C206	0.5885 (3)	0.3966 (2)	0.1595 (2)	0.0569 (6)
C207	0.7119 (3)	0.4085 (2)	0.0543 (2)	0.0558 (6)
C208	0.7550 (2)	0.5408 (2)	0.00888 (14)	0.0478 (5)
C209	0.6771 (2)	0.66199 (15)	0.06936 (11)	0.0373 (4)
C210	0.5540 (2)	0.6504 (2)	0.17654 (11)	0.0375 (4)

Table 2. *Selected geometric parameters (Å, °)*

Molecule 1		Molecule 2	
O101—C101	1.2068 (19)	O201—C201	1.2094 (18)
O102—C102	1.333 (2)	O202—C202	1.3309 (18)
O104—C104	1.2280 (18)	O204—C204	1.2293 (18)
O101—C101—C102	119.15 (16)	O201—C201—C202	118.86 (15)
O101—C101—C109	123.64 (15)	O201—C201—C209	123.65 (15)
O102—C102—C101	115.93 (13)	O202—C202—C201	116.23 (12)
O102—C102—C103	121.67 (16)	O202—C202—C203	121.77 (14)
O104—C104—C103	121.66 (15)	O204—C204—C203	121.49 (15)
O104—C104—C110	119.92 (16)	O204—C204—C210	119.88 (15)

The unit-cell parameters were checked for the presence of higher lattice symmetry (Spek, 1988). Data were collected during 78 h of X-ray exposure time. Reflections were measured with Zr-filtered Mo $K\alpha$ radiation, a scan angle $\Delta\omega$ of $(0.67 + 0.35\tan\theta)^\circ$, and horizontal and vertical apertures of 3.00 and 5.00 mm, respectively. The structure was solved by direct methods in space group $P1$, since no interpretable Fourier maps could be derived in space group $P\bar{1}$. After detection of an inversion center, the coordinates were transformed to comply with space group $P\bar{1}$. The aromatic H atoms were included in the refinement at calculated positions, riding on their carrier atoms. The hydroxyl H atom was included in the refinement in a rigid-group position, allowing for rotation along the C—O bond while retaining a C—O—H angle of 109.5°. The starting position was derived from the maximum electron density found in a circular $\Delta\rho$ map around the hydroxyl O atom. The H atoms were refined with a fixed isotropic displacement parameter related to the equivalent isotropic displacement parameter of the carrier atom by a factor of 1.2 for the aromatic H atoms and 1.5 for the hydroxyl H atoms. All non-H atoms were refined with anisotropic displacement parameters.

Data collection: locally modified *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *SET*4 (de Boer & Duisenberg, 1984). Data reduction: *HELENA* (Spek, 1993). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *PLUTON* (Spek, 1994). Software used to prepare material for publication: *PLATON* (Spek, 1990).

The X-ray data were kindly collected by A. M. M. Schreurs.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, together with a displacement ellipsoid plot of molecule 2, have been deposited with the IUCr (Reference: AB1365). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Three Testosterone Derivatives

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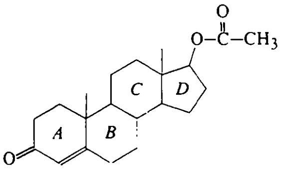
Abstract

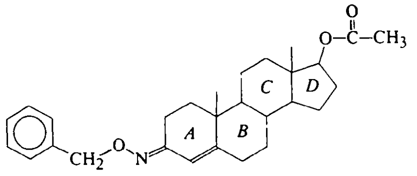
The crystal structures of three testosterone derivatives are discussed and compared. Both *anti*- and *syn*-3-benzyloxyiminoandrost-4-en-17β-yl acetate, $C_{28}H_{37}NO_3$, and their parent compound, 3-oxoandrost-4-en-17β-yl acetate, $C_{21}H_{30}O_3$, are important for the correlation between the *syn–anti* geometry of unsaturated oximes and their circular dichroism for the longest wavelength CD bands.

Comment

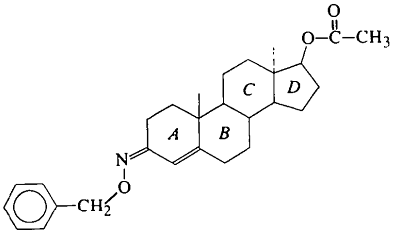
To the best of our knowledge, no crystal structures of 3-oximino steroid derivatives have been determined so far. In this study, we present the structures of *anti*- and *syn*-3-benzyloxyiminoandrost-4-en-17β-yl acetate [compounds (2) and (3), respectively] together with that of their parent compound, 3-oxoandrost-4-en-17β-yl acetate, (1).



