond angles (Table 3) of the peptide unit are comparable to the values quoted for the *cis* peptide unit (Ramachandran & Sasisekharan, 1968). The peptide unit is non-planar with  $\Delta\omega = -6.1 (4)^\circ$ ,  $\theta_N = 18.5 (2)^\circ$  and  $\theta_C = 0.9 (2)^\circ$  (Ramachandran, Lakshminarayanan & Kolaskar, 1973).

The deviation of the amide nitrogen N(3) from the plane through C(9), C(10) and C(8) is 0.128 (3) Å and that of C(8) from the plane through N(3), O(2) and C(6) is 0.006 (3) Å. According to Ramachandran *et al.* (1973), Dunitz & Winkler (1975) and Kolaskar, Lakshminarayanan, Sarathy & Sasisekharan (1975), the pyramidal distortions at N of the peptide unit are much larger than those at C', a feature also found in the present structure.

The methyl group C(7) in ring C is in the axial position as can be seen from the torsion angles given in Table 4. This conformation has been predicted by NMR studies and is presumably preferred so that the unfavourable interaction between atoms O(1) and C(7), which exists with C(7) in an equatorial position, is avoided.

Table 2. Bond lengths (Å) involving non-H atoms

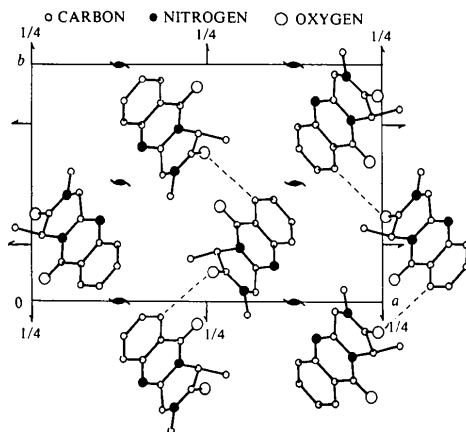
O(1)-C(5)	1.228 (3)	C(1)-C(2)	1.394 (4)
O(2)-C(8)	1.228 (3)	C(1)-C(13)	1.375 (4)
N(1)-C(5)	1.391 (3)	C(2)-C(3)	1.382 (4)
N(1)-C(6)	1.486 (3)	C(3)-C(4)	1.404 (4)
N(1)-C(11)	1.369 (3)	C(4)-C(5)	1.457 (3)
N(2)-C(11)	1.299 (3)	C(4)-C(12)	1.400 (3)
N(2)-C(12)	1.389 (3)	C(6)-C(7)	1.529 (4)
N(3)-C(8)	1.337 (3)	C(6)-C(8)	1.525 (4)
N(3)-C(9)	1.466 (4)	C(10)-C(11)	1.489 (3)
N(3)-C(10)	1.445 (3)	C(12)-C(13)	1.406 (4)

Table 3. Bond angles ( $^\circ$ ) involving non-H atoms

C(5)-N(1)-C(6)	116.1 (2)	N(1)-C(5)-C(4)	114.3 (2)
C(5)-N(1)-C(11)	122.7 (2)	N(1)-C(6)-C(7)	110.7 (2)
C(6)-N(1)-C(11)	121.2 (2)	N(1)-C(6)-C(8)	114.4 (2)
C(11)-N(2)-C(12)	117.2 (2)	C(7)-C(6)-C(8)	109.2 (2)
C(8)-N(3)-C(9)	120.4 (2)	O(2)-C(8)-N(3)	123.6 (2)
C(8)-N(3)-C(10)	122.2 (2)	O(2)-C(8)-C(6)	117.8 (2)
C(9)-N(3)-C(10)	115.2 (2)	N(3)-C(8)-C(6)	118.6 (2)
C(2)-C(1)-C(13)	120.7 (3)	N(3)-C(10)-C(11)	115.1 (2)
C(1)-C(2)-C(3)	120.0 (3)	N(1)-C(11)-N(2)	124.3 (2)
C(2)-C(3)-C(4)	119.6 (2)	N(1)-C(11)-C(10)	118.2 (2)
C(3)-C(4)-C(5)	120.3 (2)	N(2)-C(11)-C(10)	117.4 (2)
C(3)-C(4)-C(12)	120.7 (2)	N(2)-C(12)-C(4)	122.4 (2)
C(5)-C(4)-C(12)	119.0 (2)	N(2)-C(12)-C(13)	119.0 (2)
O(1)-C(5)-N(1)	120.5 (2)	C(4)-C(12)-C(13)	118.5 (2)
O(1)-C(5)-C(4)	125.2 (2)	C(1)-C(13)-C(12)	120.5 (3)

