

C34	-0.1977 (3)	0.6622 (3)	0.4451 (2)	0.0575 (14)
C35	-0.2964 (3)	0.5790 (3)	0.4707 (2)	0.0617 (16)
C36	-0.2579 (3)	0.4461 (3)	0.3908 (2)	0.0488 (13)

Table 4. Selected geometric parameters (Å, °) for (V)

S1—N1	1.6980 (18)	C1—C11	1.534 (2)
S1—C1	1.8729 (17)	C1—C21	1.533 (2)
N1—HN1	0.88	C1—C31	1.538 (2)
N1—HN2	0.91		
N1—S1—C1	104.49 (8)	S1—C1—C31	111.91 (11)
S1—N1—HN1	106	C11—C1—C21	112.44 (13)
S1—N1—HN2	107	C11—C1—C31	111.13 (13)
HN1—N1—HN2	112	C21—C1—C31	111.14 (13)
S1—C1—C11	99.82 (11)	N1—S1—HN1 ⁱ	96
S1—C1—C21	109.92 (11)		
C1—S1—N1—HN1	-125	C1—S1—N1—HN2	116

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

Both (IV) and (V) crystallized in the triclinic system; space group $P\bar{1}$ was assumed and confirmed by the successful refinement. All H atoms bonded to C atoms were clearly visible in difference maps; they were positioned geometrically (C—H 0.95 Å) and included as riding atoms in the structure-factor calculations. The coordinates of the H atoms bonded to the N atoms were obtained from difference maps and included but not refined in the calculations. Electron-density sections showing the H atoms bonded to the N atoms have been deposited. Examination of the structures with PLATON (Spek, 1992) showed that there were no solvent-accessible voids in the crystal lattices. Data collection and cell refinement: CAD-4 Software (Enraf-Nonius, 1989). Data reduction, structure solution and refinement, preparation of material for publication: NRCVAX (Gabe, Le Page, Charland, Lee & White, 1989). Figs. 1 and 2 were prepared using ORTEPII (Johnson, 1976).

GF thanks NSERC (Canada) for research grants.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, along with electron-density sections showing the H atoms bonded to the N atoms have been deposited with the IUCr (Reference: AB1184). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.
- Auricchio, S., Brückner, S., Malpezzi Giunchi, L., Kozinsky, V. A. & Zelenskaja, O. V. (1980). *J. Heterocycl. Chem.* **17**, 1217–1219.
- Enraf-Nonius (1989). *CAD-4 Software*. Enraf-Nonius, Delft, The Netherlands.
- Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.
- Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* **B46**, 256–262.
- Ferguson, G., Gallagher, J. F., Glidewell, C., Low, J. N. & Scrimgeour, S. N. (1992). *Acta Cryst.* **C48**, 1272–1275.
- Ferguson, G., Glidewell, C. & Zakaria, C. M. (1994). *Acta Cryst.* **C50**, 928–931.
- Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.
- Glidewell, C. & Ferguson, G. (1994). *Acta Cryst.* **C50**, 924–928.
- Hunter, L. R., Hunter, C. A. & Purvis, D. H. (1992). *J. Chem. Soc. Chem. Commun.* pp. 1134–1136.
- Hunter, C. A. (1991). *J. Chem. Soc. Chem. Commun.* pp. 749–751.

- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Larson, A. C. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall & C. P. Huber, pp. 291–294. Copenhagen: Munksgaard.
- Spek, A. L. (1992). *PLATON. Molecular Geometry Program*. Univ. of Utrecht, The Netherlands.

Acta Cryst. (1994). **C50**, 1366–1372

(2S,3R,4S,5R,6S,7R,8R,9S)-1,2,3,4,5,6,7,8,9-Nonaacetoxydecane

KRZYSZTOF LEWINSKI

Faculty of Chemistry, Jagiellonian University, ul. Ingardena 3, 30-060 Krakow, Poland

JAMES A. MARSHALL AND SERGE BEAUDOIN

Department of Chemistry and Biochemistry, University of South Carolina, Columbia, SC 29208, USA

(Received 15 December 1993; accepted 8 February 1994)

Abstract

The structure of the title compound, C₂₈H₄₀O₁₈, has been determined by X-ray diffraction. The acetoxy groups are planar and are approximately perpendicular to the carbon backbone, which adopts a *gauche* conformation around the C3—C4 bond.

