Table 2. *Interatomic distances* (Å) *and bond angles* (°)

N(1)–C(1)	1·333 (3)	N(1)–C(2)	1·334 (3)
N(2)–C(6)	1·333 (2)	N(2)–C(7)	1·336 (3)
N(3)–C(11)	1·329 (3)	N(3)–C(12)	1·343 (3)
C(2)–C(3)	1·372 (4)	C(3)–C(4)	1·368 (4)
C(4)–C(5)	1·374 (4)	C(5)–C(1)	1·389 (3)
C(7)–C(8)	1·375 (3)	C(8)–C(9)	1·368 (4)
C(9)–C(10)	1·378 (3)	C(10)–C(6)	1·379 (3)
C(12)–C(13)	1·364 (4)	C(13)–C(14)	1·354 (4)
C(14)–C(15)	1·380 (4)	C(15)–C(11)	1·380 (3)
C(16)–O(1)	1·427 (2)	C(16)–C(1)	1·524 (3)
C(16)–C(6)	1·530 (2)	C(16)–C(11)	1·526 (3)
O(1)–H(16)	0·88 (3)		

C(1)–N(1)–C(2)	117·3 (2)	C(6)–N(2)–C(7)	117·2 (2)
C(11)–N(3)–C(12)	117·6 (2)	N(1)–C(1)–C(5)	122·2 (2)
N(1)–C(1)–C(16)	117·4 (2)	C(5)–C(1)–C(16)	120·4 (2)
N(1)–C(2)–C(3)	124·2 (3)	C(2)–C(3)–C(4)	118·1 (2)
C(3)–C(4)–C(5)	119·2 (2)	C(4)–C(5)–C(1)	119·0 (2)
N(2)–C(6)–C(10)	122·3 (2)	N(2)–C(6)–C(16)	113·9 (1)
C(10)–C(6)–C(16)	123·8 (2)	N(2)–C(7)–C(8)	123·4 (2)
C(7)–C(8)–C(9)	118·4 (2)	C(8)–C(9)–C(10)	119·1 (2)
C(9)–C(10)–C(6)	119·0 (2)	N(3)–C(11)–C(15)	122·3 (2)
N(3)–C(11)–C(16)	116·9 (2)	C(15)–C(11)–C(16)	120·8 (2)
N(3)–C(12)–C(13)	123·7 (2)	C(12)–C(13)–C(14)	117·9 (2)
C(13)–C(14)–C(15)	120·3 (2)	C(14)–C(15)–C(11)	118·2 (2)
O(1)–C(16)–C(1)	109·8 (1)	O(1)–C(16)–C(6)	108·7 (1)
O(1)–C(16)–C(11)	106·1 (2)	C(1)–C(16)–C(6)	110·1 (1)
C(1)–C(16)–C(11)	110·4 (2)	C(6)–C(16)–C(11)	111·6 (1)
C(16)–O(1)–H(16)	106 (2)		

tridentate N,N′,N″ coordination has been noted in the cations $[M\{(py)_3COH\}_2]^{2+}$, M = Co (Szalda & Keene, 1986) and for one ligand in M = Ru (Keene, Szalda & Wilson, 1987). Asymmetric tridentate coordination has been found in $[CH_3Hg(py)_3COH)][NO_3]$ (Canty, Chaichit, Gatehouse & George, 1981) with Hg—N bond distances in the range 2·28 (1) to 2·53 (1) Å. Tridentate coordination *via* two N atoms and the hydroxyl O atom has also been observed in one of the ligands in $[Ru\{(py)_3COH\}_2]^{2+}$ (Keene, Szalda & Wilson, 1987) and in $[Ru(NH_3)_3\{(py)_3COH\}]^{2+}$ (Diamantis, Keene, Moritz, Snow & Tiekink, 1988).

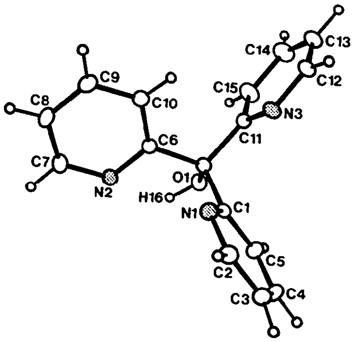

Fig. 1. The molecular structure and numbering scheme for $(C_5H_4N)_3COH$. Hydrogen atoms are numbered according to the C atom to which they are bonded (Johnson, 1971).

When deprotonated, the ligand also coordinates *via* two N atoms and the O atom as found in $[Ru\{(py)_3COH\}\{(py)_3CO\}]^+$ (Keene, Szalda & Wilson, 1987).

