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

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

	x	y	z	U_{eq}
O(1)	0.2126 (2)	0.7640 (5)	0.4976 (3)	0.0463 (10)
C(2)	0.1187 (4)	0.7041 (8)	0.4837 (5)	0.0438 (14)
C(2a)	0.0502 (4)	0.7753 (8)	0.3746 (5)	0.058 (2)
C(2b)	0.1033 (4)	0.7704 (9)	0.5898 (5)	0.064 (2)
O(3)	0.1161 (3)	0.5008 (5)	0.4873 (3)	0.0455 (10)
C(4)	0.1507 (4)	0.4038 (8)	0.4176 (4)	0.0392 (14)
O(4)	0.1249 (3)	0.2441 (6)	0.3919 (4)	0.0549 (11)
C(5)	0.2209 (3)	0.4998 (7)	0.3841 (4)	0.0347 (13)
C(6)	0.2561 (4)	0.6858 (8)	0.4318 (5)	0.0419 (14)
O(6)	0.3221 (3)	0.7639 (5)	0.4215 (4)	0.0623 (13)
C(7)	0.2626 (4)	0.4143 (7)	0.3180 (4)	0.0402 (14)
S	0.23996 (11)	0.1997 (2)	0.25649 (14)	0.0507 (5)
C(1p)	0.3251 (4)	0.1915 (8)	0.1893 (5)	0.0427 (14)
C(2p)	0.3318 (4)	0.3314 (8)	0.1153 (5)	0.051 (2)
C(3p)	0.4001 (4)	0.3198 (9)	0.0676 (5)	0.056 (2)
C(4p)	0.4577 (4)	0.1661 (10)	0.0882 (5)	0.061 (2)
C(5p)	0.4493 (4)	0.0240 (10)	0.1579 (5)	0.063 (2)
C(6p)	0.3822 (4)	0.0357 (9)	0.2083 (5)	0.050 (2)

Table 2. *Selected geometric parameters* ($\text{Å}, °$)

O(1)—C(2)	1.447 (6)	C(4)—C(5)	1.452 (7)
O(1)—C(6)	1.347 (6)	C(5)—C(7)	1.351 (7)
C(2)—C(2a)	1.484 (8)	C(5)—C(6)	1.471 (7)
C(2)—C(2b)	1.502 (7)	C(6)—O(6)	1.198 (6)
C(2)—O(3)	1.446 (7)	C(7)—S	1.688 (5)
O(3)—C(4)	1.357 (6)	S—C(1p)	1.782 (5)
C(4)—O(4)	1.208 (6)		

C(6)—O(1)—C(2)	119.2 (4)	C(7)—C(5)—C(4)	121.4 (5)
O(1)—C(2)—O(3)	109.1 (4)	C(7)—C(5)—C(6)	117.9 (5)
O(1)—C(2)—C(2a)	110.9 (5)	C(4)—C(5)—C(6)	120.4 (5)
O(3)—C(2)—C(2a)	110.5 (5)	O(6)—C(6)—O(1)	119.4 (5)
O(1)—C(2)—C(2b)	105.1 (4)	O(6)—C(6)—C(5)	124.9 (5)
O(3)—C(2)—C(2b)	105.8 (5)	O(1)—C(6)—C(5)	115.5 (5)
C(2a)—C(2)—C(2b)	115.1 (5)	C(5)—C(7)—S	128.3 (4)
C(4)—O(3)—C(2)	117.8 (4)	C(7)—S—C(1p)	100.3 (3)
O(4)—C(4)—O(3)	118.9 (5)	C(2p)—C(1p)—S	122.0 (4)
O(4)—C(4)—C(5)	124.8 (5)	C(6p)—C(1p)—S	117.9 (5)
O(3)—C(4)—C(5)	116.3 (5)		

C(4)—C(5)—C(7)—S	4.2 (8)	C(7)—S—C(1p)—C(2p)	−54.0 (5)
C(6)—C(5)—C(7)—S	177.6 (4)	C(7)—S—C(1p)—C(6p)	129.2 (5)
C(5)—C(7)—S—C(1p)	−178.6 (5)		

Data collection: *DIF*4 (Stoe & Cie, 1988a). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1988b). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1994). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1992).

