Crystal structure of *trans*-3-[4-(2-oxo-2-phenylethyl)-1,3-dioxolan-2-yl]-1-phenyl-1-propanone, $C_{20}H_{20}O_4$

T. D. Avery[I], W. P. Jensen[I II], D. K. Taylor[I] and E. R. T. Tiekink*[,I]

[I] The Univeristy of Adelaide, Department of Chemistry, Australia 5005
[II] Permanent address: South Dakota State University, Department of Chemistry, Brookings, SD 57007, USA

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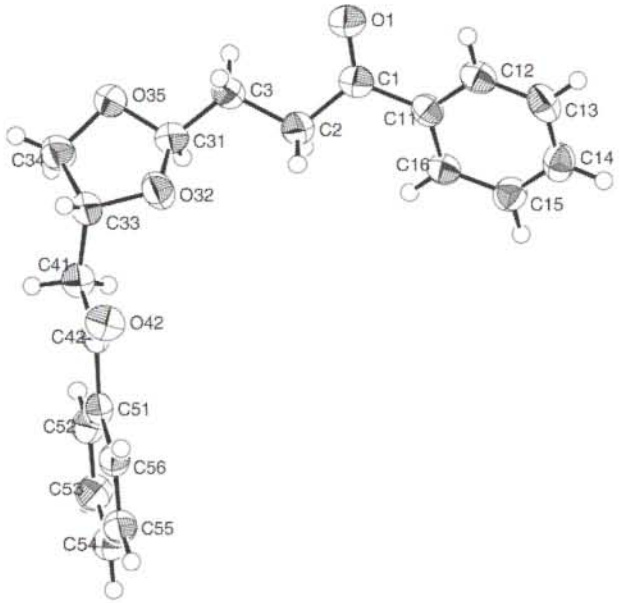

Abstract

$C_{20}H_{20}O_4$, monoclinic, $P12_1/n1$ (No. 14), a = 5.557(5) Å, b = 10.149(7) Å, c = 28.597(3) Å, β = 93.32(3)°, V = 1610.1 Å^3, Z = 4, $R_{gt}(F)$ = 0.044, $wR(F)$ = 0.053, T = 153 K.

Source of material

To a mixture of 4-oxo-4-phenylbutanal [1, 2] (1.0 g) in dichloromethane (5 ml) under a nitrogen gas atmosphere at ambient temperature benzyl 1,2-(triphenylphosphoranylidene) acetate (2.53 g) was added. After 1 day the mixture was concentrated and subjected to column chromatography. Elution with a mixture of hexane/ethyl acetate (7:3) afforded *E* and *Z* (14:1) benzyl 6-oxo-6-phenyl-2-hexenoate [2] in 91 % yield along with the title compound in 9 % yield as a mixture of *E* and *Z* isomers (2:1). Recrystallisation from heptane afforded the title compound as white crystals with mp 382 K – 384 K.

Discussion

The structure is the unsymmetrical, thermodynamically favoured, product from the self-condensation of 4-oxo-4-phenylbutanal [1, 2]. The side chains occuppy positions on opposite sides of the five-membered ring.

* Correspondence author
(E-mail: etiekink@chemistry.adelaide.edu.au)

Table 1. Data collection and handling.

Crystal:	colorless plate, size 0.03 × 0.19 × 0.37 mm
Wavelength:	Cu K_α radiation (1.5418 Å)
μ:	7.54 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC7R, ω/2θ
$2\theta_{max}$:	130.44°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3320, 2642
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 3\ \sigma(I_{obs})$, 1503
$N(param)_{refined}$:	218
Programs:	SIR97 [3], TEXSAN [4], DIFABS [5]

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

Atom	Site	x	y	z	U_{iso}
H(2)	4*e*	–0.2697	0.2484	0.2340	0.055
H(2)	4*e*	–0.0854	0.2195	0.1966	0.055
H(3a)	4*e*	–0.4989	0.0691	0.2052	0.055
H(3b)	4*e*	–0.3221	0.0544	0.1655	0.055
H(12)	4*e*	0.3029	–0.0342	0.2914	0.054
H(13)	4*e*	0.6238	0.0435	0.3396	0.061
H(14)	4*e*	0.6412	0.2675	0.3593	0.062
H(15)	4*e*	0.3386	0.4090	0.3321	0.058
H(16)	4*e*	0.0247	0.3333	0.2818	0.051
H(31)	4*e*	–0.5968	0.2778	0.1760	0.059
H(33)	4*e*	–0.5331	0.2626	0.0549	0.054
H(34a)	4*e*	–0.9210	0.3032	0.1089	0.065
H(34b)	4*e*	–0.8951	0.1943	0.0715	0.065
H(41a)	4*e*	–0.7356	0.4683	0.0572	0.051
H(41b)	4*e*	–0.6126	0.4878	0.1068	0.051
H(52)	4*e*	–0.6668	0.7021	0.0865	0.053
H(53)	4*e*	–0.6142	0.9294	0.0848	0.062
H(54)	4*e*	–0.2815	1.0211	0.0514	0.063
H(55)	4*e*	0.0050	0.8839	0.0199	0.059
H(56)	4*e*	–0.0469	0.6556	0.0207	0.052

