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## Structure of a Highly Potent Anxiolytic Dipeptoid

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**Abstract.**  $[R-(R^*,R^*)]$ -4-[[2-[[3-(1*H*-Indol-3-yl)-2-methyl-1-oxo-2-[[tricyclo[3.3.1.1<sup>3,7</sup>]dec-2-yloxy)carbonyl]amino]propyl]amino]-1-phenylethyl]amino]-4-oxobutanoic acid,  $C_{35}H_{42}N_4O_6$ ,  $M_r = 614.748$ , orthorhombic,  $P2_12_12_1$ ,  $a = 13.341$  (2),  $b = 13.643$  (2),  $c = 18.222$  (2) Å,  $V = 3316.61$  Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.24$ ,  $D_x = 1.23$  Mg m<sup>-3</sup>, Mo  $K\alpha$  radiation,  $\lambda = 0.71069$  Å,  $\mu = 0.050$  mm<sup>-1</sup>,  $F(000) = 1312$ , room temperature,  $R = 0.0497$  for 5121 observed reflections. The analysis confirms the expected (1*R*,17*R*) configuration. In addition, four hydrogen bonds (one intra- and three intermolecular) are identified.

**Experimental.** The dipeptoid (internal code CI-988, free acid) was synthesized in a ten-step synthesis starting from D-(–)-2-amino-2-phenylethanol and D- $\alpha$ -methyltryptophan methyl ester according to a procedure developed by Horwell *et al.* (1991). Prismatic colourless crystals (0.2 × 0.2 × 0.4 mm) were obtained by dissolving 300 mg of CI-988 (free acid) in 60 ml ethyl acetate at 348 K. The solution was cooled slowly from 345 to 293 K during 108 h (LAUDA RKS/RK20, LAUDA PM351). The crystals were dried in vacuum at 303 K.

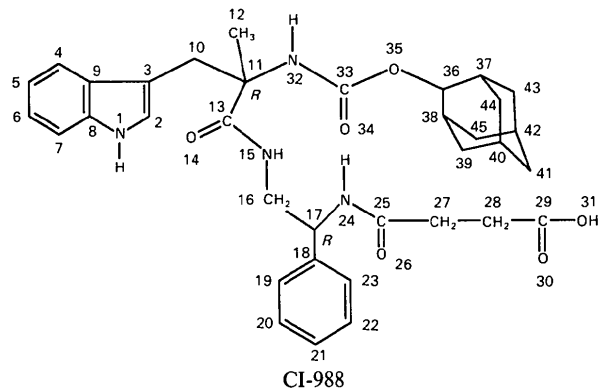


Table 1. Final fractional coordinates and equivalent isotropic thermal parameters (Å<sup>2</sup>)

