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8-[(4-Methylphenyl)methyl]-1-phenyl-1,3,8-triazaspiro[4,5]decan-4-one

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Abstract. $C_{21}H_{25}N_3O$; monoclinic, $P2_1/c$; $a=18{\cdot}834$ (5), $b=7{\cdot}553$ (5), $c=12{\cdot}850$ (5) Å, $\beta=100{\cdot}21$ (5)°; $D_m=1{\cdot}22$, $D_c=1{\cdot}24$ g cm^{-3}; $Z=4$.

Introduction. This structure was solved as part of a study on the structure–activity relationship of narcotic analgesics. The title compound is ten times more potent than morphine.

Colourless prismatic crystals were obtained by slow evaporation of a solution in methanol. The space group was determined from precession photographs. The cell dimensions and intensities were measured on a CAD-4 automatic diffractometer. The experimental conditions are given in Table 1.

Table 1. *Experimental conditions*

Crystal dimensions: 0·36 × 0·25 × 0·32 mm
Source: Cu $K\alpha$, $\lambda=1{\cdot}54178$ 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
$\Delta\theta=0{\cdot}5+0{\cdot}3$ tg θ
$\theta_{min}=2{\cdot}0°$; $\theta_{max}=72{\cdot}0°$
Aperture: $2{\cdot}5+0{\cdot}5$ tg θ
$T_{max}=120$ s
Total number of independent reflexions: 3621
Number observed: 2757

The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971). The most probable E map contained the whole structure. Full-matrix least-squares refinement was performed with the X-RAY 72 system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). Nineteen H atoms located on a difference map were included in the last cycle of refinement together with the calculated positions of the remaining six H atoms. Only their positional parameters were refined resulting in an $R=0{\cdot}06$ for all observed reflexions. The scattering factors were those of Cromer & Mann (1968) except for H for which those of Stewart, Davidson &

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

$T=\exp[-2\pi^2(U_{11}h^2a^{*2}+\ldots+2U_{12}hka^*b^*+\ldots)]$ and $\exp[-2\pi^2U(2\sin\theta/\lambda)^2]$.

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(25)	2432 (1)	1187 (3)	−1836 (1)	792	694	324	−279	96	−107
N(9)	2375 (1)	31 (2)	941 (1)	309	307	322	24	78	15
N(15)	3415 (1)	4452 (3)	−242 (1)	628	456	245	−194	69	19
N(23)	3027 (1)	3779 (3)	−1965 (1)	506	519	256	−60	64	28
C(1)	−519 (2)	−1221 (5)	3144 (3)	563	759	1124	−23	434	184
C(2)	130 (1)	−1280 (4)	2591 (3)	450	501	731	−51	218	135
C(3)	823 (2)	−1224 (5)	3169 (2)	553	861	517	−138	162	134
C(4)	1420 (1)	−1340 (5)	2681 (1)	425	856	498	−133	65	155
C(5)	1343 (1)	−1469 (3)	1592 (2)	400	401	517	−84	99	69
C(6)	649 (1)	−1479 (4)	1003 (2)	454	658	513	−60	45	35
C(7)	52 (1)	−1397 (5)	1499 (3)	412	729	685	−30	52	96
C(8)	1992 (1)	−1655 (3)	1050 (2)	453	346	636	−43	173	4
C(10)	1899 (1)	1314 (3)	294 (2)	296	429	349	37	58	45
C(11)	2290 (1)	3054 (3)	204 (2)	406	363	346	71	107	36
C(12)	2994 (1)	2812 (3)	−240 (2)	405	380	258	−33	79	−1
C(13)	3441 (1)	1285 (3)	328 (2)	323	405	372	2	107	−2
C(14)	2996 (1)	−369 (3)	425 (2)	361	353	422	48	129	−19
C(16)	3832 (1)	5290 (3)	622 (2)	385	314	334	23	57	−15
C(17)	4372 (1)	6474 (4)	457 (2)	388	452	482	−17	140	−53
C(18)	4772 (1)	7393 (4)	1298 (2)	391	587	625	−76	117	−156
C(19)	4656 (1)	7158 (4)	2324 (2)	453	568	546	−4	20	−188
C(20)	4123 (1)	5986 (4)	2489 (2)	537	440	363	72	27	−53
C(21)	3717 (1)	5054 (3)	1662 (2)	504	391	339	−25	56	−9
C(22)	3466 (1)	5038 (4)	−1298 (2)	460	481	286	−54	97	51
C(24)	2781 (1)	2461 (4)	−1434 (2)	460	491	278	−42	91	−26

