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2,3,6-Trimethylgalactono-γ-lactone

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Abstract. $C_9O_6H_{16}$, orthorhombic, $P2_12_12_1$, $a=31{\cdot}032$ (5), $b=7{\cdot}875$ (4), $c=4{\cdot}488$ (2) Å, $Z=4$, $D_m=1{\cdot}27$ (1), $D_x=1{\cdot}272$ g cm^{-3}. The crystals were supplied by Dr J. H. Robertson, University of Leeds. The structure was determined at 120°K by photographic methods, solved by direct methods and refined to a final R of 0·1022, with all hydrogen atoms placed.

Introduction. All photographs were taken at a temperature of 120°K with dry nitrogen gas as the coolant from apparatus similar to that described previously (Robertson, 1960). Cell dimensions were measured by extrapolation to $\theta=90°$ of high-order reflexions on zero-layer Weissenberg photographs, calibrated with aluminum-wire powder lines. A crystal of dimensions $0{\cdot}5\times0{\cdot}5\times0{\cdot}6$ mm was used to record multiple-film Weissenberg photographs with b and c as rotation axes for all layers up to $\mu=30°$ and the remaining reflexions were obtained by recording three layers with the crystal set to rotate about $[0\bar{1}2]$. For Cu $K\alpha$ radiation 1611 independent reflexions should be available and, of these, 1457 were recorded, 29 of which were too weak to be measured and were included in the later stages of refinement with values of half the minimum observed. The systematic absences, $h00$ for h odd, $0k0$ for k odd and $00l$ for l odd indicated that the space group is $P2_12_12_1$ (No. 19). The intensities were measured by visual comparison with calibrated intensity spots, the Lorentz–polarization factors applied, correlated by a program based on the use of an inverse scale factor (Monahan, Schiffer & Schiffer, 1967) and placed on an absolute scale. No corrections were made for absorption.

The structure was solved by direct methods, with the *MULTAN* program (Germain, Main & Woolfson, 1971). The E map produced showed clear peaks for all the carbon and oxygen atoms. Calculation of the structure factors based on these atomic positions gave a residual $(R)=0{\cdot}2985$. The atomic scattering factors used were those listed in *International Tables for X-ray Crystallography* (1962). Seven cycles of refinement, with a block-diagonal structure factors least-squares program (Cruickshank, Pilling, Bujosa, Lovell & Truter, 1961), reduced R to 0·1703 and a further five cycles of anisotropic refinement reduced it to 0·1279. A Fourier synthesis based on the phase angles of the final structure-factor calculation showed the positions of the 16 hydrogen atoms. These were introduced and nine cycles of structure-factor refinement were carried out with anisotropic temperature parameters for the non-hydrogen atoms and isotropic temperature parameters for the hydrogen atoms. The final value of R, with all planes included and the isotropic temperature parameters of the hydrogen atoms set to an average value of $U(\text{iso})=0{\cdot}024$, is 0·1022. The weighting scheme used was $w^{1/2}=1/\{1+[(|F_o|-p_2)/p_1]^2\}^{1/2}$ with $p_1=F_{MAX}/14{\cdot}3$ and $p_2=F_{MAX}/49{\cdot}7$.*

* A table of structure factors has been deposited with the National Lending Library, England, as Supplementary Publication, No. SUP 30184 (10 pp.). Copies may be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1 NZ, England.

Table 1. *Atomic coordinates and estimated standard deviations*

	x	y	z
C(1)	0·10313 (14)	0·36264 (65)	0·15478 (126)
C(2)	0·08167 (16)	0·20765 (66)	0·02129 (115)
C(3)	0·11980 (12)	0·08592 (55)	0·99076 (104)
C(4)	0·15572 (14)	0·21106 (59)	0·91527 (104)
C(5)	0·20051 (14)	0·14886 (56)	0·00322 (108)
C(6)	0·23524 (13)	0·27830 (60)	0·92415 (108)
C(7)	0·31103 (15)	0·31302 (68)	0·89712 (170)
C(8)	0·08652 (18)	−0·16621 (80)	0·81926 (168)
C(9)	0·00709 (18)	0·21483 (89)	0·12944 (207)
O(1)	0·08758 (12)	0·46999 (51)	0·31293 (117)
O(2)	0·04824 (10)	0·14197 (51)	0·19620 (103)
O(3)	0·11687 (10)	−0·03417 (44)	0·75781 (79)
O(4)	0·14541 (9)	0·36339 (43)	0·08446 (85)
O(5)	0·20156 (10)	0·11808 (44)	0·31563 (74)
O(6)	0·27573 (9)	0·20227 (41)	0·97443 (87)

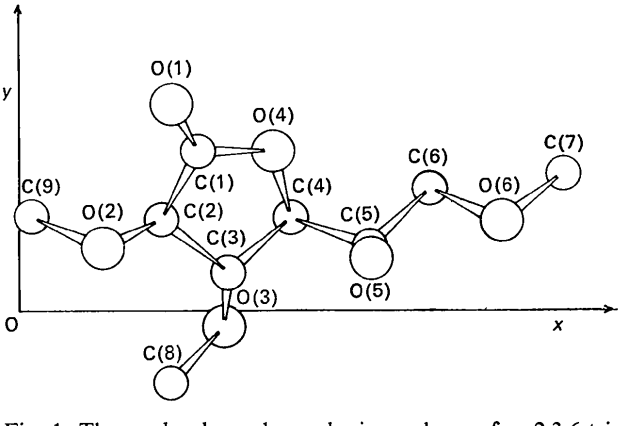

Fig. 1. The molecule and numbering scheme for 2,3,6-trimethylgalactono-γ-lactone.

