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10-Acetamido-4-methoxytricyclo[7.3.1.$0^{2,7}$]trideca-2(7),3,5-triene

BY PETER MURRAY-RUST AND JUDITH MURRAY-RUST

Department of Chemistry, University of Stirling, Stirling FK9 4LA, Scotland

AND DAVID MIDDLEMISS

Glaxo Group Research Ltd, Ware, Hertfordshire SG12 0DJ, England

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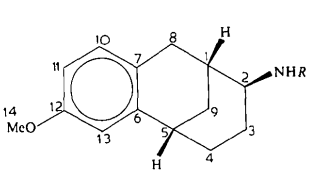
Abstract. $C_{16}H_{21}NO_2$, M_r = 259·4, orthorhombic, *Pbca*, a = 10·43 (3), b = 31·40 (9), c = 8·67 (4) Å from diffractometer measurements (Mo $K\bar{\alpha}$ radiation), V = 2839 Å^3, Z = 8, D_c = 1·213 Mg m^{-3}, $F(000)$ = 1120, μ = 0·45 mm^{-1}, approximate crystal dimensions 0·4 × 0·25 × 0·15 mm. R = 0·044 for 1070 observed reflexions. The molecules are held together in the crystal by hydrogen bonds O(2)···N(1).

Introduction. The title compound (I) was recrystallized from benzene–petrol (b.p. 353–373 K). Systematic absences (from precession photographs) 0*kl* *k* odd, *h*0*l* *l* odd and *hk*0 *h* odd indicated space group *Pbca*. Data were collected for 0–8*kl* with θ_{max} = 22·5° on a Stoe Stadi-2 two-circle diffractometer (graphite-monochromated Mo $K\bar{\alpha}$ radiation). This gave 1640 data of which 1070 unique reflexions with $I > 3\sigma(I)$ were used in subsequent calculations. Lorentz and polarization corrections (but none for extinction or absorption) were applied, and the data scaled by a Wilson plot. The structure was solved by direct-phasing methods with the *SHELX* 76 system of crystallographic programs (Sheldrick, 1976) which was used for all calculations. Complex neutral-atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Weighted full-matrix least-squares refinement (including isotropic H atoms) converged at R = 0·044 for 1070 observed reflexions; R_w = 0·042 $\{R_w = \sum(||F_o| - |F_c||.w^{1/2})/\sum(|F_o|.w^{1/2})$, $w = 2{\cdot}6431/[\sigma^2(F_o) + 0{\cdot}000269F_o^2]\}$. In the final cycle all shifts in parameters were less than their standard deviations.



(I) R = COMe
(II) R = H

Table 1. *Atomic coordinates* (×10^4) *and thermal parameters* (U_{eq} × 10^4, U_{iso} × 10^3) *with e.s.d.'s in parentheses*

For non-H atoms $U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33} + 2U_{12}\cos\gamma + 2U_{13}\cos\beta + 2U_{23}\cos\alpha)$.

	x	y	z	U_{eq} or U_{iso} (Å^2)
C(1)	2921 (4)	8960 (1)	4814 (4)	500 (26)
C(2)	2003 (4)	9311 (1)	4288 (5)	541 (24)
C(3)	622 (4)	9204 (1)	4639 (5)	613 (33)
C(4)	271 (5)	8767 (1)	3966 (5)	634 (33)
C(5)	1216 (4)	8421 (1)	4452 (4)	532 (27)
C(6)	1141 (3)	8347 (1)	6177 (4)	432 (24)
C(7)	1953 (3)	8571 (1)	7158 (4)	420 (22)
C(8)	2944 (4)	8887 (1)	6561 (4)	512 (27)
C(9)	2575 (5)	8545 (1)	3993 (5)	596 (30)
C(10)	1850 (4)	8489 (1)	8741 (4)	505 (28)
C(11)	996 (4)	8199 (1)	9331 (5)	500 (26)
C(12)	199 (4)	7977 (1)	8333 (4)	446 (24)
C(13)	286 (4)	8050 (1)	6773 (5)	459 (26)
C(14)	−1482 (6)	7464 (2)	8056 (7)	678 (32)
C(15)	3156 (4)	9991 (1)	4102 (5)	553 (26)
C(16)	3499 (7)	10399 (1)	4918 (7)	737 (29)
N(1)	2369 (4)	9729 (1)	4877 (4)	581 (24)
O(1)	−629 (3)	7693 (1)	9017 (3)	592 (17)
O(2)	3595 (3)	9903 (1)	2824 (3)	695 (19)
H(1N)	2102 (38)	9822 (11)	5760 (45)	70 (14)
H(1)	3798 (33)	9045 (9)	4542 (36)	52 (11)
H(2)	2132 (29)	9332 (9)	3197 (39)	51 (10)
H(3)	519 (29)	9226 (8)	5778 (38)	41 (9)
H(4)	30 (36)	9405 (11)	4292 (40)	68 (13)
H(5)	−616 (40)	8677 (11)	4333 (39)	68 (13)
H(6)	384 (35)	8788 (10)	2818 (46)	71 (12)
H(7)	979 (31)	8155 (10)	3918 (36)	51 (10)
H(8)	2836 (29)	9166 (10)	7084 (33)	49 (10)
H(9)	3838 (35)	8793 (11)	6858 (41)	69 (12)
H(10)	3226 (37)	8306 (12)	4268 (40)	83 (14)
H(11)	2563 (34)	8601 (9)	2881 (40)	58 (11)
H(12)	2404 (30)	8643 (9)	9410 (33)	44 (10)
H(13)	902 (30)	8153 (9)	10510 (41)	54 (10)
H(14)	−232 (34)	7920 (10)	6111 (39)	57 (12)
H(15)	−1944 (42)	7260 (14)	8721 (50)	99 (15)
H(16)	−1066 (45)	7294 (14)	7310 (56)	107 (20)
H(17)	−2000 (39)	7665 (12)	7439 (46)	85 (15)
H(18)	2724 (44)	10536 (12)	5413 (49)	94 (18)
H(19)	4169 (45)	10328 (14)	5724 (57)	115 (20)
H(20)	3928 (48)	10567 (16)	4216 (61)	126 (21)

