the sugar chain and another with the N(2) of the imino group.

We should like to express our special gratitude to Dr Sanchez-Marcos for all his help. The present work is part of a wider research project supported by the Government through the 'Comision Asesora de Investigacion Cientifica y Tecnica'.

References

Alaiz-Barragan, M. (1987). Doctoral thesis, Univ. of Seville, Spain.

Dewar, M. J. S. & Phiel, W. (1977). *J. Am. Chem. Soc.* **99**, 4899–4907; 4907–4917.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 202–207. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Klyne, W. & Prelog, V. (1960). *Experientia*, **16**, 521–523.

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.

Stewart, J. M., Kundell, F. A. & Baldwin, J. C. (1970). *The XRAY system.* Computer Science Center, Univ. of Maryland, College Park, Maryland, USA.

Acta Cryst. (1988). C**44**, 159–161

Structure of (2*S*,4*S*)-*trans*-4-Methylthio-2-phthalimido-γ-butyrolactone

By Ivanka Matijašić

Laboratory of Organic Chemistry, Faculty of Science, University of Zagreb, Strossmayerov trg 14,
41000 *Zagreb, Yugoslavia*

and Gabriele Bocelli, Franco Ugozzoli and Paolo Sgarabotto

Centro di Studio per la Strutturistica Diffrattometrica del CNR, Istituto di Strutturistica Chimica, Universita di Parma, Viale delle Science, 43100 *Parma, Italy*

(*Received 2 January* 1987; *accepted* 16 *September* 1987)

Abstract. $C_{13}H_{11}NO_4S$, $M_r = 277{\cdot}29$, monoclinic, $P2_1$, $a = 11{\cdot}211\,(3)$, $b = 6{\cdot}822\,(2)$, $c = 8{\cdot}710\,(2)$ Å, $\beta = 99{\cdot}90\,(1)°$, $V = 656{\cdot}2\,(3)$ Å^3, $Z = 2$, $D_x = 1{\cdot}40$ g cm^{-3}, $\lambda(\text{Cu}\,K\alpha) = 1{\cdot}5418$ Å, $\mu = 22{\cdot}451$ cm^{-1}, $F(000) = 288$, room temperature, $R = 0{\cdot}038$ for 1301 observed unique reflections. The lactone ring has an envelope conformation with C3 deviating by 0·498 (3) Å from the least-squares plane formed by the remaining four ring atoms. The absolute configuration of the two chiral carbon atoms was deduced as (2*S*,4*S*). The phthalimido and methylthio groups are in pseudo-equatorial and pseudo-axial position respectively, *trans* to each other.

Introduction. In order to establish the stereochemistry of the different 2,4-disubstituted γ-butyrolactones we report here the crystal and molecular structure of the title compound. Some of the other stereoisomers from this series have already been published (Bruvo, Sikirica & Vicković, 1981; Sikirica, Vicković & Viterbo, 1979).

Experimental. Colorless prismatic crystal, approximately 0·29 × 0·52 × 0·19 mm, Siemens AED single-crystal diffractometer, Ni-filtered Cu $K\alpha$ radiation, cell parameters from least-squares fit of 30 reflections ($\theta > 15°$) accurately measured on the diffractometer, room temperature, one check reflection measured every 50 counts without significant variation, profile analysis with Lehmann & Larsen (1974) procedure modified by Belletti, Ugozzoli, Cantoni & Pasquinelli (1979); $3 < \theta < 70°$, Lp correction, absorption ignored, $-13 < h < 13$, $0 < k < 8$, $0 < l < 10$, 2304 reflections collected, 1306 unique observed at $2\sigma(I)$ level ($R_{\text{int}} = 0{\cdot}012$),

