Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*Z*)-3-(3-Phenylallylidene)-1,5-dioxaspiro[5.5]undecane-2,4-dione

Wu-Lan Zeng,[a] Hua-Xiang Zhang[a] and Fang-Fang Jian[b]*

[a]MicroScale Science Institute, Department of Chemistry and Chemical Engineering, Weifang University, Weifang 261061, People's Republic of China, and [b]MicroScale Science Institute, Weifang University, Weifang 261061, People's Republic of China Correspondence e-mail: wulanzeng@163.com

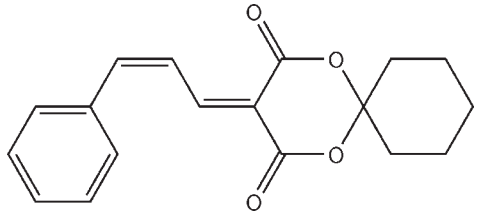
Received 18 September 2009; accepted 24 September 2009

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.034; wR factor = 0.130; data-to-parameter ratio = 17.1.

In the title compound, $C_{18}H_{18}O_4$, the 1,3-dioxane ring adopts a distorted envelope conformation with the C atom common to the cyclohexane ring forming the flap. In the crystal, inversion dimers linked by pairs of C—H···O hydrogen bonds occur.

Related literature

For background information on spiro-compounds, see: Jiang *et al.* (1998); Lian *et al.* (2008); Wei *et al.* (2008).



Experimental

Crystal data

$C_{18}H_{18}O_4$
M_r = 298.32
Triclinic, $P\overline{1}$
a = 7.1177 (14) Å
b = 9.5506 (19) Å
c = 11.734 (2) Å
α = 106.82 (3)°
β = 100.14 (3)°
γ = 93.35 (3)°
V = 746.6 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 K
0.22 × 0.18 × 0.10 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: none
7448 measured reflections
3401 independent reflections
2309 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.130
S = 1.17
3401 reflections
199 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10A···O2^{i}	0.97	2.52	3.440 (2)	158

Symmetry code: (i) $-x, -y+1, -z$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5108).

References

Bruker (1997). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Jiang, Y. Z., Xue, S., Li, Z., Deng, J. G., Mi, A. Q. & Albert, S. C. C. (1998). *Tetrahedron*, **9**, 3185–3189.
Lian, Y., Guo, J. J., Liu, X. M. & Wei, R. B. (2008). *Chem. Res. Chin. Univ.* **24**, 441–444.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Wei, R. B., Liu, B., Liu, Y., Guo, J. J. & Zhang, D. W. (2008). *Chin. J. Or. C.* **28**, 1501–1514.

supporting information

Acta Cryst. (2009). E**65**, o2586 [doi:10.1107/S1600536809038781]

(*Z*)-3-(3-Phenylallylidene)-1,5-dioxaspiro[5.5]undecane-2,4-dione

Wu-Lan Zeng, Hua-Xiang Zhang and Fang-Fang Jian

S1. Comment

Spiro compounds are widely used in medicine, catalysis and optical materials (Lian *et al.*, 2008; Jiang *et al.*, 1998; Wei *et al.*, 2008) owing to their interesting conformational features. We report here the synthesis and structure of the title compound, (I) (Fig. 1), as part of our ongoing studies on new spiro compounds with potentially higher bioactivity.

The 1,3-dioxane ring is in a distored envelope conformation with atom C11 atom common to the cyclohexane forming the flap. The crystal structure is stabilized by weak intermolecular C—H···O hydrogen bonds (Table 1).

S2. Experimental

A mixture of malonic acid (6.24 g, 0.06 mol) and acetic anhydride(9 ml) in strong sulfuric acid (0.25 ml) was stirred with water at 303K, After dissolving, cyclohexanone (5.88 g, 0.06 mol) was added dropwise into solution for 1 h. The reaction was allowed to proceed for 4 h. The mixture was cooled and filtered, and then an ethanol solution of (Z)-3-phenylacryl-aldehyde (7.92g, 0.06 mol) was added. The solution was then filtered and concentrated. Yellow blocks of (I) were obtained by evaporation of a petroleum ether–ethylacetate (3:1 *v*/*v*) solution at room temperature over a period of one week.

S3. Refinement

The H atoms were placed in calculated positions (C—H = 0.93–0.97 Å), and refined as riding with U_{iso}(H) = 1.2U_{eq}(C).

