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Structure of (±)-*N*-[5-(*tert*-Butoxycarbonylamino)-2-cyano-1-cyclopenten-1-yl]glycine Benzyl Ester

BY JOHN C. DEWAN*

Department of Chemistry, Massachusetts Institute of Technology, Cambridge, MA 02139, *USA*

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Abstract. $C_{20}H_{25}N_3O_4$, $M_r = 371{\cdot}44$, triclinic, $P\bar{1}$, $a = 10{\cdot}002\ (3)$, $b = 11{\cdot}731\ (2)$, $c = 17{\cdot}516\ (3)$ Å, $\alpha = 97{\cdot}10\ (1)$, $\beta = 97{\cdot}31\ (2)$, $\gamma = 92{\cdot}23\ (2)°$, $V = 2019{\cdot}8$ Å^3, $Z = 4$, $D_x = 1{\cdot}221$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}51$ cm^{-1}, $F(000) = 792$, $T = 295$ K, final $R = 0{\cdot}058$ for 2734 unique observed reflections. The structure determination was undertaken to establish the stereochemistry of the compound. Two independent molecules, having the same stereochemistry but differing in orientation of the side-chain benzyl groups, appear in the asymmetric unit. The largest deviation of any of the atoms defining the plane C(112), C(113), N(114), C(115), N(116) in molecule 1 is 0·01 Å. Deviations of other atoms from this plane are C(19) −0·15, C(110) 0·27, and C(111) −0·06 Å. For molecule 2, the largest deviation of any of the defining atoms from the corresponding plane is also 0·01 Å. Deviations of other atoms are C(29) −0·07, C(210) −0·02, and C(211) −0·04 Å, indicating a difference in the puckering of the cyclopentene rings in these two independent molecules. Equivalent bond lengths and angles in the two molecules agree well.

Experimental. Colorless crystal, dimensions 0·18 × 0·25 × 0·29 mm, space group $P\bar{1}$, Enraf–Nonius CAD-4F-11 κ-geometry diffractometer, monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ scan technique, variable scan width where $\Delta\omega = (0{\cdot}8 + 0{\cdot}35\tan\theta)°$, scan rate 4° min^{-1} in ω, scan extended 25% on either side for background measurement, $3 < 2\theta < 45°$, lattice parameters from 25 reflections with $2\theta > 30°$, no absorption correction, three intensity standards showed no decay. Solved by direct methods followed by difference Fourier syntheses. 5268 reflections measured (h −10→10, k −12→12, l 0→18), 2734 with $I_o > 3\sigma(I_o)$ used in structure refinement on F using *SHELX*76 (Sheldrick, 1978), two blocks refined alternately with each containing the scale factor and one of the independent molecules (238 variables/block). All non-hydrogen atoms refined anisotropically, benzene rings refined as rigid groups (C–C = 1·395 Å, C–C–C = 120°), H atoms placed in calculated positions. Neutral-atom scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). Final $R = 0{\cdot}058$, $wR = 0{\cdot}067$.

Table 1. *Atom coordinates and equivalent isotropic temperature factors* (Å^2)

$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$.

