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Structures of *endo*- and *exo*-3-Acetoxy-2,4-diethoxy-6-(2-oxo-1,3-oxazolidin-3-ylcarbonyl)-3,4-dihydro-2*H*-pyran

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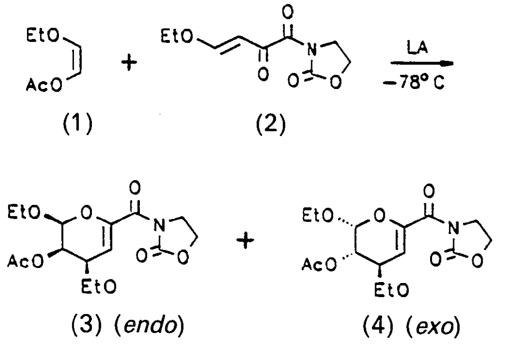
Abstract

The crystal structures of (±)-(2β,3β,4β)- and (±)-(2α,3α,4β)-2,4-diethoxy-6-(2-oxo-1,3-oxazolidin-3-ylcarbonyl)-3,4-dihydro-2*H*-pyran-3-yl acetate (the *endo* compound with half a molecule of diethyl ether in the asymmetric unit) determined at 153 and 293 K, respectively, are reported. The two structures have similar bond lengths and angles and similar conformations.

Comment

The *endo*/*exo* selectivity of the intermolecular Diels-Alder reaction of 4-ethoxy-1-(2-oxo-1,3-oxazolidin-3-yl)-3-butene-1,2-dione (2) with 2-ethoxyvinyl acetate (1) depends on the Lewis acid (LA) used. With $SnCl_4$, for example, a high *exo* selectivity is observed, whereas with Me_2AlCl, *endo* selectivity occurs. Details of the reaction have been published elsewhere (Tietze & Schneider, 1992; Schneider, 1992). In this paper we present the X-ray crystal structure of the *endo* product (3) and the *exo* product (4).



All bond lengths and angles are generally as expected. The conformations of the two structures are very similar. All H atoms were included in calculated positions and refined using a riding model. The ethoxy group in compound (3), O21 to C23, was found to be disordered. Two positions were refined with distance restraints for the 1-2 and 1-3 distances to an occupancy of

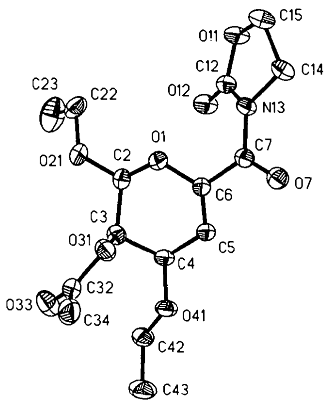


Fig. 1. Structure of compound (3) showing 50% probability displacement ellipsoids. The H atoms and the solvent molecule are omitted.

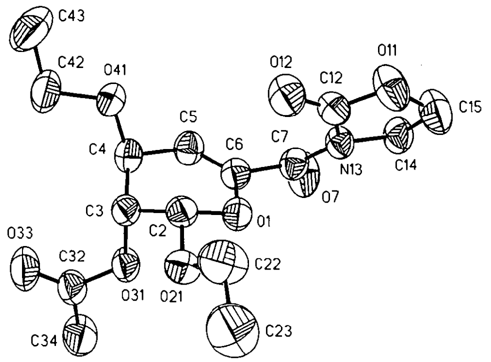


Fig. 2. Structure of compound (4) showing 50% probability displacement ellipsoids. The H atoms are omitted.

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0.85:0.15, respectively. This part of the structure was refined with rigid-bond restraints (Rollett, 1970; Hirshfeld, 1976; Trueblood & Dunitz, 1983) and similarity restraints for the anisotropic displacement parameters. Compound (3) crystallizes with half a solvent molecule in the asymmetric unit which is disordered by an inversion centre. This diethyl ether molecule was refined with the restraints described above.

