*Carbohydrate Research*, 238 (1993) 317–320
Elsevier Science Publishers B.V., Amsterdam



# Note

## Crystal and molecular structure of octa-*O*-acetyl-L-*threo*-D-*galacto*-octitol

Peter Köll [a], Jürgen Kopf [b], Martina Morf [b], Bärbel Zimmer [b] and John. S. Brimacombe [c]

[a] *Department of Chemistry, The University, Carl-von-Ossietzky-Str. 9-11, D-W 2900 Oldenburg (Germany)*
[b] *Institute of Inorganic and Applied Chemistry, The University, Martin-Luther-King-Platz 6, D-W 2000 Hamburg 13 (Germany)*
[c] *Department of Chemistry, The University of Dundee, Dundee DD1 4HN (United Kingdom)*

(Received January 23rd, 1992; accepted June 2nd, 1992)

There are 16 enantiomeric pairs and 4 *meso* forms of the octitols, but comparatively few of them are known and no crystal structures have been reported. In extending previous work on the structures of minor alditol acetates[1], 1,2,3,4,5,6,7,8-octa-*O*-acetyl-L-*threo*-D-*galacto*-octitol (**1**)[2] was subjected to X-ray crystallographic analysis.

The relevant crystallographic data for **1** are given in Table I. The structure was solved by direct methods in the usual way with the help of the programs SHELXS90[3] and SHELX76[4]. All atoms, with hydrogen atoms included at calculated positions, were refined. The final fractional co-ordinates of the carbon and oxygen atoms, together with equivalent isotropic thermal parameters, are listed in Table II*.

Fig. 1 shows a single molecule of **1** (SCHAKAL88[5] plot) in the expected zigzag conformation, with O-1 and O-8 extending the planar carbon chain; as is the case

*Correspondence to:* Professor Dr. P. Köll, Department of Chemistry, The University, Carl-von-Ossietzky-Str. 9-11, D-W 2900 Oldenburg, Germany; or to: Privatdozent Dr. J. Kopf, Institute of Inorganic and Applied Chemistry, The University, Martin-Luther-King-Platz 6, D-W 2000 Hamburg 13, Germany; or to: Professor J.S. Brimacombe, Department of Chemistry, The University of Dundee, Dundee DD1 4HN, United Kingdom.

* Lists of observed and calculated structure amplitudes, anisotropic thermal parameters, fractional co-ordinates of hydrogen atoms with isotropic thermal parameters, tables of bond distances and angles, and further information (26 pages) have been deposited with, and can be obtained from, Elsevier Science Publishers B.V., BBA Data Deposition, P.O. Box 1527, Amsterdam, The Netherlands. Reference should be made to No. BBA/DD/512/*Carbohydr. Res.*, 238 (1993) 317–320.

TABLE I

Crystallographic data for 1 [a]

| | | | |
|---|---|---|---|
| Formula | $C_{24}H_{34}O_{16}$ | Density (calcd) ($g \times cm^{-3}$) | 1.369 |
| Mol wt | 578.52 | $\lambda$ (Cu $K_\alpha$) (pm) | 154.051 |
| Mp (°C) | 134.5 | $\mu$ ($cm^{-1}$) | 9.6 |
| Crystal size (mm) | $0.3 \times 0.1 \times 0.1$ | $2\theta$ range (degrees) | 4.5–153 |
| Space group | $P2_1$ | Reflections measured | 3330 |
| Cell parameters | | Symmetry independent reflections | 3141 |
| (pm, degrees) | | Observed reflections with $F_0 > 4\sigma(F_0)$ | 3079 |
| $a$ | 1270.1(2) | Number of refined parameters | 360 |
| $b$ | 793.2(1) | Final residual parameters | |
| $c$ | 1442.8(2) | $R$ | 0.046 |
| $\beta$ | 105.12(2) | $R_w$ | 0.052 |
| Volume ($pm^3$) | $1403.2(4) \times 10^6$ | Diffractometer | Enraf–Nonius |
| $Z$ | 2 | | CAD4 |
| $F(000)$ | 612 | | |

