

## Références

- DUPONT, L., DIDEBERG, O., LIÉGEOIS, J. F. & DELARGE, J. (1991). *Acta Cryst.* **C47**, 1740–1742.  
 NORTH A. C. T., PHILLIPS, D. C. & MATHEWS, F. S. (1968). *Acta Cryst.* **A24**, 351–359.

- SHELDRICK, G. M. (1976). *SHELX76*. Programme pour la détermination des structures cristallines. Univ. de Cambridge, Angleterre.  
 SHELDRICK, G. M. (1986). *SHELXS86*. Programme pour la résolution des structures cristallines. Univ. de Göttingen, Allemagne.

*Acta Cryst.* (1991). **C47**, 2693–2695

## Structure of a Lactone, 2,4,6-Trideoxy-4,6-dimethyl-2-(2-oxopropyl)-7-O-(phenylmethyl)-L-glycero-D-manno-heptono-1,5-lactone 3-(3,5-Dinitrobenzoate)

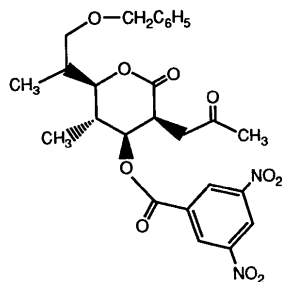
BY HERMAN L. AMMON, PHILIP DESHONG AND DAVID SIMPSON

*Department of Chemistry and Biochemistry, University of Maryland, College Park, MD 20742, USA*

(Received 19 April 1991; accepted 20 June 1991)

**Abstract.**  $C_{26}H_{28}N_2O_{10}$ ,  $M_r = 528.5$ , triclinic,  $P\bar{1}$ ,  $a = 9.524$  (1),  $b = 11.8187$  (8),  $c = 12.615$  (1) Å,  $\alpha = 66.512$  (7),  $\beta = 83.321$  (9),  $\gamma = 88.758$  (8)°,  $V = 1293.0$  (4) Å<sup>3</sup>,  $D_x = 1.357$  g cm<sup>-3</sup>,  $Z = 2$ ,  $\lambda(\text{Cu } K\alpha) = 1.54178$  Å,  $\mu = 8.99$  cm<sup>-1</sup>,  $F(000) = 556$ ,  $T = 293$  K, final  $R = 0.039$ ,  $wR = 0.048$  for 2425 reflections with  $I > 3\sigma(I)$ . The tetra-substituted valerolactone ring has a boat conformation and the relative stereochemistries of the three pairs of adjacent substituents are *cis-trans-trans*.

**Experimental.** Colorless crystals from ethyl acetate-hexane,  $0.1 \times 0.13 \times 0.16$  mm parallelepiped-shaped specimen; Enraf-Nonius CAD-4 diffractometer controlled with Digital Equipment Corporation MicroVAX II computer and modified Enraf-Nonius programs (*NRCCAD*, LePage, White & Gabe, 1986);



cell parameters and crystal orientation from 25 automatically centered reflections in the range  $12.2 < \theta < 20.3$ °;  $2\theta$ - $\theta$  step-scan over  $\Delta\theta$  range of  $1.5(0.7 + 0.14\tan\theta)$ °,  $ca$   $0.01$ ° per step, reflection profiles processed on-line;  $\theta$  scan speed of  $4.12$ ° min<sup>-1</sup>; seven standard reflections monitored at 1 h intervals of X-ray exposure,  $-1.6$  to  $0.2\%$  intensity variation,  $-0.5\%$  average, correction applied;  $3.8 < \theta < 55$ °,

$hkl$  range for data collection of  $h - 9 \rightarrow 0$ ,  $k - 12 \rightarrow 12$ ,  $l - 13 \rightarrow 13$ ; 3639 total data measured including standards, 3249 unique data, 2425 data with  $I > 3\sigma(I)$ ;  $R_{\text{int}} = 0.01$  for 243 twice-measured data; no absorption correction. All crystallographic calculations performed with the *TEXSAN* (1989) program system on DEC MicroVAX II or VAXStation II computers; structure solved with the *MITHRIL* (Gilmore, 1983) direct-methods program incorporated in *TEXSAN*. Full-matrix least-squares refinement,  $\sum[w(F_o - F_c)^2]$  minimized with  $w = 1/\sigma^2(F_o)$ , reflections with  $I < 3\sigma(I)$  excluded from refinement; correction for secondary isotropic extinction (Zachariasen, 1968) applied,  $g = 0.23$  (4)  $\times 10^{-5}$ ; C, O and N refined with anisotropic temperature factors; H atoms positioned from the C-atom framework, coordinates and individual isotropic temperature factors refined; 456 total variables; atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 155–175); min. and max.  $\Delta\rho$  of  $-0.20$  and  $0.14$  e Å<sup>-3</sup>; max.  $\Delta/\sigma$  in final least-squares cycle of  $0.15$ ;  $R$ ,  $wR$  and  $S$  of  $0.039$ ,  $0.048$  and  $1.9$ . Atomic coordinates are listed in Table 1,\* bond lengths, angles and torsion angles are given in Table 2. The *PLOTMD* program (Luo, Ammon & Gilliland, 1989) was used to display the *ORTEP* (Johnson, 1965) drawing on a VAXStation II monitor, label the drawing, and prepare a print file for a Hewlett-Packard Laser-Jet II printer (Fig. 1).