Experimental

Compound (3)

Crystal data

$C_{15}H_{21}NO_8.\frac{1}{2}C_4H_{10}O$
$M_r = 380.39$
Monoclinic
$P2_1/n$
$a = 8.078$ (2) Å
$b = 19.274$ (5) Å
$c = 12.686$ (3) Å
$\beta = 102.85$ (2)°
$V = 1925.7$ (8) Å^3
$Z = 4$

$D_x = 1.312$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 54 reflections
$\theta = 10$- 12.5°
$\mu = 0.105$ mm^{-1}
$T = 153$ (2) K
Blocks
0.6 × 0.4 × 0.3 mm
Colourless

Data collection

Stoe Siemens AED four-circle diffractometer
Profile data from $2\theta/\omega$ scans
Absorption correction: none
4761 measured reflections
3373 independent reflections
2805 observed reflections $[I > 2\sigma(I)]$

$R_{\text{int}} = 0.0315$
$\theta_{\text{max}} = 25.00°$
$h = -9 \rightarrow 9$
$k = -12 \rightarrow 22$
$l = -15 \rightarrow 15$
3 standard reflections
frequency: 90 min
intensity variation: none

Refinement

Refinement on F^2
Final $R(F) = 0.0482$ for $F > 4\sigma(F)$
$wR(F^2) = 0.1326$ for all data
$S = 1.064$
3372 reflections
289 parameters
Calculated weights $w = 1/[\sigma^2(F_o^2)+(0.0627P)^2 +1.1712P]$ where $P = (F_o^2+2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = -0.001$
$\Delta\rho_{\text{max}} = 0.331$ e Å^{-3}
$\Delta\rho_{\text{min}} = -0.304$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (3)

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

	x	y	z	U_{eq}
O1	0.9406 (2)	0.34536 (7)	1.00695 (10)	0.0303 (3)
C2	0.8100 (3)	0.39748 (11)	0.9794 (2)	0.0318 (5)
O21	0.7012 (3)	0.38760 (14)	1.0492 (2)	0.0411 (7)
C22	0.7590 (4)	0.4216 (2)	1.1516 (2)	0.0445 (8)
C23	0.6783 (5)	0.4912 (2)	1.1469 (3)	0.0690 (11)
O21′	0.7093 (15)	0.3947 (8)	1.0523 (7)	0.051 (5)
C22′	0.7021 (26)	0.4560 (9)	1.1152 (12)	0.059 (4)
C23′	0.8053 (43)	0.4470 (12)	1.2259 (13)	0.090 (8)
C3	0.7162 (2)	0.39032 (10)	0.8630 (2)	0.0303 (4)
O31	0.6554 (2)	0.32019 (7)	0.84541 (11)	0.0344 (3)
C32	0.4992 (3)	0.31165 (13)	0.7804 (2)	0.0372 (5)
C34	0.4659 (3)	0.23742 (14)	0.7515 (2)	0.0487 (6)
O33	0.4028 (2)	0.35847 (10)	0.75038 (14)	0.0510 (4)
C4	0.8421 (2)	0.40407 (10)	0.79030 (15)	0.0281 (4)
O41	0.7759 (2)	0.38052 (7)	0.68314 (11)	0.0338 (4)
C42	0.6652 (3)	0.42971 (12)	0.6176 (2)	0.0370 (5)
C43	0.5890 (4)	0.3948 (2)	0.5123 (2)	0.0584 (7)
C5	1.0061 (2)	0.36710 (10)	0.83475 (15)	0.0269 (4)
C6	1.0446 (2)	0.34264 (10)	0.93525 (15)	0.0257 (4)
C7	1.2076 (2)	0.30632 (10)	0.98089 (15)	0.0267 (4)
O7	1.2660 (2)	0.26211 (8)	0.93145 (11)	0.0369 (4)
O11	1.3742 (2)	0.37832 (8)	1.24123 (11)	0.0392 (4)
C12	1.2770 (3)	0.38364 (10)	1.1410 (2)	0.0300 (4)
O12	1.2003 (2)	0.43516 (7)	1.10708 (12)	0.0385 (4)
N13	1.2868 (2)	0.32166 (8)	1.08671 (12)	0.0273 (4)
C14	1.4262 (3)	0.27884 (11)	1.1467 (2)	0.0332 (5)
C15	1.4403 (3)	0.30788 (11)	1.2594 (2)	0.0391 (5)
C90	1.1138 (23)	0.3871 (6)	0.5247 (7)	0.071 (2)
C91	1.0028 (8)	0.4382 (3)	0.4474 (4)	0.0662 (12)
O92	1.0000	0.5000	0.5000	0.0667 (7)
C93	0.8889 (7)	0.5511 (4)	0.4435 (5)	0.0701 (13)
C94	0.8976 (24)	0.6151 (5)	0.5128 (8)	0.085 (3)

