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Crystal and molecular structures of the pentaacetates of D-gluconic acid and derived *tert*-butyl- and *tert*-butylperoxyesters and the tetra-*O*-acetyl-1,2-*O*-*tert*-butyl-peroxyorthoacetyl derivative [1]

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Abstract

The crystal structures of 2,3,4,5,6-penta-*O*-acetyl-D-gluconic acid (**1**), 2,3,4,5,6-penta-*O*-acetyl-D-gluconic acid *tert*-butylester (**2**), 2,3,4,5,6-penta-*O*-acetyl-D-gluconic acid *tert*-butylperoxyester (**3**) and 3,4,5,6-tetra-*O*-acetyl-1,2-(*tert*-butylperoxyorthoacetyl)-D-gluconic acid (**4**) were determined by X-ray crystallography, using direct methods, and refined to final conventional residual parameters of $R = 0.050$, 0.037, 0.042 and 0.042, respectively. In all cases, a planar zigzag conformation of the carbon chain is adopted with a resulting 1,3-parallel interaction between O-2 and O-4. In **2**, a second 1,3-parallel alignment between O-3 and the esterified oxygen at C-1 is observed. The peroxycarbonyl group in **3** is essentially planar with a torsion angle about the O–O bond of −119° for **3** and 145° for **4**. © 1998 Elsevier Science Ltd

Keywords: X-ray structures; 1,3-Parallel interactions; Conformation; Peroxyesters; D-Gluconic acid, protected; D-Gluconic acid esters, protected

1. Introduction

It was for a long time the almost generally accepted opinion that the carbon chain in alditols and related compounds in the crystalline state had to bend to a sickle in order to avoid 1,3-parallel interactions between oxygen atoms which would occur in planar zigzag conformations. Angyal et al. [1] were the first to doubt this point of view. Recently, we were able to show that 1,3-parallel O||O arrangements in planar zigzag chains as well as 1,3-parallel C||O orientations in sickle conformations are often tolerated [2–4].

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[1] Dedicated to Professor Manfred Weidenbruch on the occasion of his 60th birthday.

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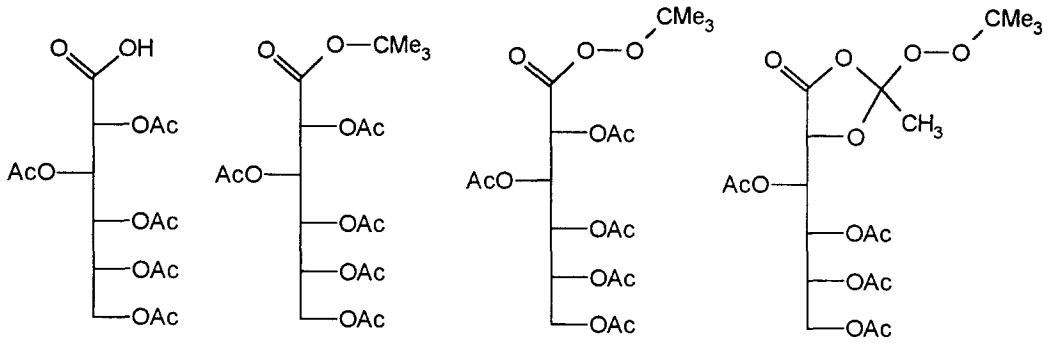
In order to broaden this still small basis of data, the title compounds were subjected to X-ray analysis. All the compounds investigated adopted planar zigzag conformations involving 1,3-parallel interactions between oxygens.



1 2 3 4

Another interesting feature of compounds **3** and **4** is the geometry of the peroxy group. Up to now, only three structure determinations of peroxyesters have been performed, namely, of *tert*-butyl 3-nitroperbenzoate [5], *tert*-butyl 4-nitroperbenzoate [5] and *tert*-butyl 2,4,6-tri-*tert*-butylperbenzoate [6]. This lack is, on the one hand, undoubtedly caused by the chemical and thermal instability of this compounds and, on the other hand, by the decay of crystals when exposed to the X-ray radiation [5].