(1)



(2)



(3)

The steroid ring systems of all three compounds are superimposable with very small r.m.s. deviations [(1) *versus* (2) = 0.078 and (1) *versus* (3) = 0.123 Å]. The conformation of the 17β-acetate group in (1) is different from that in both the oximino derivatives (2) and (3). Ring *A* of the steroid ring system of (1) is in a half-chair conformation, with the C1 atom 0.57 (1) Å beneath the plane of the other five ring atoms (Fig. 1). Rings *B* and *C* are both in chair conformations. Ring *D* has an envelope conformation with the C13 atom 0.704 (9) Å above the plane of the other four atoms of the ring. Preliminary crystal data for (1) have been reported previously (Ohrt, Haner & Norton, 1965).

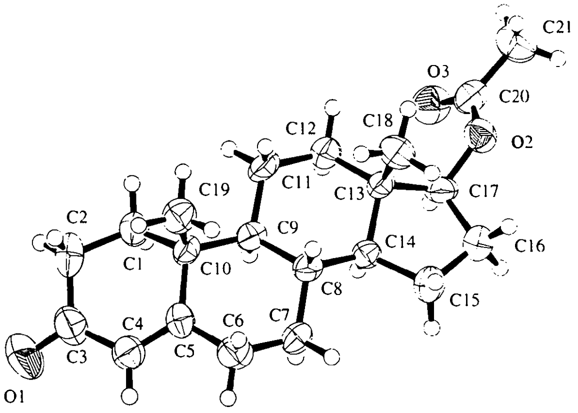


Fig. 1. The molecular structure and atomic numbering for compound (1). Displacement ellipsoids are plotted at the 50% probability level.

In compound (2) (Fig. 2), the oximino moiety is conjugated with the C4═C5 double bond of steroid ring *A*. This conjugation leads to the formation of a plane of eight atoms which makes an interplanar angle of 25.7 (3)° with the plane through atoms C20–C26. The *anti* conformation of the oximino bond is characterized by an O1—N1—C3—C4 torsion angle of 178.6 (6)°.

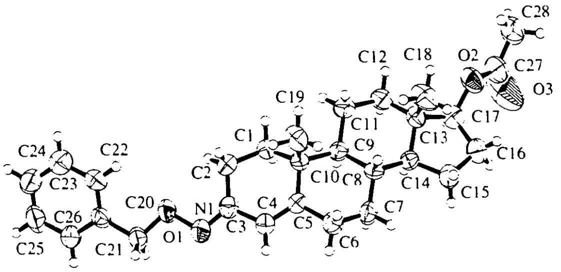


Fig. 2. The molecular structure and atomic numbering for compound (2). Displacement ellipsoids are plotted at the 50% probability level.

In compound (3) (Fig. 3), the conformation around the C3 atom is reversed compared with the situation in compound (2). This is manifested in an opening of the N1—C3—C4 angle and a slight lengthening of the N1—C3 bond as a result of the close interaction of the O1 and C4 atoms. The plane formed by the eight atoms around the oximino moiety is less perfect than that of compound (2). The corresponding interplanar angle is 81.2 (1)°. The *syn* conformation of the oximino bond is characterized by an O1—N1—C3—C4 torsion angle of 0.4 (7)°.

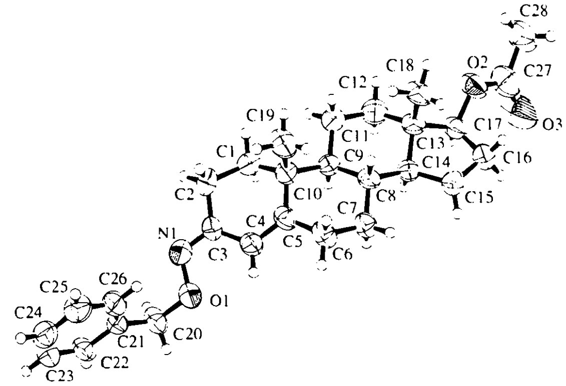


Fig. 3. The molecular structure and atomic numbering for compound (3). Displacement ellipsoids are plotted at the 50% probability level.