Table 2 (*cont.*)

	x	*y*	*z*	*U*
*H(11)	−649 (18)	−2280 (49)	3469 (27)	742
*H(12)	−956 (18)	−654 (48)	−2646 (27)	742
H(13)	−440 (19)	−199 (49)	3749 (27)	742
*H(3)	891 (16)	−1129 (43)	4018 (24)	589
H(6)	1925 (16)	−1358 (41)	3077 (24)	569
*H(4)	567 (16)	−1654 (41)	192 (23)	553
H(7)	−491 (15)	−1399 (39)	1019 (23)	567
H(81)	2367 (15)	−2453 (39)	1463 (22)	454
H(82)	1832 (14)	−2187 (38)	257 (21)	454
H(101)	1490 (13)	1536 (34)	574 (20)	367
H(102)	1663 (13)	806 (36)	−471 (20)	367
*H(111)	1962 (14)	3853 (38)	−288 (21)	440
*H(112)	2382 (14)	3606 (37)	890 (21)	440
H(131)	3822 (14)	984 (37)	−58 (20)	399
H(132)	3679 (14)	1687 (36)	1047 (20)	399
H(141)	2817 (14)	−1030 (36)	−311 (25)	381
H(142)	3309 (13)	−1193 (35)	883 (20)	381
H(17)	4434 (14)	6637 (38)	−354 (21)	455
H(18)	5164 (16)	8264 (41)	1180 (24)	560
H(19)	4939 (15)	7932 (40)	2944 (21)	484
H(20)	4123 (15)	5986 (40)	2489 (21)	463
H(21)	3327 (14)	4129 (37)	1802 (21)	413
H(221)	3293 (14)	6267 (38)	−1462 (21)	427
H(222)	3989 (14)	4993 (38)	−1396 (21)	427
H(23)	2823 (14)	3970 (36)	−2729 (20)	394

* Positions calculated before refinement.

Table 3. *Bond distances and angles including hydrogen atoms*

The e.s.d.'s for the bond lengths are 0·03 Å and for the angles 3°.

C(1)—H(11)	0·954	C(13)–H(131)	0·968
C(1)—H(12)	1·090	C(13)–H(132)	0·998
C(1)—H(13)	1·041	C(14)–H(141)	1·069
C(3)—H(3)	1·077	C(14)–H(142)	0·978
C(4)—H(4)	0·995	C(17)–H(17)	1·076
C(6)—H(6)	1·036	C(18)–H(18)	1·019
C(7)—H(7)	1·097	C(19)–H(19)	1·053
C(8)—H(81)	1·005	C(20)–H(20)	1·015
C(8)—H(82)	1·087	C(21)–H(21)	1·052
C(10)–H(101)	0·921	C(22)–H(221)	0·994
C(10)–H(102)	1·075	C(22)–H(222)	1·017
C(11)–H(111)	1·003	N(23)–H(23)	0·999
C(11)–H(112)	0·962		