2. Results and discussion

Suitable crystals of 2,3,4,5,6-penta-*O*-acetyl-D-gluconic acid (**1**) [7] were obtained from solutions in toluene. For 2,3,4,5,6-penta-*O*-acetyl-D-gluconic acid *tert*-butylester (**2**), 2,3,4,5,6-penta-*O*-acetyl-D-gluconic acid *tert*-butylperoxyester (**3**) [8] and 3,4,5,6-tetra-*O*-acetyl-1,2-(*tert*-butylperoxyorthoacetyl)-D-gluconic acid (**4**) [8], methylene chloride–petroleum ether showed to be the solvent of choice.

The structures were solved by direct methods with the program SHELXS-96 [9] and refined with the program SHELXL-96 [10]. The refinement was done on F^2 for all reflections. The validated threshold $F > 2\sigma(F)$ was used for calculating R_{obsd} only. All atoms, including hydrogens introduced at theoretical positions using AFIX option [10], were refined. Table 1 covers the relevant crystallographic data of all compounds investigated. [2] The final fractional atomic coordinates of C and O with equivalent isotropic thermal parameters are listed in Table 2 for compounds **1** and **2**, and in Table 3 for **3** and **4**. ORTEP equivalent illustrations of the title compounds including the atom numbering scheme were obtained with PLATON96 [11] and are presented in Figs. 1–4, respectively.

In all cases, a planar zigzag conformation of the carbon skeleton is observed with a resulting 1,3-parallel orientation of O-2 and O-4. Only in the case of **1**, the carbon chain is extended by O-6 at one end. Interestingly, in **2** O-3||O-11 are found in a 1,3-parallel arrangement as well, despite the fact that this could be avoided easily by a simple rotation around the C-1 C-2 bond, which would not change the overall geometry of the carbon chain. From the values of the O||O interactions presented in Table 4, it is obvious that the distances between O-2 and O-4 are considerably lower than the sum of the van der Waals radii [12] of 304 pm. Therefore, these compounds are other examples of substances with a gluco or xylo configuration found with this topology [3,4].

More specifically, it can be stated that gluconic acid derivatives (salts, esters and amides) are very prone to adopt the zigzag conformation in the solid state. The most recent release of the Cambridge File [13] of spring 1997 reports about 28 structures of this kind. Only, in one third of these structures, the gluconic acid moiety is found in a sickle conformation with D-gluconic acid as monohydrate [14] the

[2] Tables of atomic coordinates, bond lengths, and bond angles have been deposited with the Cambridge Crystallographic Data Centre. These tables may be obtained, on request, from the Director, Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge, CB2 1EZ, UK.

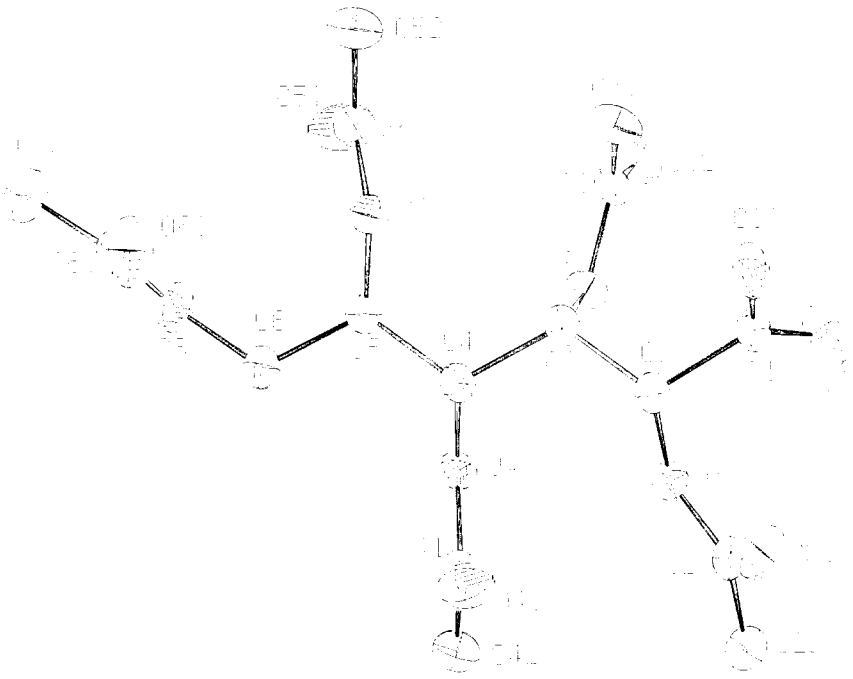


Fig. 1. ORTEP equivalent representation [11] of a molecule of 2,3,4,5,6-penta-*O*-acetyl-D-gluconic acid (**1**).

