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

O(1)—C(1)	1·263 (1)	C(3)—C(4)	1·380 (1)
O(2)—C(11)	1·312 (1)	C(4)—C(5)	1·383 (2)
O(3)—C(12)	1·324 (1)	C(5)—C(6)	1·385 (2)
O(3)—C(13)	1·459 (1)	C(6)—C(7)	1·390 (1)
O(4)—C(12)	1·199 (1)	C(7)—C(8)	1·507 (1)
C(1)—C(2)	1·471 (1)	C(8)—C(9)	1·517 (1)
C(1)—C(10)	1·447 (1)	C(9)—C(10)	1·515 (1)
C(2)—C(3)	1·404 (1)	C(10)—C(11)	1·371 (1)
C(2)—C(7)	1·398 (1)	C(11)—C(12)	1·511 (1)
O(2)—H(20)	1·16 (3)		
C(12)—O(3)—C(13)	116·39 (8)	C(6)—C(7)—C(8)	122·00 (9)
O(1)—C(1)—C(2)	119·30 (8)	C(7)—C(8)—C(9)	111·82 (8)
O(1)—C(1)—C(10)	121·21 (8)	C(8)—C(9)—C(10)	111·34 (8)
C(2)—C(1)—C(10)	119·45 (7)	C(1)—C(10)—C(9)	117·81 (8)
C(1)—C(2)—C(3)	119·88 (8)	C(1)—C(10)—C(11)	117·80 (7)
C(1)—C(2)—C(7)	119·94 (8)	C(9)—C(10)—C(11)	124·31 (8)
C(3)—C(2)—C(7)	120·18 (8)	O(2)—C(11)—C(10)	123·70 (8)
C(2)—C(3)—C(4)	119·6 (1)	O(2)—C(11)—C(12)	114·04 (7)
C(3)—C(4)—C(5)	120·1 (1)	C(10)—C(11)—C(12)	122·22 (7)
C(4)—C(5)—C(6)	120·8 (1)	O(3)—C(12)—O(4)	124·64 (8)
C(5)—C(6)—C(7)	120·0 (1)	O(3)—C(12)—C(11)	111·61 (7)
C(2)—C(7)—C(6)	119·29 (9)	O(4)—C(12)—C(11)	123·72 (8)
C(2)—C(7)—C(8)	118·67 (8)		
C(11)—O(2)—H(20)	104 (1)		
C(13)—O(3)—C(12)—O(4)	1·0 (2)	C(8)—C(9)—C(10)—C(1)	−34·0 (2)
C(13)—O(3)—C(12)—C(11)	−177·29 (12)	C(8)—C(9)—C(10)—C(11)	149·46 (14)
O(1)—C(1)—C(2)—C(7)	−164·59 (13)	C(1)—C(10)—C(11)—O(2)	5·1 (2)
C(10)—C(1)—C(2)—C(7)	17·6 (2)	C(1)—C(10)—C(11)—C(12)	−172·59 (12)
O(1)—C(1)—C(10)—C(9)	−178·37 (13)	C(9)—C(10)—C(11)—O(2)	−178·36 (14)
O(1)—C(1)—C(10)—C(11)	−1·6 (2)	C(9)—C(10)—C(11)—C(12)	3·95 (21)
C(2)—C(1)—C(10)—C(9)	−0·6 (2)	O(2)—C(11)—C(12)—O(3)	28·87 (17)
C(2)—C(1)—C(10)—C(11)	176·15 (12)	O(2)—C(11)—C(12)—O(4)	−149·47 (14)
C(1)—C(2)—C(7)—C(8)	2·74 (20)	C(10)—C(11)—C(12)—O(3)	−153·24 (13)
C(2)—C(7)—C(8)—C(9)	−38·0 (2)	C(10)—C(11)—C(12)—O(4)	28·42 (21)
C(7)—C(8)—C(9)—C(10)	52·2 (2)		

Related literature. Structure of 2-acetyl-1-tetralone: Geoffroy, Jain, Celalyan & Bernardinelli (1983); structures of several tetracycline derivatives: Stezowski (1976).