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Structural Investigation of Some Lactides

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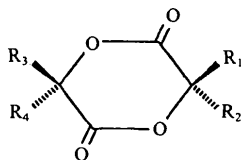
Abstract

We determined the crystal structures of four lactides: (1) (3S,6S)-3-benzyl-6-methyl-1,4-dioxane-2,5-dione ($C_{12}H_{12}O_4$), (2) (3RS,6SR)-6-(1,1-dimethyl-2-meth-oxyethyl)-3-[(SR)-α-hydroxybenzyl]-3-methyl-1,4-di-oxane-2,5-dione ($C_{17}H_{22}O_6$), (3) (3RS,6SR)-6-(1,1-dimethyl-2-methoxyethyl)-3-(1-hydroxy-1-methyl-ethyl)-3-methyl-1,4-dioxane-2,5-dione ($C_{13}H_{22}O_6$) and (4) (3S,6R)-6-(1,1-dimethyl-2-methoxyethyl)-3-methyl-3-[(S)-3-oxocyclopentyl]-1,4-dioxane-2,5-dione ($C_{15}H_{22}O_6$). The structure of the lactide heterocycle in the title compounds is compared with the only four structures of this type previously reported. The geometrical parameters of this hetero-cycle agree with those found previously for carboxylic esters.

Comment

The structures of carboxylic ester groups have been investigated thoroughly (Schweizer & Dunitz, 1982). Lactides contain this fragment twice within a six-membered heterocycle. We have determined the crystal structures of four lactides and compared their structural parameters with those of carboxylic esters. We also studied the influence of substituents on the conformation of the dilactone ring. Compounds (2) and (3) crystallized as racemic mixtures, while (1)

and (4) were available as pure enantiomers for which the absolute configuration at C(6) was known from the chemical synthesis.

	R_1	R_2	R_3	R_4
(1)	H	CH₂—C₆H₅	H	CH₃
(2)	CH₃	CH(OH)—C₆H₅	C(CH₃)₂—CH₂—O—CH₃	H
(3)	CH₃	C(CH₃)₂—OH	C(CH₃)₂—CH₂—O—CH₃	H
(4)	CH₃		C(CH₃)₂—CH₂—O—CH₃	H

The lactide heterocycle of (1) adopts a boat conformation with O(1), C(2), O(4) and C(5) in the plane ($\sigma = 0.04$ Å) and C(3) and C(6) above it by 0.47 and 0.43 Å, respectively. The H atoms at C(3) and C(6) are in axial positions and thus parallel to each other. The methyl and benzyl groups adopt equatorial positions, minimizing repulsive interactions.

Fig. 2. Perspective view of (2) with the atom numbering.

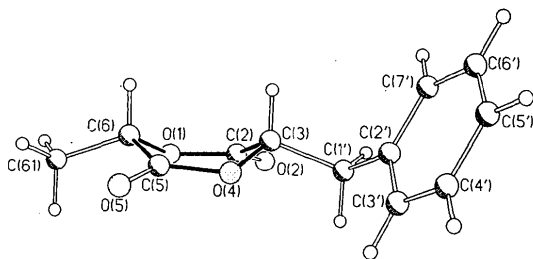Fig. 3. Perspective view of (3) with the atom numbering.

Fig. 1. Perspective view of (1) with the atom numbering.

The heterocycles in (2) and (3) exhibit twisted boat conformations (Figs. 2 and 3). In contrast to (1), the C atoms form the base ($\sigma = 0.01$, 0.08 Å, respectively) and O(1) and O(4) deviate from it by 0.12 and 0.35 Å, respectively, in (2) and by 0.19 and 0.37 Å, respectively, in (3). In both structures, the intermolecular packing is stabilized by a hydrogen bond from the hydroxyl group (which is situated below the heterocycle) to O(2) [in (2) O(1')···O(2) = 2.973 (5) Å, O(1')—H···O(2) = 161 (2)° and in (3) O(32)···O(2) = 2.767 (4) Å, O(32)—H···O(2) = 161 (1)°].