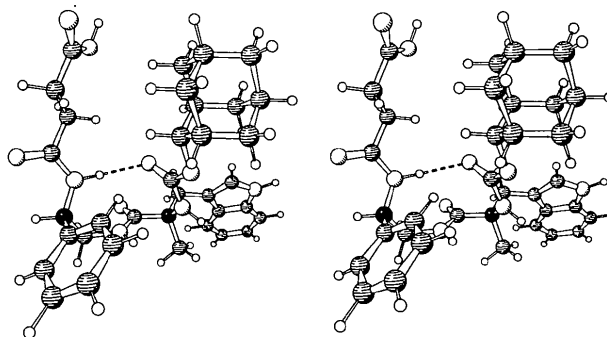
	x	y	z	$U_{eq}$
N(1)	0.6931 (1)	-0.2760 (1)	0.9902 (1)	0.055 (2)
C(2)	0.6200 (2)	-0.2078 (1)	0.9742 (1)	0.050 (2)
C(3)	0.5580 (1)	-0.1952 (1)	1.0330 (1)	0.038 (1)
C(4)	0.5633 (1)	-0.2832 (1)	1.1602 (1)	0.045 (2)
C(5)	0.6150 (2)	-0.3526 (2)	1.2001 (1)	0.057 (2)
C(6)	0.7005 (2)	-0.3999 (2)	1.1697 (1)	0.060 (2)
C(7)	0.7316 (1)	-0.3799 (1)	1.1005 (1)	0.054 (2)
C(8)	0.6794 (1)	-0.3094 (1)	1.0599 (1)	0.043 (2)
C(9)	0.5947 (1)	-0.2600 (1)	1.0894 (1)	0.037 (1)
C(10)	0.4695 (1)	-0.1275 (1)	1.0396 (1)	0.040 (1)
C(11)	0.4990 (1)	-0.0210 (1)	1.0601 (1)	0.034 (1)
C(12)	0.5671 (1)	-0.0197 (1)	1.1283 (1)	0.043 (2)
C(13)	0.4049 (1)	0.0396 (1)	1.0782 (1)	0.035 (1)
O(14)	0.3238 (1)	0.0003 (1)	1.0938 (1)	0.049 (1)
N(15)	0.4184 (1)	0.1371 (1)	1.0815 (1)	0.039 (1)
C(16)	0.3365 (2)	0.2039 (1)	1.1002 (1)	0.045 (2)
C(17)	0.3078 (1)	0.2711 (1)	1.0356 (1)	0.042 (2)
C(18)	0.3931 (2)	0.3438 (1)	1.0187 (1)	0.054 (2)
C(19)	0.4023 (4)	0.4350 (2)	1.0572 (2)	0.095 (5)
C(20)	0.4917 (4)	0.4924 (3)	1.0421 (3)	0.103 (5)
C(21)	0.5593 (3)	0.4657 (4)	0.9947 (2)	0.096 (5)
C(22)	0.5512 (2)	0.3826 (3)	0.9578 (2)	0.087 (4)
C(23)	0.4688 (2)	0.3211 (2)	0.9697 (2)	0.064 (3)
N(24)	0.2814 (1)	0.2130 (1)	0.9716 (1)	0.038 (1)
C(25)	0.2097 (1)	0.2400 (1)	0.9245 (1)	0.044 (2)
O(26)	0.1555 (1)	0.3120 (1)	0.9348 (1)	0.069 (2)
C(27)	0.2001 (2)	0.1786 (1)	0.8559 (1)	0.047 (2)
C(28)	0.2087 (3)	0.2440 (2)	0.7895 (1)	0.066 (3)
C(29)	0.2075 (2)	0.1938 (1)	0.7158 (1)	0.045 (2)
O(30)	0.2255 (2)	0.2377 (1)	0.6597 (1)	0.079 (2)
O(31)	0.1836 (2)	0.1010 (1)	0.7177 (1)	0.069 (2)
N(32)	0.5546 (1)	0.0246 (1)	0.9995 (1)	0.037 (1)
C(33)	0.5085 (1)	0.0505 (1)	0.9369 (1)	0.036 (1)
O(34)	0.4179 (1)	0.0441 (1)	0.9261 (0)	0.039 (1)
O(35)	0.5745 (1)	0.0871 (1)	0.8877 (0)	0.048 (1)
C(36)	0.5323 (1)	0.1159 (1)	0.8160 (1)	0.045 (2)
C(37)	0.6115 (3)	0.1803 (2)	0.7792 (1)	0.067 (3)
C(38)	0.5109 (1)	0.0262 (2)	0.7690 (1)	0.052 (2)
C(39)	0.6079 (2)	-0.0295 (2)	0.7528 (2)	0.068 (3)
C(40)	0.6824 (2)	0.0369 (2)	0.7129 (1)	0.064 (3)
C(41)	0.6374 (2)	0.0731 (3)	0.6413 (1)	0.071 (3)
C(42)	0.5431 (2)	0.1295 (3)	0.6572 (1)	0.072 (3)
C(43)	0.5634 (3)	0.2165 (2)	0.7063 (2)	0.087 (4)
C(44)	0.7059 (2)	0.1240 (3)	0.7631 (1)	0.073 (3)
C(45)	0.4664 (2)	0.0630 (3)	0.6963 (1)	0.081 (4)

$D_m$  was determined by the flotation method. Intensity data were collected with an automatic Philips PW1100 four-circle diffractometer with graphite monochromator, using  $2\theta$  scans to  $2\theta_{max} = 50^\circ$ . Cell parameters were determined by least-squares fit of 88

Table 2. Bond lengths (Å) and angles (°)