	x	y	z	U_{eq}
C(11)	0·4525 (6)	0·6645 (4)	0·8966 (4)	0·081
C(12)	0·2367 (6)	0·5747 (5)	0·8227 (4)	0·093
C(13)	0·2971 (6)	0·5570 (5)	0·9646 (4)	0·087
C(14)	0·3491 (5)	0·5652 (4)	0·8884 (3)	0·052
O(15)	0·4270 (3)	0·4651 (3)	0·86760 (19)	0·054
C(16)	0·3748 (5)	0·3585 (4)	0·8543 (3)	0·045
O(17)	0·2556 (3)	0·3294 (3)	0·8529 (2)	0·062
N(18)	0·4711 (3)	0·2849 (3)	0·8411 (2)	0·048
C(19)	0·4499 (4)	0·1617 (4)	0·8382 (3)	0·044
C(110)	0·5110 (5)	0·1117 (4)	0·9101 (3)	0·059
C(111)	0·5402 (5)	−0·0107 (4)	0·8804 (3)	0·063
C(112)	0·5576 (4)	−0·0034 (4)	0·7973 (3)	0·041
C(113)	0·6130 (5)	−0·0952 (4)	0·7520 (3)	0·049
N(114)	0·6554 (4)	−0·1717 (4)	0·7173 (3)	0·068
C(115)	0·5111 (4)	0·0938 (4)	0·7732 (3)	0·037
N(116)	0·5030 (4)	0·1343 (3)	0·7045 (2)	0·052
C(117)	0·5219 (5)	0·0680 (5)	0·6321 (3)	0·063
C(118)	0·6697 (6)	0·0490 (5)	0·6238 (3)	0·058
O(119)	0·7655 (4)	0·0865 (3)	0·6681 (2)	0·072
O(120)	0·6720 (3)	−0·0200 (3)	0·5579 (2)	0·072
C(121)	0·8065 (6)	−0·0528 (6)	0·5404 (4)	0·102
C(122)	0·7842 (4)	−0·1594 (5)	0·4851 (3)	0·078
C(123)	0·8106 (4)	−0·1584 (5)	0·4089 (3)	0·116
C(124)	0·7918 (4)	−0·2595 (5)	0·3567 (3)	0·155
C(125)	0·7466 (4)	−0·3615 (5)	0·3808 (3)	0·171
C(126)	0·7201 (4)	−0·3624 (5)	0·4571 (3)	0·158
C(127)	0·7389 (4)	−0·2614 (5)	0·5092 (3)	0·104
C(21)	0·8845 (6)	0·5763 (5)	0·6973 (3)	0·076
C(22)	0·6638 (5)	0·4888 (5)	0·7186 (3)	0·074
C(23)	0·8166 (6)	0·6040 (5)	0·8288 (3)	0·076
C(24)	0·8089 (5)	0·5236 (4)	0·7545 (3)	0·051
O(25)	0·8858 (3)	0·4218 (3)	0·76810 (19)	0·056
C(26)	0·8543 (5)	0·3539 (4)	0·8205 (3)	0·047
O(27)	0·7487 (3)	0·3545 (3)	0·8483 (2)	0·066
N(28)	0·9552 (4)	0·2850 (3)	0·8374 (2)	0·051
C(29)	0·9510 (5)	0·2151 (4)	0·8997 (3)	0·050
C(210)	1·0396 (6)	0·2637 (4)	0·9753 (3)	0·068
C(211)	1·1287 (5)	0·1691 (4)	0·9997 (3)	0·060
C(212)	1·0963 (4)	0·0726 (4)	0·9344 (3)	0·042
C(213)	1·1635 (5)	−0·0316 (5)	0·9367 (3)	0·052
N(214)	1·2185 (5)	−0·1153 (4)	0·9415 (3)	0·080
C(215)	1·0003 (5)	0·0974 (4)	0·8792 (3)	0·043
N(216)	0·9414 (4)	0·0346 (4)	0·8129 (3)	0·056
C(217)	0·9801 (5)	−0·0780 (4)	0·7831 (3)	0·053
C(218)	1·1122 (5)	−0·0745 (5)	0·7489 (3)	0·055
O(219)	1·1704 (4)	0·0108 (4)	0·7386 (3)	0·091
O(220)	1·1496 (3)	−0·1806 (3)	0·7325 (2)	0·068
C(221)	1·2756 (5)	−0·1928 (6)	0·6994 (3)	0·083
C(222)	1·2509 (4)	−0·2343 (3)	0·61509 (19)	0·061
C(223)	1·1219 (4)	−0·2498 (3)	0·57322 (19)	0·104
C(224)	1·1059 (4)	−0·2824 (3)	0·49311 (19)	0·134
C(225)	1·2190 (4)	−0·2996 (3)	0·45489 (19)	0·109
C(226)	1·3481 (4)	−0·2841 (3)	0·49676 (19)	0·115
C(227)	1·3641 (4)	−0·2515 (3)	0·57686 (19)	0·090

* Present address: Department of Chemistry, New York University, New York, NY 10003, USA.