Compound (4) crystallizes with two molecules in the asymmetric unit. The heterocycle in molecule 1 can be described as a flat boat with the C atoms forming the base ($\sigma = 0.02$ Å) and the O atoms 0.23 [O(1)] and 0.07 Å [O(4)] above that plane. The dilactone ring of molecule 2 is planar ($\sigma = 0.02$ Å). The two molecules differ mainly in the dihedral angle C(6)—C(61)—C(64)—O(65) [56.9 (2) and −59.1 (2)°
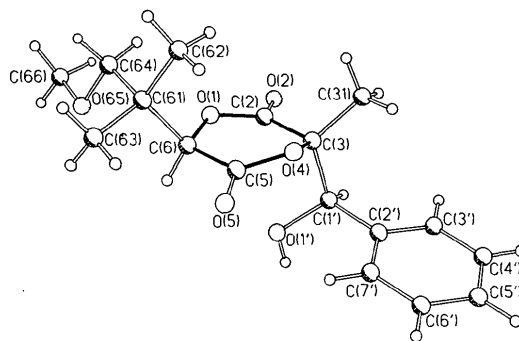

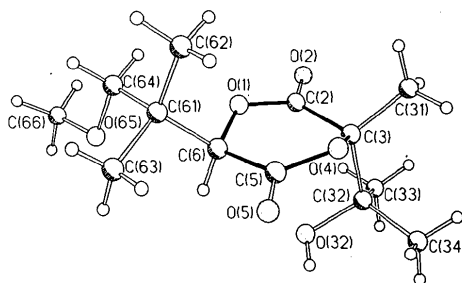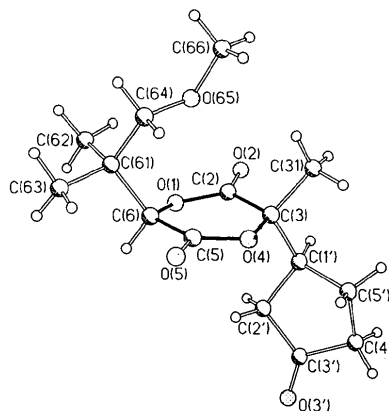Fig. 4. Perspective view of molecule 1 of (4) with the atom numbering.

for molecules 1 and 2, respectively]; this means that the terminal methoxy group is rotated by approximately 120°. The repulsion between O(65) and O(1) in molecule 1 induces non-planarity in the heterocycle (Fig. 4).

The geometrical parameters of the lactide heterocycle were calculated (Table 3) for the four title structures and the four found in the Cambridge Structural Database (1992) [BICVIS (van Hummel, Harkema, Kohn & Feijen, 1982), JACBRL (Mathieson & Taylor, 1963), KEMNIZ (Lynch, Pojman, Whitesell & Davis, 1990) and SCELER (Coetzer & Wiechers, 1973)] using the convention of Schweizer & Dunitz (1982). The bond lengths show

small variations and agree well with the published average values. The standard deviations drop significantly when the two bridged lactides (JACBRL, SCELER) are not included. The bond angles tend to lie closer to 120° than was found by Schweizer & Dunitz [especially when omitting JACBRL ($bc =$ 110, 112, $cd = 128$, 131°) and SCELER ($bc = 107$, 114, $cd = 127$, 132°) which are distorted by the bridge]. The observation that $bd < cd$ is true for all eight structures.

While the bond lengths and angles do not vary much, the dihedral angles within the dilactone ring depend markedly on the relative position (cis or trans) of the substituents. The root-mean-square deviations of the six ring atoms from the respective best plane (σ) show a clear gap between the two classes: two substituents, even if small, on the same side of the ring induce a pronounced boat conformation ($\sigma \geq 0.20$ Å) in which they can occupy equatorial positions. On the other hand, lactides with the largest substituents on opposite sides of the ring are much less puckered, or even planar ($\sigma \leq 0.13$ Å).