Table 2. *Selected geometric parameters* (Å, °) *for* (3)

O1—C6	1.370 (2)	C7—N13	1.384 (2)
O1—C2	1.442 (2)	O11—C12	1.341 (2)
C2—C3	1.509 (3)	O11—C15	1.458 (3)
C3—C4	1.540 (3)	C12—O12	1.199 (2)
C4—C5	1.500 (3)	C12—N13	1.390 (3)
C5—C6	1.330 (3)	N13—C14	1.465 (2)
C6—C7	1.490 (3)	C14—C15	1.515 (3)
C7—O7	1.214 (2)		
C6—O1—C2	112.92 (14)	N13—C7—C6	117.0 (2)
O1—C2—C3	110.8 (2)	C12—O11—C15	109.7 (2)
C2—C3—C4	108.4 (2)	O12—C12—O11	123.4 (2)
C5—C4—C3	109.8 (2)	O12—C12—N13	128.0 (2)
C6—C5—C4	121.9 (2)	O11—C12—N13	108.6 (2)
C5—C6—O1	125.1 (2)	C7—N13—C12	126.8 (2)
C5—C6—C7	122.5 (2)	C7—N13—C14	121.3 (2)
O1—C6—C7	112.3 (2)	C12—N13—C14	110.4 (2)
O7—C7—N13	120.2 (2)	N13—C14—C15	99.8 (2)
O7—C7—C6	122.7 (2)	O11—C15—C14	104.3 (2)

Compound (4)

Crystal data

$C_{15}H_{21}NO_8$
$M_r = 343.33$
Triclinic
$P\overline{1}$
$a = 9.016$ (2) Å
$b = 9.038$ (2) Å
$c = 10.850$ (2) Å
$\alpha = 104.60$ (2)°
$\beta = 91.60$ (2)°
$\gamma = 91.48$ (2)°
$V = 854.8$ (3) Å^3
$Z = 2$

$D_x = 1.334$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 46 reflections
$\theta = 10$- 12.5°
$\mu = 0.109$ mm^{-1}
$T = 293$ (2) K
Plates
0.5 × 0.4 × 0.2 mm
Colourless

Data collection

Stoe Siemens AED-2 four-circle diffractometer
Profile data from $2\theta/\omega$ scans
Absorption correction: none

$R_{\text{int}} = 0.0213$
$\theta_{\text{max}} = 22.50°$
$h = -9 \rightarrow 9$
$k = -9 \rightarrow 9$
$l = -11 \rightarrow 11$

3293 measured reflections
2220 independent reflections
1931 observed reflections
  $[I > 2\sigma(I)]$
3 standard reflections
  frequency: 90 min
  intensity variation: none

Refinement

Refinement on F^2
Final $R(F) = 0.0401$ for
  $F > 4\sigma(F)$
$wR(F^2) = 0.1124$ for all data
$S = 1.058$
2217 reflections
220 parameters
Calculated weights
  $w = 1/[\sigma^2(F_o^2)+(0.0496P)^2 +0.3690P]$
  where $P = (F_o^2+2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.000$
$\Delta\rho_{\max} = 0.177$ e Å^{-3}
$\Delta\rho_{\min} = -0.211$ e Å^{-3}
Extinction correction: none
Atomic scattering factors
  from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (4)