Table 2. *Anisotropic temperature parameters* ($\times 10^4$) *and estimated standard deviations*

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	161 (18)	266 (21)	286 (24)	39 (48)	63 (37)	107 (35)
C(2)	230 (20)	269 (23)	182 (21)	−53 (43)	24 (37)	−21 (38)
C(3)	104 (16)	197 (19)	154 (19)	−46 (35)	−8 (32)	−29 (30)
C(4)	192 (18)	199 (19)	135 (19)	−95 (37)	54 (34)	26 (33)
C(5)	180 (18)	169 (19)	165 (20)	−40 (36)	51 (32)	−31 (31)
C(6)	142 (17)	220 (19)	165 (19)	39 (38)	−11 (32)	60 (32)
C(7)	138 (18)	238 (22)	597 (38)	−165 (57)	81 (51)	31 (35)
C(8)	263 (23)	361 (29)	493 (37)	−312 (62)	149 (54)	−362 (44)
C(9)	200 (22)	478 (33)	695 (48)	117 (81)	119 (59)	199 (48)
O(1)	209 (15)	281 (18)	535 (27)	−333 (44)	89 (38)	−3 (28)
O(2)	129 (13)	325 (18)	416 (23)	28 (42)	118 (31)	−11 (27)
O(3)	169 (13)	243 (16)	199 (16)	−169 (30)	63 (27)	−140 (24)
O(4)	130 (13)	205 (14)	261 (17)	−140 (31)	52 (26)	41 (23)
O(5)	204 (14)	215 (14)	131 (14)	−54 (28)	−1 (24)	97 (24)
O(6)	118 (13)	186 (14)	291 (18)	−71 (31)	−8 (27)	10 (23)

Discussion. A diagram of the molecule, showing numbering system, is given in Fig. 1. The atomic coordinates are in Table 1 and the thermal vibration parameters in Table 2. Table 3 contains the calculated bond lengths and Table 4 the calculated bond angles. The average C–C bond length is 1·523 Å with no bond showing a significant difference from this value and the average C–O bond length, excluding C(1)–O(4), is 1·426 Å. The bond angles in the furanose ring are similar to those measured for D-galactono-γ-lactone, and the C(1)–O(1) bond length of 1·2048 Å is similar to the figure of 1·198 Å quoted for the carbonyl bond (Jeffrey, Rosenstein & Vlasse, 1967). One difference, possibly due to the presence of the methyl group on O(6), is the bond length C(6)–O(6) with a value of 1·410 Å. This suggests that the length of 1·447 Å for the same bond in D-galactono-γ-lactone may be related to the hydrogen bonding of the atom O(6).

Table 3. *Bond lengths and estimated standard deviations*

C(1)–O(1)	1·2048 (52) Å
C(1)–C(2)	1·5140 (73)
C(1)–O(4)	1·3495 (52)
C(2)–O(2)	1·4000 (62)
C(2)–C(3)	1·5260 (65)
C(3)–O(3)	1·4128 (56)
C(3)–C(4)	1·5260 (61)
C(4)–O(4)	1·4554 (56)
C(4)–C(5)	1·5257 (63)
C(5)–O(5)	1·4234 (59)
C(5)–C(6)	1·5253 (62)
C(6)–O(6)	1·4100 (50)
C(7)–O(6)	1·4426 (57)
C(8)–O(3)	1·4298 (62)
C(9)–O(2)	1·4317 (68)

Table 4. *Bond angles and estimated standard deviations*

O(1)–C(1)–C(2)	128·51 (0·51)
O(1)–C(1)–O(4)	121·61 (0·45)
C(2)–C(1)–O(4)	109·79 (0·42)
C(1)–C(2)–O(2)	113·70 (0·43)
C(1)–C(2)–C(3)	101·57 (1·08)
O(2)–C(2)–C(3)	113·08 (1·33)
C(2)–C(3)–O(3)	115·86 (1·27)
C(2)–C(3)–C(4)	100·38 (0·91)
O(3)–C(3)–C(4)	108·36 (0·35)
C(3)–C(4)–O(4)	104·83 (0·76)
C(3)–C(4)–C(5)	113·62 (0·91)
O(4)–C(4)–C(5)	109·26 (1·61)
C(1)–O(4)–C(4)	109·40 (1·00)
C(4)–C(5)–O(5)	109·29 (1·16)
C(4)–C(5)–C(6)	111·65 (1·79)
O(5)–C(5)–C(6)	109·08 (1·16)
C(5)–C(6)–O(6)	107·97 (0·89)
C(6)–O(6)–C(7)	112·42 (0·36)
C(3)–O(3)–C(8)	112·74 (0·41)
C(2)–O(2)–C(9)	113·30 (0·48)

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