H(1)	0.021 (8)	1/4	−0.470 (7)	0.066 (9)
H(2)	0.215 (5)	0.375 (4)	−0.286 (4)	0.077 (7)
H(4)	0.135 (8)	1/4	−0.810 (7)	0.054 (8)
H(5)	0.398 (8)	1/4	−0.810 (7)	0.065 (10)
H(6)	0.619 (7)	1/4	−0.304 (8)	0.066 (11)

Table 2. *Selected geometric parameters* (Å, °)

C(1)—C(2)	1.473 (5)	C(2)—O	1.297 (4)
C(1)—H(1)	0.96 (4)	N—H(4)	0.92 (4)
C(1)—H(2)	1.02 (3)	N—H(5)	0.86 (5)
C(2)—N	1.287 (4)	O—H(6)	0.80 (5)
Cl···H(4^i)	2.29 (4)	Cl···H(6)	2.09 (5)
Cl···H(5^{ii})	2.39 (5)		
C(1)—C(2)—N	122.6 (3)	N—C(2)—O	116.6 (3)
C(1)—C(2)—O	120.8 (3)		
H(4^i)···Cl···H(5^{ii})	134.1 (14)	Cl^{iii}···H(4)—N	174 (3)
H(4^i)···Cl···H(6)	118.0 (15)	Cl^{iv}···H(5)—N	171 (4)
H(5^{ii})···Cl···H(6)	108.0 (15)	Cl···H(6)—O	177 (4)

Symmetry codes: (i) $1 + x, y, 1 + z$; (ii) $x, y, 1 + z$; (iii) $x - 1, y, z - 1$; (iv) $x, y, z - 1$.

Computations were carried out on a MicroVAX 3600 computer with the *NRC* package (Gabe, Lee & Le Page, 1985). Data collection: *CAD*-4 *Software* (Enraf-Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *NRC*. Program(s) used to solve structure: *MULTAN*11/82 (Main *et al.*, 1982). Program(s) used to refine structure: *NRC*. Molecular graphics: *ORTEP* (Johnson, 1976). Software used to prepare material for publication: *NRC*.

The authors are grateful to the National Science Council of Taiwan, ROC, and Academia Sinica for partial financial support. Thanks are due to Ms S.-C. Chou for assistance in the X-ray intensity data collection.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1105). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Gabe, E. J., Lee, F. L. & Le Page, Y. (1985). *Crystallograhic Computing* 3, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 167–174. Oxford: Clarendon Press.

Gubin, A. I., Buranbaev, M. Zh. & Nurakhmetov, N. N. (1988). *Kristallografiya*, 33, 506–508.

Gubin, A. I., Yanovskii, A. I., Struchkov, Yu. T., Beremzhanov, B. A., Nurakhmetov, N. N. & Buranbaev, M. Zh. (1980). *Cryst. Struct. Commun.* **9**, 745–748.

Gubin, A. I., Yanovskii, A. I., Struchkov, Yu. T., Nurakhmetov, N. N., Buranbaev, M. Zh. & Beremzhanov, B. A. (1986). *Izv. Akad. Nauk Kaz. SSR Ser. Khim.* pp. 78–81.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

Acta Cryst. (1994). C**50**, 1335–1337

2,3,4,5,6-Penta-*O*-acetyl-D-galactonic Acid 1′-Phenyl-2′-ethoxycarbonylvinyl Ester

Jürgen Kopf and Dirk Abeln

Institut für Anorganische und Angewandte Chemie,
Universität Hamburg, Martin-Luther-King-Platz 6,
D-20146 Hamburg, Germany

Peter Köll

Fachbereich Chemie, Organische Chemie,
Universität Oldenburg, Carl-von-Ossietzky-Strasse
9-11, D-26111 Oldenburg, Germany

Roland Meisel and Klaus Peseke

Fachbereich Chemie, Universität Rostock,
Buchbinderstrasse 9, D-18051 Rostock, Germany

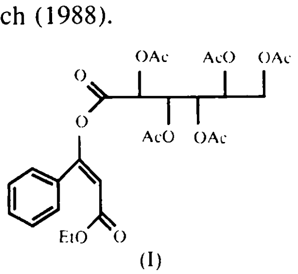
(*Received* 8 *November* 1993; *accepted* 27 *January* 1994)

Abstract

In the galactonic acid part of the title compound, $C_{27}H_{32}O_{14}$, two C—O bonds are aligned 1,3-parallel with an O···O distance of 2.884 (2) Å. The absolute structure, determined crystallographically, corresponds to the known chirality of the title compound.