Comment

This work is part of a project concerning the stereoselective synthesis of long-chain polyols from tartrate derivatives (Marshall, Beaudoin & Lewinski, 1993). The chain C1 to C10 in the title compound (I) contains two regions of extended conformation, C1 to C4 and C3 to C10, in a *gauche* orientation to each other. The average C—C bond length in the chain is 1.523 (9) Å and the average C—C—C angle is 113.5 (15)°, in agreement with the values found in other structures, especially with the average values of 1.523 (8) Å and 113.2 (5)° found in hexadecyltrimethylammonium bromide (Cimpanelli & Scaramuzza, 1986). All nine acetoxy groups are planar (average r.m.s. deviation 0.004 Å) and adopt a typical conformation with the C=O bonds pointing in the same direction as the C—H bonds (Abboud, Simonsen, Voll & Younathan, 1990). The torsion angles C—O—C—H vary from 0.1 to 28.1°. The average bond distances in the acetoxy groups are C_{sp³}—O 1.442 (9), O—C_{sp²} 1.355 (6), C_{sp²}—C_{methyl} 1.482 (10)

and $C_{sp^2}=O$ 1.118 (8) Å and do not differ significantly from those reported previously (Jiménez, González, Lira-Rocha, Toscano & Soriano-García, 1989; Conde & Conde, 1989).

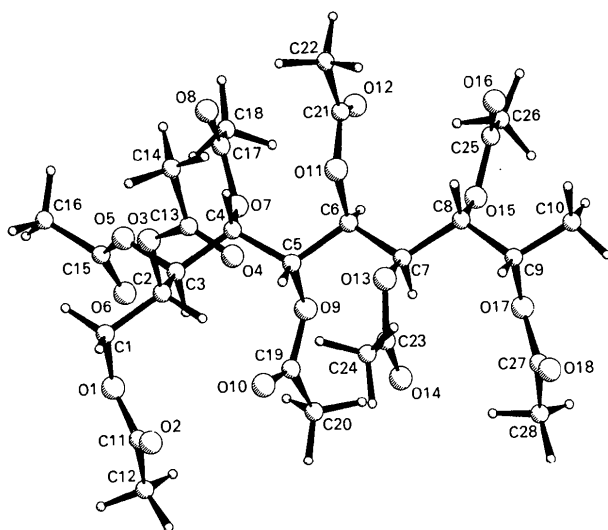
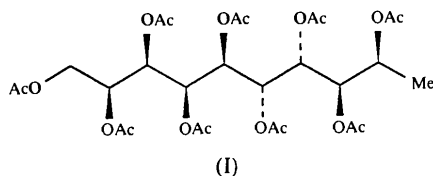


Fig. 1. *PLUTO* (Motherwell & Clegg, 1976) drawing of the title compound showing the atomic numbering scheme.

Experimental

Crystal data

$C_{28}H_{40}O_{18}$

$M_r = 664.6$

Monoclinic

$P2_1$

$a = 9.314$ (3) Å

$b = 18.889$ (4) Å

$c = 10.036$ (1) Å

$\beta = 95.47$ (2)°

$V = 1758$ (1) Å³

$Z = 2$

$D_x = 1.256$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71069$ Å

Cell parameters from 25 reflections

$\theta = 10-13^\circ$

$\mu = 0.0993$ mm⁻¹

$T = 293$ K

Plate

$0.6 \times 0.4 \times 0.2$ mm

Colorless

Crystal source: ethanol solution

Data collection

Enraf-Nonius CAD-4 diffractometer

$\omega/2\theta$ scans

Absorption correction: none

$R_{int} = 0.022$

$\theta_{max} = 24.0^\circ$

$h = 0 \rightarrow 10$

$k = -21 \rightarrow 21$

$l = -11 \rightarrow 11$

5901 measured reflections
5113 independent reflections
4409 observed reflections
[$F > 4\sigma(F)$]