H(11)—C(1)—C(2)	118·0	H(12)—C(1)—C(2)	109·7
H(13)—C(1)—C(2)	109·7	H(3)—C(3)—C(4)	120·1
H(3)—C(3)—C(2)	118·5	H(4)—C(4)—C(3)	123·2
H(4)—C(4)—C(5)	115·8	H(6)—C(6)—C(5)	120·4
H(6)—C(6)—C(7)	118·6	H(7)—C(7)—C(6)	109·4
H(7)—C(7)—C(5)	110·2	H(81)—C(8)—N(9)	104·9
H(81)—C(8)—C(5)	111·2	H(82)—C(8)—N(9)	106·7
H(82)—C(8)—C(5)	110·6	H(101)–C(10)–C(11)	108·8
H(101)–C(10)–N(9)	111·5	H(102)–C(10)–C(11)	111·5
H(102)–C(10)–N(9)	113·1	H(111)–C(11)–C(10)	108·4
H(111)–C(11)–C(12)	108·1	H(112)–C(11)–C(10)	108·4
H(112)–C(11)–C(12)	111·2	H(131)–C(13)–C(12)	109·1
H(131)–C(13)–C(14)	108·5	H(132)–C(13)–C(12)	109·2
H(132)–C(13)–C(14)	109·8	H(141)–C(14)–C(13)	113·7
H(141)–C(14)–N(9)	110·0	H(142)–C(14)–C(13)	106·8
H(142)–C(14)–N(9)	107·6	H(17)—C(17)–C(16)	115·2
H(17)—C(17)–C(18)	124·0	H(18)—C(18)–C(17)	120·9
H(18)—C(18)–C(19)	117·7	H(19)—C(19)–C(18)	120·3
H(19)—C(19)–C(20)	121·6	H(20)—C(20)–C(19)	122·0
H(20)—C(20)–C(21)	116·3	H(21)—C(21)–C(20)	120·9
H(21)—C(21)–C(16)	118·5	H(221)–C(22)–N(15)	114·3
H(221)–C(22)–N(23)	110·9	H(222)–C(33)–N(15)	109·5
H(222)–C(22)–N(23)	111·8	H(23)—N(23)–C(22)	124·8
H(23)—N(23)–C(24)	120·1		

Simpson (1965) were used. The final positional and thermal parameters are given in Table 2.*

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31822 (40 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

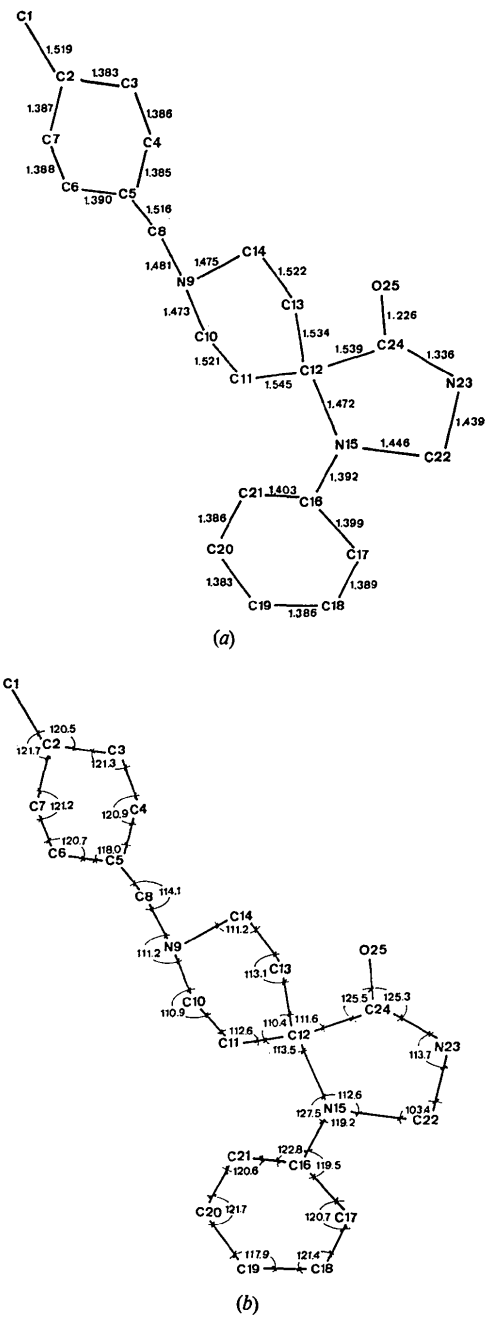

Fig. 1. (*a*) Bond distances (Å) and (*b*) angles (°). The e.s.d.'s for the bond lengths are in the range 0·002–0·005 Å and for the angles 0·1–0·3°.