most prominent representative. But the majority of the compounds prefers the planar conformation in the crystal. It should be mentioned that, in almost all previously reported cases, the hydroxy groups are unprotected. Due to the comparably much lower steric demand of an acetoxy group, acetylated derivatives of gluconic acid, as compounds **1**–**4**, most probably will adopt this conformation generally.

An intermolecular hydrogen bond between the donor O-11 H-11 and the acceptor oxygen O-51 is only observed in compound **1**. The distance between the hydrogen and the donor, the hydrogen and the

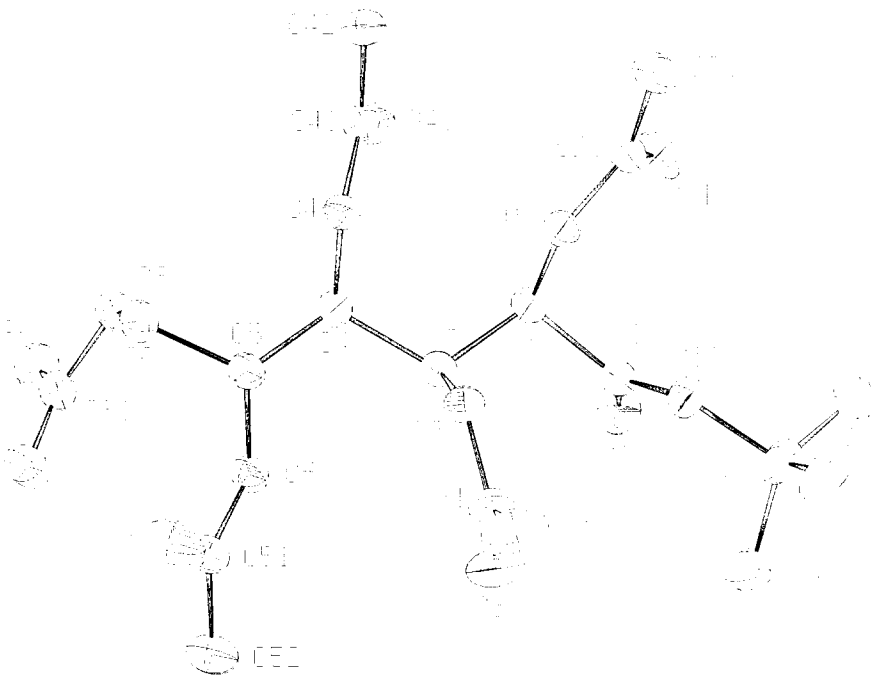


Fig. 2. ORTEP equivalent representation [11] of a molecule of 2,3,4,5,6-penta-*O*-acetyl-D-gluconic acid *tert*-butylester (**2**).