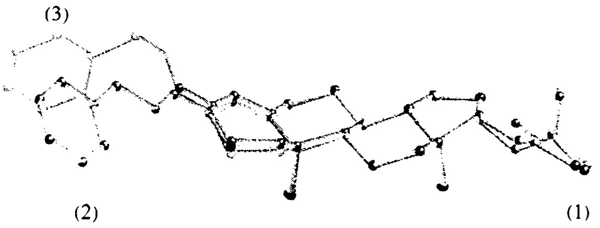


Fig. 4. Superposition of molecules (1)–(3).

Experimental

All three title compounds were synthesized according to Bodor & Barabás (1979).

Compound (1)

Crystal data

$C_{21}H_{30}O_3$
$M_r = 330.45$
Orthorhombic
$P2_12_12_1$
$a = 12.801\ (4)$ Å
$b = 18.168\ (4)$ Å
$c = 7.866\ (4)$ Å
$V = 1829.4\ (11)$ Å^3
$Z = 4$
$D_x = 1.200$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 16 reflections
$\theta = 40.29$–$53.32°$
$\mu = 0.616$ mm^{-1}
$T = 296\ (2)$ K
Needle
0.70 × 0.15 × 0.10 mm
Transparent

Data collection

Rigaku AFC-6*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2106 measured reflections
2106 independent reflections
775 observed reflections $[I > 2\sigma(I)]$

$\theta_{\max} = 75.13°$
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 22$
$l = 0 \rightarrow 9$
3 standard reflections monitored every 150 reflections intensity decay: 2.33%

Refinement

Refinement on F^2
$R(F) = 0.0492$
$wR(F^2) = 0.1746$
$S = 1.079$
2101 reflections
224 parameters
Only H-atom *U*'s refined
$w = 1/[\sigma^2(F_o^2) + (0.0509P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.003$
$\Delta\rho_{\max} = 0.191$ e Å^{-3}
$\Delta\rho_{\min} = -0.199$ e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0031 (5)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983)
Flack parameter = 0.32 (89)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (1)*

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	$U_{\rm eq}$
O1	0.2814 (5)	0.6731 (3)	−0.1225 (8)	0.114 (3)
O2	0.8803 (4)	0.2796 (2)	0.2204 (6)	0.0542 (13)
O3	0.8922 (4)	0.3630 (2)	0.4305 (7)	0.0688 (15)
C1	0.4017 (6)	0.5310 (4)	0.1232 (9)	0.059 (2)
C2	0.3072 (5)	0.5775 (4)	0.0759 (11)	0.069 (2)
C3	0.3222 (6)	0.6138 (4)	−0.0926 (12)	0.068 (2)
C4	0.3829 (5)	0.5730 (4)	−0.2168 (10)	0.054 (2)
C5	0.4314 (5)	0.5099 (3)	−0.1848 (9)	0.044 (2)
C6	0.4840 (6)	0.4697 (4)	−0.3259 (9)	0.059 (2)
C7	0.5955 (5)	0.4466 (4)	−0.2789 (8)	0.052 (2)
C8	0.5978 (5)	0.4026 (3)	−0.1141 (8)	0.041 (2)
C9	0.5467 (5)	0.4475 (3)	0.0302 (8)	0.041 (2)
C10	0.4337 (5)	0.4743 (3)	−0.0106 (8)	0.042 (2)
C11	0.5554 (5)	0.4061 (4)	0.1973 (8)	0.052 (2)
C12	0.6684 (5)	0.3844 (3)	0.2448 (8)	0.052 (2)
C13	0.7191 (5)	0.3417 (3)	0.1004 (8)	0.040 (2)
C14	0.7103 (5)	0.3859 (3)	−0.0637 (8)	0.041 (2)
C15	0.7827 (6)	0.3467 (4)	−0.1896 (9)	0.063 (2)
C16	0.8709 (6)	0.3173 (4)	−0.0751 (9)	0.062 (2)
C17	0.8384 (5)	0.3351 (4)	0.1091 (9)	0.048 (2)
C18	0.6721 (5)	0.2631 (3)	0.0861 (10)	0.062 (2)
C19	0.3541 (5)	0.4100 (4)	−0.0106 (10)	0.060 (2)
C20	0.9062 (6)	0.3015 (4)	0.3780 (10)	0.056 (2)
C21	0.9534 (6)	0.2401 (4)	0.4790 (11)	0.076 (2)

Compound (2)