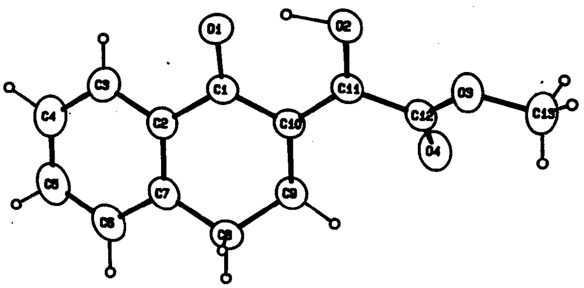

Fig. 1. Numbering scheme of title compound; thermal ellipsoids are drawn at the 40% probability level. H atoms are drawn as circles with the same arbitrary radius.

Support for this work is provided by a grant from the National Institutes of Health.

References

Brown, E., Touet, J. & Ragault, M. (1972). *Bull. Soc. Chim. Fr.* pp. 212–220.

Cromer, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Frenz, B. A. & Okaya, Y. (1980). *Enraf–Nonius Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.

Geoffroy, M., Jain, A., Celalyan, A. & Bernardinelli, G. (1983). *Z. Naturforsch. Teil B*, **38**, 830–834.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

Stezowski, J. J. (1976). *J. Am. Chem. Soc.* **98**, 6012–6018.

Acta Cryst. (1989). C**45**, 690–692

Structure of *N-tert*-Butoxycarbonyl-L-tyrosine 4-Bromophenacyl Ester

By J. Matsoukas, M. Semertzidis, J. Hondrelis, V. Nastopoulos and S. Voliotis*

Department of Chemistry, University of Patras, GR-26110 Patras, Greece

and Ivan Leban

Department of Chemistry and Chemical Technology, Edvard Kardelj University, Murnikova 6, PO Box 537, 61001 Ljubljana, Yugoslavia

(*Received* 12 *May* 1988; *accepted* 4 *November* 1988)

Abstract. $C_{22}H_{24}BrNO_6$, $M_r = 478{\cdot}3$, monoclinic, $P2_1$, $a = 5{\cdot}456\ (1)$, $b = 10{\cdot}158\ (1)$, $c = 20{\cdot}239\ (5)$ Å, $\beta = 95{\cdot}83\ (2)°$, $V = 1115{\cdot}9\ (6)$ Å^3, $Z = 2$, $D_x = 1{\cdot}423$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 19{\cdot}89$ cm^{-1}, $F(000) = 492$, room temperature, $R = 0{\cdot}085$ for 962 unique observed reflections. The urethane amide bond

---

* To whom all correspondence should be addressed.

0108-2701/89/040690-03$03.00

adopts the *trans* conformation [C(14)—N(23)—C(24)—O(26) = 170 (4)°]. Neighbouring molecules are interconnected along **b** by hydrogen bonds [O(22)···O(13) = 2·75 (5) Å] between the ester C=O and the tyrosine hydroxyl group. The butoxycarbonyl (Boc) moiety is directed away from the tyrosine aromatic ring and is close to the ester CO group, in contrast to the arrangement observed in Boc-phenylalanine phenacyl ester [Vlassi, Germain, Matsoukas, Psachoulia, Voliotis & Leban (1987). *Acta Cryst.* C**43**, 2173–2175]. The orientation assumed by the Boc group may be the result of steric restrictions imposed by the presence of the tyrosine hydroxyl group and affects the reactivity during esterification of Boc-amino acids.