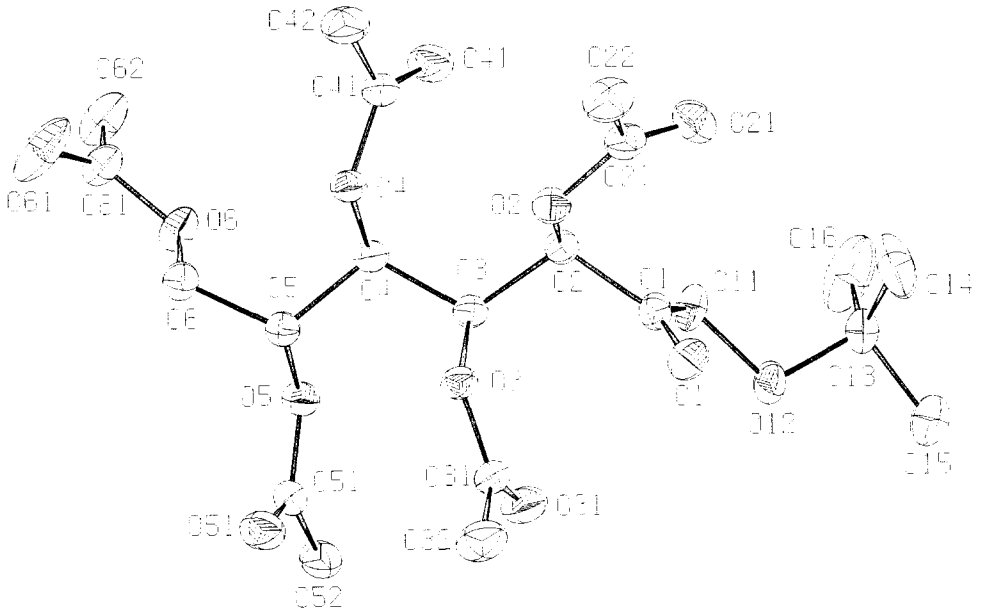


Fig. 3. ORTEP equivalent representation [11] of a molecule of 2,3,4,5,6-penta-*O*-acetyl-D-gluconic acid *tert*-butylperoxyester (**3**).

acceptor, and the donor and the acceptor are 88.0, 249.8 and 331.7 pm, respectively. The angle between O-11 H-11 O-51 is 155.4°. The symmetry operation on the acceptor oxygen O-51 is $1-x$, $1-y$, $1-z$.

In all compounds investigated, no unusual bond lengths and angles were found.

Compounds **3** and **4** were very stable to X-rays at −100 °C, thus, enabling a satisfactory accuracy in

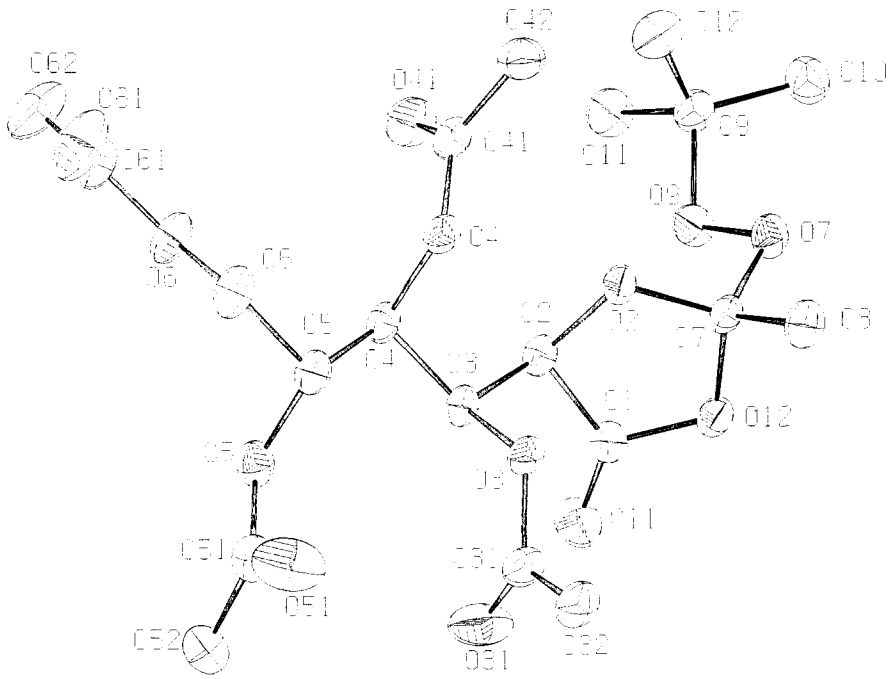


Fig. 4. ORTEP equivalent representation [11] of a molecule of 3,4,5,6-tetra-*O*-acetyl-1,2-(*tert*-butylperoxyorthoacetyl)-D-gluconic acid (**4**).