3 standard reflections
frequency: 120 min
intensity variation: 2%

Refinement

Refinement on F

$R = 0.046$

$wR = 0.050$

$S = 0.878$

4409 reflections

512 parameters

Calculated weights

$w = 1/[\sigma^2(F) + 0.002F^2]$

$(\Delta/\sigma)_{max} = 0.034$

$\Delta\rho_{max} = 0.34$ e Å⁻³

$\Delta\rho_{min} = -0.31$ e Å⁻³

Extinction correction: empirical (Sheldrick, 1976)

Extinction coefficient: 0.0009 (5)

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
O1	0.0274 (3)	0.5029†	0.0103 (3)	0.058 (2)
O2	-0.0043 (4)	0.4192 (2)	0.1629 (4)	0.096 (3)
O3	-0.1179 (3)	0.6403 (2)	0.2110 (3)	0.068 (2)
O4	-0.0632 (6)	0.6095 (5)	0.4233 (5)	0.170 (5)
O5	0.0370 (3)	0.6843 (2)	0.0037 (2)	0.049 (1)
O6	0.2098 (4)	0.6543 (3)	-0.1240 (3)	0.099 (3)
O7	0.2694 (3)	0.7430 (1)	0.1504 (2)	0.044 (1)
O8	0.1144 (4)	0.8328 (2)	0.1685 (3)	0.076 (2)
O9	0.3969 (2)	0.6247 (1)	0.2752 (2)	0.040 (1)
O10	0.2849 (3)	0.5199 (2)	0.2545 (3)	0.062 (2)
O11	0.2253 (2)	0.7496 (1)	0.4832 (2)	0.042 (1)
O12	0.3442 (4)	0.8475 (2)	0.4291 (3)	0.070 (2)
O13	0.2924 (2)	0.6157 (1)	0.5996 (2)	0.043 (1)
O14	0.4208 (4)	0.5191 (2)	0.5593 (4)	0.074 (2)
O15	0.5672 (2)	0.7514 (1)	0.6326 (2)	0.042 (1)
O16	0.4811 (4)	0.8427 (2)	0.7423 (3)	0.078 (2)
O17	0.6429 (2)	0.6186 (1)	0.7350 (2)	0.043 (1)
O18	0.6001 (4)	0.5256 (2)	0.8646 (3)	0.073 (2)
C1	-0.0786 (4)	0.5465 (2)	0.0652 (4)	0.059 (2)
C2	-0.0052 (4)	0.5967 (2)	0.1681 (3)	0.047 (2)
C3	0.1080 (3)	0.6439 (2)	0.1125 (3)	0.040 (2)
C4	0.1802 (3)	0.6954 (2)	0.2162 (3)	0.038 (2)
C5	0.2724 (3)	0.6576 (2)	0.3270 (3)	0.035 (2)
C6	0.3380 (3)	0.7050 (2)	0.4395 (3)	0.036 (2)
C7	0.4027 (3)	0.6614 (2)	0.5566 (3)	0.036 (2)
C8	0.4613 (3)	0.7040 (2)	0.6806 (3)	0.037 (2)
C9	0.5299 (4)	0.6579 (2)	0.7926 (3)	0.042 (2)
C10	0.5916 (5)	0.6978 (3)	0.9134 (4)	0.065 (3)
C11	0.0565 (5)	0.4401 (3)	0.0723 (5)	0.067 (3)
C12	0.1723 (6)	0.4023 (3)	0.0054 (6)	0.089 (4)
C13	-0.1360 (6)	0.6416 (4)	0.3433 (7)	0.099 (4)
C14	-0.2454 (8)	0.6947 (5)	0.3710 (10)	0.147 (7)
C15	0.0982 (4)	0.6839 (3)	-0.1125 (4)	0.060 (3)
C16	0.0101 (6)	0.7228 (3)	-0.2192 (5)	0.080 (4)
C17	0.2230 (5)	0.8106 (2)	0.1297 (4)	0.056 (2)
C18	0.3241 (7)	0.8512 (3)	0.0558 (6)	0.088 (4)
C19	0.3903 (4)	0.5544 (2)	0.2479 (4)	0.046 (2)
C20	0.5297 (5)	0.5278 (3)	0.2076 (6)	0.072 (3)
C21	0.2421 (5)	0.8212 (2)	0.4725 (4)	0.049 (2)
C22	0.1162 (7)	0.8585 (3)	0.5179 (6)	0.083 (4)
C23	0.3148 (5)	0.5451 (2)	0.5965 (4)	0.053 (2)
C24	0.1928 (7)	0.5059 (3)	0.6445 (7)	0.094 (4)
C25	0.5637 (4)	0.8201 (2)	0.6697 (4)	0.051 (2)
C26	0.6733 (5)	0.8617 (3)	0.6044 (5)	0.070 (3)
C27	0.6629 (4)	0.5508 (2)	0.7779 (4)	0.049 (2)
C28	0.7691 (5)	0.5133 (3)	0.7024 (5)	0.068 (3)