Experimental
Compound (1)
Crystal data

$C_{12}H_{12}O_4$
$M_r = 220.2$
Orthorhombic
$P2_12_12_1$
$a = 5.995$ (1) Å
$b = 9.080$ (1) Å
$c = 20.379$ (2) Å
$V = 1109.3$ (4) Å³
$Z = 4$
$D_x = 1.319$ Mg m⁻³

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 55 reflections
$\theta = 10\text{--}12.5°$
$\mu = 0.09$ mm⁻¹
$T = 293$ K
Transparent plate
$0.6 \times 0.4 \times 0.1$ mm
Colourless
Crystal source: ether/acetone

Data collection

Stoe Siemens four-circle diffractometer
$\omega/2\theta$ profile-fitting (Clegg, 1981) scans
Absorption correction: none
1236 measured reflections
1157 independent reflections
892 observed reflections
[$|F| > 3\sigma(F)$]

$R_{int} = 0.018$
$\theta_{max} = 25°$
$h = -7 \rightarrow 1$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 24$
3 standard reflections monitored every 100 reflections
intensity variation: none

Refinement

Refinement on F
$R = 0.049$
$wR = 0.045$
$S = 0.99$
892 reflections
148 parameters
$w = 1/[\sigma^2(F) + 0.0005F^2]$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.16$ e Å⁻³
$\Delta\rho_{min} = -0.22$ e Å⁻³
Atomic scattering factors from International Tables for X-ray Crystallography (1974, Vol. IV)

Compound (2)
Crystal data

$C_{17}H_{22}O_6$
$M_r = 322.4$
Orthorhombic
$Pca2_1$
$a = 11.405$ (1) Å
$b = 8.354$ (1) Å
$c = 17.142$ (1) Å
$V = 1633.2$ (3) Å³
$Z = 4$
$D_x = 1.311$ Mg m⁻³

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 60 reflections
$\theta = 10\text{--}12.5°$
$\mu = 0.09$ mm⁻¹
$T = 293$ K
Transparent block
$0.8 \times 0.8 \times 0.8$ mm
Colourless
Crystal source: ether/cyclohexane

Data collection

Stoe Siemens four-circle diffractometer
$\omega/2\theta$ profile-fitting scans
Absorption correction: none
1500 measured reflections
1490 independent reflections
1361 observed reflections
[$|F| > 3\sigma(F)$]

$R_{int} = 0.008$
$\theta_{max} = 25°$
$h = 0 \rightarrow 13$
$k = -9 \rightarrow 13$
$l = 0 \rightarrow 20$
3 standard reflections monitored every 100 reflections
intensity variation: none

Refinement

Refinement on F
$R = 0.034$
$wR = 0.041$
$S = 1.14$
1361 reflections
224 parameters
$w = 1/[\sigma^2(F) + 0.0005F^2]$

$(\Delta/\sigma)_{max} = 0.02$
$\Delta\rho_{max} = 0.14$ e Å⁻³
$\Delta\rho_{min} = -0.12$ e Å⁻³
Atomic scattering factors from International Tables for X-ray Crystallography (1974, Vol. IV)

Compound (3)
Crystal data

$C_{13}H_{22}O_6$
$M_r = 274.3$
Monoclinic
$P2_1/c$
$a = 10.899$ (1) Å
$b = 11.056$ (1) Å
$c = 12.174$ (2) Å
$\beta = 99.74$ (2)°
$V = 1445.8$ (3) Å³
$Z = 4$
$D_x = 1.260$ Mg m⁻³

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 58 reflections
$\theta = 10\text{--}12.5°$
$\mu = 0.09$ mm⁻¹
$T = 293$ K
Transparent needle
$1.0 \times 0.3 \times 0.3$ mm
Colourless
Crystal source: ether/cyclohexane

Data collection

Stoe Siemens four-circle diffractometer
$\omega/2\theta$ profile-fitting scans
Absorption correction: none
3878 measured reflections
2541 independent reflections
2168 observed reflections
[$|F| > 3\sigma(F)$]

$R_{int} = 0.022$
$\theta_{max} = 30°$
$h = -12 \rightarrow 12$
$k = 0 \rightarrow 13$
$l = -14 \rightarrow 6$
3 standard reflections monitored every 100 reflections
intensity variation: none

Refinement

Refinement on F
$R = 0.046$
$wR = 0.052$
$S = 1.34$
2168 reflections
193 parameters
$w = 1/[\sigma^2(F) + 0.0004F^2]$

$(\Delta/\sigma)_{max} = 0.03$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}
Atomic scattering factors
  from *International Tables*
  for X-ray Crystallography
  (1974, Vol. IV)