Table 1
Crystallographic data for **1**, **2**, **3** and **4**[a,b]

	1	**2**	**3**	**4**
Formula	$C_{16}H_{22}O_{12}$	$C_{20}H_{30}O_{12}$	$C_{20}H_{30}O_{13}$	$C_{20}H_{30}O_{13}$
Molecular weight	406.34	462.44	478.44	478.44
Crystal dimensions (mm)	0.7 × 0.4 × 0.4	0.6 × 0.4 × 0.2	0.6 × 0.4 × 0.3	0.6 × 0.4 × 0.4
mp (°C)	110	63–64	68–69	158
Space group	$P2_1$	$P2_12_12_1$	$P2_12_12_1$	$P2_1$
Cell parameters (pm, deg)				
a	1021.0(1)	817.1(1)	1013.9(1)	1231.5(1)
b	843.6(1)	1205.6(1)	1393.2(1)	785.6(1)
c	1193.9(1)	2404.9(2)	1731.3(1)	1242.7(1)
β	104.38(1)			102.37(1)
Volume V (pm^3)	$966.1(2) \times 10^6$	$2369.1(4) \times 10^6$	$2445.6(3) \times 10^6$	$1174.4(2) \times 10^6$
Z	2	4	4	2
F (000)	428	984	1016	508
Calculated density, D_x ($g\ cm^{-3}$)	1.355	1.297	1.299	1.353
μ (cm^{-1})	10.2	9.2	9.4	9.8
λ (Cu K_α) (pm)	154.178	154.178	154.178	154.178
2θ range (deg)	7.6–152.9	7.4–152.8	8.1–152.7	7.3–153.3
Reflections measured	2383	2873	2941	2804
Symmetry independent reflections	2239	2844	2903	2659
Reflections with ($F > 2\sigma(F)$)	2185	2764	2665	2623
Number of refined parameters	284	328	337	337
Ratio of valued reflections to parameters	7.7	8.4	7.9	7.8
Final residual factors $R^{c,d}$				
$R_{1(obsd)}$	0.050	0.037	0.042	0.042
$wR_{2(all)}$	0.136	0.102	0.121	0.115
$wR_{2(obsd)}$	0.134	0.100	0.110	0.114
Goodness of fit S^e				
S_{all}	1.07	1.09	1.07	1.05
S_{obsd}	1.07	1.08	1.02	1.05
Largest difference peak ($e\ pm^{-3}$)	0.35×10^{-6}	0.30×10^{-6}	0.25×10^{-6}	0.33×10^{-6}
Largest difference hole ($e\ pm^{-3}$)	-0.36×10^{-6}	-0.24×10^{-6}	-0.29×10^{-6}	-0.31×10^{-6}
Flack parameter	0.0(3)	0.05(2)	−0.4(2)	−0.1(2)

[a] Standard deviations in parentheses.
[b] Diffractometer Enraf-Nonius CAD4.
[c] Definitions: $R_1 = \Sigma||F_0| - |F_c||/\Sigma|F_0|$; $wR_2 = [\Sigma w(F_0^2 - F_c^2)^2/\Sigma w(F_0^2)^2]^{1/2}$.
[d] Weighting scheme: **1**: $w = 1/[\sigma^2(F_0^2) + (0.0982P)^2 + 0.3228P]$ where $P = (F_0^2 + 2F_c^2)/3$; **2**: $w = 1/[\sigma^2(F_0^2) + (0.0735P)^2 + 0.3183P]$ where $P = (F_0^2 + 2F_c^2)/3$; **3**: $w = 1/[\sigma^2(F_0^2) + (0.0773P)^2 + 0.7400P]$ where $P = (F_0^2 + 2F_c^2)/3$; **4**: $w = 1/[\sigma^2(F_0^2) + (0.0794P)^2 + 0.3978P]$ where $P = (F_0^2 + 2F_c^2)/3$.
[e] Definitions: $S = [\Sigma w(F_0^2 - F_c^2)^2/(n-p)]^{1/2}$.

the structure determinations. The geometry of the peroxy group is particularly interesting. The O–O bond distances for **3** and **4** of 146.3(2) pm and 147.8(2) pm, respectively, are within the range of 145 pm to 149 pm observed for the previously published compounds [5,6]. The peroxycarbonyl group in **3** is almost planar with a torsion angle of 8.0(3)° and, therefore, well comparable with the previously examined substances [5,6]. The torsion angle about O-11 and O-12 of −119.3(2)° in **3** agrees with the values of the nitroperbenzoates [5], whereas the torsional angle around the peroxy bond in **4** of 145.0(2)° is somewhat greater.