Table 4. Torsion angles (°) involving non-H atoms

C(6)–N(1)–C(5)–O(1)	2.3 (3)	C(11)–N(2)–C(12)–C(13)	–175.4 (2)	C(3)–C(4)–C(5)–N(1)	175.1 (2)
C(11)–N(1)–C(5)–O(1)	179.4 (2)	C(9)–N(3)–C(8)–O(2)	9.9 (4)	C(12)–C(4)–C(5)–N(1)	–3.0 (3)
C(6)–N(1)–C(5)–C(4)	–175.4 (2)	C(10)–N(3)–C(8)–O(2)	172.3 (2)	C(3)–C(4)–C(12)–N(2)	–176.8 (2)
C(11)–N(1)–C(5)–C(4)	1.7 (3)	C(9)–N(3)–C(8)–C(6)	–168.4 (2)	C(5)–C(4)–C(12)–N(2)	1.3 (3)
C(5)–N(1)–C(6)–C(7)	75.0 (3)	C(10)–N(3)–C(8)–C(6)	–6.1 (4)	C(3)–C(4)–C(12)–C(13)	0.6 (4)
C(11)–N(1)–C(6)–C(7)	–102.2 (2)	C(8)–N(3)–C(10)–C(11)	30.7 (3)	C(5)–C(4)–C(12)–C(13)	178.8 (2)
C(5)–N(1)–C(6)–C(8)	–161.1 (2)	C(9)–N(3)–C(10)–C(11)	–166.1 (2)	N(1)–C(6)–C(8)–O(2)	161.5 (2)
C(11)–N(1)–C(6)–C(8)	21.7 (3)	C(13)–C(1)–C(2)–C(3)	–0.1 (4)	C(7)–C(6)–C(8)–O(2)	–73.8 (3)
C(5)–N(1)–C(11)–N(2)	1.8 (3)	C(2)–C(1)–C(13)–C(12)	0.5 (4)	N(1)–C(6)–C(8)–N(3)	–20.1 (3)
C(6)–N(1)–C(11)–N(2)	178.8 (2)	C(1)–C(2)–C(3)–C(4)	–0.1 (4)	C(7)–C(6)–C(8)–N(3)	104.6 (3)
C(5)–N(1)–C(11)–C(10)	–174.7 (2)	C(2)–C(3)–C(4)–C(5)	–178.4 (2)	N(3)–C(10)–C(11)–N(1)	–28.3 (3)
C(6)–N(1)–C(11)–C(10)	2.3 (3)	C(2)–C(3)–C(4)–C(12)	–0.2 (4)	N(3)–C(10)–C(11)–N(2)	155.0 (2)
C(12)–N(2)–C(11)–N(1)	–3.7 (3)	C(3)–C(4)–C(5)–O(1)	–2.5 (4)	N(2)–C(12)–C(13)–C(1)	176.8 (2)
C(12)–N(2)–C(11)–C(10)	172.8 (2)	C(12)–C(4)–C(5)–O(1)	179.3 (2)	C(4)–C(12)–C(13)–C(1)	–0.8 (4)
C(11)–N(2)–C(12)–C(4)	2.1 (3)				

Fig. 3. The packing of the molecules as viewed down the *c* axis.

Of the three six-membered rings, *A* and *B* are essentially planar and ring *C* has a flattened boat conformation. The torsion angles of ring *C* are 31, –28, 2, 22, –20, –6°; the corresponding angles for the cyclohexane boat form are 54, –54, 0, 54, –54, 0° (Bucourt, 1974).

The molecular packing is shown in Fig. 3. The structure is stabilized by two intermolecular C–H···O interactions C(7)–H(13)···O(1) and C(3)–H(3)···O(2) with C···O = 3.283 (3), 3.483 (3) Å; ∠C–H···O = 137 (3), 162 (3)° respectively.

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