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References

Canty, A. J., Chaichit, N., Gatehouse, B. M. & George, E. E. (1981). *Inorg. Chem.* **20**, 4293–4300.

Diamantis, A. A., Keene, F. R., Moritz, P. S., Snow, M. R. & Tiekink, E. R. T. (1988). In preparation.

Johnson, C. K. (1971). *ORTEPII*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Keene, F. R., Szalda, D J. & Wilson, T. A. (1987). *Inorg. Chem.* **26**, 2211–2216.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Szalda, D. J. & Keene, F. R. (1986). *Inorg. Chem.* **25**, 2795–2799.

Wibaut, J. P., De Jonge, A. P., Van Der Voort, H. G. P. & Otto, P. Ph. H. L. (1951). *Recl Trav. Chim. Pays Bas*, **70**, 1054–1066.

Acta Cryst. (1988). C**44**, 938–939

Structure of 4β-Hydroxy-6-methoxymethoxy-*N*,*N*-dimethyl-1,2,3,4α,4a,9b-hexahydro-9bβ-dibenzofuranacetamide

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Abstract. $C_{18}H_{25}NO_5$, $M_r = 335·4$, triclinic, $P\bar{1}$, $a = 11·155$ (4), $b = 9·732$ (3), $c = 9·150$ (3) Å, $\alpha = 110·81$ (3), $\beta = 79·86$ (2), $\gamma = 112·11$ (3)°, $V = 859·21$ Å^3, $Z = 2$, $D_x = 1·30$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 6·87$ cm^{-1}, $F(000) = 360$, room temperature, final $R = 0·068$ for 2683 observed reflections. The title compound is an intermediate in the synthesis of a morphinic analogue. The relative configuration was established. There are no unusual bond distances or angles.

0108-2701/88/050938-02$03.00

Experimental. Crystal 0·50 × 0·38 × 0·35 mm. Data collected on a Philips PW1100 diffractometer using graphite-monochromated Cu $K\alpha$ radiation. Unit-cell parameters from setting angles of 72 accurately centered reflections ($5{\cdot}20 \leq 2\theta \leq 42{\cdot}56°$). 2942 intensities obtained by the θ–2θ scan technique up to $\theta = 68°$ ($-12 \leq h \leq 12$, $-11 \leq k \leq 10$, $0 \leq l \leq 10$) from which 2683 observed with $I \geq 3\sigma(I)$. Three reference reflections monitored every 120 min showed no significant changes in intensities. No correction for absorption. Structure solved by direct methods (program *DEVIN;* Riche, 1982) and refined by blocked full-matrix least squares minimizing the function $\sum w(F_o - |F_c|)^2$ where $w = 1/\sigma^2(F_o)$. Non-H atoms refined anisotropically. All H atoms located on difference Fourier maps and refined with an isotropic thermal factor equivalent to that of the bonded atom. Convergence was reached at $R = 0{\cdot}068$ and $wR = 0{\cdot}059$. $(\Delta/\sigma)_{\max} = 0{\cdot}03$. On the final difference map $\Delta\rho_{\max} = 0{\cdot}38$ and $\Delta\rho_{\min} = -0{\cdot}57$ e Å^{-3}. Calculations performed with program *SHELX*76 (Sheldrick, 1976), which also provided atomic scattering factors. A perspective view of the molecule is shown in Fig. 1. The absolute configuration of morphine has been adopted. Atomic coordinates are given in Table 1, bond distances and angles in Table 2.*

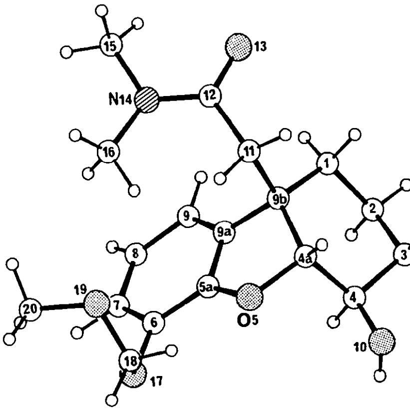

Fig. 1. Perspective view of the molecule with atom numbering.