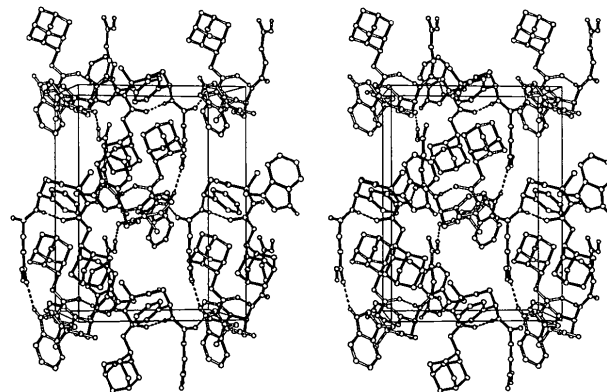
N(1)—C(2)	1.379 (4)	N(1)—C(8)	1.360 (4)
C(2)—C(3)	1.363 (4)	C(3)—C(10)	1.505 (3)
C(3)—C(9)	1.441 (3)	C(4)—C(5)	1.379 (4)
C(4)—C(9)	1.394 (3)	C(5)—C(6)	1.423 (4)
C(6)—C(7)	1.355 (5)	C(7)—C(8)	1.400 (4)
C(8)—C(9)	1.422 (3)	C(10)—C(11)	1.551 (3)
C(11)—C(12)	1.540 (3)	C(11)—C(13)	1.538 (3)
C(11)—N(32)	1.468 (3)	C(13)—N(15)	1.344 (3)
C(13)—O(14)	1.240 (3)	N(15)—C(16)	1.463 (3)
C(16)—C(17)	1.540 (3)	C(17)—C(18)	1.540 (4)
C(17)—N(24)	1.454 (3)	C(18)—C(19)	1.381 (5)
C(18)—C(23)	1.384 (5)	C(19)—C(20)	1.487 (7)
C(20)—C(21)	1.301 (7)	C(21)—C(22)	1.323 (7)
C(22)—C(23)	1.400 (5)	N(24)—C(25)	1.337 (3)
C(25)—C(27)	1.509 (4)	C(25)—O(26)	1.234 (3)
C(27)—C(28)	1.508 (4)	C(28)—C(29)	1.508 (4)
C(29)—O(30)	1.209 (3)	C(29)—O(31)	1.307 (3)
N(32)—C(33)	1.343 (3)	C(33)—O(34)	1.228 (3)
C(33)—O(35)	1.351 (3)	O(35)—C(36)	1.476 (3)
C(36)—C(37)	1.528 (4)	C(36)—C(38)	1.521 (4)
C(37)—C(44)	1.504 (5)	C(37)—C(43)	1.555 (5)
C(38)—C(45)	1.536 (4)	C(38)—C(39)	1.530 (4)
C(40)—C(39)	1.529 (5)	C(40)—C(41)	1.519 (5)
C(40)—C(44)	1.533 (5)	C(41)—C(42)	1.503 (5)
C(42)—C(43)	1.511 (6)	C(42)—C(45)	1.543 (5)
C(2)—N(1)—C(8)	109.2 (2)	N(1)—C(2)—C(3)	110.3 (2)
C(9)—C(3)—C(10)	125.8 (2)	C(2)—C(3)—C(10)	128.0 (2)
C(2)—C(3)—C(9)	106.1 (2)	C(5)—C(4)—C(9)	119.6 (2)
C(4)—C(5)—C(6)	120.5 (3)	C(5)—C(6)—C(7)	121.1 (3)
C(6)—C(7)—C(8)	118.6 (3)	N(1)—C(8)—C(7)	131.1 (2)
N(1)—C(8)—C(9)	107.5 (2)	C(7)—C(8)—C(9)	121.4 (2)
C(3)—C(9)—C(4)	134.3 (2)	C(3)—C(9)—C(8)	106.9 (2)
C(4)—C(9)—C(8)	118.8 (2)	C(3)—C(10)—C(11)	113.3 (2)