Table 1. *Atomic fractional coordinates* (×10^4) *and equivalent isotropic thermal parameters* (Å^2 × 10^4)

	x	y	z	U_{eq}*
S	6340 (1)	8204	6918 (1)	588 (2)
O1	5862 (2)	10343 (3)	9313 (2)	536 (6)
O2	6969 (2)	12761 (4)	10572 (3)	688 (7)
O3	7170 (2)	9660 (4)	13413 (2)	592 (6)
O4	10478 (2)	9650 (5)	11003 (2)	731 (8)
N	8640 (2)	9631 (4)	11855 (2)	462 (6)
C1	6895 (2)	11088 (4)	10132 (3)	477 (7)
C2	7856 (2)	9506 (4)	10349 (3)	461 (7)
C3	7127 (3)	7624 (5)	10019 (3)	580 (10)
C4	6039 (2)	8318 (4)	8876 (3)	496 (7)
C5	4921 (4)	9013 (8)	5840 (5)	822 (13)
C6	8229 (2)	9661 (4)	13273 (3)	437 (7)
C7	9335 (2)	9705 (4)	14491 (3)	418 (6)
C8	9456 (3)	9706 (5)	16098 (3)	506 (8)
C9	10628 (3)	9669 (5)	16949 (3)	537 (7)
C10	11620 (2)	9666 (5)	16220 (3)	531 (8)
C11	11506 (2)	9665 (5)	14592 (3)	509 (7)
C12	10333 (2)	9679 (4)	13760 (3)	433 (6)
C13	9909 (2)	9661 (5)	12042 (3)	487 (7)

* Hamilton (1959).

0108-2701/88/010159-03$03.00

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

S—C4	1·796 (3)	C2—C3	1·523 (4)
S—C5	1·790 (4)	C3—C4	1·512 (4)
O1—C1	1·350 (3)	C6—C7	1·487 (3)
O1—C4	1·455 (3)	C7—C8	1·383 (4)
O2—C1	1·202 (4)	C7—C12	1·378 (4)
O3—C6	1·214 (3)	C8—C9	1·392 (4)
O4—C13	1·193 (3)	C9—C10	1·372 (4)
N—C2	1·450 (3)	C10—C11	1·402 (4)
N—C6	1·391 (3)	C11—C12	1·388 (3)
N—C13	1·404 (3)	C12—C13	1·490 (4)
C1—C2	1·513 (4)		
C4—S—C5	100·9 (1)	N—C6—C7	105·7 (2)
C1—O1—C4	110·6 (2)	O3—C6—C7	129·6 (2)
C6—N—C13	112·4 (1)	C6—C7—C12	108·2 (2)
C2—N—C13	123·4 (1)	C6—C7—C8	130·3 (2)
C2—N—C6	124·2 (2)	C8—C7—C12	121·4 (2)
O1—C1—O2	122·1 (2)	C7—C8—C9	117·3 (2)
O2—C1—C2	128·9 (2)	C8—C9—C10	121·2 (2)
O1—C1—C2	109·0 (2)	C9—C10—C11	121·9 (2)
N—C2—C1	112·3 (2)	C10—C11—C12	116·2 (2)
C1—C2—C3	103·3 (2)	C7—C12—C11	122·0 (2)
N—C2—C3	116·2 (2)	C11—C12—C13	129·3 (2)
C2—C3—C4	101·9 (2)	C7—C12—C13	108·7 (2)
O1—C4—C3	104·8 (2)	N—C13—C12	104·9 (2)
S—C4—C3	110·5 (2)	O4—C13—C12	130·0 (2)
S—C4—O1	110·2 (1)	O4—C13—N	125·1 (2)
O3—C6—N	124·6 (2)		
C5—S—C4—O1	−66·3 (2)	O1—C1—C2—N	−143·9 (2)
C5—S—C4—C3	178·3 (2)	N—C2—C3—C4	152·8 (2)
C1—O1—C4—S	−97·3 (2)	C2—C3—C4—O1	−31·2 (3)
C1—O1—C4—C3	21·6 (3)	C2—C3—C4—S	87·6 (2)
C13—N—C2—C1	−128·5 (3)	O2—C1—C2—N	36·4 (4)
C6—N—C2—C1	54·0 (3)	O2—C1—C2—C3	162·3 (3)
C6—N—C2—C3	−64·6 (3)	O1—C1—C2—C3	−18·0 (3)
C13—N—C2—C3	112·9 (3)	C1—C2—C3—C4	29·4 (3)