3. Experimental

General methods.—NMR spectroscopy data were recorded with a Bruker AMX 500 spectrometer.

Table 2
Fractional positional parameters of C and O atoms and the temperature factors U_{eq} for **1** and **2**[a]

Atom	**1**				Atom	**2**			
	10^4 x	10^4 y	10^4 z	10^3 U_{eq}		10^4 x	10^4 y	10^4 z	10^3 U_{eq}
O-11	7112(2)	2394(4)	1913(2)	40(1)	O-1	10777(2)	6540(1)	2096(1)	32(1)
O-12	6665(3)	726(3)	3216(3)	47(1)	O-2	9569(1)	6905(1)	701(1)	23(1)
O-2	4594(2)	2574(3)	3473(2)	27(1)	O-3	7154(2)	5533(1)	1080(1)	24(1)
O-21	6348(2)	4027(4)	4447(2)	45(1)	O-4	9274(2)	4825(1)	152(1)	23(1)
O-3	3580(2)	813(2)	1421(2)	25(1)	O-5	7841(2)	3019(1)	1233(1)	24(1)
O-31	4692(2)	120(3)	82(2)	35(1)	O-6	10176(2)	2035(1)	495(1)	29(1)
O-4	2053(2)	3317(3)	2067(2)	26(1)	O-11	8687(2)	7549(1)	1735(1)	27(1)
O-41	2864(3)	5633(3)	2881(2)	44(1)	O-21	11872(2)	7844(1)	897(1)	36(1)
O-5	2374(2)	3028(3)	−864(2)	26(1)	O-31	6648(2)	5227(2)	1989(1)	40(1)
O-51	1189(3)	823(4)	−1516(2)	49(1)	O-41	11996(2)	5109(1)	217(1)	32(1)
O-6	−344(2)	4170(3)	−1211(2)	35(1)	O-51	5373(2)	2326(2)	1017(1)	46(1)
O-61	489(2)	5901(4)	−2272(3)	52(1)	O-61	8895(2)	703(1)	992(1)	36(1)
C-1	6379(3)	1845(4)	2592(2)	28(1)	C-1	9867(2)	6796(1)	1725(1)	24(1)
C-2	5104(2)	2831(3)	2476(2)	24(1)	C-2	9957(2)	6201(1)	1165(1)	21(1)
C-3	4015(2)	2429(3)	1386(2)	23(1)	C-3	8828(2)	5192(1)	1151(1)	21(1)
C-4	2783(2)	3502(3)	1192(2)	23(1)	C-4	9272(2)	4349(1)	702(1)	22(1)
C-5	1709(2)	3156(4)	62(2)	26(1)	C-5	8041(2)	3392(1)	667(1)	24(1)
C-6	720(3)	4524(5)	−200(3)	38(1)	C-6	8624(2)	2454(2)	300(1)	29(1)
C-21	5351(3)	3267(4)	4445(3)	32(1)	C-12	8449(3)	8265(2)	2236(1)	36(1)
C-22	4787(4)	2965(5)	5461(3)	42(1)	C-13	9900(4)	9032(2)	2282(1)	52(1)
C-31	4002(3)	−239(3)	721(2)	25(1)	C-14	6894(4)	8881(3)	2088(1)	56(1)
C-32	3474(3)	−1858(4)	874(3)	34(1)	C-15	8198(4)	7552(2)	2754(1)	44(1)
C-41	2162(3)	4494(4)	2866(3)	31(1)	C-21	10689(2)	7713(1)	603(1)	26(1)
C-42	1317(3)	4150(5)	3687(3)	42(1)	C-22	10256(3)	8384(2)	103(1)	34(1)
C-51	1996(3)	1805(4)	−1619(3)	31(1)	C-31	6186(2)	5508(2)	1540(1)	29(1)
C-52	2718(4)	1886(5)	−2558(3)	41(1)	C-32	4490(3)	5876(3)	1394(1)	48(1)
C-61	−339(3)	4934(4)	−2186(3)	31(1)	C-41	10742(2)	5183(2)	−44(1)	25(1)
C-62	−1509(3)	4458(5)	−3154(3)	41(1)	C-42	10579(3)	5631(2)	−621(1)	32(1)
					C-51	6444(2)	2468(2)	1353(1)	29(1)
					C-52	6436(3)	2087(2)	1941(1)	37(1)
					C-61	10134(3)	1151(2)	844(1)	29(1)
					C-62	11829(3)	807(2)	1000(1)	37(1)

[a]Standard deviations in parentheses.