Experimental. Recrystallization from ethyl acetate/petroleum ether, m.p. = 420 K. Transparent prismatic crystal 0·32 × 0·32 × 0·48 mm with prominent axis [100]; Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation. Lattice parameters determined from 25 reflections ($7 < \theta < 11°$); $\omega/2\theta$ scan technique up to $2\theta_{\max} = 56°$; 2θ scan width $(0{\cdot}8 + 0{\cdot}3\tan\theta)°$, scan rate 1·18–5·49° min^{-1}, background 1/4 of scan time at each scan limit, max. scan time 60 s, aperture $(2{\cdot}4 + 0{\cdot}9\tan\theta)$ mm. Measured reflections 6375, $-5 < h < 5$, $0 < k < 9$, $-19 < l < 19$, averaged 2843, $R_{\rm int} = 0{\cdot}053$. Reference reflections (210, 11$\bar{3}$, 12$\bar{4}$ after 2 h), orientation control reflections (12$\bar{2}$, 053, 131) measured in every 300, intensity decrease 0·05%. Structure solved with *MULTAN*. $R = 0{\cdot}085$ for 962 unique observed reflections with $I > 1{\cdot}5\sigma(I)$; $wR = 0{\cdot}075$; $\sum w(\Delta F)^2$ minimized, $w = 1{\cdot}2859/[\sigma^2(F) + 0{\cdot}002651F^2]$; max. $\Delta/\sigma < 0{\cdot}4$; max. and min. electron densities in final difference map within $\pm 0{\cdot}15$ e Å^{-3}. All non-H atoms refined with anisotropic thermal parameters, H atoms were calculated, riding model (C—H = 1·08 Å). Intensities corrected for Lp, absorption and extinction corrections not used; atomic scattering factors from *SHELX*76. Computer programs used: *MULTAN*87 (Debaerdemaeker, Germain, Main, Tate & Woolfson, 1987), *DIRDIF* (Beurskens, Bosman, Doesburg, Gould, van den Hark, Prick, Noordik, Beurskens & Parthasarathi, 1981), *PATSEE* (Egert, 1985), *SHELX*76 (Sheldrick, 1976), *PLUTO* (Motherwell & Clegg, 1978). The low precision of the structure and the high level of some thermal parameters are due to poor crystal quality yielding a weak diffraction pattern. The atomic coordinates and temperature factors are listed in Table 1.* Selected distances and angles are presented in Table 2. A perspective drawing of the molecule and the

Table 1. *Atomic parameters*

$$B_{\rm eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
Br(1)	8705 (6)	2018	4408 (1)	9·73
C(2)	8424 (57)	3984 (17)	4221 (10)	8·38
C(3)	6589 (44)	4614 (23)	4525 (11)	6·45
C(4)	6341 (44)	5904 (28)	4397 (11)	6·83
C(5)	7827 (33)	6512 (24)	3994 (11)	5·30
C(6)	9613 (39)	5824 (21)	3702 (10)	5·02
C(7)	9925 (40)	4620 (30)	3858 (7)	7·84
C(8)	7365 (62)	7957 (29)	3779 (11)	7·00
O(9)	5624 (39)	8496 (19)	4011 (9)	9·91
C(10)	9059 (47)	8673 (33)	3344 (12)	8·47
O(11)	7885 (28)	9777 (15)	3049 (7)	5·93
C(12)	6061 (43)	9605 (28)	2581 (12)	6·05
O(13)	5200 (30)	8521 (14)	2461 (8)	5·84
C(14)	5159 (32)	10852 (12)	2249 (12)	4·36
C(15)	3237 (49)	11483 (20)	2639 (13)	5·76
C(16)	2789 (34)	12900 (30)	2423 (10)	5·57
C(17)	4476 (32)	13896 (28)	2601 (10)	5·81
C(18)	4045 (49)	15225 (21)	2443 (12)	5·77
C(19)	1780 (49)	15545 (27)	2027 (9)	6·96
C(20)	221 (42)	14510 (32)	1850 (12)	9·25
C(21)	800 (43)	13215 (17)	2046 (11)	5·45
O(22)	1535 (24)	16879 (18)	1901 (7)	7·13
N(23)	4354 (30)	10645 (17)	1567 (7)	4·79
C(24)	6111 (47)	10207 (18)	1192 (12)	4·52
O(25)	8313 (28)	10204 (13)	1384 (6)	5·43
O(26)	5118 (23)	9846 (15)	604 (7)	4·22
C(27)	6507 (40)	9212 (20)	128 (11)	5·00
C(28)	8408 (40)	10230 (30)	−99 (11)	7·97
C(29)	7666 (53)	7998 (26)	405 (13)	10·15
C(30)	4698 (40)	8876 (27)	−461 (11)	6·48