Comment

The title compound (I) was synthesized by reacting penta-*O*-acetyl-D-galactonyl chloride with ethyl benzoylacetate in the presence of a base in an aprotic solvent (Meisel, 1991). In order to characterize the resulting reaction product unambiguously, an X-ray structure determination was undertaken. Suitable single crystals, melting at 403–405 K, were obtained from ethanol. The positions of all H atoms were refined with distance restraints for the C—H distances (Sheldrick, 1993). All other distances and angles are generally as expected. Friedel opposites were collected after each reflection. These Friedel pairs are regarded as symmetry independent and were not merged. Since the title compound crystallizes in a polar space group, polar axis restraints were applied by the method of Flack & Schwarzenbach (1988).



(I)

Only recently it has been realized that acetylated open-chain carbohydrates show a high tendency towards aligning primary acetoxy groups 1,3-parallel to one another (Köll *et al.*, 1993). The 1,3-parallel interactions between the heavy atoms O and C are usually designated as O//O and C//O. The O//O interaction between O4 and O6 in the title compound could easily have been avoided by a rotation of 120° around the C5—C6 bond resulting in a conformation that is free of such an interaction. The angle between the C4—O4 and C6—O6 vectors is −12.5 (1)°.

Besides the title compound, other reported cases are: xylitol pentaacetate (Park, Park & Shin, 1990), the hexaacetates of D-glucitol (Park *et al.*, 1990; Kopf, Topf, Morf, Zimmer & Köll, 1991), D,L-glucitol and galactitol (Kopf, Morf, Zimmer, Bischoff & Köll, 1992). Two acetylated derivatives of mannitol have also been described, in which such an interaction is also observed (Galan *et al.*, 1988).

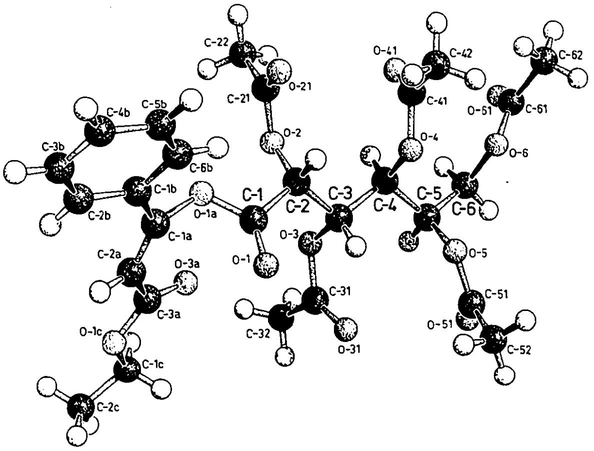


Fig. 1. *SCHAKAL*88 drawing (Keller, 1986) of the molecular conformation and atomic numbering scheme.

Experimental

Crystal data

$C_{27}H_{32}O_{14}$
$M_r = 580.53$
Monoclinic
$P2_1$
$a = 11.775\ (1)$ Å
$b = 7.763\ (1)$ Å
$c = 16.283\ (1)$ Å
$\beta = 93.79\ (1)°$
$V = 1485.2\ (2)$ Å^3
$Z = 2$
$D_x = 1.298$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 41.4–53.0°$
$\mu = 0.903$ mm^{-1}
$T = 293\ (1)$ K
Transparent blocks
0.5 × 0.4 × 0.3 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$2\theta/\omega$ scans
Absorption correction: none
7117 measured reflections
6233 independent reflections
5921 observed reflections $[I > 2\sigma(I)]$

$R_{\rm int} = 0.0279$
$\theta_{\rm max} = 76.5°$
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 9$
$l = -20 \rightarrow 20$
3 standard reflections
frequency: 120 min
intensity variation: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0412$
$wR(F^2) = 0.1124$
$S = 1.042$
6233 reflections
409 parameters
Calculated weights
$w = 1/[\sigma^2(F_o^2) + (0.0779P)^2 + 0.1346P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.430$
$\Delta\rho_{\rm max} = 0.201$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.201$ e Å^{-3}

