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Structure of (2'R*,5S*)-(±)-5-(Tetrahydro-2'H-pyran-2'-yl)-2(5H)-furanone

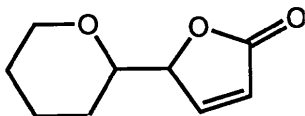
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Abstract. C₉H₁₂O₃, $M_r = 168.2$, triclinic, $P\bar{1}$, $a = 5.5385$ (6), $b = 8.543$ (1), $c = 10.373$ (1) Å, $\alpha = 113.329$ (9), $\beta = 100.78$ (1), $\gamma = 99.82$ (1)°, $V = 426.2$ (2) Å³, $D_x = 1.31$ g cm⁻³, $Z = 2$, Cu $K\alpha$ ($\lambda = 1.54178$ Å, graphite monochromator), $\mu = 8.2$ cm⁻¹, $F(000) = 180$, $T = 293$ K. Final $R = 0.036$, $wR = 0.081$ for 1559 reflections with $I > 3\sigma(I)$. The relative configurations at the two chiral centers are (S,R). The dihedral angle between the five- and six-membered rings (O1—C4—C5—O3) is 69.9 (1)°. The tetrahydropyran is in a chair conformation with the furanone ring equatorial.

Experimental. Colorless crystals from pentane, 0.3 × 0.4 × 0.5 mm block-like specimen; Enraf–Nonius CAD-4 diffractometer; cell parameters and crystal orientation from 25 automatically centered reflections in the range $8.9 < \theta < 48.8$ °; 2θ – θ scans over



$\Delta\theta$ range of $1.5(1.0 + 0.14\tan\theta)$ °; variable θ scan speed of 8.24 – 1.18 ° min⁻¹; each scan recorded in 96 steps with two outermost 16 step intensity blocks used for background determination; diffractometer controlled with Digital Equipment Corporation MicroVAX II computer and standard Enraf–Nonius programs (version 5.0); seven standard reflections monitored at 1 h intervals of X-ray exposure, -9.7

to -2.0 % intensity variation, -5.0 % average, correction applied; ψ -scan absorption correction with data from five reflections, transmission-factor range of 0.973 – 1.000 , average of 0.985 ; $4.8 < \theta < 70$ °, hkl 's selected to maintain diffractometer χ angle within 0 – 90 ° range; 1703 total data measured, 1624 unique data, 1559 data with $I > 3\sigma(I)$, $R_{\text{int}} = 0.0$ for nine twice-measured data. All crystallographic calculations performed with the *TEXSAN* (Molecular Structure Corporation, 1989) program system on DEC MicroVAX II or VAXStation II computers; structure solved with the *MITHRIL* (Gilmore, 1983) direct-methods program incorporated in *TEXSAN*. Full-matrix least-squares refinement, $\sum[w(F_o - F_c)^2]$ minimized with $w = 1/\sigma^2(F_o)$, reflections with $I < 3\sigma(I)$ excluded from refinement, correction for secondary isotropic extinction (Zachariasen, 1968) applied, $g = 0.88$ (13) × 10^{-4} ; C and O refined with anisotropic temperature factors; H atoms initially positioned from the C-atom framework and refined with individual isotropic temperature factors; 158 total variables; atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 155–175); minimum and maximum $\Delta\rho$ of -0.13 and 0.19 e Å⁻³; maximum Δ/σ in final least-squares cycle of 0.08 ; R , wR and S of 0.036 , 0.081 and 2.93 . Atomic coordinates are listed in Table 1;*

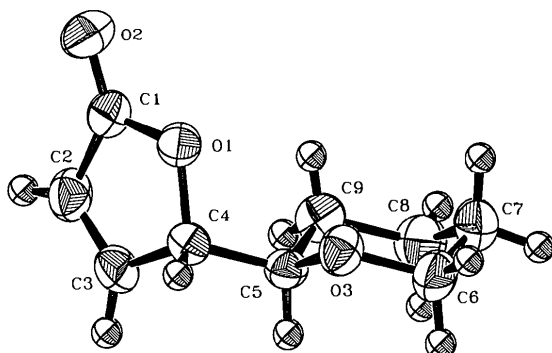
* Lists of structure factors, anisotropic temperature factors, and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54296 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional coordinates, equivalent isotropic temperature factors (\AA^2) and *e.s.d.*'s in parentheses
$$B_{\text{eq}} = (8\pi^2/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq}
O1	0.9996 (2)	0.4495 (1)	0.1477 (1)	3.59 (4)
O2	1.1431 (3)	0.7414 (1)	0.2238 (1)	5.21 (5)
O3	0.7787 (2)	0.1162 (1)	0.1430 (1)	3.72 (3)
C1	1.1877 (3)	0.6000 (2)	0.1902 (1)	3.69 (4)
C2	1.4297 (3)	0.5511 (2)	0.1845 (2)	4.22 (5)
C3	1.3843 (2)	0.3798 (2)	0.1418 (2)	3.92 (4)
C4	1.1072 (2)	0.3006 (2)	0.1133 (1)	3.22 (4)
C5	1.0450 (2)	0.2001 (1)	0.2010 (1)	3.09 (4)
C6	0.6918 (3)	0.0082 (2)	0.2104 (2)	4.41 (5)
C7	0.7462 (3)	0.1131 (2)	0.3726 (2)	5.02 (6)
C8	1.0281 (4)	0.2045 (2)	0.4404 (2)	4.93 (6)
C9	1.1196 (3)	0.3145 (2)	0.3651 (1)	3.99 (4)