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

Br(1)—C(2)	2·03 (2)	C(24)—N(23)—C(14)	116 (2)
C(8)—O(9)	1·23 (3)	C(28)—C(27)—O(26)	108 (2)
C(12)—O(13)	1·21 (3)	C(29)—C(27)—O(26)	110 (2)
C(19)—O(22)	1·39 (3)	C(29)—C(27)—C(28)	113 (2)
C(24)—O(25)	1·23 (2)	C(30)—C(27)—O(26)	106 (2)
C(27)—O(26)	1·44 (2)	C(30)—C(27)—C(28)	109 (2)
C(27)—C(28)	1·56 (3)	C(30)—C(27)—C(29)	109 (2)
C(27)—C(29)	1·47 (3)	C(14)—N(23)—C(24)—O(26)	170 (4)
C(27)—C(30)	1·50 (3)	O(11)—C(12)—C(14)—N(23)	146 (3)
O(22)—O(13$^{\rm i}$)	2·75 (5)	C(16)—C(15)—C(14)—N(23)	−69 (4)

Symmetry code: (i) $x, y-1, z$.

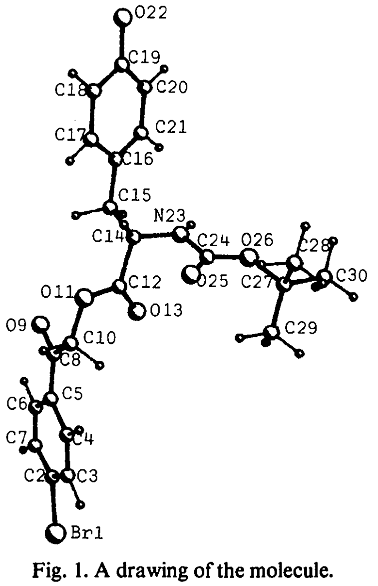

Fig. 1. A drawing of the molecule.

---

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and complete molecular geometry tables have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51566 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

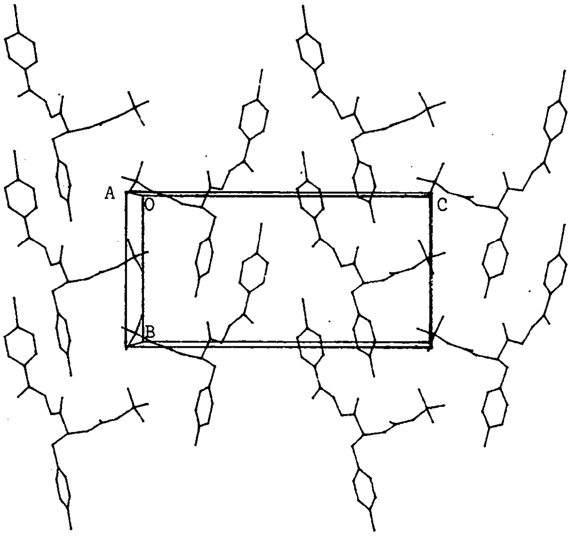

Fig. 2. A view of the unit-cell packing.

crystal packing arrangement are shown in Figs. 1 and 2.

Related literature. For the preparation and properties of the title compound and related derivatives see Nagasawa, Kuroiwa, Narita & Isowa (1973); Stelakatos, Panagou & Zervas (1966); Matsoukas, Moharir & Findlay (1983); Findlay, Dalzeet, Matsoukas & Moharir (1984); Vlassi, Germain, Matsoukas, Psachoulia, Voliotis & Leban (1987); Matsoukas, Goghari, Scanlon, Franklin & Moore (1985); Moore & Matsoukas (1985). Knowledge of the conformation of the title compound and related tyrosine derivatives is desirable for the design and synthesis of effective inhibitors for diagnostic and therapeutic applications.