Table 1. *Atomic coordinates* (× 10⁴) *and equivalent isotropic thermal factors* (× 10³) *with e.s.d.'s in parentheses*

$$U_{\text{eq}} = \tfrac{1}{3}\textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}(Å^2)
C1	3090 (2)	6094 (3)	7895 (2)	51 (2)
C2	3659 (2)	7053 (3)	9484 (3)	54 (2)
C3	2617 (2)	6802 (3)	10776 (3)	51 (2)
C4	2161 (2)	5110 (3)	10725 (2)	44 (2)
C4a	1573 (2)	4024 (2)	9164 (2)	40 (2)
O5	1460 (1)	2427 (2)	8998 (1)	45 (1)
C5a	2582 (2)	2239 (2)	8178 (2)	40 (2)
C6	2994 (2)	1002 (3)	7977 (2)	50 (2)
C7	4173 (3)	1032 (3)	7142 (3)	66 (3)
C8	4876 (2)	2218 (3)	6525 (3)	64 (3)
C9	4419 (2)	3401 (3)	6661 (2)	50 (2)
C9a	3253 (2)	3390 (2)	7484 (2)	39 (2)
C9b	2400 (2)	4354 (2)	7698 (2)	38 (2)
O10	1188 (2)	4806 (2)	11929 (2)	60 (2)
C11	1459 (2)	3551 (3)	6334 (2)	43 (2)
C12	2102 (2)	3488 (2)	4718 (2)	43 (2)
O13	2414 (2)	4666 (2)	4301 (2)	62 (2)
N14	2330 (2)	2202 (2)	3777 (2)	47 (2)
C15	2928 (3)	2192 (4)	2222 (3)	72 (3)
C16	1908 (3)	719 (3)	4085 (3)	67 (3)
O17	2319 (2)	−226 (2)	8552 (2)	66 (2)
C18	981 (3)	−958 (3)	8193 (3)	66 (3)
O19	805 (1)	−1348 (2)	6618 (2)	59 (2)
C20	1286 (3)	−2561 (3)	5674 (3)	63 (3)

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

C1–C2	1·518 (3)	C7–C8	1·375 (4)
C1–C9b	1·530 (3)	C8–C9	1·386 (4)
C2–C3	1·522 (4)	C9–C9a	1·381 (3)
C3–C4	1·515 (4)	C9a–C9b	1·516 (3)
C4–C4a	1·524 (3)	C9b–C11	1·556 (3)
C4–O10	1·431 (3)	C11–C12	1·516 (3)
C4a–O5	1·465 (3)	C12–O13	1·242 (3)
C4a–C9b	1·550 (3)	C12–N14	1·330 (3)
O5–C5a	1·381 (3)	N14–C15	1·459 (3)
C5a–C6	1·387 (3)	N14–C16	1·455 (4)
C5a–C9a	1·388 (3)	O17–C18	1·432 (4)
C6–C7	1·395 (4)	C18–O19	1·383 (3)
C6–O17	1·380 (3)	O19–C20	1·421 (4)
C2–C1–C9b	113·9 (2)	C5a–C9a–C9	120·4 (2)
C1–C2–C3	109·9 (2)	C5a–C9a–C9b	107·1 (2)
C2–C3–C4	109·3 (2)	C9–C9a–C9b	132·3 (2)
C3–C4–C4a	110·6 (2)	C1–C9b–C4a	114·0 (2)
C3–C4–O10	111·9 (2)	C1–C9b–C9a	116·7 (2)
C4a–C4–O10	107·2 (2)	C1–C9b–C11	111·3 (2)
C4–C4a–O5	108·6 (2)	C4a–C9b–C9a	99·0 (2)
C4–C4a–C9b	115·0 (2)	C4a–C9b–C11	106·1 (2)
O5–C4a–C9b	104·7 (2)	C9a–C9b–C11	108·7 (2)
C4a–O5–C5a	105·0 (2)	C9b–C11–C12	115·2 (2)
O5–C5a–C6	125·0 (2)	C11–C12–O13	118·4 (2)
O5–C5a–C9a	113·0 (2)	C11–C12–N14	120·5 (2)
C6–C5a–C9a	121·9 (2)	O13–C12–N14	121·0 (2)
C5a–C6–C7	116·7 (2)	C12–N14–C15	118·4 (2)
C5a–C6–O17	124·2 (2)	C12–N14–C16	125·6 (2)
C7–C6–O17	119·1 (2)	C15–N14–C16	115·6 (2)
C6–C7–C8	121·4 (3)	C6–O17–C18	114·8 (2)
C7–C8–C9	121·2 (3)	O17–C18–O19	112·3 (2)
C8–C9–C9a	118·1 (2)	C18–O19–C20	112·4 (2)

Related literature. The title compound was synthesized according to Labidalle, Zhang, Reynet, Thal & Moskowitz (1986).

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* Lists of structure factors, H-atom coordinates and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44678 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Labidalle, S., Zhang, Y.-M., Reynet, A., Thal, C. & Moskowitz, M. (1986). *Tetrahedron Lett.* **27**, 2861–2862.

Riche, C. (1982). Abstr. 7th Eur. Crystallogr. Meet., Jerusalem, 29 August–3 September, p. 25.

Sheldrick, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.