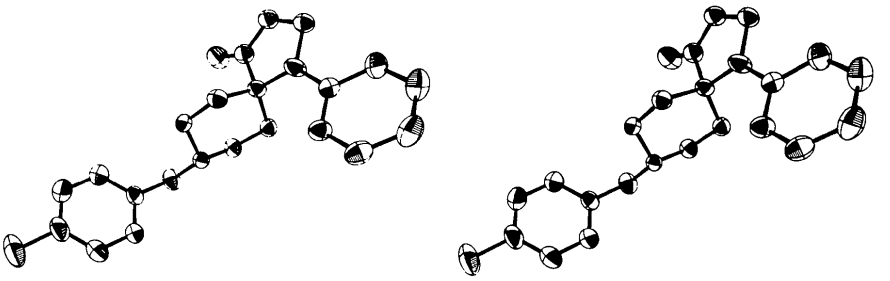

Fig. 2. Stereoscopic view of the molecule with 50% probability thermal ellipsoids (Johnson, 1965).

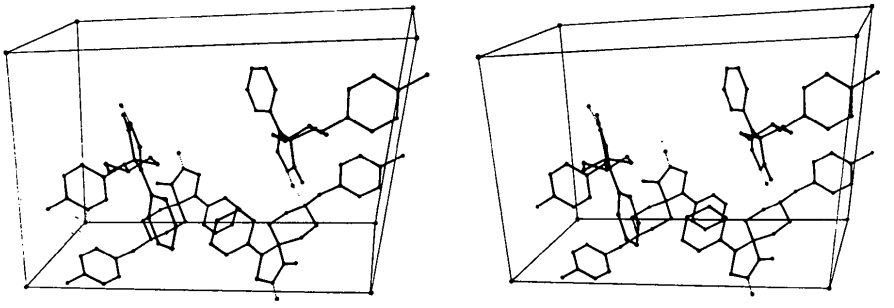

Fig. 3. Stereoscopic view of the molecular packing. The N(23)–H(23)···N(9) hydrogen bond is indicated by dotted lines.

Discussion. The atomic numbering scheme and the bond distances and angles are given in Fig. 1; Table 3 gives the bond distances and angles involving H atoms; Fig. 2 shows the conformation of the molecule. The five-membered and the 1-phenyl groups are nearly coplanar (17°) and perpendicular to the mean plane of the piperidine ring (93°).

Potential-energy calculations allowing rotation about C(5)–C(8) and C(8)–N(9) show that the structure corresponds to one of the energy minima of the isolated molecule.

Each molecule is hydrogen-bonded, N(9)···H(23)–N(23), to two neighbours as shown in Fig. 3; N(9)–N(23): 2·89 Å [N(23) $\bar{x},\frac{1}{2}+y,\frac{1}{2}-z$].

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endo-7-Chloro-7-phenyl-2-oxabicyclo[4,1,0]heptane

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Abstract. $C_{12}H_{13}OCl$, F.W. 208·7; monoclinic, $P2_1/c$; $a=11{\cdot}929$ (1), $b=7{\cdot}469$ (1), $c=11{\cdot}900$ (2) Å, $\beta=96{\cdot}25$ (1)°; $D_x=1{\cdot}315$ g cm^{-3} for $Z=4$, $D_o=1{\cdot}317$ g cm^{-3}; $V=1054$ Å^3; $\mu=28{\cdot}9$ cm^{-1}. The structure was solved by the heavy-atom method and refined to $R=0{\cdot}074$. The tetrahydropyran ring is in an envelope conformation with only one atom being significantly out of the plane defined by the other five non-hydrogen atoms of that moiety. The phenyl ring is oriented parallel to the bridgehead bond of the bicyclic system.