\* Lists of structure factors, anisotropic temperature factors and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54298 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional coordinates and equivalent isotropic temperature factors (Å<sup>2</sup>) with e.s.d.'s in parentheses

$$B_{eq} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	B <sub>eq</sub>
O1	0.6900 (2)	0.6886 (2)	0.5824 (2)	4.0 (1)
O2	0.5936 (2)	0.5177 (2)	0.5889 (2)	4.4 (1)
O3	0.8606 (2)	0.3674 (2)	0.5529 (2)	5.7 (2)
O4	0.6352 (2)	0.7545 (2)	0.3145 (1)	3.5 (1)
O5	0.6921 (2)	0.9432 (2)	0.1756 (2)	4.4 (1)
O6	0.2470 (3)	0.5538 (2)	0.2020 (3)	8.0 (2)
O7	0.0535 (3)	0.6548 (2)	0.1815 (2)	7.6 (2)
O8	0.0931 (3)	1.0991 (2)	0.0183 (2)	6.9 (2)
O9	0.2957 (3)	1.1872 (2)	-0.0005 (2)	5.7 (2)
O10	0.9571 (2)	0.7571 (2)	0.7299 (2)	5.5 (2)
N1	0.1813 (3)	0.6469 (3)	0.1859 (2)	5.5 (2)
N2	0.2176 (3)	1.0961 (2)	0.0324 (2)	4.6 (2)
C1	0.6823 (3)	0.5986 (3)	0.5427 (2)	3.5 (2)
C2	0.7855 (3)	0.6129 (2)	0.4378 (2)	3.3 (2)
C3	0.7790 (3)	0.7448 (2)	0.3496 (3)	3.1 (2)
C4	0.8076 (3)	0.8395 (2)	0.4001 (2)	3.5 (2)
C5	0.8076 (3)	0.7773 (3)	0.5311 (2)	3.6 (2)
C6	0.7583 (4)	0.5159 (3)	0.3924 (3)	3.8 (2)
C7	0.7853 (3)	0.3867 (3)	0.4762 (3)	4.2 (2)
C8	0.7164 (7)	0.2834 (4)	0.4596 (5)	6.6 (4)
C9	0.6096 (3)	0.8568 (3)	0.2252 (2)	3.5 (2)
C10	0.4655 (3)	0.8565 (3)	0.1896 (2)	3.3 (2)
C11	0.3908 (3)	0.7496 (3)	0.2092 (3)	3.9 (2)
C12	0.2605 (3)	0.7598 (3)	0.1684 (2)	3.8 (2)
C13	0.2006 (3)	0.8710 (3)	0.1119 (2)	4.0 (2)
C14	0.2773 (3)	0.9748 (2)	0.0942 (2)	3.5 (2)
C15	0.4085 (3)	0.9705 (3)	0.1300 (2)	3.4 (2)
C16	0.9463 (4)	0.9116 (4)	0.3414 (3)	5.1 (3)
C17	0.7922 (4)	0.8632 (3)	0.5952 (3)	4.6 (3)
C18	0.6510 (5)	0.9265 (5)	0.5865 (5)	6.6 (4)
C19	0.8154 (4)	0.7947 (4)	0.7208 (3)	5.6 (3)
C20	0.9765 (5)	0.6780 (5)	0.8479 (3)	6.7 (4)
C21	1.1311 (4)	0.6581 (3)	0.8566 (3)	5.0 (3)
C22	1.1861 (5)	0.5448 (4)	0.8737 (3)	5.8 (3)
C23	1.3264 (6)	0.5225 (5)	0.8829 (4)	7.3 (4)
C24	1.4137 (6)	0.6149 (6)	0.8742 (4)	8.6 (5)
C25	1.3614 (6)	0.7278 (5)	0.8586 (5)	9.1 (5)
C26	1.2209 (5)	0.7496 (4)	0.8505 (4)	6.9 (4)