Table 2. Bond lengths (\AA), angles ($^\circ$), torsion angles ($^\circ$) and *e.s.d.*'s in parentheses

O1—C1	1.362 (2)	C3—C4	1.490 (2)
O1—C4	1.442 (1)	C4—C5	1.522 (2)
O2—C1	1.201 (2)	C5—C9	1.518 (2)
O3—C5	1.420 (1)	C6—C7	1.499 (2)
O3—C6	1.434 (2)	C7—C8	1.510 (3)
C1—C2	1.475 (2)	C8—C9	1.522 (2)
C2—C3	1.312 (2)		
C1—O1—C4	109.4 (1)	C3—C4—C5	115.1 (1)
C5—O3—C6	112.4 (1)	O3—C5—C9	111.9 (1)
O2—C1—O1	121.5 (1)	O3—C5—C4	105.7 (1)
O2—C1—C2	130.6 (1)	C9—C5—C4	114.3 (1)
O1—C1—C2	107.9 (1)	O3—C6—C7	112.0 (1)
C3—C2—C1	108.6 (1)	C6—C7—C8	110.0 (1)
C2—C3—C4	109.9 (1)	C7—C8—C9	110.3 (1)
O1—C4—C3	104.2 (1)	C5—C9—C8	109.8 (1)
O1—C4—C5	111.0 (1)		
O1—C1—C2—C3	-0.5 (3)	C1—C2—C3—C4	0.9 (2)
O1—C4—C3—C2	-1.0 (2)	C2—C3—C4—C5	-122.8 (1)
O1—C4—C5—O3	69.9 (1)	C2—C1—O1—C4	0 (1)
O1—C4—C5—C9	-53.7 (1)	C3—C4—C5—C9	64.3 (1)
O2—C1—O1—C4	179.7 (4)	C4—C5—O3—C6	177.2 (1)
O2—C1—C2—C3	179.7 (3)	C4—C5—C9—C8	175.0 (1)
O3—C5—C9—C8	54.9 (2)	C5—O3—C6—C7	58.6 (2)
O3—C5—C4—C3	-172.1 (1)	C5—C9—C8—C7	-53.2 (2)
O3—C6—C7—C8	-56.3 (2)	C6—O3—C5—C9	-57.7 (1)
C1—O1—C4—C3	0.7 (2)	C6—C7—C8—C9	54.1 (2)
C1—O1—C4—C5	125.1 (1)		

Fig. 1. ORTEP drawing. The C and O atoms are shown as 50% ellipsoids, and the H atoms are depicted as spheres with $B = 1.5 \text{\AA}^2$. The configurations at the C4 and C5 chiral centers are *S* and *R*, respectively.

bond lengths, angles and torsion angles are given in Table 2. The *PLOTMD* program (Luo, Ammon & Gilliland, 1989) was used to display the *ORTEP* drawing (Fig. 1, Johnson, 1965) on a VAXStation II monitor, label the drawing, and prepare a print file for a Hewlett-Packard Laser-Jet II printer.

Related literature. The compound was prepared in three steps from 2-chlorotetrahydropyran, phenyl vinyl sulfone and potassium manganese pentacarbonyl (similar preparations have been described by DeShong, Sidler, Rybczynski, Slough & Rheingold, 1988). The determination of the relative stereochemistries of the two chiral centers was the principal objective of this analysis. A search of the January 1991 Cambridge Structural Database located only three other structures with a 5-substituted-2(5*H*)-furanone moiety {5-[2-hydroxy-4-(2,6,6-trimethylcyclohexenyl)butan-2-yl]-furan-2-one (Jefford, Jaggi, Bernardinelli & Boukouvalas, 1987); (5*R*,6*S*)-6-(*tert*-butylmethylsilyloxymethyl)-2-(2*R*)-2,5-dihydro-5-oxofuran-2-ylmethyl-2-penem-3-carboxylic acid allyl ester (Caparo, Francotte, Kohler, Rihs, Schneider, Scartazzini, Zak & Tosch, 1988); 3-[2,5-dihydro-5-oxo-2(*R*)-furan-yl]acetyl-4(*S*)-isopropyl-1,3-thiazolidine-2-thione (Nagao, Dai, Ochiai & Shiro, 1989)}.

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