Crystal structure of ethyl (–)-(1*S*,2*R*,5*S*,7*S*,8*R*,4′*S*,5′*S*)-2-methoxy-8-(2′,2′-dimethyl-5′-phenyl-1′,3′-dioxolane-4′-yl)-6-oxo-tricyclo[3.2.1.$0^{2,7}$]octane-1-carboxylate, $C_{23}H_{28}O_6$

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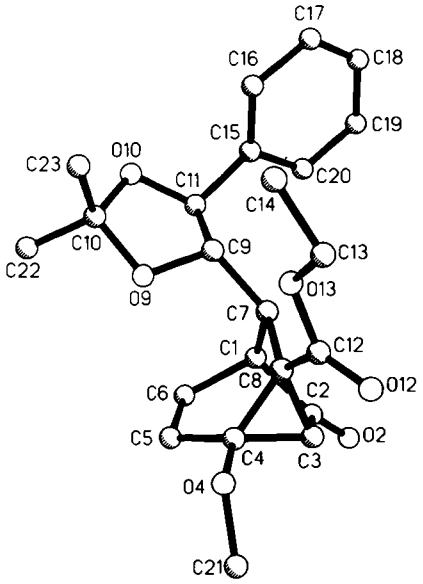

Source of material: The title compound was formed as a single isomer (d.e.>95%) from enantiomerically pure ethyl (*Z*)-(+)-2-bromo-3-[4*S*,5*S*)-2,2-dimethyl-5-phenyl-1,3-dioxolane-4-yl]-2-propenoate (**1**) and the kinetically controlled generated lithium 5-methoxy-1,5-cyclohexadiene-1-olate.
(**1**) was prepared from the prochiral ethyl 2-bromo-5-phenyl-2,4-pentadienoate (see ref. 1) by Sharpless-dihydroxylation (see ref. 2) and consecutive protection of the diol.

$C_{23}H_{28}O_6$, monoclinic, *C*121 (No. 5), a =19.873(1) Å, b =9.294(1) Å, c =14.068(1) Å, β =122.09°, V =2201.4 Å^3, Z =4, $R(F)$ =0.071, $R_w(F)$ =0.073.

Table 1. Parameters used for the X-ray data collection

Crystal:	colorless prism, size 0.8 x 0.9 x 0.6 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.90 cm^{-1}
Diffractometer:	Siemens P4
Scan mode:	ω
$T_{measurement}$:	293 K
$2\theta_{max}$:	55°
N(hkl)$_{unique}$:	5061
Criterion for F_o:	$F_o > 3\,\sigma(F_o)$
N($param$)$_{refined}$:	262
Program:	SHELXTL-plus

Table 2. Final atomic coordinates and displacement parameters (in Å^2)

Atom	Site	x	y	z	U_{iso}
H(1)	4*c*	0.3080(2)	0.46226	–0.3518(3)	0.08
H(3)	4*c*	0.4032(2)	0.3195(6)	–0.0412(3)	0.08
H(5A)	4*c*	0.1902(2)	0.3608(6)	–0.2079(3)	0.08
H(5B)	4*c*	0.1830(2)	0.5286(6)	–0.2197(3)	0.08
H(6A)	4*c*	0.1888(2)	0.5099(6)	–0.3688(3)	0.08
H(6B)	4*c*	0.2038(2)	0.3438(6)	–0.3506(3)	0.08
H(7)	4*c*	0.3912(2)	0.6261(6)	–0.2103(3)	0.08
H(9)	4*c*	0.3428(2)	0.8086(5)	–0.2093(2)	0.08
H(11)	4*c*	0.2622(2)	0.6896(6)	–0.4304(3)	0.08
H(13A)	4*c*	0.4416(3)	0.8433(6)	0.1455(3)	0.08
H(13B)	4*c*	0.5038(3)	0.8632(6)	0.1102(3)	0.08
H(14A)	4*c*	0.4518(3)	1.0850(6)	0.1107(5)	0.08
H(14B)	4*c*	0.3669(3)	1.0251(6)	0.0241(5)	0.08
H(14C)	4*c*	0.4290(3)	1.0450(6)	–0.0112(5)	0.08
H(16)	4*c*	0.3437(3)	1.0352(7)	–0.3567(3)	0.08
H(17)	4*c*	0.4605(3)	1.1038(8)	–0.3556(4)	0.08
H(18)	4*c*	0.5309(3)	0.939(1)	–0.3833(4)	0.08
H(19)	4*c*	0.4942(3)	0.7010(9)	–0.4146(4)	0.08
H(20)	4*c*	0.3778(3)	0.6255(7)	–0.4220(3)	0.08
H(21A)	4*c*	0.2801(3)	0.3365(7)	0.0961(4)	0.08
H(21B)	4*c*	0.3280(3)	0.2674(7)	0.0481(4)	0.08
H(21C)	4*c*	0.2353(3)	0.2786(7)	–0.0275(4)	0.08
H(22A)	4*c*	0.0725(2)	0.9081(8)	–0.4716(5)	0.08
H(22B)	4*c*	0.0820(2)	0.8183(8)	–0.3708(5)	0.08
H(22C)	4*c*	0.0980(2)	0.7461(8)	–0.4577(5)	0.08
H(23A)	4*c*	0.1795(3)	1.0091(6)	–0.2438(5)	0.08
H(23B)	4*c*	0.1767(3)	1.0921(6)	–0.3428(5)	0.08
H(23C)	4*c*	0.2577(3)	1.0331(6)	–0.2433(5)	0.08