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Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(1)–C(2)	1·530 (5)	C(7)–C(8)	1·522 (5)
C(1)–C(8)	1·533 (5)	C(7)–C(10)	1·400 (5)
C(1)–C(9)	1·527 (5)	C(10)–C(11)	1·371 (5)
C(2)–C(3)	1·510 (5)	C(11)–C(12)	1·388 (5)
C(2)–N(1)	1·458 (5)	C(12)–C(13)	1·375 (5)
C(3)–C(4)	1·535 (6)	C(12)–O(1)	1·377 (4)
C(4)–C(5)	1·528 (5)	C(14)–O(1)	1·414 (5)
C(5)–C(6)	1·516 (5)	C(15)–C(16)	1·508 (6)
C(5)–C(9)	1·523 (5)	C(15)–N(1)	1·342 (5)
C(6)–C(7)	1·392 (4)	C(15)–O(2)	1·230 (4)
C(6)–C(13)	1·390 (5)		

C(8)–C(1)–C(2)	114·3 (3)	C(10)–C(7)–C(6)	117·3 (3)
C(9)–C(1)–C(2)	109·1 (4)	C(10)–C(7)–C(8)	120·4 (3)
C(9)–C(1)–C(8)	109·7 (3)	C(7)–C(8)–C(1)	115·0 (3)
C(3)–C(2)–C(1)	112·1 (3)	C(5)–C(9)–C(1)	108·5 (4)
N(1)–C(2)–C(1)	112·3 (3)	C(11)–C(10)–C(7)	122·6 (4)
N(1)–C(2)–C(3)	112·3 (4)	C(12)–C(11)–C(10)	119·3 (4)
C(4)–C(3)–C(2)	110·4 (4)	C(13)–C(12)–C(11)	119·4 (4)
C(5)–C(4)–C(3)	112·2 (4)	O(1)–C(12)–C(11)	115·7 (3)
C(6)–C(5)–C(4)	110·3 (3)	O(1)–C(12)–C(13)	125·0 (3)
C(9)–C(5)–C(4)	110·2 (4)	C(12)–C(13)–C(6)	121·3 (4)
C(9)–C(5)–C(6)	110·2 (3)	N(1)–C(15)–C(16)	115·6 (4)
C(7)–C(6)–C(5)	119·6 (3)	O(2)–C(15)–C(16)	121·6 (4)
C(13)–C(6)–C(5)	120·2 (3)	O(2)–C(15)–N(1)	122·8 (4)
C(13)–C(6)–C(7)	120·2 (3)	C(15)–N(1)–C(2)	122·4 (4)
C(8)–C(7)–C(6)	122·3 (3)	C(14)–O(1)–C(12)	118·0 (3)

Atomic parameters are given in Table 1 and bond distances and angles in Table 2.*

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36228 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

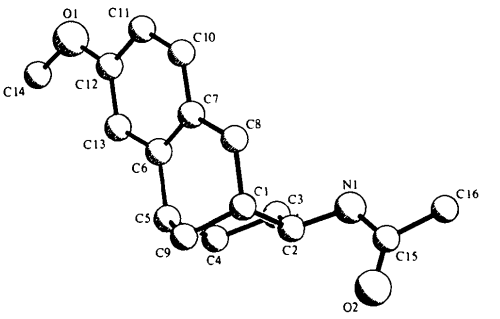

Fig. 1. General view of the molecule (excluding H atoms).

Discussion. The primary amine (II) is one of a series of compounds showing analgesic activity (Garry, Middlemiss, Reynolds & Sims, unpublished results). The structure determination of the amide (I) was undertaken to confirm the configuration at C(2).

The molecules are held together in the crystal by hydrogen bonds [$O(2)\cdots N(1)(\frac{1}{2}-x,\ 2-y,\ z-\frac{1}{2}) = 2{\cdot}98$ Å]. The methoxy group is coplanar with the benzene ring (Fig. 1) and otherwise the geometry seems much as expected, with the central bicyclo[3.3.1]-nonane system having a sofa [C(7)]–chair [C(3)] conformation.

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[*p*-(Bromo-2 éthoxy)phényl]-1 Chloro-2 Diphényl-1,2 Ethylène

PAR G. PRÉCIGOUX, M. HOSPITAL ET F. LEROY

Laboratoire de Cristallographie associé au CNRS LA 144, *Université de Bordeaux* I, 33405 *Talence, France*

ET A. DELBARRE ET B. P. ROQUES

Département de Chimie Organique, ERA 613 *du CNRS et SCN* 21 *de l'INSERM, Université René Descartes,* 75006 *Paris, France*

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Abstract. $C_{22}H_{18}BrClO$, *Cc*, $a = 12{\cdot}317\ (2)$, $b = 15{\cdot}238\ (3)$, $c = 9{\cdot}982\ (2)$ Å, $\beta = 94{\cdot}32\ (5)°$, $Z = 4$, $d_x = 1{\cdot}47$ Mg m^{-3}; $R = 0{\cdot}056$ for 2046 observed reflexions. X-ray intensity data were measured on a Nonius CAD-4 diffractometer and the structure was solved by direct methods. The relative angles between aromatic rings differ from those of other triphenyl derivatives. The bromoethoxy chain is not in a *trans* conformation and the Br–C–C–O dihedral angle is 71·8 (8)°.

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