Compound (4)

Crystal data

$C_{15}H_{22}O_6$
$M_r = 298.3$
Monoclinic
$P2_1$
$a = 7.503$ (1) Å
$b = 19.330$ (2) Å
$c = 10.957$ (2) Å
$\beta = 102.91$ (2)°
$V = 1549.0$ (5) Å^3
$Z = 4$
$D_x = 1.279$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 40
  reflections
$\theta = 10–12.5°$
$\mu = 0.09$ mm^{-1}
$T = 293$ K
Transparent block
$0.9 \times 0.5 \times 0.4$ mm
Colourless
Crystal source: ether/petrol
  ether

Data collection

Stoe Siemens four-circle
  diffractometer
$\omega/2\theta$ profile-fitting scans
Absorption correction:
  none
5511 measured reflections
4997 independent reflections
4440 observed reflections
  $[|F| > 4\sigma(F)]$

$R_{int} = 0.016$
$\theta_{max} = 25°$
$h = -8 \rightarrow 8$
$k = -22 \rightarrow 22$
$l = 0 \rightarrow 13$
3 standard reflections
  monitored every 100
  reflections
  intensity variation: none

Refinement

Refinement on F
$R = 0.041$
$wR = 0.048$
$S = 1.27$
4440 reflections
404 parameters
$w = 1/[\sigma^2(F) + 0.0005F^2]$