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

C(11)–C(14)	1·508 (7)	C(21)–C(24)	1·507 (8)
C(12)–C(14)	1·521 (8)	C(22)–C(24)	1·525 (7)
C(13)–C(14)	1·505 (9)	C(23)–C(24)	1·501 (7)
C(14)–O(15)	1·468 (6)	C(24)–O(25)	1·473 (6)
O(15)–C(16)	1·318 (6)	O(25)–C(26)	1·346 (6)
C(16)–O(17)	1·224 (6)	C(26)–O(27)	1·217 (6)
C(16)–N(18)	1·341 (6)	C(26)–N(28)	1·343 (6)
N(18)–C(19)	1·446 (6)	N(28)–C(29)	1·448 (7)
C(19)–C(110)	1·523 (7)	C(29)–C(210)	1·529 (7)
C(19)–C(115)	1·516 (7)	C(29)–C(215)	1·502 (6)
C(110)–C(111)	1·518 (7)	C(210)–C(211)	1·518 (8)
C(111)–C(112)	1·501 (7)	C(211)–C(212)	1·501 (6)
C(112)–C(113)	1·429 (7)	C(212)–C(213)	1·420 (7)
C(112)–C(115)	1·343 (7)	C(212)–C(215)	1·340 (6)
C(113)–N(114)	1·148 (7)	C(213)–N(214)	1·151 (8)
C(115)–N(116)	1·341 (7)	C(215)–N(216)	1·350 (6)
N(116)–C(117)	1·441 (6)	N(216)–C(217)	1·444 (6)
C(117)–C(118)	1·526 (8)	C(217)–C(218)	1·520 (7)
C(118)–O(119)	1·187 (6)	C(218)–O(219)	1·183 (7)
C(118)–O(120)	1·330 (7)	C(218)–O(220)	1·322 (7)
O(120)–C(121)	1·472 (7)	O(220)–C(221)	1·457 (7)
C(121)–C(122)	1·475 (8)	C(221)–C(222)	1·483 (6)
C(11)–C(14)–C(12)	110·8 (5)	C(21)–C(24)–C(22)	110·4 (4)
C(11)–C(14)–C(13)	110·3 (4)	C(21)–C(24)–C(23)	110·5 (4)
C(11)–C(14)–O(15)	102·9 (4)	C(21)–C(24)–O(25)	102·2 (4)
C(12)–C(14)–C(13)	112·7 (5)	C(22)–C(24)–C(23)	112·5 (4)
C(12)–C(14)–O(15)	109·4 (4)	C(22)–C(24)–O(25)	111·1 (4)
C(13)–C(14)–O(15)	110·3 (4)	C(23)–C(24)–O(25)	109·7 (4)
C(14)–O(15)–C(16)	123·5 (4)	C(24)–O(25)–C(26)	120·8 (4)
O(15)–C(16)–O(17)	125·6 (4)	O(25)–C(26)–O(27)	124·6 (4)
O(15)–C(16)–N(18)	110·3 (4)	O(25)–C(26)–N(28)	110·5 (4)
O(17)–C(16)–N(18)	124·1 (4)	O(27)–C(26)–N(28)	124·9 (5)
C(16)–N(18)–C(19)	123·0 (4)	C(26)–N(28)–C(29)	120·0 (4)
N(18)–C(19)–C(110)	115·3 (4)	N(28)–C(29)–C(210)	114·4 (4)
N(18)–C(19)–C(115)	113·5 (4)	N(28)–C(29)–C(215)	112·0 (4)
C(110)–C(19)–C(115)	103·2 (4)	C(210)–C(29)–C(215)	104·6 (4)
C(19)–C(110)–C(111)	104·7 (4)	C(29)–C(210)–C(211)	107·4 (4)
C(110)–C(111)–C(112)	102·9 (4)	C(210)–C(211)–C(212)	104·1 (4)
C(111)–C(112)–C(113)	120·8 (4)	C(211)–C(212)–C(213)	120·0 (4)
C(111)–C(112)–C(115)	112·1 (4)	C(211)–C(212)–C(215)	112·7 (4)
C(113)–C(112)–C(115)	127·0 (5)	C(213)–C(212)–C(215)	127·3 (4)
C(112)–C(113)–N(114)	177·5 (6)	C(212)–C(213)–N(214)	177·6 (5)
C(112)–C(115)–C(19)	109·2 (4)	C(212)–C(215)–C(29)	110·0 (4)
C(112)–C(115)–N(116)	132·6 (4)	C(212)–C(215)–N(216)	131·3 (4)
C(19)–C(115)–N(116)	118·1 (4)	C(29)–C(215)–N(216)	117·7 (4)
C(115)–N(116)–C(117)	125·0 (4)	C(215)–N(216)–C(217)	124·8 (4)
N(116)–C(117)–C(118)	113·5 (4)	N(216)–C(217)–C(218)	113·1 (4)
C(117)–C(118)–O(119)	127·0 (5)	C(217)–C(218)–O(219)	124·4 (5)
C(117)–C(118)–O(120)	107·3 (4)	C(217)–C(218)–O(220)	109·4 (5)
O(119)–C(118)–O(120)	125·7 (5)	O(219)–C(218)–O(220)	126·3 (5)
C(118)–O(120)–C(121)	115·8 (4)	C(218)–O(220)–C(221)	116·4 (4)
O(120)–C(121)–C(122)	106·1 (4)	O(220)–C(221)–C(222)	111·6 (4)
C(121)–C(122)–C(123)	119·8 (5)	C(221)–C(222)–C(223)	122·8 (4)
C(121)–C(122)–C(127)	120·2 (5)	C(221)–C(222)–C(227)	117·1 (4)
C(123)–C(122)–C(127)	120·0 (5)	C(223)–C(222)–C(227)	120·0 (3)

