recently reported (Lynch, Stamford, Magnus & Davis, 1991).

The National Institutes of Health (GM 29801 to PM) are thanked for their support of this research.

### References

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

Gadol, S. M. & Davis, R. E. (1982). *Organometallics*, **1**, 1607–1613.

Kuehne, M. E. & Marko, I. (1990). *The Alkaloids*, Vol. 37, edited by A. Brossi & M. Suffness, pp. 77–132.

Lynch, V. M., Stamford, A., Magnus, P. & Davis, B. E. (1991). *Acta Cryst.* Accepted.

Magnus, P, Ladlow, M., Elliot, J. & Kim, C. S. (1989). *J. Chem. Soc. Chem. Commun.* pp. 518–520.

Magnus, P., Stamford, A. & Ladlow, M. (1990). *J. Am. Chem. Soc.* In the press.

Moncrief, J. W. & Lipscomb, W. N. (1966). *Acta Cryst.* **21**, 322–331.

Sheldrick, G. M. (1988). *SHELXTL-Plus88 Structure Determination Software Programs.* Nicolet Instrument Corporation, Madison, Wisconsin, USA.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

---

*Acta Cryst.* (1991). C**47**, 1345–1347

# Determination of the Absolute Configuration of a Novel Dipeptide Isostere

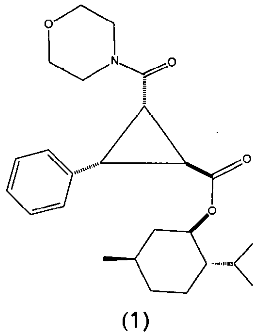
By Vincent M. Lynch, Richard E. Austin, Stephen F. Martin and Thaddeus George

*Department of Chemistry and Biochemistry, University of Texas at Austin, Austin, TX* 78712, *USA*

(*Received* 15 *October* 1990; *accepted* 20 *November* 1990)

**Abstract.** {1*R*-[1α(1*R**,2*R**,3*S**),2β,5α]}-2-(4-Morpholinocarbonyl)-3-phenylcyclopropane 2-(1-methylethyl)-5-methylcyclohexyl ester, $C_{25}H_{35}NO_4$, $M_r$ = 413·56, orthorhombic, $P2_12_12_1$, $a$ = 9·359 (2), $b$ = 10·1172 (14), $c$ = 24·710 (4) Å, $V$ = 2339·8 (6) Å$^3$, $Z$ = 4, $D_x$ = 1·17 g cm$^{-3}$, $\mu$ = 0·7332 cm$^{-1}$, Mo $K\alpha$ radiation, $\lambda$ = 0·7107 Å, $F(000)$ = 896, $T$ = 198 K, $R$ = 0·0475 for 2460 reflections [$F_o \geq 4\sigma(F_o)$]. The assignment of the absolute configuration was based on internal comparison to the (−)-menthol moiety. The morpholenyl and cyclohexyl rings are in the chair conformation. The amide group is essentially planar [max. deviation 0·025 (2) Å for N19] resulting in a close non-bonded contact between the amide oxygen, O18, and H24*a* of 2·24 (4) Å.