[a] Standard deviations in parentheses.

with most of the alditol acetates examined[1], each of these atoms is arranged *gauche* to the neighbouring oxygen atom. Only two of the twenty diastereomeric octitols are free from parallel 1,3-interactions between oxygen atoms (designated O//O interactions) in a planar zigzag conformation, notably the parent compound of **1** (or its enantiomer) and *meso-erythro-manno*-octitol. If such O//O interactions occur, rotation can take place about a neighbouring bond to give a sickle

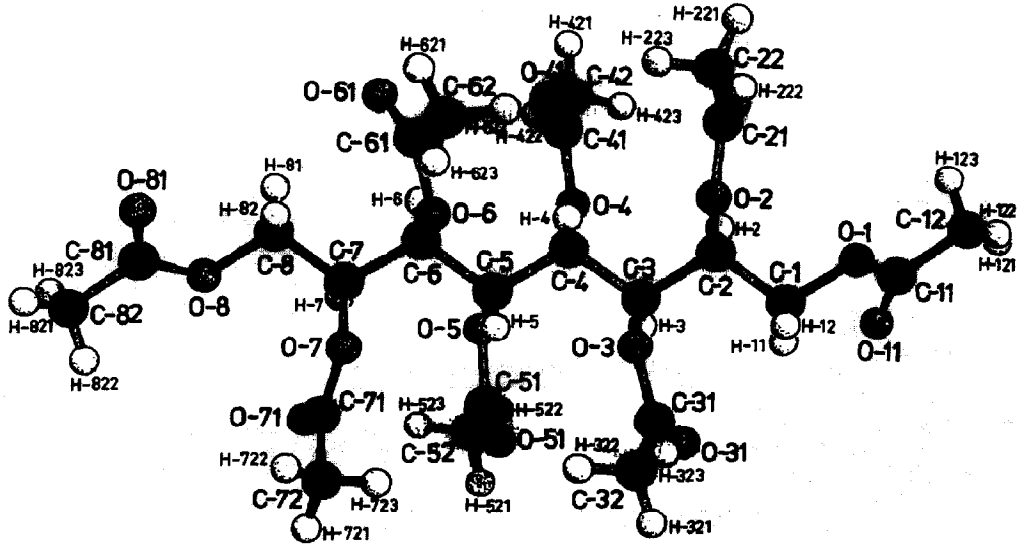


Fig. 1. SCHAKAL88[5] plot of a molecule of octa-*O*-acetyl-L-*threo*-D-*galacto*-octitol (**1**) showing atom numbering.

TABLE II

Fractional positional parameters of C and O atoms ($\times 10^4$) [a] and the temperature factors $U_{eq}$ [b] for 1

