Acta Cryst. (1990). C**46**, 333–334

tert-Butyl *N*-[1-(3-*tert*-Butyl-2-hydroxyphenyl)-1-hydroxy-3-phenyl-2-propyl]-carbamate

By Gabriele Bocelli and Andrea Cantoni

Centro di Studio per la Strutturistica Diffrattometrica del CNR, Viale delle Scienze, 43100 *Parma, Italy*

(*Received* 3 *July* 1989; *accepted* 14 *August* 1989)

Abstract. $C_{24}H_{33}NO_4$, $M_r = 399{\cdot}5$, orthorhombic, $P2_12_12$, $a = 23{\cdot}575\ (2)$, $b = 10{\cdot}490\ (2)$, $c = 9{\cdot}558\ (2)$ Å, $V = 2363{\cdot}7\ (7)$ Å^3, $Z = 4$, $D_x = 1{\cdot}12$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu = 5{\cdot}72$ cm^{-1}, $F(000) = 864$, room temperature, $R = 0{\cdot}046$ for 1416 observed reflections. Intermolecular contacts involve hydrogen bonds of the type $\mathrm{HO2}\cdots\mathrm{O3}^{\mathrm{i}} = 1{\cdot}88\ (5)$ Å with $\mathrm{O2—HO2}\cdots\mathrm{O3}^{\mathrm{i}} = 163\ (5)°$ ($\mathrm{i} = -x + \frac{3}{2}, y + \frac{1}{2}, z$).

Experimental. Siemens AED single-crystal diffractometer controlled by an IBM PS2/30 personal computer. Elongated prismatic crystals of about 0·3 × 0·3 × 0·6 mm, accurate unit-cell parameters from a least-squares fit to 30 reflections ($11 \leq \theta \leq 28°$), intensities measured using a modified version (Belletti, Cantoni & Pasquinelli, 1988) of the Lehmann & Larsen (1974) method, nickel-filtered Cu $K\alpha$ radiation, θ range 3–70°, one check reflection monitored every 50 measurements showed a decrease in intensity of about 11%, intensities corrected for this decay, for Lorentz and polarization effects, but not for absorption. 2588 reflections collected, index range h 0/28, k 0/12, l 0/11, 2569 independent reflections ($R_{\mathrm{int}} = 0{\cdot}049$), 1423 with $I > 2\sigma(I)$ observed. The structure was solved by direct methods with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), block-matrix anisotropic least-squares refinements with *SHELX*76 (Sheldrick, 1976), H atoms found in difference Fou-

Table 1. *Atomic fractional coordinates* (× 10^4) *and* U_{eq} (× 10^4 Å^2)

	x	y	z	U_{eq}*
O1	8200 (1)	2262 (3)	− 763 (4)	502 (11)
O2	7198 (1)	3335 (3)	− 952 (4)	451 (11)
O3	7886 (2)	393 (3)	2653 (4)	519 (12)
O4	7786 (2)	2372 (3)	3571 (3)	492 (12)
N	7303 (2)	1854 (4)	1676 (5)	396 (13)
C1	7956 (2)	4085 (4)	605 (4)	360 (12)
C2	8358 (2)	3236 (4)	99 (4)	374 (13)
C3	8937 (2)	3358 (4)	436 (5)	419 (14)
C4	9087 (2)	4368 (5)	1294 (6)	514 (19)
C5	8689 (2)	5274 (5)	1764 (6)	529 (20)
C6	8131 (2)	5104 (4)	1429 (5)	434 (15)
C7	9389 (2)	2391 (5)	− 77 (6)	539 (19)
C8	9230 (3)	1046 (6)	442 (9)	716 (25)
C9	9410 (3)	2402 (10)	− 1679 (7)	874 (32)
C10	9968 (3)	2736 (8)	487 (11)	933 (31)
C11	7319 (2)	3903 (5)	365 (5)	393 (15)
C12	7043 (2)	3108 (4)	1548 (5)	406 (16)
C13	7673 (2)	1465 (4)	2635 (5)	391 (15)
C14	8159 (2)	2168 (5)	4789 (5)	442 (16)
C15	7922 (4)	1109 (8)	5686 (7)	677 (29)
C16	8124 (4)	3442 (7)	5526 (8)	746 (25)
C17	8755 (3)	1889 (10)	4325 (9)	740 (33)
C18	6398 (2)	2971 (6)	1317 (7)	509 (19)
C19	6094 (2)	2546 (5)	2581 (6)	530 (19)
C20	5970 (3)	1242 (7)	2788 (8)	699 (24)
C21	5684 (3)	881 (11)	3995 (12)	993 (41)
C22	5510 (4)	1726 (14)	4960 (9)	1092 (46)
C23	5622 (4)	2994 (11)	4751 (11)	1188 (47)
C24	5907 (3)	3378 (9)	3556 (9)	890 (32)