**Experimental.** (1) was synthesized by the dicyclohexylcarbodiimide/4-dimethylaminopyridine mediated esterification of the corresponding optically active carboxylic acid with (−)-menthol (Doyle, Pieters, Martin, Austin, Oalmann & Muller, 1991; Martin, Austin & Oalmann, 1990). Crystals of (1) were obtained by slow evaporation from a carbon tetrachloride–diethyl ether solution. The data crystal was a large block of approximate dimensions 0·56 × 0·70 × 0·76 mm. Data were collected on a Nicolet *R*3 diffractometer using a graphite monochromator and a Nicolet LT-2 low-temperature delivery system. Lattice parameters were obtained from the least-squares refinement of 40 reflections with 25·2 < 2θ < 27·8°. Data were collected using the ω-scan technique, with a 1·2° ω scan at 4–8° min$^{-1}$ and a 2θ range from 4·0–55·0° ($h$ = 0→11, $k$ = 0→12, $l$ = 0→32). 3068 reflections were collected. Four reflections (3$\bar{5}$4, $\bar{3}$,2,$\overline{14}$, $\bar{3}$,2,14, 354) were remeasured every 96 reflections to monitor instrument and crystal stability. A smoothed curve of the intensities of these check reflections was used to scale the data. The scaling factor ranged from 0·995–1·01. Data were also corrected for Lp effects but not absorption. 608 reflections were considered unobserved, $F_o < 4\sigma(F_o)$. Data reduction and decay correction were performed using the Nicolet XRD *SHELXTL-Plus* software package (Sheldrick, 1988). The structure was solved by direct methods and refined by full-matrix least squares (Sheldrick, 1988). In all, 399 parameters were refined. The non-H atoms were refined with anisotropic thermal parameters. All H-atom positions were obtained from a Δ*F* map and refined with isotropic thermal parameters. The bond lengths



(1)

0108-2701/91/061345-03$03.00

Table 1. *Fractional coordinates and equivalent isotropic thermal parameters* (Å$^2$) *for the non-*H *atoms of* (1)

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

| | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| C1 | 0·5344 (3) | 0·1830 (3) | 0·33725 (10) | 0·0359 (7) |
| C2 | 0·4372 (3) | 0·3011 (3) | 0·33906 (10) | 0·0347 (7) |
| C3 | 0·5279 (3) | 0·2759 (2) | 0·28912 (10) | 0·0342 (7) |
| C4 | 0·6645 (3) | 0·1866 (3) | 0·37111 (10) | 0·0378 (8) |
| O5 | 0·7417 (2) | 0·2811 (2) | 0·37637 (8) | 0·0531 (7) |
| O6 | 0·6822 (2) | 0·0711 (2) | 0·39622 (8) | 0·0469 (6) |
| C7 | 0·8083 (3) | 0·0528 (3) | 0·43061 (11) | 0·0469 (9) |
| C8 | 0·7565 (4) | −0·0172 (3) | 0·48224 (12) | 0·0523 (10) |
| C9 | 0·8905 (5) | −0·0486 (4) | 0·51564 (14) | 0·0731 (14) |
| C10 | 0·9956 (5) | −0·1336 (4) | 0·4836 (2) | 0·0747 (14) |
| C11 | 1·0440 (4) | −0·0667 (4) | 0·4329 (2) | 0·0630 (12) |
| C12 | 0·9144 (3) | −0·0266 (3) | 0·39911 (13) | 0·0494 (10) |
| C13 | 1·1472 (6) | −0·1489 (6) | 0·4001 (3) | 0·093 (2) |
| C14 | 0·6426 (5) | 0·0555 (4) | 0·5169 (2) | 0·0775 (15) |
| C15 | 0·4916 (5) | 0·0338 (5) | 0·4988 (2) | 0·101 (2) |
| C16 | 0·6778 (7) | 0·2022 (4) | 0·5250 (2) | 0·094 (2) |
| C17 | 0·2785 (3) | 0·2719 (3) | 0·33767 (10) | 0·0386 (8) |
| O18 | 0·2318 (2) | 0·1621 (2) | 0·34827 (8) | 0·0534 (7) |
| N19 | 0·1944 (2) | 0·3734 (3) | 0·32296 (10) | 0·0472 (8) |
| C20 | 0·2417 (3) | 0·5049 (3) | 0·30703 (13) | 0·0479 (9) |
| C21 | 0·1864 (4) | 0·5376 (4) | 0·25168 (15) | 0·0584 (11) |
| O22 | 0·0349 (2) | 0·5255 (3) | 0·24950 (10) | 0·0684 (9) |
| C23 | −0·0059 (4) | 0·3937 (4) | 0·2616 (2) | 0·0666 (13) |
| C24 | 0·0395 (3) | 0·3533 (4) | 0·3177 (2) | 0·0623 (12) |
| C25 | 0·4683 (3) | 0·2372 (2) | 0·23564 (9) | 0·0354 (7) |
| C26 | 0·3630 (3) | 0·1409 (3) | 0·22892 (12) | 0·0452 (9) |
| C27 | 0·3125 (4) | 0·1107 (3) | 0·17717 (12) | 0·0549 (10) |
| C28 | 0·3646 (4) | 0·1743 (3) | 0·13310 (13) | 0·0576 (11) |
| C29 | 0·4680 (4) | 0·2688 (3) | 0·13873 (12) | 0·0572 (11) |
| C30 | 0·5206 (3) | 0·3002 (3) | 0·18979 (11) | 0·0474 (9) |