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

	x	y	z	$U_{\rm eq}$
O1	0.2055 (2)	0.7505 (2)	0.68972 (14)	0.0501 (4)
C2	0.2617 (2)	0.8493 (2)	0.6136 (2)	0.0474 (5)
C3	0.4263 (2)	0.8814 (2)	0.6367 (2)	0.0483 (5)
C4	0.4617 (2)	0.9575 (3)	0.7771 (2)	0.0536 (6)
C5	0.3705 (2)	0.8831 (3)	0.8588 (2)	0.0542 (6)
C6	0.2538 (2)	0.7932 (2)	0.8140 (2)	0.0475 (5)
C7	0.1587 (3)	0.7241 (2)	0.8965 (2)	0.0513 (6)
O7	0.2061 (2)	0.6433 (2)	0.9611 (2)	0.0793 (6)
O11	−0.1992 (2)	0.8743 (2)	0.8846 (2)	0.0762 (5)
C12	−0.0545 (3)	0.8757 (3)	0.8605 (2)	0.0518 (6)
O12	0.0040 (2)	0.9716 (2)	0.8188 (2)	0.0698 (5)
N13	0.0086 (2)	0.7527 (2)	0.8953 (2)	0.0473 (5)
C14	−0.0947 (3)	0.6830 (3)	0.9665 (2)	0.0602 (6)
C15	−0.2389 (3)	0.7444 (3)	0.9325 (3)	0.0811 (8)
O21	0.2290 (2)	0.7756 (2)	0.48796 (14)	0.0536 (4)
C22	0.0808 (3)	0.7943 (4)	0.4467 (3)	0.0892 (9)
C23	0.0438 (4)	0.6970 (5)	0.3220 (3)	0.1018 (11)
O31	0.5021 (2)	0.7388 (2)	0.6029 (2)	0.0577 (4)
C32	0.6369 (3)	0.7428 (3)	0.5531 (2)	0.0607 (6)
O33	0.6898 (2)	0.8546 (2)	0.5300 (2)	0.0875 (7)
C34	0.7080 (3)	0.5925 (3)	0.5339 (3)	0.0880 (10)
O41	0.4281 (2)	1.1152 (2)	0.8063 (2)	0.0636 (5)
C42	0.5407 (3)	1.2102 (3)	0.7716 (3)	0.0836 (9)
C43	0.4884 (5)	1.3693 (4)	0.8016 (4)	0.133 (2)

Table 4. *Selected geometric parameters* (Å, °) *for* (4)

O1—C6	1.361 (3)	C7—N13	1.384 (3)
O1—C2	1.452 (2)	O11—C12	1.338 (3)
C2—C3	1.506 (3)	O11—C15	1.440 (3)
C3—C4	1.525 (3)	C12—O12	1.192 (3)
C4—C5	1.492 (3)	C12—N13	1.391 (3)
C5—C6	1.316 (3)	N13—C14	1.455 (3)
C6—C7	1.492 (3)	C14—C15	1.499 (4)
C7—O7	1.210 (3)		
C6—O1—C2	113.7 (2)	N13—C7—C6	117.2 (2)
O1—C2—C3	111.5 (2)	C12—O11—C15	110.6 (2)
C2—C3—C4	110.5 (2)	O12—C12—O11	122.9 (2)
C5—C4—C3	110.0 (2)	O12—C12—N13	128.8 (2)
C6—C5—C4	122.6 (2)	O11—C12—N13	108.2 (2)
C5—C6—O1	125.5 (2)	C7—N13—C12	125.5 (2)
C5—C6—C7	122.7 (2)	C7—N13—C14	121.4 (2)
O1—C6—C7	111.8 (2)	C12—N13—C14	110.8 (2)
O7—C7—N13	119.6 (2)	N13—C14—C15	101.1 (2)
O7—C7—C6	123.2 (2)	O11—C15—C14	105.5 (2)

Both compounds were crystallized from diethyl ether at room temperature. Data were collected with a learnt profile method (Clegg, 1981). Data collection: *DIF*4 (Stoe & Cie, 1988). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1988). Program(s) used to solve structure: *SHELXS*-90 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*-92 (Sheldrick, 1992). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXL*-92.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71290 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1040]

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Structure of Methyltris(2,4,6-trimethoxyphenyl)phosphonium Iodide Ethanol Solvate

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Abstract

The structure of methyltris(2,4,6-trimethoxyphenyl)phosphonium iodide, [CH_3TMPP]I, shows slightly-distorted tetrahedral geometry at phosphorus. There are no close anion–cation contacts.

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