| Atom | x | y | z | $U_{eq}$ |
|---|---|---|---|---|
| O-1 | 6511(2) | 3600(5) | 8178(2) | 258(8) |
| O-2 | 4205(2) | 4000(5) | 7305(2) | 229(7) |
| O-3 | 3572(2) | 2180(5) | 8830(2) | 210(7) |
| O-4 | 3017(2) | 396 [c] | 6456(2) | 211(7) |
| O-5 | 2127(2) | −1430(5) | 7709(2) | 211(7) |
| O-6 | 539(2) | 2333(4) | 6923(2) | 170(7) |
| O-7 | −133(2) | −102(5) | 8084(2) | 196(7) |
| O-8 | −2009(2) | −1237(5) | 6743(2) | 254(8) |
| O-11 | 7574(3) | 1315(5) | 8597(3) | 438(11) |
| O-21 | 4635(3) | 3551(6) | 5904(2) | 473(11) |
| O-31 | 4765(2) | 504(5) | 9852(2) | 343(10) |
| O-41 | 2099(2) | 2373(5) | 5435(2) | 328(9) |
| O-51 | 2406(2) | −1132(5) | 9313(2) | 331(9) |
| O-61 | −363(2) | 2559(5) | 5354(2) | 285(8) |
| O-71 | −129(3) | −2875(5) | 8469(2) | 337(9) |
| O-81 | −3285(3) | 795(6) | 6296(3) | 450(11) |
| C-1 | 5673(3) | 2789(6) | 8524(3) | 262(11) |
| C-2 | 4700(3) | 2414(6) | 7692(3) | 206(10) |
| C-3 | 3846(3) | 1383(6) | 8022(2) | 193(9) |
| C-4 | 2768(3) | 1266(6) | 7254(2) | 189(9) |
| C-5 | 1871(3) | 357(6) | 7573(3) | 176(9) |
| C-6 | 760(3) | 535(5) | 6850(3) | 170(9) |
| C-7 | −120(3) | −506(6) | 7114(3) | 194(10) |
| C-8 | −1236(3) | −166(6) | 6454(3) | 244(10) |
| C-11 | 7431(3) | 2732(6) | 8255(3) | 257(11) |
| C-12 | 8236(3) | 3703(7) | 7884(3) | 329(11) |
| C-21 | 4217(3) | 4396(6) | 6389(3) | 306(12) |
| C-22 | 3609(4) | 6016(7) | 6083(3) | 387(14) |
| C-31 | 4055(3) | 1552(7) | 9714(3) | 274(11) |
| C-32 | 3588(4) | 2320(7) | 10460(3) | 413(14) |
| C-41 | 2662(3) | 1123(6) | 5573(3) | 253(11) |
| C-42 | 3026(4) | 148(7) | 4837(3) | 401(16) |
| C-51 | 2367(3) | −2010(6) | 8633(3) | 250(11) |
| C-52 | 2573(4) | −3889(6) | 8660(3) | 356(14) |
| C-61 | 43(3) | 3197(6) | 6111(3) | 196(10) |
| C-62 | 79(3) | 5058(6) | 6336(3) | 260(11) |
| C-71 | −151(3) | −1402(6) | 8698(3) | 258(11) |
| C-72 | −189(4) | −780(7) | 9659(3) | 369(14) |
| C-81 | −3021(3) | −606(7) | 6628(3) | 321(13) |
| C-82 | −3763(4) | −1804(8) | 6934(4) | 506(19) |

[a] Standard deviations in parentheses. [b] $U_{eq} = 1/3\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i \mathbf{a}_j$. [c] This co-ordinate was fixed.

conformation although this appears to be less prevalent in the case of alditol acetates[1] (for example, D-glucitol hexa-acetate[6,7]), whose crystal structures are able to tolerate such interactions.

Compound **1** represents one of the four diastereomeric octitols with potential $C_2$ symmetry. This is not realised in the crystal structure.

ACKNOWLEDGMENTS

The Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie are thanked for financial support (to P.K. and J.K.).

REFERENCES

1 J. Kopf, M. Morf, B. Zimmer, M. Bischoff, and P. Köll, *Carbohydr. Res.*, 229 (1992) 17–32; J. Kopf, M. Morf, B. Zimmer, and P. Köll, *ibid.*, 233 (1992) 35–43; P. Köll, M. Bischoff, M. Morf, B. Zimmer, and J. Kopf, *ibid.*, submitted.
2 J.S. Brimacombe, R. Hanna, A.K.M.S. Kabir, F. Bennett, and I.D. Taylor, *J. Chem. Soc., Perkin Trans. 1*, (1986) 815–821.
3 G.M. Sheldrick, *Acta Crystallogr., Sect. A*, 46 (1990) 467–473.
4 G.M. Sheldrick, SHELX76, Programs for Crystal Structure Determination, Cambridge, 1976.
5 E. Keller, *Chem. Unserer Zeit*, 14 (1980) 56–60.
6 Y.J. Park, M.H. Park, and J.M. Shin, *Daehan Hwahak Hwoejee*, 34 (1990) 517–526; *Chem. Abstr.*, 114 (1991) 82345a.
7 J. Kopf, C. Topf, M. Morf, B. Zimmer, and P. Köll, *Acta Crystallogr., Sect. C*, 47 (1991) 2425–2428.