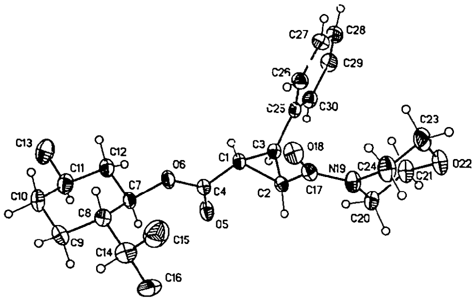


Fig. 1. View of (1) showing the atom-labeling scheme. The non-H atoms are scaled to the 30% probability level while the H atoms are drawn to an arbitrary size. Methyl H atoms are omitted for clarity.

Table 2. *Bond lengths* (Å) *and angles* (°) *for the non-*H *atoms of* (1)

| 1 | 2 | 3 | 1–2 | 1–2–3 |
|---|---|---|---|---|
| C2 | C1 | C3 | 1·503 (4) | 60·4 (2) |
| C3 | C1 | C4 | 1·517 (4) | 117·5 (2) |
| C4 | C1 | C2 | 1·478 (4) | 117·5 (2) |
| C3 | C2 | C17 | 1·519 (3) | 119·8 (2) |
| C3 | C2 | C1 | | 60·3 (2) |
| C17 | C2 | C1 | 1·514 (3) | 115·9 (2) |
| C25 | C3 | C1 | 1·487 (3) | 123·3 (2) |
| C25 | C3 | C2 | | 123·8 (2) |
| C1 | C3 | C2 | | 59·3 (2) |
| O5 | C4 | O6 | 1·205 (3) | 124·8 (2) |
| O5 | C4 | C1 | | 125·1 (2) |
| O6 | C4 | C1 | 1·334 (3) | 110·1 (2) |
| C7 | O6 | C4 | 1·466 (4) | 118·7 (2) |
| C8 | C7 | C12 | 1·537 (4) | 113·2 (2) |
| C8 | C7 | O6 | | 106·6 (2) |
| C12 | C7 | O6 | 1·496 (4) | 107·5 (2) |
| C9 | C8 | C14 | 1·535 (5) | 111·3 (3) |
| C9 | C8 | C7 | | 106·5 (3) |
| C14 | C8 | C7 | 1·553 (5) | 117·1 (3) |
| C10 | C9 | C8 | 1·528 (6) | 111·3 (3) |
| C11 | C10 | C9 | 1·494 (6) | 112·0 (3) |
| C12 | C11 | C13 | 1·527 (5) | 111·2 (4) |
| C12 | C11 | C10 | | 109·7 (3) |
| C13 | C11 | C10 | 1·510 (7) | 113·2 (4) |
| C7 | C12 | C11 | | 112·7 (3) |
| C15 | C14 | C16 | 1·499 (7) | 112·6 (4) |
| C15 | C14 | C8 | | 114·4 (3) |
| C16 | C14 | C8 | 1·533 (6) | 112·5 (4) |
| O18 | C17 | N19 | 1·222 (3) | 122·9 (2) |
| O18 | C17 | C2 | | 121·5 (2) |
| N19 | C17 | C2 | 1·344 (4) | 115·6 (2) |
| C20 | N19 | C24 | 1·457 (4) | 113·7 (3) |
| C20 | N19 | C17 | | 126·3 (2) |
| C24 | N19 | C17 | 1·469 (4) | 119·8 (3) |
| C21 | C20 | N19 | 1·500 (5) | 110·1 (3) |
| O22 | C21 | C20 | 1·424 (4) | 111·1 (3) |
| C23 | O22 | C21 | 1·419 (5) | 109·9 (3) |
| C24 | C23 | O22 | 1·508 (6) | 111·9 (3) |
| N19 | C24 | C23 | | 108·8 (3) |
| C26 | C25 | C30 | 1·396 (4) | 118·1 (2) |
| C26 | C25 | C3 | | 123·7 (2) |
| C30 | C25 | C3 | 1·389 (4) | 118·2 (2) |
| C27 | C26 | C25 | 1·397 (4) | 120·0 (3) |
| C28 | C27 | C26 | 1·356 (5) | 120·6 (3) |
| C29 | C28 | C27 | 1·367 (5) | 120·3 (3) |
| C30 | C29 | C28 | 1·391 (4) | 120·1 (3) |
| C25 | C30 | C29 | | 120·7 (3) |

