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Propionic Acid 1,2-Dimethyl-3-phenyl-3-pyrrolidynyl Ester Hydrochloride: Prodilidine

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Abstract. $C_{15}O_2NH_{21}$.HCl, monoclinic, $P2_1/c$, $a = 14{\cdot}321$ (5), $b = 8{\cdot}325$ (5), $c = 13{\cdot}717$ (5) Å, $\beta = 96{\cdot}93$ (5)°; $D_m = 1{\cdot}15$, $D_c = 1{\cdot}16$ g cm^{-3}, $Z = 4$. The orientation of the phenyl group to the pyrrolidine ring is equatorial.

Introduction. The title structure was solved as part of a study on the structure–activity relationships of narcotic analgesics.

Colourless prismatic crystals were obtained from a diethyl ether/acetone solution. The space group was determined from rotation and Weissenberg photographs. The cell dimensions and intensities were measured on a Nonius CAD-4 diffractometer with the experimental conditions given in Table 1.

The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971). The *E* map obtained from the second most probable set contained the whole structure. Full-matrix least-squares refinement was performed with the *SDP* program system (Okaya & Frenz, 1975). The positions of all H atoms were calculated and included in the last cycle of refinement, except those of the methyl groups. The final positional (Table 2) and thermal parameters converged to an R of 0·036.*

Table 1. *Experimental conditions*

Source: Cu $K\bar{\alpha}$ $\lambda = 1{\cdot}54178$ Å
Scan: ω–2θ
Graphite monochromator
Confidence level: $2{\cdot}5\sigma$, with $\sigma^2(I) = S + B + (0{\cdot}035)^2$
(S being the scan and B the background count)
$\Delta\theta = 0{\cdot}6 + 0{\cdot}3$ tg θ
$2{\cdot}0° \leq \theta \leq 65{\cdot}0°$
Aperture: $2{\cdot}5 + 0{\cdot}5$ tg θ
$t_{max} = 120$ s
Total number of independent reflexions: 2828
Number observed: 1048

Discussion. The atomic numbering, bond distances and angles are given in Fig. 1; Table 3 gives the bond

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32459 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

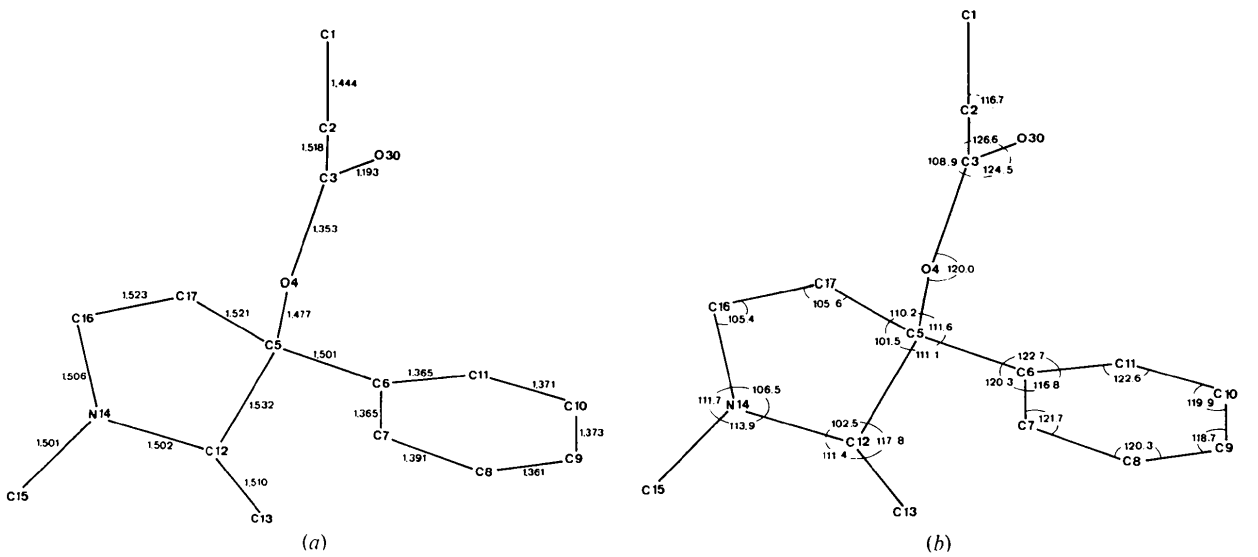

Fig. 1. (*a*) Bond distances (Å) and (*b*) angles (°). The maximum e.s.d.'s are 0·012 Å for the bond lengths and 1° for the angles.

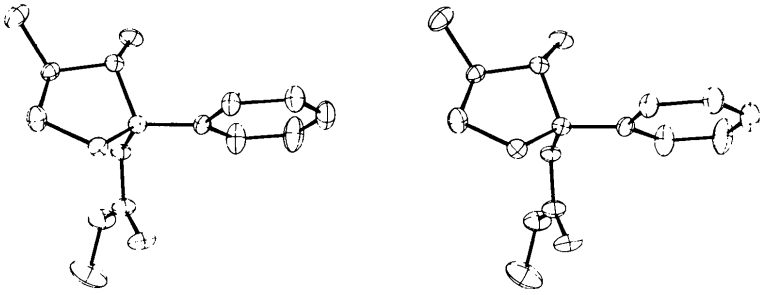

Fig. 2. Stereoscopic view of the molecule with 50% probability thermal ellipsoids (Johnson, 1965).