Table 3. Final atomic coordinates and displacement parameters (in $Å^2$)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	4*c*	0.3081(2)	0.4616	–0.2835(3)	0.072(2)	0.049(2)	0.050(2)	–0.008(2)	0.037(2)	–0.010(2)
C(2)	4*c*	0.3575(3)	0.3456(6)	–0.1989(3)	0.071(2)	0.045(2)	0.062(2)	–0.000(2)	0.045(2)	–0.005(2)
O(2)	4*c*	0.3851(2)	0.2385(5)	–0.2130(3)	0.133(3)	0.055(2)	0.094(2)	0.014(2)	0.075(2)	–0.007(2)
C(3)	4*c*	0.3675(2)	0.3915(6)	–0.0917(3)	0.059(2)	0.039(2)	0.057(2)	0.007(2)	0.034(2)	0.005(2)
C(4)	4*c*	0.2935(2)	0.4627(6)	–0.1050(3)	0.048(2)	0.038(2)	0.044(2)	0.000(1)	0.027(1)	0.003(1)
O(4)	4*c*	0.2888(2)	0.4669(5)	–0.0090(2)	0.067(2)	0.054(1)	0.053(1)	–0.001(1)	0.041(1)	–0.002(1)
C(5)	4*c*	0.2151(2)	0.4471(6)	–0.2119(3)	0.049(2)	0.060(2)	0.053(2)	–0.017(2)	0.022(2)	–0.006(2)
C(6)	4*c*	0.2214(2)	0.4366(6)	–0.3163(3)	0.063(2)	0.052(2)	0.044(2)	–0.018(2)	0.017(2)	–0.008(2)
C(7)	4*c*	0.3437(2)	0.5989(6)	–0.2127(3)	0.040(2)	0.042(2)	0.041(2)	0.001(1)	0.024(1)	0.001(1)
C(8)	4*c*	0.3574(2)	0.5543(6)	–0.0993(3)	0.037(2)	0.036(2)	0.039(2)	0.003(1)	0.020(1)	0.004(1)
C(9)	4*c*	0.3033(2)	0.7428(5)	–0.2622(2)	0.036(1)	0.047(2)	0.032(1)	–0.002(1)	0.018(1)	0.000(1)
O(9)	4*c*	0.2290(1)	0.7570(5)	–0.2709(2)	0.039(1)	0.053(1)	0.051(1)	0.002(1)	0.025(1)	0.005(1)
C(10)	4*c*	0.1886(2)	0.8719(6)	–0.3482(3)	0.045(2)	0.061(2)	0.052(2)	0.009(2)	0.021(2)	0.010(2)
O(10)	4*c*	0.2218(2)	0.8814(6)	–0.4164(2)	0.052(2)	0.100(2)	0.054(2)	0.024(2)	0.025(1)	0.031(2)
C(11)	4*c*	0.2809(2)	0.7735(6)	–0.3835(3)	0.046(2)	0.055(2)	0.034(2)	0.001(2)	0.017(1)	0.004(1)
C(12)	4*c*	0.4103(2)	0.6344(6)	0.0061(3)	0.040(2)	0.052(2)	0.037(2)	0.002(1)	0.017(1)	0.005(2)
O(12)	4*c*	0.4570(2)	0.5848(5)	0.0935(2)	0.075(2)	0.059(2)	0.048(2)	0.003(2)	0.007(1)	0.011(1)
C(13)	4*c*	0.4486(3)	0.8704(6)	0.0854(3)	0.066(2)	0.057(2)	0.043(2)	–0.005(2)	0.013(2)	–0.013(2)
O(13)	4*c*	0.4018(2)	0.7778(5)	–0.0124(2)	0.056(1)	0.047(1)	0.036(1)	0.002(1)	0.015(1)	–0.004(1)
C(14)	4*c*	0.4222(3)	1.0196(6)	0.0493(5)	0.086(3)	0.060(3)	0.075(3)	–0.008(2)	0.028(3)	–0.025(2)
C(15)	4*c*	0.3497(2)	0.8253(6)	–0.3893(3)	0.052(2)	0.058(2)	0.030(1)	–0.005(2)	0.023(1)	0.002(1)
C(16)	4*c*	0.3737(3)	0.9651(7)	–0.3692(3)	0.073(3)	0.072(3)	0.039(2)	–0.009(2)	0.029(2)	0.002(2)
C(17)	4*c*	0.4433(3)	1.0053(8)	–0.3679(4)	0.082(3)	0.097(4)	0.054(2)	–0.038(3)	0.038(2)	–0.008(2)
C(18)	4*c*	0.4841(3)	0.909(1)	–0.3849(4)	0.066(3)	0.160(6)	0.056(3)	–0.027(4)	0.040(2)	0.000(3)
C(19)	4*c*	0.4628(3)	0.7685(9)	–0.4029(4)	0.079(3)	0.145(6)	0.077(3)	0.030(4)	0.058(3)	0.027(4)
C(20)	4*c*	0.3943(3)	0.7244(7)	–0.4076(3)	0.076(3)	0.081(3)	0.054(2)	0.007(2)	0.044(2)	0.011(2)
C(21)	4*c*	0.2827(3)	0.3251(7)	0.0303(4)	0.100(3)	0.070(3)	0.086(3)	0.003(3)	0.071(3)	0.020(2)
C(22)	4*c*	0.1025(2)	0.8335(8)	–0.4184(5)	0.044(2)	0.114(4)	0.095(4)	–0.001(3)	0.022(2)	0.017(3)
C(23)	4*c*	0.2017(3)	1.0151(6)	–0.2898(5)	0.099(4)	0.057(3)	0.086(4)	0.010(3)	0.050(3)	0.013(2)

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