Chemical shifts are expressed in ppm downfield from the signal for internal Me_4Si. Optical rotations was determined with a Perkin Elmer 343 polarimeter at 20 °C. Column chromatography was performed on Silica Gel F_{254} from E. Merck. Mass spectra was recorded on a Finnigan MAT 95 using CI with isobutane as reactant gas. Melting points are uncorrected. The X-ray structure determinations were performed at −100 °C (173 K) by using an Enraf-Nonius CAD-4 diffractometer. The relevant options of the SCHAKAL-92 program [15] were used for the calculation of the geometrical parameters given in Table 4.

2,3,4,5,6-Penta-O-*acetyl*-D-*gluconic acid* tert-*butyl ester* (**2**).—2,3,4,5,6-Penta-*O*-acetyl-D-gluconic acid chloride [7] (1 g, 2.4 mmol) in dry CH_2Cl_2 (5 mL) was added at 0 °C during 10 min to a stirred mixture of dry pyridine (0.3 mL, 3 mmol) and *tert*-butanol (0.4 mL, 4 mmol) in CH_2Cl_2 (10 mL). The mixture was stirred for 1.5 h and then washed with water and concentrated. Column chromatography (2:1 EtOAc–petroleum ether) of the crude product afforded **2** (0.62 g, 57%); mp 63–64 °C; $[\alpha]_D$ +13.1° (*c* 1.39, $CHCl_3$); ^{1}H NMR ($CDCl_3$): δ 5.67 (dd, 1 H, $J_{2,3}$ 3.8 Hz, $J_{3,4}$ 5.5 Hz, H-3), 5.48 (dd, 1 H, $J_{4,5}$ 5.5 Hz, H-4), 5.20 (d, 1 H, H-2), 5.03 (ddd, 1 H, $J_{5,6}$ 6.1 Hz, $J_{5,6'}$ 4.4 Hz, H-5), 4.30 (dd, 1 H, $J_{6',6}$ −12.1 Hz,

Table 3
Fractional positional parameters of C and O atoms and the temperature factors U_{eq} for **3** and **4**[a]