Table 2. *Final positional parameters* ($\times 10^4$) *with standard deviations in parentheses*

	x	*y*	*z*
C(1)	2724 (9)	2939 (9)	8842 (6)
C(2)	2821 (6)	4410 (9)	9314 (5)
C(3)	2771 (5)	4408 (6)	10396 (4)
O(30)	2891 (4)	3283 (5)	10935 (3)
O(4)	2558 (4)	5906 (4)	10681 (3)
C(5)	2394 (4)	6209 (7)	11708 (4)
C(6)	3296 (4)	6186 (7)	12391 (4)
C(7)	3281 (4)	6283 (7)	13382 (4)
C(8)	4105 (5)	6379 (7)	14029 (5)
C(9)	4953 (5)	6399 (10)	13683 (6)
C(10)	4979 (5)	6313 (13)	12687 (6)
C(11)	4158 (5)	6206 (11)	12064 (5)
C(12)	1957 (4)	7890 (7)	11649 (3)
C(13)	2463 (5)	9192 (7)	11167 (5)
N(14)	997 (3)	7587 (6)	11108 (3)
C(15)	291 (5)	8834 (9)	11274 (5)
C(16)	699 (4)	5940 (8)	11419 (4)
C(17)	1579 (4)	5193 (7)	11969 (4)
Cl(18)	741 (1)	7572 (2)	13903 (1)
H(11)	3291 (59)	2115 (102)	9200 (58)
H(12)	2051 (55)	2382 (107)	8982 (56)
H(13)	2768 (56)	2903 (103)	8063 (57)
H(21)	3500 (50)	4955 (95)	9155 (52)
H(22)	2264 (51)	5222 (95)	8937 (52)
H(7)	2669 (45)	6393 (84)	13607 (46)
H(8)	4062 (49)	6436 (92)	14814 (50)
H(9)	5573 (48)	6503 (94)	14161 (50)
H(10)	5597 (52)	6365 (98)	12464 (55)
H(11)	4171 (49)	6102 (89)	11419 (50)
H(12)	1853 (42)	8148 (77)	12368 (42)
H(131)	3183 (47)	9312 (86)	11532 (49)
H(132)	2507 (47)	8829 (87)	10395 (49)
H(133)	2163 (49)	10418 (88)	11120 (49)
H(151)	209 (50)	8951 (91)	12057 (50)
H(152)	535 (50)	10040 (92)	11033 (51)
H(153)	−414 (49)	8659 (95)	10874 (51)
H(161)	166 (46)	6043 (83)	11935 (45)
H(162)	572 (45)	5388 (84)	10783 (46)
H(171)	1509 (43)	5265 (79)	12698 (43)
H(172)	1667 (42)	4166 (77)	11893 (44)

distances and angles involving H atoms; Fig. 2 shows the conformation of the molecule.

This weakly active analgesic differs from the more potent compounds by having a pyrrolidine group instead of the usual piperidine ring. Nevertheless, it can be compared with alphaprodine (Kartha, Ahmed & Barnes, 1960). The equatorial orientation of the phenyl group relative to the piperidine or pyrrolidine rings contradicts the receptor theory of Beckett & Casy (1954).

Table 3. *Bond distances* (Å) *and angles* (°) *involving hydrogen atoms*

The e.s.d.'s are 0·05 Å for the bond lengths and 4° for the angles.

H(11)–C(1)	1·034	H(12)–C(1)	1·112
H(13)–C(2)	1·102	H(21)–C(2)	1·116
H(22)–C(2)	1·101	H(71)–C(7)	0·971
H(81)–C(8)	1·086	H(91)–C(9)	1·039
H(101)–C(10)	0·967	H(111)–C(11)	0·891
H(121)–C(12)	1·036	H(131)–C(13)	1·082
H(132)–C(13)	1·100	H(133)–C(13)	1·120
H(151)–C(15)	1·093	H(152)–C(15)	1·114
H(153)–C(15)	1·099	H(161)–C(16)	1·105
H(162)–C(16)	0·983	H(171)–C(17)	1·019
H(172)–C(17)	0·873		
H(11)–C(1)–C(2)	107·9	H(12)–C(1)–C(2)	109·2
H(13)–C(1)–C(2)	108·2	H(21)–C(2)–C(3)	109·9
H(22)–C(2)–C(3)	110·0	H(7)–C(7)–C(8)	121·5
H(8)–C(8)–C(9)	120·4	H(9)–C(9)–C(10)	120·5
H(10)–C(10)–C(11)	123·6	H(11)–C(11)–C(6)	117·0
H(12)–C(12)–C(5)	104·1	H(131)–C(13)–C(12)	110·0
H(132)–C(13)–C(12)	108·0	H(133)–C(13)–C(12)	109·2
H(151)–C(15)–N(14)	111·5	H(152)–C(15)–N(14)	109·7
H(153)–C(15)–N(14)	110·6	H(161)–C(16)–C(17)	108·0
H(162)–C(16)–C(17)	107·6	H(171)–C(17)–C(5)	111·6
H(172)–C(17)–C(5)	113·0		

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