$(\Delta/\sigma)_{max} = 0.03$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}
Atomic scattering factors
  from *International Tables*
  for X-ray Crystallography
  (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)
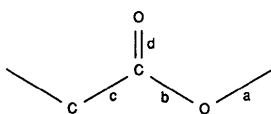
$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
(1)				
O(1)	0.0713 (4)	0.5764 (3)	0.5739 (1)	0.047 (1)
C(2)	0.2099 (6)	0.6140 (4)	0.5249 (2)	0.044 (1)
O(2)	0.3274 (4)	0.7194 (3)	0.5315 (1)	0.070 (1)
C(3)	0.2095 (5)	0.5149 (4)	0.4652 (2)	0.040 (1)
O(4)	−0.0122 (4)	0.4576 (2)	0.4519 (1)	0.040 (1)
C(5)	−0.1339 (5)	0.4092 (3)	0.5018 (2)	0.039 (1)
O(5)	−0.3058 (4)	0.3453 (3)	0.4910 (1)	0.053 (1)
C(6)	−0.0496 (6)	0.4388 (4)	0.5699 (2)	0.041 (1)
C(61)	−0.2356 (8)	0.4452 (5)	0.6196 (2)	0.062 (2)
C(1′)	0.2868 (7)	0.5954 (4)	0.4043 (2)	0.055 (1)
C(2′)	0.3088 (6)	0.4978 (4)	0.3448 (2)	0.044 (1)
C(3′)	0.1403 (7)	0.4840 (5)	0.2993 (2)	0.060 (2)
C(4′)	0.1633 (8)	0.3937 (6)	0.2451 (2)	0.077 (2)
C(5′)	0.3546 (9)	0.3161 (5)	0.2355 (2)	0.070 (2)
C(6′)	0.5232 (8)	0.3286 (5)	0.2797 (2)	0.067 (2)
C(7′)	0.5022 (7)	0.4188 (4)	0.3342 (2)	0.054 (1)
(2)				
O(1)	0.4493 (2)	0.6874 (2)	0.4184 (1)	0.047 (1)
C(2)	0.4290 (2)	0.7866 (3)	0.3593 (2)	0.038 (1)
O(2)	0.3553 (2)	0.8893 (2)	0.3673 (1)	0.053 (1)
C(3)	0.4946 (2)	0.7680 (3)	0.2833 (2)	0.035 (1)
O(4)	0.5603 (1)	0.6191 (2)	0.2802 (1)	0.040 (1)
C(5)	0.6092 (2)	0.5570 (3)	0.3442 (2)	0.040 (1)
O(5)	0.6949 (2)	0.4747 (2)	0.3375 (1)	0.061 (1)
C(6)	0.5471 (2)	0.5767 (3)	0.4209 (2)	0.038 (1)
C(31)	0.4083 (2)	0.7596 (3)	0.2159 (2)	0.050 (1)
C(61)	0.5001 (2)	0.4129 (3)	0.4514 (2)	0.042 (1)
C(62)	0.4388 (3)	0.3209 (4)	0.3862 (2)	0.064 (1)
C(63)	0.6019 (3)	0.3162 (4)	0.4854 (2)	0.064 (1)
C(64)	0.4103 (3)	0.4443 (3)	0.5154 (2)	0.047 (1)
O(65)	0.4594 (2)	0.5393 (2)	0.5752 (1)	0.057 (1)
C(66)	0.3746 (3)	0.5866 (4)	0.6300 (2)	0.059 (1)
C(1′)	0.5836 (2)	0.9065 (3)	0.2773 (2)	0.040 (1)
O(1′)	0.6510 (2)	0.8938 (2)	0.3465 (1)	0.054 (1)
C(2′)	0.6566 (2)	0.9019 (3)	0.2032 (1)	0.037 (1)
C(3′)	0.6244 (2)	0.9917 (3)	0.1391 (2)	0.048 (1)
C(4′)	0.6901 (3)	0.9906 (4)	0.0717 (2)	0.059 (1)
C(5′)	0.7893 (3)	0.8984 (3)	0.0670 (2)	0.059 (1)
C(6′)	0.8220 (3)	0.8064 (4)	0.1296 (2)	0.057 (1)
C(7′)	0.7577 (2)	0.8088 (3)	0.1978 (2)	0.046 (1)
(3)				
O(1)	0.2245 (1)	0.1988 (1)	0.1175 (1)	0.041 (1)
C(2)	0.1551 (2)	0.2976 (2)	0.1002 (1)	0.034 (1)
O(2)	0.0944 (1)	0.3145 (1)	0.0091 (1)	0.055 (1)
C(3)	0.1563 (2)	0.3896 (1)	0.1925 (1)	0.030 (1)
O(4)	0.2672 (1)	0.3739 (1)	0.2762 (1)	0.036 (1)
C(5)	0.3088 (2)	0.2632 (2)	0.3097 (1)	0.036 (1)
O(5)	0.3688 (2)	0.2525 (1)	0.4003 (1)	0.065 (1)
C(6)	0.2855 (2)	0.1597 (1)	0.2268 (1)	0.032 (1)
C(31)	0.1678 (2)	0.5162 (2)	0.1465 (2)	0.046 (1)
C(32)	0.0406 (2)	0.3720 (1)	0.2507 (1)	0.031 (1)
O(32)	0.0460 (1)	0.2481 (1)	0.2859 (1)	0.039 (1)
C(33)	−0.0811 (2)	0.3888 (2)	0.1705 (2)	0.046 (1)
C(34)	0.0457 (2)	0.4561 (2)	0.3504 (2)	0.046 (1)
C(61)	0.4065 (2)	0.0935 (1)	0.2113 (1)	0.034 (1)
C(62)	0.5031 (2)	0.1826 (2)	0.1816 (2)	0.051 (1)
C(63)	0.4578 (2)	0.0233 (2)	0.3182 (2)	0.049 (1)
C(64)	0.3776 (2)	0.0040 (2)	0.1147 (1)	0.040 (1)
O(65)	0.2865 (1)	−0.0796 (1)	0.1362 (1)	0.052 (1)
C(66)	0.2640 (2)	−0.1712 (2)	0.0549 (2)	0.060 (1)
(4)				
Molecule 1				
O(1)	0.4324 (2)	0.4448 (1)	0.7207 (1)	0.046 (1)
C(2)	0.4941 (3)	0.4696 (1)	0.6244 (2)	0.046 (1)
O(2)	0.4934 (3)	0.5309 (1)	0.6073 (2)	0.073 (1)
C(3)	0.5456 (3)	0.4205 (1)	0.5306 (2)	0.046 (1)
O(4)	0.5562 (2)	0.3485 (1)	0.5719 (1)	0.056 (1)
C(5)	0.5378 (3)	0.3269 (1)	0.6832 (2)	0.042 (1)
O(5)	0.5657 (2)	0.2673 (1)	0.7100 (1)	0.061 (1)
C(6)	0.4769 (3)	0.3767 (1)	0.7720 (2)	0.040 (1)
C(31)	0.7311 (3)	0.4386 (2)	0.5058 (2)	0.070 (1)
C(61)	0.6089 (3)	0.3815 (1)	0.9034 (2)	0.043 (1)
C(62)	0.5631 (3)	0.4451 (1)	0.9716 (2)	0.059 (1)
C(63)	0.5835 (4)	0.3166 (1)	0.9794 (2)	0.068 (1)
C(64)	0.8076 (3)	0.3835 (1)	0.8929 (2)	0.050 (1)
O(65)	0.8356 (2)	0.4393 (1)	0.8168 (1)	0.052 (1)
C(66)	1.0235 (3)	0.4456 (2)	0.8131 (3)	0.073 (1)
C(1′)	0.3932 (3)	0.4249 (1)	0.4104 (2)	0.040 (1)
C(2′)	0.2023 (3)	0.4059 (1)	0.4245 (2)	0.054 (1)
C(3′)	0.1021 (4)	0.3829 (2)	0.2954 (2)	0.067 (1)
O(3′)	−0.0564 (3)	0.3691 (2)	0.2637 (2)	0.111 (1)
C(4′)	0.2374 (4)	0.3795 (2)	0.2132 (2)	0.068 (1)
C(5′)	0.4238 (3)	0.3800 (1)	0.3020 (2)	0.057 (1)
Molecule 2				
O(1a)	0.0041 (2)	0.2274 (1)	0.7460 (1)	0.050 (1)
C(2a)	0.0431 (3)	0.2353 (1)	0.6358 (2)	0.046 (1)
O(2a)	0.0821 (3)	0.2922 (1)	0.6079 (2)	0.079 (1)