Atom	**3**				Atom	**4**			
	10^4 x	10^4 y	10^4 z	10^3 U_{eq}		10^4 x	10^4 y	10^4 z	10^3 U_{eq}
O-1	1625(2)	10911(1)	77(1)	34(1)	O-11	4368(2)	1188(3)	−339(2)	37(1)
O-2	2665(2)	10084(1)	1416(1)	26(1)	O-12	4136(1)	2889(2)	1036(1)	25(1)
O-3	3011(2)	8942(1)	160(1)	24(1)	O-2	5281(1)	1614(2)	2492(1)	24(1)
O-4	3247(2)	8159(1)	1786(1)	24(1)	O-3	6988(1)	2034(2)	1360(1)	23(1)
O-5	1972(2)	6812(1)	179(1)	25(1)	O-31	6873(2)	1870(3)	−468(2)	48(1)
O-6	2203(2)	5863(1)	1608(1)	30(1)	O-4	7308(1)	−32(2)	3314(1)	24(1)
O-11	−342(2)	10258(1)	428(1)	30(1)	O-41	6075(2)	−1953(3)	3681(2)	38(1)
O-12	−886(2)	10777(2)	−233(1)	39(1)	O-5	8366(1)	−1421(2)	987(2)	27(1)
O-21	1354(2)	11248(2)	1889(1)	37(1)	O-51	9639(2)	508(4)	752(2)	53(1)
O-31	1639(2)	8910(2)	−867(1)	35(1)	O-6	8686(2)	−3499(2)	2990(2)	31(1)
O-41	1629(2)	8593(2)	2614(1)	35(1)	O-61	10179(2)	−4260(3)	4260(2)	54(1)
O-51	3528(2)	6789(2)	−756(1)	35(1)	O-7	3559(1)	2637(2)	2665(1)	26(1)
O-61	3531(2)	5034(2)	2383(2)	60(1)	O-9	3103(2)	953(2)	2276(1)	27(1)
C-1	982(3)	10380(2)	462(1)	25(1)	C-1	4564(2)	1524(4)	619(2)	24(1)
C-2	1511(2)	9679(2)	1064(1)	22(1)	C-2	5304(2)	577(3)	1556(2)	23(1)
C-3	1913(2)	8744(2)	670(1)	22(1)	C-3	6479(2)	372(3)	1364(2)	21(1)
C-4	2283(2)	7909(2)	1205(1)	22(1)	C-31	7193(2)	2602(3)	389(2)	27(1)
C-5	2939(2)	7097(2)	749(1)	22(1)	C-32	7874(2)	4181(4)	546(2)	32(1)
C-6	3360(3)	6246(2)	1229(2)	26(1)	C-4	7221(2)	−728(3)	2233(2)	23(1)
C-13	−1825(3)	11493(2)	43(2)	31(1)	C-41	6675(2)	−733(4)	3959(2)	27(1)
C-14	−1117(4)	12235(3)	519(3)	60(1)	C-42	6812(3)	212(4)	5017(2)	35(1)
C-15	−2297(4)	11897(3)	−719(2)	55(1)	C-5	8420(2)	−792(3)	2081(2)	25(1)
C-16	−2937(4)	11013(4)	472(3)	74(2)	C-51	8989(2)	−605(4)	374(2)	30(1)
C-21	2448(3)	10898(2)	1829(2)	29(1)	C-52	8749(2)	−1204(5)	−777(2)	37(1)
C-22	3679(3)	11257(2)	2191(2)	42(1)	C-6	9187(2)	−1855(4)	2931(2)	30(1)
C-31	2728(3)	9029(2)	−603(1)	28(1)	C-61	9284(2)	−4605(4)	3714(2)	33(1)
C-32	3933(3)	9288(3)	−1052(2)	42(1)	C-62	8689(3)	−6263(5)	3727(3)	49(1)
C-41	2786(3)	8466(2)	2482(1)	27(1)	C-7	4482(2)	2911(3)	2209(2)	22(1)
C-42	3890(3)	8615(3)	3036(2)	38(1)	C-8	4937(2)	4628(3)	2607(2)	30(1)
C-51	2392(3)	6699(2)	−561(2)	27(1)	C-9	2674(2)	182(4)	3163(2)	27(1)
C-52	1254(3)	6488(2)	−1079(2)	35(1)	C-10	1707(2)	1232(4)	3384(2)	34(1)
C-61	2434(3)	5232(2)	2179(2)	31(1)	C-11	2273(2)	−1540(4)	2653(2)	34(1)
C-62	1202(3)	4837(3)	2507(2)	45(1)	C-12	3595(2)	−42(4)	4186(2)	37(1)

[a]Standard deviations in parentheses.

Table 4
1,3-Dihedral angles and distances of the O||O interactions in **1**, **2**, **3** and **4**

Compound	Interaction	Distance O||O (pm)	1,3-Dihedral angle (deg)
1	O-2||O-4	278.8	0.3
2	O-2||O-4	284.4	15.6
2	O-11||O-3	315.5	8.2
3	O-2||O-4	281.9	18.2
4	O-2||O-4	280.2	9.1

H-6′), 4.14 (dd, 1 H, H-6), 2.20, 2.09 (2 ×), 2.06 and 2.05 (4 s, 15 H, 5 Ac), 1.44 (s, 9 H, *tert*-butyl); ^{13}C NMR: 170.3, 169.9, 169.6, 169.5, 169.3, 165.3, 83.5, 71.2, 69.7, 68.7, 68.6, 61.3, 27.7, 20.7 (3 ×), 20.4 (2 ×); CIMS: 463 MH^+. Anal. Calcd. for $C_{20}H_{30}O_{12}$: C, 51.95; H, 6.54. Found: C, 52.10, H, 6.23.

Compounds **1**, **3** and **4** were prepared following reported procedures [7,8].

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