C(10)—C(11)—C(12)	110.8 (2)	C(12)—C(11)—C(13)	107.6 (2)
N(32)—C(11)—C(13)	110.2 (2)	C(10)—C(11)—C(13)	110.4 (2)
C(12)—C(11)—N(32)	107.7 (2)	C(10)—C(11)—N(32)	110.1 (2)
C(11)—C(13)—N(15)	115.6 (2)	O(14)—C(13)—N(15)	122.3 (2)
C(11)—C(13)—O(14)	121.9 (2)	C(13)—N(15)—C(16)	121.8 (2)
N(15)—C(16)—C(17)	112.3 (2)	N(24)—C(17)—C(16)	110.4 (2)
C(16)—C(17)—C(18)	110.7 (2)	N(24)—C(17)—C(18)	111.7 (2)
C(23)—C(18)—C(17)	121.6 (2)	C(23)—C(18)—C(19)	117.0 (3)
C(17)—C(18)—C(19)	121.1 (3)	C(20)—C(19)—C(18)	117.7 (4)
C(21)—C(20)—C(19)	121.3 (4)	C(20)—C(21)—C(22)	121.4 (5)
C(21)—C(22)—C(23)	120.0 (4)	C(22)—C(23)—C(18)	122.6 (3)
C(25)—N(24)—C(17)	122.6 (2)	N(24)—C(25)—C(27)	116.0 (2)
O(26)—C(25)—C(27)	121.3 (2)	N(24)—C(25)—O(26)	122.7 (2)
C(25)—C(27)—C(28)	109.2 (2)	C(27)—C(28)—C(29)	116.4 (2)
O(31)—C(29)—C(28)	114.8 (2)	O(30)—C(29)—C(28)	121.8 (3)
O(30)—C(29)—O(31)	123.4 (2)	C(11)—N(32)—C(33)	121.3 (2)
N(32)—C(33)—O(34)	124.6 (2)	N(32)—C(33)—O(35)	111.3 (2)
O(34)—C(33)—O(35)	124.1 (2)	C(33)—O(35)—C(36)	115.9 (2)
O(35)—C(36)—C(37)	106.2 (2)	C(37)—C(36)—C(38)	110.2 (2)
O(35)—C(36)—C(38)	110.9 (2)	C(43)—C(37)—C(36)	105.8 (3)
C(44)—C(37)—C(36)	111.8 (3)	C(43)—C(37)—C(44)	110.0 (3)
C(45)—C(38)—C(36)	107.2 (3)	C(45)—C(38)—C(39)	108.9 (3)
C(36)—C(38)—C(39)	110.4 (2)	C(40)—C(39)—C(38)	110.3 (3)
C(44)—C(40)—C(39)	107.9 (3)	C(41)—C(40)—C(39)	110.2 (3)
C(41)—C(40)—C(44)	110.0 (3)	C(40)—C(41)—C(42)	109.4 (3)
C(41)—C(42)—C(43)	111.5 (3)	C(43)—C(42)—C(45)	107.9 (3)
C(41)—C(42)—C(45)	110.0 (3)	C(42)—C(43)—C(37)	109.2 (3)
C(40)—C(44)—C(37)	109.9 (3)	C(42)—C(45)—C(38)	109.6 (3)