Table 2. Bond lengths (Å), angles (°) and selected torsion angles (°) with e.s.d.'s in parentheses

O1	C1	1.351 (3)	C4	C16	1.529 (4)				
O1	C5	1.454 (3)	C5	C17	1.524 (4)				
O2	C1	1.202 (3)	C6	C7	1.508 (4)				
O3	C7	1.217 (3)	C7	C8	1.494 (5)				
O4	C3	1.476 (3)	C9	C10	1.493 (4)				
O4	C9	1.326 (3)	C10	C11	1.383 (4)				
O5	C9	1.209 (3)	C10	C15	1.389 (4)				
O6	N1	1.211 (3)	C11	C12	1.383 (4)				
O7	N1	1.224 (3)	C12	C13	1.370 (4)				
O8	N2	1.218 (3)	C13	C14	1.368 (4)				
O9	N2	1.222 (3)	C14	C15	1.371 (4)				
O10	C19	1.415 (4)	C17	C18	1.518 (5)				
O10	C20	1.440 (4)	C17	C19	1.506 (5)				
N1	C12	1.472 (4)	C20	C21	1.496 (5)				
N2	C14	1.470 (4)	C21	C22	1.371 (5)				
C1	C2	1.508 (4)	C21	C26	1.366 (5)				
C2	C3	1.517 (4)	C22	C23	1.366 (6)				
C2	C6	1.509 (4)	C23	C24	1.349 (6)				
C3	C4	1.534 (4)	C24	C25	1.361 (7)				
C4	C5	1.517 (4)	C25	C26	1.365 (6)				
C1	O1	C5	117.1 (2)	O4	C9	O5	124.9 (2)		
C3	O4	C9	116.2 (2)	O4	C9	C10	113.2 (2)		
C19	O10	C20	111.2 (3)	O5	C9	C10	121.9 (2)		
O6	N1	O7	124.8 (3)	C9	C10	C11	123.2 (2)		
O6	N1	C12	117.9 (3)	C9	C10	C15	117.1 (3)		
O7	N1	C12	117.3 (3)	C11	C10	C15	119.7 (3)		
O8	N2	O9	124.3 (3)	C10	C11	C12	118.5 (3)		
O8	N2	C14	117.7 (3)	N1	C12	C11	119.2 (3)		
O9	N2	C14	118.0 (3)	N1	C12	C13	117.9 (3)		
O1	C1	O2	119.4 (3)	C11	C12	C13	122.9 (3)		
O1	C1	C2	115.3 (2)	C12	C13	C14	117.0 (3)		
O2	C1	C2	125.3 (3)	N2	C14	C13	118.7 (3)		
C1	C2	C3	107.7 (2)	N2	C14	C15	118.6 (3)		
C1	C2	C6	111.2 (2)	C13	C14	C15	122.7 (3)		
C3	C2	C6	114.6 (2)	C10	C15	C14	119.2 (3)		
O4	C3	C2	104.6 (2)	C5	C17	C18	113.1 (3)		
O4	C3	C4	111.8 (2)	C5	C17	C19	110.7 (3)		
C2	C3	C4	112.4 (2)	C18	C17	C19	110.2 (4)		
C3	C4	C5	110.9 (2)	O10	C19	C17	110.5 (3)		
C3	C4	C16	111.0 (3)	O10	C20	C21	109.0 (3)		
C5	C4	C16	111.2 (2)	C20	C21	C22	120.1 (4)		
O1	C5	C4	110.2 (2)	C20	C21	C26	121.9 (4)		
O1	C5	C17	105.4 (3)	C22	C21	C26	118.0 (4)		
C4	C5	C17	115.5 (2)	C21	C22	C23	121.9 (4)		
C2	C6	C7	113.2 (3)	C22	C23	C24	119.1 (5)		
O3	C7	C6	121.6 (3)	C23	C24	C25	120.1 (5)		
O3	C7	C8	121.6 (3)	C24	C25	C26	120.8 (5)		
C6	C7	C8	116.8 (3)	C21	C26	C25	120.0 (4)		
O1	C1	C2	C3	-47.6 (3)	O10	C20	C21	C22	-110.0 (4)
O1	C1	C2	C6	-173.9 (2)	O10	C20	C21	C26	71.5 (5)
O1	C5	C4	C3	-43.7 (3)	N1	C12	C11	C10	178.5 (3)
O1	C5	C4	C16	-167.7 (3)	N1	C12	C13	C14	-178.6 (3)
O1	C5	C17	C18	-57.7 (4)	N2	C14	C13	C12	179.1 (3)
O1	C5	C17	C19	66.5 (3)	N2	C14	C15	C10	179.3 (3)
O2	C1	O1	C5	174.1 (2)	C1	O1	C5	C4	56.9 (3)
O2	C1	C2	C3	129.6 (3)	C1	O1	C5	C17	-177.8 (2)
O2	C1	C2	C6	3.3 (4)	C1	C2	C3	C4	55.8 (3)
O3	C7	C6	C2	-21.0 (4)	C1	C2	C6	C7	-65.7 (3)
O4	C3	C2	C1	-65.7 (3)	C2	C1	O1	C5	-8.5 (3)
O4	C3	C2	C6	58.5 (3)	C2	C3	O4	C9	-170.8 (2)
O4	C3	C4	C5	106.6 (3)	C2	C3	C4	C5	-10.7 (3)
O4	C3	C4	C16	-129.3 (3)	C2	C3	C4	C16	113.4 (3)
O4	C9	C10	C11	-26.1 (4)	C2	C6	C7	C8	159.6 (4)
O4	C9	C10	C15	156.5 (2)	C3	O4	C9	C10	176.1 (2)
O5	C9	O4	C3	-3.9 (4)	C3	C2	C6	C7	172.0 (3)
O5	C9	C10	C11	153.9 (3)	C3	C4	C5	C17	-163.1 (3)
O5	C9	C10	C15	-23.5 (4)	C4	C3	O4	C9	67.3 (3)
O6	N1	C12	C11	-22.1 (4)	C4	C3	C2	C6	-179.9 (3)
O6	N1	C12	C13	157.9 (3)	C4	C5	C17	C18	64.2 (4)
O7	N1	C12	C11	158.8 (3)	C4	C5	C17	C19	-171.6 (3)
O7	N1	C12	C13	-21.1 (4)	C9	C10	C11	C12	-177.3 (3)
O8	N2	C14	C13	13.1 (4)	C9	C10	C15	C14	179.1 (2)
O8	N2	C14	C15	-168.0 (3)	C16	C4	C5	C17	73.0 (4)
O9	N2	C14	C13	-167.7 (3)	C17	C19	O10	C20	-172.5 (3)
O9	N2	C14	C15	11.2 (4)	C19	O10	C20	C21	-170.6 (3)
O10	C19	C17	C5	66.2 (4)	C20	C21	C22	C23	-179.5 (4)
O10	C19	C17	C18	-168.0 (3)	C20	C21	C26	C25	-179.9 (4)