Crystal data

$C_{28}H_{37}NO_3$
$M_r = 435.59$
Orthorhombic
$P2_12_12_1$
$a = 10.207\ (5)$ Å
$b = 40.174\ (7)$ Å
$c = 6.074\ (13)$ Å
$V = 2491\ (6)$ Å^3
$Z = 4$
$D_x = 1.162$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 14 reflections
$\theta = 42.18$–$49.27°$
$\mu = 0.582$ mm^{-1}
$T = 296\ (2)$ K
Plate
1.0 × 0.5 × 0.1 mm
Transparent

Data collection

Rigaku AFC-6*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2798 measured reflections
2798 independent reflections
1044 observed reflections $[I > 2\sigma(I)]$

$\theta_{\max} = 75.17°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 50$
$l = 0 \rightarrow 7$
3 standard reflections monitored every 150 reflections intensity decay: 0.10%

Refinement

Refinement on F^2
$R(F) = 0.0520$
$wR(F^2) = 0.2097$
$S = 1.075$
2789 reflections
294 parameters
Only H-atom *U*'s refined
$w = 1/[\sigma^2(F_o^2) + (0.0545P)^2 + 1.0367P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = -0.027$
$\Delta\rho_{\max} = 0.182$ e Å^{-3}
$\Delta\rho_{\min} = -0.182$ e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0013 (3)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983)
Flack parameter = −0.46 (99)

Table 2. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (2)*

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	$U_{\rm eq}$
O1	0.3490 (5)	0.81429 (10)	0.8803 (9)	0.073 (2)
O2	0.0704 (5)	0.54522 (11)	0.3830 (9)	0.075 (2)
O3	−0.0311 (10)	0.5138 (2)	0.6270 (15)	0.171 (4)
N1	0.4325 (6)	0.78668 (13)	0.9160 (11)	0.066 (2)
C1	0.2309 (7)	0.72631 (14)	0.6098 (14)	0.069 (2)
C2	0.2872 (7)	0.76176 (15)	0.6344 (14)	0.074 (2)
C3	0.3981 (7)	0.7621 (2)	0.7943 (13)	0.054 (2)

C4	0.4776 (7)	0.73215 (14)	0.8101 (12)	0.049 (2)
C5	0.4522 (6)	0.70412 (15)	0.6996 (11)	0.046 (2)
C6	0.5453 (6)	0.67513 (14)	0.7193 (14)	0.063 (2)
C7	0.4728 (7)	0.64256 (14)	0.7628 (14)	0.063 (2)
C8	0.3692 (6)	0.63589 (14)	0.5926 (11)	0.044 (2)
C9	0.2704 (7)	0.66482 (14)	0.5849 (11)	0.047 (2)
C10	0.3346 (7)	0.70016 (15)	0.5528 (11)	0.047 (2)
C11	0.1587 (7)	0.6580 (2)	0.4276 (15)	0.084 (3)
C12	0.0941 (7)	0.62397 (15)	0.4688 (14)	0.073 (2)
C13	0.1918 (7)	0.59626 (15)	0.4642 (11)	0.050 (2)
C14	0.2976 (6)	0.60366 (13)	0.6343 (11)	0.046 (2)
C15	0.3701 (7)	0.57073 (14)	0.6565 (15)	0.071 (2)
C16	0.2652 (7)	0.5441 (2)	0.6273 (15)	0.077 (3)
C17	0.1419 (7)	0.5628 (2)	0.5509 (13)	0.063 (2)
C18	0.2463 (9)	0.5904 (2)	0.2333 (13)	0.092 (3)
C19	0.3807 (9)	0.7042 (2)	0.3094 (12)	0.084 (3)
C20	0.3870 (7)	0.83998 (14)	1.0270 (12)	0.061 (2)
C21	0.3103 (7)	0.8708 (2)	0.9737 (13)	0.053 (2)
C22	0.2469 (7)	0.8744 (2)	0.7785 (15)	0.070 (2)
C23	0.1799 (9)	0.9035 (2)	0.7330 (16)	0.086 (3)
C24	0.1754 (8)	0.9290 (2)	0.8806 (16)	0.080 (3)
C25	0.2378 (8)	0.9252 (2)	1.0763 (17)	0.081 (3)
C26	0.3042 (7)	0.8962 (2)	1.1216 (14)	0.070 (2)
C27	−0.0148 (9)	0.5222 (2)	0.4394 (20)	0.085 (3)
C28	−0.0798 (8)	0.5072 (2)	0.2445 (19)	0.102 (3)

Compound (3)