Extinction correction: *SHELXL* (Sheldrick, 1994)
Extinction coefficient: 0.004 (0)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack parameter = −0.07 (13) (Flack, 1983)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

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

	x	y	z	$U_{\rm eq}$
O1	0.6184 (2)	0.1119 (2)	0.3827 (1)	0.0720 (5)
O2	0.6965 (1)	0.5128 (2)	0.2972 (1)	0.0435 (2)
O3	0.5401 (1)	0.3144 (2)	0.2082 (1)	0.0484 (3)
O4	0.8375 (1)	0.2543 (2)	0.1722 (1)	0.0402 (2)
O5	0.7003 (1)	0.0327 (1)	0.0826 (1)	0.0395 (2)
O6	0.8460 (1)	0.2703 (2)	−0.0043 (1)	0.0498 (3)
O21	0.8721 (1)	0.5331 (2)	0.3568 (1)	0.0757 (5)
O31	0.4742 (2)	0.0467 (3)	0.1844 (2)	0.0979 (7)
O41	0.8983 (1)	0.5236 (2)	0.1506 (1)	0.0703 (4)
O51	0.5479 (1)	−0.0337 (2)	−0.0010 (1)	0.0652 (4)
O61	0.8506 (2)	0.5026 (3)	−0.0828 (1)	0.0947 (7)
C1	0.6449 (1)	0.2596 (2)	0.3766 (1)	0.0458 (3)
C2	0.7026 (1)	0.3298 (2)	0.3031 (1)	0.0393 (3)
C3	0.6536 (1)	0.2506 (2)	0.2232 (1)	0.0383 (3)
C4	0.7216 (1)	0.3028 (2)	0.1500 (1)	0.0350 (3)
C5	0.6780 (1)	0.2143 (2)	0.0710 (1)	0.0356 (3)
C6	0.7238 (1)	0.2823 (2)	−0.0072 (1)	0.0459 (3)
C21	0.7901 (2)	0.6005 (3)	0.3260 (1)	0.0504 (4)
C22	0.7732 (2)	0.7894 (3)	0.3131 (2)	0.0726 (6)
C31	0.4555 (2)	0.1973 (4)	0.1915 (1)	0.0658 (6)
C32	0.3432 (2)	0.2841 (6)	0.1832 (2)	0.089 (1)
C41	0.9182 (1)	0.3787 (3)	0.1722 (1)	0.0479 (4)
C42	1.0314 (2)	0.3100 (4)	0.2022 (2)	0.0717 (6)
C51	0.6241 (1)	−0.0769 (2)	0.0465 (1)	0.0461 (4)
C52	0.6486 (2)	−0.2570 (3)	0.0733 (2)	0.0786 (8)
C61	0.9003 (2)	0.3903 (3)	−0.0461 (1)	0.0507 (4)
C62	1.0256 (2)	0.3660 (3)	−0.0397 (2)	0.0679 (6)
O1*a*	0.6379 (1)	0.3786 (2)	0.4360 (1)	0.0514 (3)
O1*c*	0.2942 (1)	0.2816 (3)	0.4740 (1)	0.0684 (4)
O3*a*	0.4123 (2)	0.3462 (4)	0.3769 (1)	0.0889 (6)
C1*a*	0.5949 (2)	0.3263 (3)	0.5106 (1)	0.0505 (4)
C2*a*	0.4843 (2)	0.2969 (3)	0.5155 (1)	0.0577 (5)
C3*a*	0.3976 (2)	0.3131 (3)	0.4471 (1)	0.0602 (5)
C1*b*	0.6824 (2)	0.3181 (3)	0.5793 (1)	0.0552 (4)
C2*b*	0.6540 (2)	0.3381 (4)	0.6593 (1)	0.0766 (7)
C3*b*	0.7359 (3)	0.3235 (5)	0.7238 (2)	0.0929 (9)
C4*b*	0.8467 (2)	0.2917 (5)	0.7087 (2)	0.0916 (9)
C5*b*	0.8765 (2)	0.2735 (6)	0.6295 (2)	0.098 (1)
C6*b*	0.7954 (2)	0.2900 (5)	0.5645 (2)	0.0788 (7)
C1*c*	0.2002 (2)	0.2894 (5)	0.4120 (1)	0.0800 (8)
C2*c*	0.0934 (2)	0.2823 (8)	0.4558 (2)	0.111 (1)