and/or angles for the H atoms of C14 and C15 did not refine to chemically reasonable values and, therefore, were idealized. The function $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = 1/[\sigma(F_o)]^2$ and $\sigma(F_o) = 0{\cdot}5kI^{-1/2}\{[\sigma(I)]^2 + (0{\cdot}02I)^2\}^{1/2}$. The intensity, $I$, is given by $(I_{\rm peak} - I_{\rm background}) \times$ (scan rate), where 0·02 is a factor to downweight intense reflections and to account for instrument instability and $k$ is the correction due to Lp effects and decay. $\sigma(I)$ was estimated from counting statistics; $\sigma(I) = [(I_{\rm peak} + I_{\rm background})^{1/2} \times$ (scan rate)]. The final $R = 0{\cdot}0475$ for 2460 reflections, with $wR = 0{\cdot}0520$ ($R_{\rm all} = 0{\cdot}0601$, $wR_{\rm all} = 0{\cdot}0540$) and goodness of fit, $S = 1{\cdot}764$. The maximum $|\Delta/\sigma| < 0{\cdot}1$ in the final refinement cycle and the min. and max. peaks in the final $\Delta F$ map were $-0{\cdot}22$ and $0{\cdot}20$ e Å$^{-3}$, respectively. The scattering factors for the non-H atoms were taken from Cromer & Mann (1968), with the anomalous-dispersion corrections taken from the work of Cromer & Liberman (1970). The scattering factors for the H atoms were obtained from Stewart, Davidson & Simpson (1965). Values used to calculate the linear absorption coefficient are from *International Tables for X-ray Crystallography* (1974, Vol. IV, p. 55).* Figures were generated using *SHELXTL-Plus* (Sheldrick, 1988). The positional and thermal parameters for non-H atoms are listed in Table 1, while the bond lengths and angles for the non-H atoms are listed in Table 2. The atom-labeling scheme is shown in Fig. 1. Other computer

---

* Lists of structure factors, anisotropic thermal parameters, bond distances and angles involving H atoms, torsion angles, least-squares planes and H-atom parameters and a unit-cell packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53775 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

programs used in this work are listed in reference 11 of Gadol & Davis (1982).

**Related literature.** Appropriately substituted 1,2,3-cyclopropanes have potential uses as replacements for the dipeptide unit in the design of new enzyme inhibitors and peptide hormone antagonists. A general method for the stereoselective synthesis of 1,2,3-trisubstituted cyclopropanes has been reported (Martin, Austin & Oalmann, 1990).

Funding for this project was supplied by the Robert A. Welch Foundation (F-652) and the National Institutes of Health (GM 25439) to SFM.

### References

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

Doyle, M. P., Pieters, R. J., Martin, S. F., Austin, R. E., Oalmann, C. J. & Muller, P. (1991). *J. Am. Chem. Soc.* **113**, 1423–1424.