Crystal data

$C_{28}H_{37}NO_3$
$M_r = 435.59$
Monoclinic
$P2_1$
$a = 13.507$ (3) Å
$b = 6.117$ (5) Å
$c = 15.250$ (3) Å
$\beta = 92.582$ (15)°
$V = 1258.7$ (10) Å^3
$Z = 2$
$D_x = 1.149$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 20 reflections
$\theta = 30.57–54.26°$
$\mu = 0.576$ mm^{-1}
$T = 296$ (2) K
Plate
1.0 × 0.5 × 0.3 mm
Transparent

Data collection

Rigaku AFC-6*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2842 measured reflections
2733 independent reflections
1397 observed reflections $[I > 2\sigma(I)]$

$R_{\text{int}} = 0.0454$
$\theta_{\max} = 75.19°$
$h = -16 \rightarrow 16$
$k = 0 \rightarrow 7$
$l = 0 \rightarrow 19$
3 standard reflections monitored every 150 reflections intensity decay: 0.95%

Refinement

Refinement on F^2
$R(F) = 0.0476$
$wR(F^2) = 0.1629$
$S = 1.072$
2729 reflections
293 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(F_o^2) + (0.0496P)^2 + 0.4758P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.139$ e Å^{-3}
$\Delta\rho_{\min} = -0.168$ e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0033 (6)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983)
Flack parameter = −0.27 (70)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (3)*

$$U_{\text{eq}} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O1	0.7212 (2)	0.7906	0.0583 (2)	0.0730 (11)
O2	0.1830 (3)	1.2880 (11)	0.5605 (2)	0.0779 (12)
O3	0.1253 (5)	1.0126 (14)	0.6368 (4)	0.136 (2)
N1	0.7514 (3)	0.9682 (12)	0.1114 (3)	0.0745 (14)
C1	0.6215 (4)	1.2652 (15)	0.2673 (4)	0.078 (2)
C2	0.6948 (4)	1.2680 (15)	0.1938 (4)	0.088 (2)
C3	0.6749 (4)	1.0769 (13)	0.1364 (3)	0.066 (2)
C4	0.5717 (3)	1.0231 (12)	0.1137 (3)	0.0587 (14)
C5	0.4969 (4)	1.1170 (12)	0.1560 (3)	0.0569 (14)
C6	0.3910 (3)	1.0716 (14)	0.1254 (3)	0.068 (2)
C7	0.3259 (3)	1.0082 (14)	0.2013 (3)	0.0617 (14)
C8	0.3347 (3)	1.1728 (12)	0.2764 (3)	0.0504 (12)
C9	0.4446 (4)	1.2001 (12)	0.3080 (3)	0.0524 (12)
C10	0.5122 (4)	1.2721 (12)	0.2322 (4)	0.0629 (15)
C11	0.4553 (4)	1.3481 (14)	0.3886 (4)	0.079 (2)
C12	0.3895 (4)	1.2784 (16)	0.4629 (3)	0.080 (2)
C13	0.2811 (4)	1.2598 (12)	0.4314 (3)	0.0581 (14)
C14	0.2759 (3)	1.1007 (12)	0.3535 (3)	0.0496 (12)
C15	0.1646 (3)	1.0484 (13)	0.3404 (3)	0.0650 (15)
C16	0.1251 (4)	1.0619 (14)	0.4338 (3)	0.072 (2)
C17	0.2132 (4)	1.1423 (12)	0.4922 (3)	0.0602 (14)
C18	0.2385 (5)	1.4850 (12)	0.4078 (4)	0.082 (2)
C19	0.4876 (5)	1.5028 (13)	0.1995 (5)	0.098 (2)
C20	0.8083 (4)	0.6678 (14)	0.0378 (4)	0.070 (2)
C21	0.8641 (3)	0.7648 (12)	−0.0374 (3)	0.0502 (12)
C22	0.9454 (3)	0.6496 (13)	−0.0636 (3)	0.0617 (15)
C23	1.0002 (4)	0.7246 (14)	−0.1312 (4)	0.074 (2)
C24	0.9742 (5)	0.9144 (15)	−0.1737 (4)	0.082 (2)
C25	0.8933 (5)	1.0334 (15)	−0.1475 (4)	0.079 (2)
C26	0.8376 (4)	0.9565 (13)	−0.0792 (3)	0.065 (2)
C27	0.1393 (5)	1.2066 (18)	0.6287 (5)	0.089 (2)
C28	0.1100 (5)	1.3803 (17)	0.6910 (4)	0.105 (3)

For all compounds, data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985, 1992); program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); software used to prepare material for publication: *TEXSAN FINISH*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the IUCr (Reference: KA1197). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Boc–Phe–Aminomalonaldehyde