† Coordinate fixed to define origin.

Table 2. Selected geometric parameters (Å, °)

O1—C1	1.435 (5)	O16—C25	1.187 (6)
O1—C11	1.355 (5)	O17—C9	1.453 (4)
O2—C11	1.184 (7)	O17—C27	1.359 (5)
O3—C2	1.433 (5)	O18—C27	1.192 (5)
O3—C13	1.354 (7)	C1—C2	1.516 (6)
O4—C13	1.169 (9)	C2—C3	1.526 (5)
O5—C3	1.440 (4)	C3—C4	1.533 (5)
O5—C15	1.347 (5)	C4—C5	1.517 (4)
O6—C15	1.195 (6)	C5—C6	1.522 (4)
O7—C4	1.428 (4)	C6—C7	1.513 (4)
O7—C17	1.358 (5)	C7—C8	1.538 (5)
O8—C17	1.194 (6)	C8—C9	1.515 (5)
O9—C5	1.455 (4)	C9—C10	1.495 (6)
O9—C19	1.356 (5)	C11—C12	1.504 (8)
O10—C19	1.186 (5)	C13—C14	1.475 (11)
O11—C6	1.447 (4)	C15—C16	1.481 (7)
O11—C21	1.368 (5)	C17—C18	1.469 (8)
O12—C21	1.192 (6)	C19—C20	1.484 (6)
O13—C7	1.439 (4)	C21—C22	1.476 (8)
O13—C23	1.349 (5)	C23—C24	1.475 (8)
O14—C23	1.195 (6)	C25—C26	1.488 (7)
O15—C8	1.448 (4)	C27—C28	1.483 (6)
O15—C25	1.352 (5)		
C1—O1—C11	116.1 (3)	O17—C9—C8	105.5 (2)
C2—O3—C13	117.9 (3)	C8—C9—C10	114.4 (3)
C3—O5—C15	116.8 (3)	O17—C9—C10	110.1 (3)
C4—O7—C17	118.2 (3)	O1—C11—O2	123.6 (4)
C5—O9—C19	117.9 (2)	O2—C11—C12	126.9 (4)
C6—O11—C21	117.4 (2)	O1—C11—C12	109.3 (4)
C7—O13—C23	117.9 (2)	O3—C13—O4	123.0 (6)
C8—O15—C25	117.8 (2)	O4—C13—C14	126.0 (7)
C9—O17—C27	115.9 (2)	O3—C13—C14	110.4 (5)
O1—C1—C2	109.7 (3)	O5—C15—O6	122.0 (3)
O3—C2—C1	105.6 (3)	O6—C15—C16	126.1 (4)
C1—C2—C3	113.5 (3)	O5—C15—C16	111.8 (3)
O3—C2—C3	108.9 (3)	O7—C17—O8	123.1 (3)
O5—C3—C2	107.6 (2)	O8—C17—C18	125.8 (4)
C2—C3—C4	113.3 (2)	O7—C17—C18	110.9 (4)
O5—C3—C4	108.5 (2)	O9—C19—O10	123.2 (3)
O7—C4—C3	108.8 (2)	O10—C19—C20	125.4 (3)
C3—C4—C5	112.3 (2)	O9—C19—C20	111.2 (3)
O7—C4—C5	108.7 (2)	O11—C21—O12	122.8 (3)
O9—C5—C4	110.7 (2)	O12—C21—C22	126.8 (3)
C4—C5—C6	115.2 (2)	O11—C21—C22	110.3 (3)
O9—C5—C6	103.8 (2)	O13—C23—O14	123.2 (3)
O11—C6—C5	108.4 (2)	O14—C23—C24	125.4 (4)
C5—C6—C7	110.9 (2)	O13—C23—C24	111.3 (4)
O11—C6—C7	109.1 (2)	O15—C25—O16	123.2 (3)
O13—C7—C6	108.4 (2)	O16—C25—C26	126.3 (3)
C6—C7—C8	115.3 (2)	O15—C25—C26	110.4 (3)
O13—C7—C8	106.2 (2)	O17—C27—O18	123.0 (3)
O15—C8—C7	104.7 (2)	O18—C27—C28	125.5 (4)
C7—C8—C9	113.0 (2)	O17—C27—C28	111.4 (3)
O15—C8—C9	110.4 (2)		