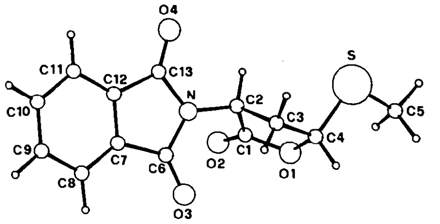


Fig. 1. A perspective view of the molecule with the atomic numbering.

direct methods with *SHELX*76 (Sheldrick, 1976), full-matrix least-squares refinement, heavy atoms anisotropic, H isotropic, located in a ΔF map, five reflections omitted in last cycles, 215 parameters refined. Absolute configuration was obtained through anomalous atomic dispersion (Cromer & Waber, 1974). The discrepancy between the R factors for the two possible absolute configurations is significant (0·0377 as compared with 0·0416 for the other enantiomer). The (2*S*,4*S*) configuration was confirmed; this assignment was checked by use of Hamilton's method in which the significance of the discrepancy between the R factors for the two absolute configurations is estimated (Hamilton, 1965). Final $R = 0{\cdot}0377$, $wR = 0{\cdot}0379$ with $w = (\sigma_F^2 + 0{\cdot}0646F^2)^{-1}$, $\sum w(\Delta F)^2$ minimized, $(\Delta/\sigma)_{\max} = 0{\cdot}18$ for all atoms, $(\Delta\rho)_{\max} = 0{\cdot}24$ e Å^{-3}, scattering factors of *SHELX*76, all calculations on a Gould SEL 32/77 computer. The final atomic parameters are given in Table 1.[*]

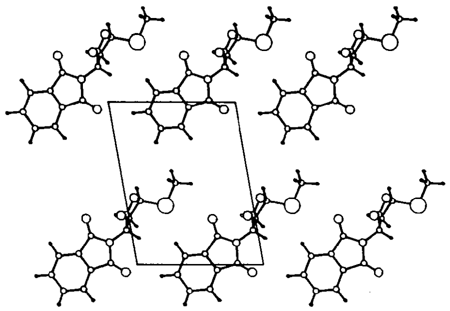


Fig. 2. A projection of the structure viewed along **b**.

Discussion. The molecular structure with the atom numbering is shown in Fig. 1. Bond distances, bond angles and selected torsion angles are listed in Table 2.

The conformation of the γ-butyrolactone ring is an envelope, where, because of the carbonyl group and the partial sp^2 character of the hetero-oxygen atom, atoms C2, C1, O1 and C4 are nearly planar, while C3 deviates considerably from this plane (0·0498 Å). γ-Lactone derivatives generally have an envelope conformation (Harlow & Simonsen, 1976; Bocelli & Grenier-Loustalot, 1981, 1982; Bruvo, Sikirica & Vicković, 1981).

In Cremer & Pople (1975) notation the puckering parameters calculated for the lactone ring are $Q = 0{\cdot}315\ (3)$ Å and $\varphi = -69{\cdot}5\ (5)°$. The values of O—C($sp^3$) and O—C($sp^2$) bond distances [1·455 (3) and 1·351 (3) Å respectively] are in accordance with those reported for the other γ-butyrolactones.

The substituents on the lactone ring can be classified as pseudo-axial and pseudo-equatorial by virtue of the resemblance between the envelope form of a five-membered ring system and the chair conformation of the cyclohexane (Eliel, 1962). In our case the bulkier phthalimido group occupies the pseudo-equatorial and the methylthio group the pseudo-axial position *trans* to each other.

The least-squares plane through the lactone ring makes a dihedral angle of 79·7 (1)° with the plane of the phthalimido group and 161·0 (2)° with the best plane through the C4, S, C5 atoms.

The six- and five-membered rings of the phthalimido group are planar within 0·007 (3) and 0·009 (3) Å. The

---

[*] Lists of structure factors, thermal parameters, H-atom coordinates and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44377 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

dihedral angle between these two rings is 1·5 (1)°. The dimensions of the phthalimido group are consistent with those in other phthalimido derivatives (Iwasaki & Mazuko, 1986; Bats & Teuber, 1985; Voliotis, Arrieta & Germain, 1984).

A view of the molecular packing is given in Fig. 2.

The authors are indebted to Professor A. Deljac for supplying the crystals and for helpful discussion.