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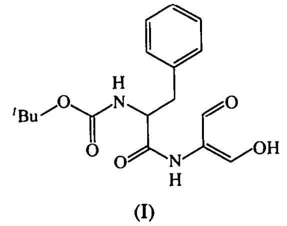
Abstract

The crystal structure of the title compound, (*N-tert*-butoxycarbonylphenylalanyl)aminomalonaldehyde {*tert*-butyl [1-(diformylmethylaminocarbonyl)-2-phenyl-1-ethyl]carbamate, $C_{17}H_{22}N_2O_5$}, was determined in order to examine the aminomalonaldehyde group in a peptide chain. The planarity constraints of the peptide bond and the aminomalonaldehyde group result in the formation of a pseudo-seven-membered ring through one intramolecular hydrogen bond (O17—H17···O11). The bond lengths in this ring indicate delocalization of the π electrons and the formation of a weak conjugated π-electron system.

Comment

Aminomalonaldehyde (Ama) contains both a primary amino group and an acidic group, namely an enolized malonaldehyde moiety, with acidity comparable to that of a carboxylic acid group; it can thus be considered as an anomalous amino acid. Following the idea of Ama incorporation into the peptide chain, we have condensed Boc–Phe–OSu with an alkaline solution of Ama (Arnold, Šauliová & Krchňák, 1973) and obtained the title compound, (I).



(I)

The molecular structure of Boc–Phe–aminomalonaldehyde, together with the atom-numbering scheme, is shown in Fig. 1. In the molecule, there are several potentially planar groups, of which we are interested mainly in the spatial arrangement of the peptide and Ama groups. The planarity constraints induced by these groups result in the eight non-H atoms C9, C10, O11, N12, C13, C14, O15, C16 and O17 lying in the same plane. The structure determination shows that they form a pseudo-seven-membered ring (comprising atoms O11, C10, N12, C13, C16, O17 and H17) through the creation of an intramolecular hydrogen bond between the O11 atom as a proton acceptor and O17 as a proton donor. The observed parameters indicate a relatively strong hydrogen bond (O11···O17 2.51 Å and O11···H17—O17 164.7°) in comparison with published values (Glusker, Lewis & Rossi, 1994). The closure of the chain to form the ring provoked some deviations from the predicted planar arrangement of the participating groups. The five atoms pertaining to the C13═C16 double bond (N12, C13, C16, O17 and C14) are not perfectly coplanar (the r.m.s. deviation of the fitted least-squares plane through these atoms is about 0.014 Å). The peptide group also deviates from a planar arrangement (r.m.s. deviation of about 0.018 Å). This non-planarity is formed by a twist about the C—N bond ($\Delta\omega = -3°$) and by a pyramidal deformation about the N atom ($\tau_N = 1.5°$) (Ramachandran & Kolaskar, 1973). Furthermore, in the seven-membered ring, the observed lengths of the single bonds are shorter and those of the double bonds and the peptide bond are longer than theoretical lengths (Glusker, Lewis & Rossi, 1994), which indicates the formation of a weak conjugated π-electron system. The phenyl ring and the seven-membered ring are approximately parallel to one another; the dihedral angle between them is 5.3 (1)°.

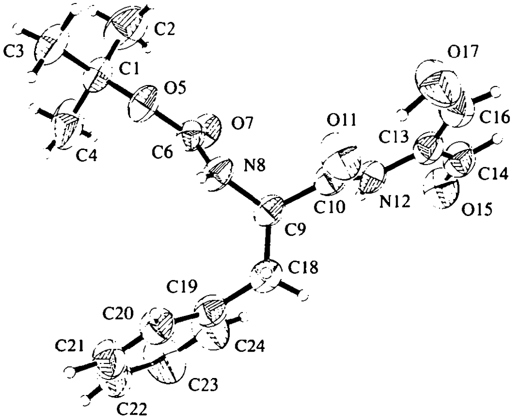


Fig. 1. A view of Boc–Phe–Ama showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn as small circles of arbitrary radii.

The crystal packing is realised by a number of intermolecular hydrogen bonds: N8—H8···O15($\frac{3}{2}-y$, $\frac{1}{2}+x$, $\frac{1}{4}+z$) with N8···O15 3.19 Å and N8—H8···O15 175.6°; N12—H12···O7(y, x, $-z$) with N12···O7 3.06 Å and N12—H12···O7 160.0°. Furthermore, the