Weights given by $w = 0{\cdot}8150/[\sigma^2(F_o) + 0{\cdot}000625F_o^2]$. In final cycles maximum and average LS shift/e.s.d. 0·15 and 0·01, respectively. Final difference Fourier map maximum and minimum peaks 0·25 and $-0{\cdot}37$ e Å^{-3}, respectively. Further details of data collection, reduction, and refinement procedures given by Silverman, Dewan, Giandomenico & Lippard (1980). Table 1* gives atomic positional parameters and Table 2 bond lengths and angles. Fig. 1 shows the molecular geometry and atom-labeling scheme.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51254 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

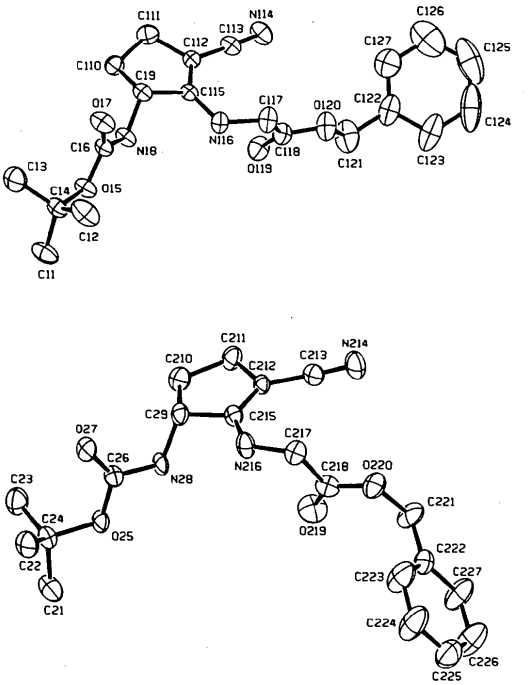
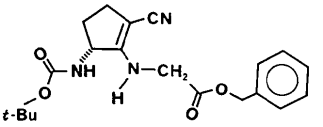


Fig. 1. *ORTEP*II diagrams (Johnson, 1976) of the two crystallographically independent molecules showing the atom-labeling scheme and 30% probability thermal ellipsoids. H atoms are omitted for clarity.

Related literature. The structure determination has established the stereochemistry of the compound, synthesized by Kemp & Carter (1987), to be that displayed in Fig. 1 and represented by the structural formula:



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