Table 2. *Selected geometric parameters* (Å, °)

O1—C1	1.194 (2)	C5—C6	1.511 (2)
O2—C21	1.353 (2)	C21—C22	1.493 (3)
O2—C2	1.425 (2)	C31—C32	1.482 (4)
O3—C31	1.363 (2)	C41—C42	1.488 (3)
O3—C3	1.431 (2)	C51—C52	1.487 (3)
O4—C41	1.354 (2)	C61—C62	1.484 (3)
O4—C4	1.439 (2)	O1*a*—C1*a*	1.407 (2)
O5—C51	1.343 (2)	O1*c*—C3*a*	1.343 (2)
O5—C5	1.444 (2)	O1*c*—C1*c*	1.449 (3)
O6—C61	1.341 (2)	O3*a*—C3*a*	1.195 (3)
O6—C6	1.440 (2)	C1*a*—C2*a*	1.330 (3)
O21—C21	1.180 (3)	C1*a*—C1*b*	1.471 (3)
O31—C31	1.197 (3)	C2*a*—C3*a*	1.466 (3)
O41—C41	1.197 (3)	C1*b*—C2*b*	1.375 (3)
O51—C51	1.194 (2)	C1*b*—C6*b*	1.385 (3)
O61—C61	1.189 (3)	C2*b*—C3*b*	1.383 (4)
C1—O1*a*	1.343 (2)	C3*b*—C4*b*	1.367 (4)
C1—C2	1.517 (2)	C4*b*—C5*b*	1.367 (4)
C2—C3	1.518 (2)	C5*b*—C6*b*	1.383 (4)
C3—C4	1.533 (2)	C1*c*—C2*c*	1.487 (4)
C4—C5	1.518 (2)		
C21—O2—C2	116.26 (13)	O41—C41—O4	123.1 (2)
C31—O3—C3	117.8 (2)	O41—C41—C42	125.9 (2)
C41—O4—C4	117.84 (13)	O4—C41—C42	110.9 (2)
C51—O5—C5	116.78 (12)	O51—C51—O5	123.8 (2)
C61—O6—C6	116.52 (14)	O51—C51—C52	125.3 (2)
O1—C1—O1*a*	125.0 (2)	O5—C51—C52	110.87 (14)
O1—C1—C2	122.9 (2)	O61—C61—O6	122.0 (2)
O1*a*—C1—C2	111.88 (15)	O61—C61—C62	125.6 (2)
O2—C2—C1	112.81 (13)	O6—C61—C62	112.4 (2)
O2—C2—C3	109.37 (13)	C1—O1*a*—C1*a*	117.72 (15)
C1—C2—C3	111.53 (13)	C3*a*—O1*c*—C1*c*	115.6 (2)
O3—C3—C2	107.67 (12)	C2*a*—C1*a*—O1*a*	120.7 (2)
O3—C3—C4	107.85 (12)	C2*a*—C1*a*—C1*b*	125.9 (2)
C2—C3—C4	111.78 (12)	O1*a*—C1*a*—C1*b*	113.3 (2)
O4—C4—C5	110.93 (11)	C1*a*—C2*a*—C3*a*	125.2 (2)
O4—C4—C3	105.73 (11)	O3*a*—C3*a*—O1*c*	122.9 (2)
C5—C4—C3	111.98 (12)	O3*a*—C3*a*—C2*a*	127.4 (2)
O5—C5—C6	112.28 (12)	O1*c*—C3*a*—C2*a*	109.7 (2)
O5—C5—C4	106.40 (12)	C2*b*—C1*b*—C6*b*	118.6 (2)
C6—C5—C4	115.88 (13)	C2*b*—C1*b*—C1*a*	120.9 (2)
O6—C6—C5	111.16 (12)	C6*b*—C1*b*—C1*a*	120.5 (2)
O21—C21—O2	123.3 (2)	C1*b*—C2*b*—C3*b*	120.6 (2)
O21—C21—C22	126.3 (2)	C4*b*—C3*b*—C2*b*	120.3 (2)
O2—C21—C22	110.4 (2)	C5*b*—C4*b*—C3*b*	119.7 (2)
O31—C31—O3	122.3 (2)	C4*b*—C5*b*—C6*b*	120.3 (2)
O31—C31—C32	127.2 (2)	C1*b*—C6*b*—C5*b*	120.3 (2)
O3—C31—C32	110.5 (3)	O1*c*—C1*c*—C2*c*	107.2 (2)