Gadol, S. M. & Davis, R. E. (1982). *Organometallics*, **1**, 1607–1613.

Martin, S. F., Austin, R. E. & Oalmann, C. J. (1990). *Tetrahedron Lett.* **31**, 4731–4734.

Sheldrick, G. M. (1988). *SHELXTL-Plus*88. *Structure Determination Software Programs.* Nicolet Instrument Corporation, Madison, Wisconsin, USA.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

*Acta Cryst.* (1991). C**47**, 1347–1348

# Structure of 1-(2-Methyl-2,3-dihydrobenzothiazol-2-yl)-2-propanone

By Guan-Yeow Yeap

*School of Chemical Sciences, Universiti Sains Malaysia, Minden,* 11800 *Penang, Malaysia*

Hoong-Kun Fun

*School of Physics, Universiti Sains Malaysia, Minden,* 11800 *Penang, Malaysia*

and Soon-Beng Teo and Siang-Guan Teoh*

*School of Chemical Sciences, Universiti Sains Malaysia, Minden,* 11800 *Penang, Malaysia*

(*Received* 30 *October* 1990; *accepted* 10 *December* 1990)

**Abstract.** $C_{11}H_{13}NOS$, $M_r = 207{\cdot}29$, monoclinic, $P2_1/c$, $a \doteq 8{\cdot}433$ (2), $b = 12{\cdot}932$ (1), $c = 10{\cdot}503$ (3) Å, $\beta = 111{\cdot}77$ (1)°, $V = 1063{\cdot}7$ (7) Å$^3$, $Z = 4$, $D_m = 1{\cdot}307$ (flotation in nitromethane/chloroform), $D_x \doteq 1{\cdot}294$ Mg m$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}26$ mm$^{-1}$, $F(000) = 440$, $T = 298$ K, $R = 0{\cdot}0574$ for 1752 observed reflections [$I > 2{\cdot}5\sigma(I)$]. The phenyl ring is coplanar with the S and N atoms of the fused five-membered ring with torsion angles S—C(6)—C(1)—C(2) of −178·0 (2) and N—C(1)—C(6)—C(5) of 176·1 (3)°, but C(7) is displaced from the mean plane of the phenyl ring by 0·322 (2) Å. The S—C(6) distance [1·748 (2) Å] is less than that of S—C(7) [1·846 (2) Å] owing to the fact that C(7) is $sp^3$ hybridized whereas C(6) is part of the aromatic ring. Similar arguments pertain to the difference in the N—C distances; N—C(1) = 1·388 (3) and N—C(7) = 1·463 (3) Å.

**Experimental.** The title compound was prepared by a previously reported method (Cefalu, Bosco, Bonati, Maggio & Barbieri, 1970). Preliminary data obtained from Weissenberg and Buerger precession photographs yielded approximate cell dimensions and showed monoclinic symmetry, space group $P2_1/c$. Data collection on a crystal 0·3 × 0·2 × 0·2 mm was performed on a Syntex $P2_1$ diffractometer in $2\theta/\theta$ mode ($0 < h < 10$, $0 < k < 14$, $-12 < l < 12$) out to $2\theta$(max.) of 52·2°. Variable scan speed of 5·0–29·3° min$^{-1}$ was used. Lattice parameters refined using 30 reflections in the range $35 < 2\theta < 45°$. Standard reflection $0\bar{1}1$ checked every 50 reflections: no significant deviation. Data were corrected for Lorentz and polarization effects. 2317 reflections were collected, 2070 unique ($R_{\rm int} = 0{\cdot}0807$), of which 1752 observed reflections with $I > 2{\cdot}5\sigma(I)$ were used for refinement of the structure. Structure solved by direct methods, *SHELXS*86 (Sheldrick, 1986) and refined using *SHELX*76 (Sheldrick, 1976). Scattering factors for C, H, N, O and S inlaid in *SHELX*76.

---

* Author to whom all correspondence should be addressed.

0108-2701/91/061347-02$03.00