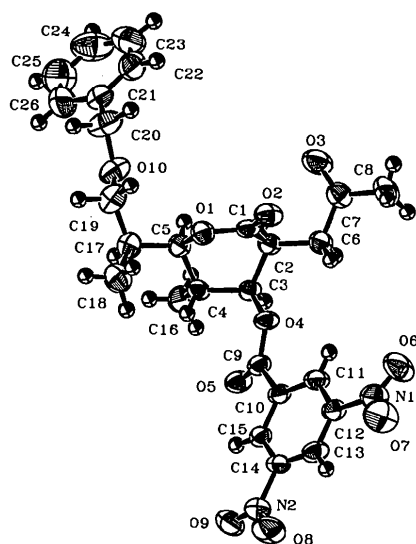


Fig. 1. ORTEP (Johnson, 1965) drawing. The C, N and O atoms are shown as 50% ellipsoids and the H atoms are depicted as spheres with  $B = 1.5 \text{ \AA}^2$ .

**Related literature.** An acid-catalyzed rearrangement of a pyranone derivative gave the title compound with three new stereogenic centers. This determina-

tion was performed to confirm the stereochemistry of the rearrangement product. A search of the January 1991 Cambridge Structural Database (Allen,

Kennard & Taylor, 1983) revealed only one X-ray structure of a tetra-substituted valerolactone in which the lactone was not bridged or fused to other rings (D-glucono-1,5-lactone; Hackert & Jacobson, 1971).

This work was supported at the University of Maryland by National Science Foundation Award No. CHE-85-02155, which provided funds for the purchase of a diffractometer system and by National Institutes of Health for Shared Instrumentation Award No. RR-03354 for the purchase of a graphics workstation/computer system.