References

Bats, W. J. & Teuber, H. J. (1985). *Acta Cryst.* **C41**, 298–300.

Belleti, D., Ugozzoli, F., Cantoni, A. & Pasquinelli, G. (1979). *Gestione on line di Diffrattometro a Cristallo Singolo Siemens AED con Sistema General Automation Jumbo* 220. Internal Report 1.3/79. Centro di Studio per la Strutturistica Diffrattometrica del CNR, Parma, Italy.

Bocelli, G. & Grenier-Loustalot, M. F. (1981). *Acta Cryst.* **B37**, 2106–2108.

Bocelli, G. & Grenier-Loustalot, M. F. (1982). *J. Mol. Struct.* **81**, 113–119; 295–299.

Bruvo, M., Sikirica, M. & Vicković, I. (1981). *Acta Cryst.* **B37**, 700–701.

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Tables 2.2A and 2.3.1. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Eliel, E. L. (1962). *Stereochemistry of Carbon Compounds*. New York: McGraw-Hill.

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

Hamilton, W. C. (1965). *Acta Cryst.* **18**, 502–510.

Harlow, R. L. & Simonsen, S. H. (1976). *Acta Cryst.* **B32**, 2137–2144.

Iwasaki, F. & Mazuko, Y. (1986). *Acta Cryst.* **C42**, 124–127.

Lehmann, M. S. & Larsen, F. K. (1974). *Acta Cryst.* **A30**, 580–589.

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

Sikirica, M., Vicković, I. & Viterbo, D. (1979). Proc. III Italian–Yugoslav Crystallogr. Congr. Parma, Italy, June 1979, 124–127.

Voliotis, S., Arrieta, J. M. & Germain, G. (1984). *Acta Cryst.* **C40**, 1946–1948.

Acta Cryst. (1988). C**44**, 161–163

Structure of *O*-Methylbaccharocephol

By A. P. Rivera and M. Castillo

Departamento de Química, Facultad de Ciencias, Universidad de Chile, Casilla 653, *Santiago, Chile*

and M. L. Rodríguez

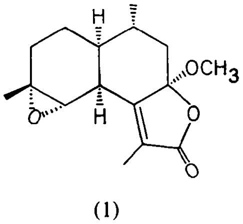
Instituto Universitario de Química Orgánica, Universidad de La Laguna, Carretera La Esperanza 2, *La Laguna, Tenerife, Spain*

(*Received* 9 *July* 1987; *accepted* 16 *September* 1987)

Abstract. $C_{16}H_{22}O_4$, $M_r = 278{\cdot}3$, orthorhombic, $P2_12_12_1$, $a = 8{\cdot}078\,(7)$, $b = 10{\cdot}476\,(9)$, $c = 17{\cdot}639\,(7)$ Å, $V = 1493$ Å^3, $Z = 4$, $D_x = 1{\cdot}24$ g cm^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}5418$ Å, $\mu = 6{\cdot}8$ cm^{-1}, $F(000) = 600$, $T = 293$ K, final $R = 0{\cdot}057$ for 883 observed reflections. The title compound, isolated from *Baccharis sphaerocephala*, corresponds to a new amorphane sesquiterpene γ-lactol derivative, a very rare type of carbon skeleton, with two *cis*-fused six-membered rings, a five-membered-ring γ-lactone and an epoxide.

Introduction. *O*-Methylbaccharocephol was isolated from *B. sphaerocephala* from southern Chile. Its spectroscopic data pointed to a sesquiterpene lactone without a hydrogen at the lactone-ring closure. A tertiary methoxyl group, an epoxide, and secondary, tertiary and vinylic methyl groups were also shown to be present (Rivera, González, Castillo & Rodríguez, 1987).

The structure (1) (without stereochemical assignments) was tentatively arrived at among other less likely alternatives. This X-ray crystallographic study was undertaken to verify the structure and relative configuration of *O*-methylbaccharocephol.



(1)

Experimental. Colourless crystal, 0·6 × 0·1 × 0·1 mm. Diffraction maxima with $2\theta < 100°$ were collected on a computer-controlled four-circle Siemens AED-4 diffractometer, using graphite-monochromated Cu $K\alpha$

0108-2701/88/010161-03$03.00