References

BEURSKENS, P. T., BOSMAN, W. P., DOESBURG, H. M., GOULD, R. O., VAN DEN HARK, TH. E. M., PRICK, P. A. J., NOORDIK, J. H., BEURSKENS, G. & PARTHASARATHI, V. (1981). *DIRDIF*. Tech. Rep. 1981/2. Crystallography Laboratory, Toernooiveld, 6525 ED Nijmegen, The Netherlands.

DEBAERDEMAEKER, T., GERMAIN, G., MAIN, P., TATE, C. & WOOLFSON, M. M. (1987). *MULTAN87. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

EGERT, E. (1985). *PATSEE. Fragment Search by Integrated Patterson and Direct Methods*. Univ. of Göttingen, Federal Republic of Germany.

FINDLAY, J., DALZEET, A., MATSOUKAS, J. & MOHARIR, Y. E. (1984). *J. Nat. Prod.* **47**, 560–561.

MATSOUKAS, J., GOGHARI, M., SCANLON, M., FRANKLIN, K. & MOORE, G. (1985). *J. Med. Chem.* **28**, 780–783.

MATSOUKAS, J., MOHARIR, Y. E. & FINDLAY, J. (1983). *J. Nat. Prod.* **46**, 582–585.

MOORE, G. & MATSOUKAS, J. (1985). *Biosci. Rep.* **5**, 407–416.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

NAGASAWA, T., KUROIWA, K., NARITA, K. & ISOWA, Y. (1973). *Bull. Chem. Soc. Jpn*, **46**, 1269–1272.

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

STELAKATOS, A., PANAGOU, A. & ZERVAS, L. (1966). *J. Chem. Soc. C*, pp. 1191–1199.

VLASSI, M., GERMAIN, G., MATSOUKAS, J., PSACHOULIA, C., VOLIOTIS, S. & LEBAN, I. (1987). *Acta Cryst.* C**43**, 2173–2175.

Acta Cryst. (1989). C**45**, 692–694

1-Mesyl-4-[2-(methoxycarbonyl)ethyl]-3-(methoxycarbonyl)methyl-*N*,*N*-dimethylpyrrole-2-carboxamide

BY FRANK H. ALLEN, ALAN R. BATTERSBY, JAMES J. DE VOSS, MICHAEL J. DOYLE AND PAUL R. RAITHBY

University Chemical Laboratory, Lensfield Road, Cambridge CB2 1EW, England

(*Received* 31 *July* 1988; *accepted* 19 *October* 1988)

Abstract. Methyl 3-[5-(*N*,*N*-dimethylamino)carbonyl-1-mesyl-4-(methoxycarbonyl)methyl-3-pyrrolyl]propionate, $C_{15}H_{22}N_2O_7S$, $M_r = 374{\cdot}42$, orthorhombic, *Pcab* (non-standard setting of *Pbca*, No. 61), $a = 14{\cdot}015\,(3)$, $b = 14{\cdot}259\,(3)$, $c = 18{\cdot}444\,(4)$ Å, $V = 3685{\cdot}6\,(5)$ Å^3, $Z = 8$, $D_x = 1{\cdot}35$ Mg m^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}54178$ Å, $\mu = 1{\cdot}864$ mm^{-1}, $F(000) = 1584$, $T = 293$ K. Final $R = 0{\cdot}073$ for 2250 unique observed $[F > 4\sigma(F)]$ reflexions and 229 parameters. The pyrrole ring is planar [maximum torsion angle = 1·8 (4)°]. The carboxamide group is twisted out of the ring plane by 67·5 (6)°. The *N*-mesyl unit is rare and this is the first crystallographic study of an *N*-mesylpyrrole. The N—S bond is long at 1·668 (3) Å and corresponds to a shortened mean S=O distance of 1·418 (3) Å as expected from the study of Kálmán, Párkányi & Schawartz [*Acta Cryst.* (1977), B**33**, 3097–3102]. The S(mesyl) tetrahedron is normal [O=S=O = 119·5 (3)°, N—S—C = 104·6 (2)°, N—S=O(mean) = 106·4 (3)°, C—S=O(mean) = 109·5 (3)°] and adopts a pseudo-

0108-2701/89/040692-03$03.00