*Acta Cryst.* (1991). **C47**, 2695–2696

## Structure of 1-(2,6-Dimethylphenoxy)-2-propanamine Hydrochloride

By JÚLIUS SIVÝ, VIKTOR KETTMANN AND EVA FREŠOVÁ

*Department of Analytical Chemistry, Faculty of Pharmacy, Comenius University, Odbojárov 10, 832 32 Bratislava, Czechoslovakia*

(Received 1 December 1990; accepted 30 May 1991)

**Abstract.**  $C_{11}H_{18}NO^+ \cdot Cl^-$ ,  $M_r = 215.7$ , triclinic,  $P\bar{1}$ ,  $a = 8.796$  (15),  $b = 10.601$  (18),  $c = 14.229$  (24) Å,  $\alpha = 78.74$  (13),  $\beta = 79.89$  (14),  $\gamma = 68.69$  (12)°,  $V = 1204.4$  (4) Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.12$ ,  $D_x = 1.19$  Mg m<sup>-3</sup>,  $\lambda(Mo K\alpha) = 0.71069$  Å,  $\mu = 0.29$  mm<sup>-1</sup>,  $F(000) = 464$ ,  $T = 293$  K, final  $R = 0.062$  for 1261 unique observed reflections. There are two independent molecules in the asymmetric unit with identical geometries (within the limits of experimental error). The propoxy side chain is in a fully extended conformation (*i.e.* the arrangement of the ether O atom and the protonated amino group is *gauche*) and approximately perpendicular to the plane of the phenyl ring. The positive charge of the cations is compensated by three neighbouring chloride anions; two of the three  $N^+ - H$  moieties donate their protons to different  $Cl^-$  anions, with  $N^+ \cdots Cl^-$  distances ranging from 3.001 to 3.175 (7) Å, while the third is involved in bifurcated hydrogen bonding with the ether O atom (within the same molecule) and another  $Cl^-$  ion.

**Introduction.** The title compound (trivial name mexiletine) is an anti-arrhythmic agent belonging to the *Ib* category, with considerable local anaesthetic activity (Remko & Mackov, 1990). The present structure determination is part of our programme to study structure–activity relationships in local anaesthetics.

### References

- ALLEN, F. H., KENNARD, O. & TAYLOR, R. (1983). *Acc. Chem. Res.* **16**, 146–153.  
 GILMORE, C. J. (1983). *MITHRIL*. Univ. of Glasgow, Scotland.  
 HACKERT, M. L. & JACOBSON, R. A. (1971). *Acta Cryst.* **B27**, 203–209.  
 JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.  
 LEPAGE, Y., WHITE, P. S. & GABE, E. J. (1986). *NRCCAD User's Manual*. National Research Council, Ottawa, Canada.  
 LUO, J., AMMON, H. L. & GILLILAND, G. J. (1989). *J. Appl. Cryst.* **22**, 186.  
 TEXSAN (1989). *TEXRAY* single crystal structure analysis software, version 5.0. Molecular Structure Corp., 3200A Research Forest Drive, The Woodlands, TX 77381, USA.  
 ZACHARIASEN, W. H. (1968). *Acta Cryst.* **A24**, 212–216.

**Experimental.** Single crystals from butanol, crystal used 0.4 × 0.2 × 0.1 mm,  $D_m$  by flotation. Weissenberg photographs did not exhibit any symmetry. Lattice parameters refined on diffractometer from the positional angles of 16 reflections,  $4 < 2\theta < 15^\circ$ . Intensity measurements on Syntex *P2*<sub>1</sub> diffractometer and graphite-monochromated  $Mo K\alpha$  radiation. Correction for Lorentz and polarization factors using *XP2*<sub>1</sub> (Pavelčík, 1987), corrections for absorption and extinction neglected. Intensities of reflections in the range  $0 < 2\theta \leq 55^\circ$  using  $\theta$ – $2\theta$  scanning mode, each reflection scanned 1° (in  $2\theta$ ) above and below  $K\alpha$  doublet, background-to-scan-time ratio 1.0. Two standard reflections ( $0\bar{1}\bar{1}$ ,  $\bar{2}11$ ) every 98 reflections, 5354 unique reflections,  $R_{int} = 0.027$ , 1283 with  $I \geq 1.96\sigma(I)$  considered observed and 1261 included in the refinement ( $|\Delta F|/|F_o| < 0.53$ ). Index ranges  $h$  0–11,  $k$  –12–13,  $l$  –17–18.

The structure was solved by the heavy-atom method in the space group  $P\bar{1}$  using *SHELXS86* (Sheldrick, 1986). Refinement on  $|F_o|$  by a block-diagonal least-squares method, anisotropic thermal parameters for non-H atoms, only coordinates and thermal parameters of nine H atoms found in the difference Fourier map were refined; the remaining H atoms fixed at calculated positions, with the thermal parameters set to 0.5 higher than  $B_{eq}$  of the associated C or N atoms. Function minimized