Data collection and cell refinement: Enraf–Nonius CAD-4 software. Data reduction: *CADSHEL* (Kopf, 1987). Program(s) used to solve structure: *SHELXS*90 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL* (Sheldrick, 1994). Molecular graphics: *SCHAKAL*88 (Keller, 1986). Software used to prepare material for publication: *CIF2TEX* (Kopf, 1992*a*); *FCF2FOC* (Kopf, 1992*b*).

We gratefully acknowledge financial support from the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1135). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Galan, E. R., Hodgson, D. J., Yokomori, Y., Eliel, E. L., Martinez, M. B. & Blazquez, J. A. S. (1988). *Carbohydr. Res.* **180**, 263–276.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Flack, H. D. & Schwarzenbach, D. (1988). *Acta Cryst.* A**44**, 499–506.

Keller, E. (1986). *Chem. Unserer Zeit*, **20**, 178–181.

Kopf, J. (1987). *CADSHEL. Program for Data Reduction of Enraf–Nonius CAD-4 Data.* Univ. of Hamburg, Germany.

Kopf, J. (1992*a*). *CIF2TEX. Program for Automatic Preparation of Deposition Data.* Univ. of Hamburg, Germany.

Kopf, J. (1992*b*). *FCF2FOC. Program for Printing F_c^2 and F_o^2.* Univ. of Hamburg, Germany.

Kopf, J., Topf, C., Morf, M., Zimmer, B. & Köll, P. (1991). *Acta Cryst.* C**47**, 2425–2428.

Kopf, J., Morf, M., Zimmer, B., Bischoff, M. & Köll, P. (1992). *Carbohydr. Res.* **229**, 17–32.

Köll, P., Morf, M., Zimmer, B., Kopf, J., Berger, A., Dax, K. & Stütz, A. E. (1993). *Carbohydr. Res.* **242**, 21–28.

Meisel, R. (1991). Diploma thesis, Univ. of Rostock, Germany.

Park, Y. J., Park, M. H. & Shin, J. M. (1990). *Daehan Hwahak Hwoejee*, **34**, 517–526 (in Korean); *Chem. Abstr.* (1991). **114**, 82345a.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1994). *J. Appl. Cryst.* In preparation.

Acta Cryst. (1994). C**50**, 1337–1339

Spiro[3-germabicyclo[3.1.0]hexane-3,9′-[9]germafluorene]

Keiichiro Ogawa and Yoshito Takeuchi

Department of Chemistry,
The College of Arts and Sciences,
The University of Tokyo, Komaba, Meguro-ku,
Tokyo 153, Japan

Georges Manuel and Rabah Boukherroub

Laboratoire des Organometalliques,
Université Paul-Sabatier, 118 route de Narbonne,
31062 Toulouse CEDEX, France

(*Received* 20 *January* 1994; *accepted* 15 *March* 1994)

Abstract

The 3-germabicyclo[3.1.0]hexane moiety in the title compound, $C_{17}H_{16}Ge$, adopts a boat form even though the ring bears bulky substituents. This stresses the difference in the structures of germa- *versus* carbocycles.

Comment

The preference for the boat conformation of the bicyclo[3.1.0]hexane (BCH) ring system, shown in Fig. 1, has been well documented (Mastryukov, Osina, Vikov & Hildebrandt, 1977; Kisiel & Legon, 1978). It was shown that the origin of this rather unexpected conformation is the enhanced eclipsing effect in the chair form, which