Table 3. 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}
O(1)	4*e*	–0.1171(3)	–0.0278(2)	0.25035(7)	0.059(1)	0.040(1)	0.061(1)	–0.0022(9)	–0.002(1)	0.0012(9)
O(32)	4*e*	–0.4100(3)	0.2666(2)	0.12109(7)	0.041(1)	0.076(1)	0.058(1)	–0.0046(9)	–0.0021(9)	0.026(1)
O(35)	4*e*	–0.7398(3)	0.1473(2)	0.13329(6)	0.047(1)	0.049(1)	0.054(1)	–0.0052(8)	–0.0045(9)	0.0095(9)
O(42)	4*e*	–0.2290(3)	0.4403(2)	0.03865(6)	0.048(1)	0.052(1)	0.054(1)	0.0053(9)	0.0147(9)	0.0016(9)
C(1)	4*e*	–0.0663(4)	0.0889(3)	0.24945(9)	0.045(1)	0.041(2)	0.038(2)	0.001(1)	0.010(1)	–0.001(1)
C(2)	4*e*	-0.1983(4)	0.1812(3)	0.21626(9)	0.052(2)	0.043(2)	0.042(2)	–0.003(1)	–0.001(1)	0.001(1)
C(3)	4*e*	–0.3950(4)	0.1151(3)	0.18559(9)	0.054(2)	0.043(2)	0.041(2)	–0.002(1)	0.001(1)	0.005(1)
C(11)	4*e*	0.1329(4)	0.1417(2)	0.28130(8)	0.045(1)	0.043(1)	0.033(2)	0.003(1)	0.007(1)	0.003(1)
C(12)	4*e*	0.3116(5)	0.0568(2)	0.29916(9)	0.052(2)	0.041(2)	0.042(2)	0.005(1)	0.009(1)	0.003(1)
C(13)	4*e*	0.5013(5)	0.1022(3)	0.32796(9)	0.047(2)	0.065(2)	0.042(2)	0.007(1)	0.004(1)	0.014(1)
C(14)	4*e*	0.5108(5)	0.2349(3)	0.3397(1)	0.047(2)	0.069(2)	0.038(2)	–0.007(1)	0.004(1)	0.000(1)
C(15)	4*e*	0.3330(5)	0.3188(3)	0.32320(9)	0.052(2)	0.048(2)	0.044(2)	–0.005(1)	0.003(1)	–0.002(1)
C(16)	4*e*	0.1453(4)	0.2738(2)	0.29366(9)	0.047(1)	0.041(1)	0.041(2)	0.000(1)	0.006(1)	0.003(1)
C(31)	4*e*	–0.5407(5)	0.2100(3)	0.1565(1)	0.049(1)	0.044(2)	0.053(2)	–0.008(1)	–0.001(1)	0.008(1)
C(33)	4*e*	–0.5805(4)	0.3018(3)	0.08306(9)	0.043(1)	0.048(2)	0.044(2)	–0.002(1)	–0.006(1)	0.006(1)
C(34)	4*e*	–0.8160(5)	0.2387(3)	0.0973(1)	0.047(1)	0.059(2)	0.058(2)	–0.003(1)	0.000(1)	0.012(1)
C(41)	4*e*	–0.5966(4)	0.4490(2)	0.07692(9)	0.039(1)	0.049(2)	0.040(2)	0.001(1)	0.003(1)	0.000(1)
C(42)	4*e*	–0.3816(4)	0.5094(3)	0.05562(9)	0.038(1)	0.048(2)	0.031(1)	0.002(1)	0.001(1)	0.000(1)
C(51)	4*e*	–0.3595(4)	0.6562(2)	0.05402(8)	0.036(1)	0.043(1)	0.032(1)	0.001(1)	–0.002(1)	0.001(1)
C(52)	4*e*	–0.5298(4)	0.7386(3)	0.0729(1)	0.034(1)	0.052(2)	0.047(2)	0.001(1)	0.004(1)	–0.003(1)
C(53)	4*e*	–0.4983(5)	0.8735(3)	0.0718(1)	0.046(2)	0.047(2)	0.061(2)	0.005(1)	0.000(1)	–0.004(1)
C(54)	4*e*	–0.3011(5)	0.9282(3)	0.0521(1)	0.052(2)	0.046(2)	0.060(2)	–0.001(1)	–0.002(1)	0.003(1)
C(55)	4*e*	–0.1321(5)	0.8469(3)	0.0333(1)	0.045(1)	0.053(2)	0.048(2)	–0.007(1)	0.000(1)	0.008(1)
C(56)	4*e*	–0.1624(4)	0.7111(3)	0.03404(9)	0.039(1)	0.049(2)	0.040(2)	–0.001(1)	–0.001(1)	0.002(1)

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