* Hamilton (1959).

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

O1—C2	1·364 (5)	C7—C8	1·542 (9)
O2—C11	1·422 (6)	C7—C9	1·532 (9)
O3—C13	1·232 (6)	C7—C10	1·512 (9)
O4—C13	1·333 (6)	C11—C12	1·548 (7)
O4—C14	1·475 (6)	C12—C18	1·543 (7)
N—C12	1·456 (6)	C14—C15	1·510 (10)
N—C13	1·330 (7)	C14—C16	1·513 (9)
C1—C2	1·387 (6)	C14—C17	1·502 (9)
C1—C6	1·390 (6)	C18—C19	1·474 (8)
C1—C11	1·531 (7)	C19—C20	1·413 (9)
C2—C3	1·408 (7)	C19—C24	1·351 (10)
C3—C4	1·386 (7)	C20—C21	1·389 (13)
C3—C7	1·551 (7)	C21—C22	1·343 (16)
C4—C5	1·409 (7)	C22—C23	1·371 (18)
C5—C6	1·366 (7)	C23—C24	1·385 (13)

C13—O4—C14	123·1 (3)	O2—C11—C12	109·7 (3)
C12—N—C13	127·7 (4)	N—C12—C11	111·8 (3)
C6—C1—C11	118·1 (3)	C11—C12—C18	111·1 (4)
C2—C1—C11	122·5 (3)	N—C12—C18	110·0 (3)
C2—C1—C6	119·2 (4)	O4—C13—N	112·0 (3)
O1—C2—C1	120·3 (3)	O3—C13—N	123·9 (4)
C1—C2—C3	121·6 (3)	O3—C13—O4	124·1 (4)
O1—C2—C3	118·1 (3)	O4—C14—C17	110·7 (4)
C2—C3—C7	122·3 (4)	O4—C14—C16	101·9 (4)
C2—C3—C4	116·9 (4)	O4—C14—C15	109·5 (4)
C4—C3—C7	120·8 (4)	C16—C14—C17	111·1 (5)
C3—C4—C5	122·3 (4)	C15—C14—C17	111·7 (5)
C4—C5—C6	118·6 (4)	C15—C14—C16	111·4 (5)
C1—C6—C5	121·3 (4)	C12—C18—C19	113·0 (4)
C3—C7—C10	110·6 (5)	C18—C19—C24	121·9 (6)
C3—C7—C9	109·5 (4)	C18—C19—C20	120·6 (5)
C3—C7—C8	109·3 (4)	C20—C19—C24	117·5 (5)
C9—C7—C10	109·0 (5)	C19—C20—C21	118·7 (7)
C8—C7—C10	108·9 (5)	C20—C21—C22	122·6 (10)
C8—C7—C9	109·6 (5)	C21—C22—C23	118·8 (8)
O2—C11—C1	112·4 (3)	C22—C23—C24	119·7 (9)
C1—C11—C12	111·7 (3)	C19—C24—C23	122·6 (8)