The structure was solved by direct methods using the *XY* procedure (Debaerdemaeker & Woolfson, 1989) in *MULTAN87* (Debaerdemaeker, Germain, Main, Tate & Woolfson, 1987). The structure refinement was carried out for both enantiomers with isotropic thermal parameters. At that stage the present enantiomer was chosen because its *R* value was 0.41% better than the other one (*R* = 7.86%, *wR* = 8.51% and *R* = 8.27%, *wR* = 8.99% respectively). Least-squares refinement used *SHELX76* (Sheldrick, 1976) with anisotropic thermal parameters for non-H atoms; H-atom positions were determined by difference Fourier synthesis and refined with isotropic thermal parameters. The atomic parameters were refined until the shifts were less than their corresponding standard deviation;  $(\Delta/\sigma)_{\max} = 0.04$ ;  $(\Delta\rho)_{\max} = 0.8$ ,  $(\Delta\rho)_{\min} = 0.7 \text{ e } \text{Å}^{-3}$ . Final *R* = 4.97%, *wR* = 5.51%  $\{w = 1.0/[\sigma^2(F) + 0.019399F^2]\}$  for 575

Fig. 1. Stereoview (*SCHAKAL*; Keller, 1988) with intramolecular hydrogen bond N(24)—H...O(34)Table 3. Intra- and intermolecular hydrogen-bond lengths (Å, *e.s.d.* 0.004 Å)

Intramolecular		Intermolecular	
N(24)—H...O(34)	3.058	N(1)—H...O(30)	2.946 (1.0 - x, -0.5 + y, 1.5 - z)
		N(32)—H...O(26)	2.866 (0.5 + x, 0.5 - y, 2.0 - z)
		O(31)—H...O(14)	2.649 (0.5 - x, -y, -0.5 + z)

high-order reflections ( $10 < \theta < 25^\circ$ ). No absorption correction but Lorentz-polarization corrections were applied. Index range:  $-15 \leq h \leq 15$ ,  $k \leq 16$ ,  $l \leq 21$ . Three standard reflections, measured every 60 min, showed no significant change during data collection. 6324 reflections were measured, 6294 were unique, of which 5121 were observed with  $I \geq 3.0\sigma(I)$ .

Fig. 2. Stereoview (*SCHAKAL*; Keller, 1988) of the unit cell.

parameters refined. Atomic scattering factors were obtained from Cromer & Mann (1968).

The final positional parameters are given in Table 1,\* bond lengths and angles in Table 2. Inter- and intramolecular hydrogen bonds are listed in Table 3. Fig. 1 shows a stereoview of the molecule. The intermolecular hydrogen bonds are shown in Fig. 2.

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55468 (37 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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### 3-Cyano-3-dimethylamino-4-ethoxy-1-phenyl-2-azetidinone

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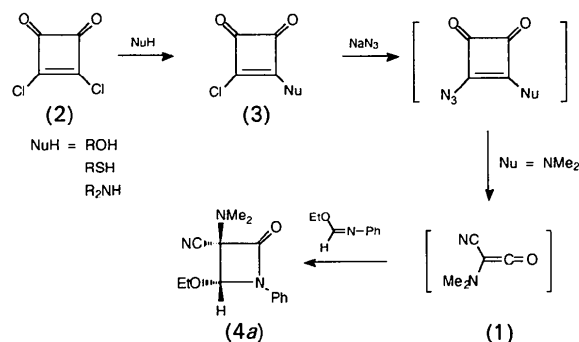
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**Abstract.** 3-Dimethylamino-4-ethoxy-2-oxo-1-phenyl-3-azetidincarbonitrile,  $C_{14}H_{17}N_3O_2$ ,  $M_r = 259.3$ , monoclinic,  $P2_1/n$ ,  $a = 6.105$  (2),  $b = 21.184$  (5),  $c = 11.356$  (7) Å,  $\beta = 91.14$  (3)°,  $V = 1468.4$  (11) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.17$  g cm<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.54178$  Å,  $\mu = 6.63$  cm<sup>-1</sup>,  $F(000) = 552$ ,  $T = 291$  K,  $R = 0.061$  for 1864 observed reflections. The azetidinone ring is nearly planar with endocyclic angles of 2, -2, 2 and -2° (maximum deviation from the plane: 0.015 Å for the N atom). The relative configurations at the chiral centres are *R,R* (or *S,S*).

**Experimental.** Captodative cyanoketenes (1) have become accessible from dichlorocyclobutenedione (2) (squaric acid dichloride) by two successive nucleophilic substitutions (Labille, Janousek & Viehe, 1991). Monochlorocyclobutenediones (3) treated with sodium azide decompose immediately with a loss of carbon monoxide and nitrogen to produce (1). Ketene intermediates (1) can be trapped with imidates to give novel  $\beta$ -lactam derivatives.



The title compound (4a) was obtained in 42% yield and has been characterized by <sup>1</sup>H, <sup>13</sup>C NMR, IR and MS. Its stereochemistry is now elucidated by X-ray analysis.

A parallelepiped crystal with dimensions 0.25 × 0.37 × 0.40 mm was used for data collection. Lattice parameters were refined using 17 reflections in the range 16 ≤ 2θ ≤ 64°. A Huber four-circle diffractom-