The data were corrected for Lorentz and polarization effects but not for absorption. The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1985). Difference Fourier maps showed rotational disorder in all the acetylic methyl groups. For each CH₃ group the positions of uniformly distributed H atoms were calculated and refined with fixed occupancies of 0.5 as a rigid group. The positions of the other H atoms were calculated and included using a riding model in the structure-factor calculations. Individual isotropic displacement factors for the H atoms and anisotropic displacement factors for all other atoms were used in a block-diagonal-matrix refinement. The absolute configuration was assigned to agree with the known chirality of the substrates. Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS86*. Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976). Molecular graphics:

PLUTO (Motherwell & Clegg, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1073). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Abboud, K. A., Simonsen, S. H., Voll, R. J. & Younathan, E. S. (1990). *Acta Cryst.* **C46**, 2208–2210.
- Campanelli, A. R. & Scaramuzza, L. (1986). *Acta Cryst.* **C42**, 1380–1383.
- Conde, C. F. & Conde, A. (1989). *Acta Cryst.* **C45**, 1563–1565.
- Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
- Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.
- Jiménez, M., González, M. A., Lira-Rocha, A., Toscano, R. A. & Soriano-García, M. (1989). *Acta Cryst.* **C45**, 1054–1057.
- Marshall, J. A., Beaudoin, S. & Lewinski, K. (1993). *J. Org. Chem.* **58**, 5876–5877.
- Motherwell, W. D. S. & Clegg, W. (1976). *PLUTO. Program for Plotting Molecular and Crystal Structures*. Univ. of Cambridge, England.
- Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
- Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination*. Univ. of Cambridge, England.
- Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.

Acta Cryst. (1994). **C50**, 1368–1370

Refinement of a Twinned Structure with *SHELXL93*: meso-2,2'-[1,6-Bis(methylamino)-3,4-dithia-1,6-hexanediyl]diphenol

MICHAEL BOLTE

Institut für Organische Chemie der Universität Frankfurt, Marie-Curie-Strasse 11, 60439 Frankfurt/Main, Germany

(Received 8 October 1993; accepted 24 December 1993)

Abstract

Crystals of the title compound appeared to be twinned, but a new option in the program *SHELXL93* [Sheldrick (1993). Univ. of Göttingen, Germany] has made a successful refinement possible. One hydroxyl O atom forms an intramolecular hydrogen bond to an amino N atom and a short contact to an aromatic H atom can be found. The other hydroxyl O atom acts as an acceptor for one intra- and one intermolecular hydrogen bond from the NH groups.