0108-2701/90/020333-02$03.00

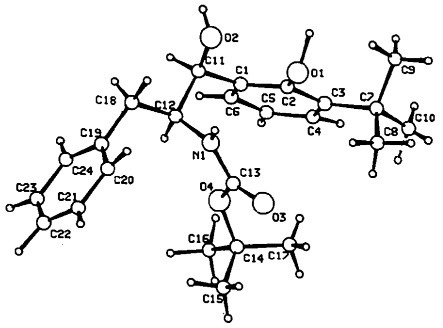


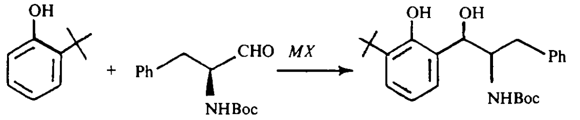
Fig. 1. Projection of the molecule.

rier maps and refined isotropically, $\sum w\Delta F^2$ minimized with weighting scheme $w = 0{\cdot}4035/(\sigma^2 F + 0{\cdot}00621F^2)$, seven reflections omitted because of extinction, $(\Delta/\sigma)_{\max} = 0{\cdot}82$, $\Delta\rho_{\max}$ 0·19 e Å^{-3}, 395 variables, final refinement converged to $R = 0{\cdot}046$ and $wR = 0{\cdot}051$. Atomic scattering factors from *SHELX76*.*

Final atomic fractional coordinates are listed in Table 1, bond distances and bond angles are presented in Table 2. A projection of the molecule with the numbering scheme is shown in Fig. 1.

All the calculations were performed on an IBM PS2/80 personal computer with the *CRYSRULER* package (Rizzoli, Sangermano, Calestani & Andreetti, 1987).

Related literature. The synthesis of the title compound forms part of a series of studies on direct diastereoselective synthesis of ephedrine-like compounds by hydroxylalkylation of metal phenolates with N-protected α-amino aldehydes (Bigi, Casnati, Sartori & Araldi, 1989).



Boc = *tert*-butoxycarbonyl MX = methylene chloride

---

* Lists of structure-factor amplitudes, H-atom coordinates with isotropic thermal parameters and anisotropic thermal parameters of heavy atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52205 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Belletti, D., Cantoni, A. & Pasquinelli, G. (1988). *Gestione on Line di Diffrattometro a Cristallo Singolo Siemens AED con Sistema IBM PS2/30*. Internal report 1/88. Centro di Studio per la Strutturistica Diffrattometrica del CNR, Parma, Italy.

Bigi, F., Casnati, G., Sartori, G. & Araldi, G. (1989). *Gazz. Chim. Ital.* Submitted.

Hamilton, W. C. (1959). *Acta Cryst.* **12**, 609–610.

Lehmann, M. S. & Larsen, F. K. (1974). *Acta Cryst.* A**30**, 580–589.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Rizzoli, C., Sangermano, V., Calestani, G. & Andreetti, G. D. (1987). *J. Appl. Cryst.* **20**, 436–440.

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

Acta Cryst. (1990). C**46**, 334–336

Structure of 3β-Hydroxy-5-androsten-17-one (DHEA) Monohydrate

By Philip J. Cox and Stephen M. MacManus

School of Pharmacy, Robert Gordon's Institute of Technology, Schoolhill, Aberdeen AB9 1FR, Scotland

Bruce C. Gibb and Ian W. Nowell

School of Chemistry, Robert Gordon's Institute of Technology, St Andrew St, Aberdeen AB1 1HG, Scotland

and R. Alan Howie

Chemistry Department, University of Aberdeen, Meston Walk, Old Aberdeen AB9 2UE, Scotland

(*Received* 29 *June* 1989; *accepted* 27 *September* 1989)

Abstract. $C_{19}H_{28}O_2.H_2O$, $M_r = 306{\cdot}4$, orthorhombic, $P2_12_12_1$, $a = 22{\cdot}545\,(7)$, $b = 22{\cdot}673\,(22)$, $c = 6{\cdot}819\,(2)$ Å, $V = 3485{\cdot}6$ Å^3, $Z = 8$, $D_x = 1{\cdot}17$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}43$ cm^{-1}, $F(000) = 1344$, $T = 293$ K, $R = 0{\cdot}071$ for 1308 observed reflexions. The asymmetric unit contains two steroid molecules of similar conformation and two water molecules. Extensive hydrogen bonding is present

0108-2701/90/020334-03$03.00