C(3a)	0.0373 (3)	0.1747 (1)	0.5471 (2)	0.046 (1)
O(4a)	−0.0157 (2)	0.1099 (1)	0.5961 (1)	0.051 (1)
C(5a)	−0.0614 (3)	0.1028 (1)	0.7067 (2)	0.041 (1)
O(5a)	−0.1248 (2)	0.0491 (1)	0.7307 (1)	0.064 (1)
C(6a)	−0.0419 (3)	0.1627 (1)	0.7964 (2)	0.038 (1)
C(31a)	0.2282 (3)	0.1641 (2)	0.5257 (3)	0.071 (1)
C(61a)	0.0906 (3)	0.1519 (1)	0.9246 (2)	0.044 (1)
C(62a)	0.0780 (4)	0.2150 (1)	1.0061 (2)	0.070 (1)
C(63a)	0.0412 (4)	0.0875 (1)	0.9901 (2)	0.061 (1)
C(64a)	0.2859 (3)	0.1469 (1)	0.9087 (2)	0.053 (1)
O(65a)	0.3033 (2)	0.0916 (1)	0.8292 (1)	0.054 (1)
C(66a)	0.4859 (3)	0.0820 (2)	0.8188 (3)	0.077 (1)
C(1'a)	−0.1041 (3)	0.1890 (1)	0.4251 (2)	0.048 (1)
C(2'a)	−0.3014 (3)	0.1941 (1)	0.4377 (2)	0.054 (1)
C(3'a)	−0.4108 (3)	0.1820 (1)	0.3064 (2)	0.060 (1)
O(3'a)	−0.5691 (3)	0.1972 (1)	0.2680 (2)	0.098 (1)
C(4'a)	−0.2914 (4)	0.1474 (2)	0.2319 (2)	0.073 (1)
C(5'a)	−0.1118 (4)	0.1331 (1)	0.3243 (2)	0.066 (1)

Table 2. *Selected bond lengths (Å), bond angles (°) and dihedral angles (°)*

	(1)	(2)	(3)	(4) Molecule 1	(4) Molecule 2
O(1)—C(2)	1.343 (4)	1.329 (3)	1.325 (2)	1.333 (3)	1.313 (3)
C(2)—C(3)	1.515 (5)	1.510 (4)	1.514 (2)	1.512 (3)	1.515 (3)
C(3)—O(4)	1.453 (4)	1.453 (3)	1.453 (2)	1.459 (3)	1.453 (3)
O(4)—C(5)	1.326 (4)	1.337 (3)	1.344 (2)	1.326 (2)	1.337 (3)
C(5)—C(6)	1.503 (5)	1.502 (4)	1.518 (2)	1.510 (3)	1.504 (3)
C(6)—O(1)	1.447 (4)	1.450 (3)	1.449 (2)	1.440 (2)	1.441 (2)
C(2)—O(2)	1.196 (4)	1.209 (3)	1.206 (2)	1.199 (3)	1.196 (3)
C(5)—O(5)	1.203 (4)	1.200 (3)	1.189 (2)	1.195 (3)	1.195 (3)
O(1)—C(2)—C(3)	116.4 (3)	120.5 (2)	120.2 (1)	119.9 (2)	121.4 (2)
C(2)—C(3)—O(4)	111.4 (3)	112.0 (2)	110.0 (1)	113.1 (2)	113.6 (2)
C(3)—O(4)—C(5)	118.6 (3)	121.1 (2)	121.3 (1)	125.2 (2)	124.8 (2)
O(4)—C(5)—C(6)	117.7 (3)	118.6 (2)	118.3 (1)	120.0 (2)	120.4 (2)
C(5)—C(6)—O(1)	112.0 (3)	114.0 (2)	112.6 (1)	114.1 (2)	114.5 (2)
C(6)—O(1)—C(2)	119.2 (3)	123.7 (2)	123.5 (1)	123.2 (2)	125.0 (2)
O(1)—C(2)—C(3)—O(4)	35.3 (4)	10.5 (3)	19.3 (2)	−11.9 (3)	0.5 (3)
C(2)—C(3)—O(4)—C(5)	−43.5 (4)	−34.2 (3)	−41.3 (2)	−5.1 (3)	2.0 (3)
C(3)—O(4)—C(5)—C(6)	9.6 (4)	34.7 (3)	30.6 (2)	8.6 (3)	−6.4 (3)
O(4)—C(5)—C(6)—O(1)	32.4 (4)	−10.5 (3)	2.9 (2)	3.8 (2)	7.9 (3)
C(5)—C(6)—O(1)—C(2)	−40.4 (4)	−12.6 (3)	−24.2 (2)	−21.3 (2)	−5.8 (3)
C(6)—O(1)—C(2)—C(3)	6.3 (4)	11.7 (3)	12.3 (2)	25.8 (3)	1.9 (3)

Table 3. *Geometrical parameters (Å, °) of the lactide heterocycle*

	Schweizer & Dunitz, 1982	All lactides	Without bridged lactides
a	1.447 (13)	1.449 (18)	1.450 (8)
b	1.340 (14)	1.344 (22)	1.335 (10)
c	1.495 (19)	1.510 (8)	1.508 (8)
d	1.195 (7)	1.193 (13)	1.197 (7)
ab	117 (2)	120 (4)	121 (3)
bd	123 (1)	119 (1)	119 (1)
bc	111 (1)	117 (4)	118 (2)
cd	125 (1)	124 (4)	123 (2)
ac		112 (3)	112 (2)

The data were corrected for Lorentz and polarization effects. The structures were solved by direct methods and refined with *SHELXTL* (Sheldrick, 1983). All H atoms were located by difference syntheses and refined with fixed individual displacement parameters using a riding model [except for the hydroxyl H atoms in (2) and (3), which were refined without constraints]. C and O atoms were refined anisotropically by blocked-cascade least-squares methods.

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Heterocyclic Tautomerism. VI.
A Redetermination and Reassignment of the Structure of 2-Aminothiazol-4(5*H*)-one (Pseudothiohydantoin)

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Abstract

The structure of 2-aminothiazol-4(5*H*)-one, $C_3H_4N_2OS$, has been redetermined at 128 K. This compound is shown to exist as the amino tautomer rather than the previously reported imino tautomer [Amirthalingam & Muralidharan (1972). *Acta Cryst.* B28, 2421–2423; Ananthamurthy, Udupa & Murthy (1972). *Z. Kristallogr.* 137, 316–317].

Comment

The title compound can exist as five possible tautomers, (1)–(5). In the solid state its infrared spectrum shows