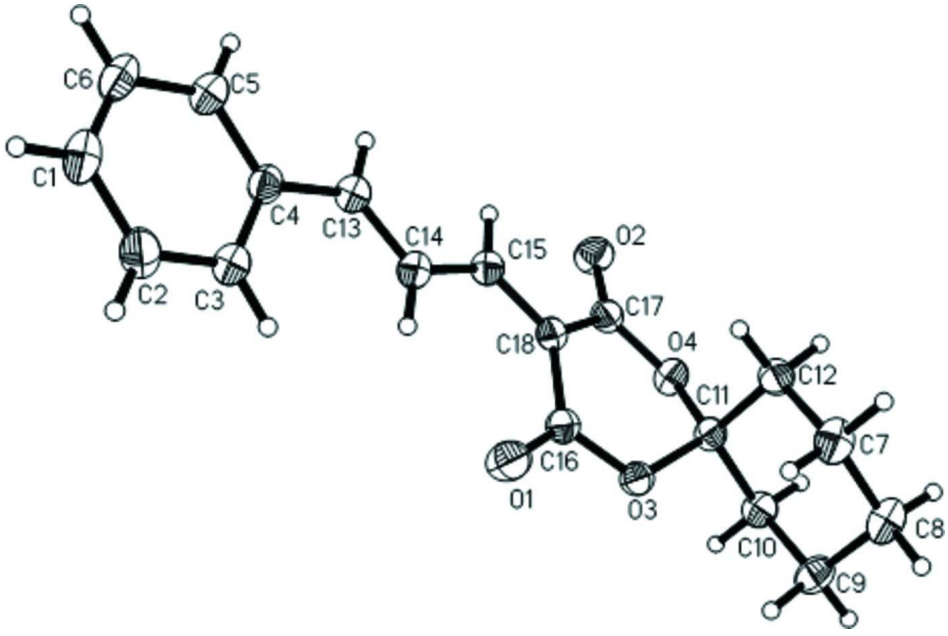


Figure 1

The molecular structure of (I), drawn with 30% probability ellipsoids and spheres of arbitrary size for the H atoms.

(*Z*)-3-(3-Phenylallylidene)-1,5-dioxaspiro[5.5]undecane-2,4-dione

Crystal data

$C_{18}H_{18}O_4$
$M_r = 298.32$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.1177\ (14)$ Å
$b = 9.5506\ (19)$ Å
$c = 11.734\ (2)$ Å
$\alpha = 106.82\ (3)°$
$\beta = 100.14\ (3)°$
$\gamma = 93.35\ (3)°$
$V = 746.6\ (3)$ Å^3
$Z = 2$
$F(000) = 316$
$D_x = 1.327$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3401 reflections
$\theta = 3.1–27.5°$
$\mu = 0.09$ mm^{-1}
$T = 293$ K
Block, yellow
0.22 × 0.18 × 0.10 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
7448 measured reflections
3401 independent reflections
2309 reflections with $I > 2\sigma(I)$
$R_{int} = 0.016$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
$h = -8\rightarrow9$
$k = -12\rightarrow12$
$l = -15\rightarrow15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.130$
$S = 1.17$
3401 reflections
199 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0724P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O4	0.07551 (13)	0.64682 (9)	0.13593 (9)	0.0440 (3)
O3	0.27862 (12)	0.86963 (9)	0.18966 (9)	0.0435 (3)
C18	−0.01600 (18)	0.86915 (14)	0.26210 (12)	0.0386 (3)
O2	−0.18747 (14)	0.63080 (11)	0.20875 (11)	0.0555 (3)
C17	−0.05451 (19)	0.70896 (14)	0.20042 (13)	0.0408 (3)
C11	0.19207 (18)	0.73899 (13)	0.09196 (12)	0.0375 (3)
C12	0.07076 (19)	0.77924 (15)	−0.01133 (13)	0.0443 (3)
H12A	−0.0251	0.8396	0.0195	0.053*
H12B	0.0042	0.6902	−0.0723	0.053*
C10	0.35558 (19)	0.65608 (14)	0.05319 (14)	0.0440 (3)
H10A	0.3043	0.5604	−0.0042	0.053*
H10B	0.4353	0.6405	0.1235	0.053*
C16	0.16915 (19)	0.94609 (15)	0.26336 (13)	0.0443 (3)
C4	−0.2995 (2)	1.28109 (15)	0.52506 (13)	0.0430 (3)
C14	−0.1418 (2)	1.08555 (15)	0.39392 (13)	0.0450 (3)
H14A	−0.0315	1.1500	0.4069	0.054*
C15	−0.15033 (19)	0.93622 (15)	0.32054 (13)	0.0426 (3)
H15A	−0.2635	0.8767	0.3116	0.051*
O1	0.23340 (15)	1.06908 (12)	0.32633 (13)	0.0728 (4)
C13	−0.2902 (2)	1.13463 (16)	0.44451 (13)	0.0453 (3)
H13A	−0.3994	1.0674	0.4260	0.054*
C5	−0.4695 (2)	1.31394 (17)	0.56535 (14)	0.0511 (4)
H5A	−0.5745	1.2418	0.5408	0.061*
C9	0.4779 (2)	0.74002 (16)	−0.00542 (15)	0.0520 (4)
H9A	0.5422	0.8302	0.0551	0.062*
H9B	0.5757	0.6807	−0.0346	0.062*
C3	−0.1442 (2)	1.39115 (17)	0.56447 (14)	0.0505 (4)
H3A	−0.0287	1.3716	0.5399	0.061*
C6	−0.4845 (2)	1.45175 (18)	0.64115 (15)	0.0576 (4)
H6A	−0.5987	1.4717	0.6676	0.069*

C8	0.3571 (2)	0.77756 (18)	−0.11085 (16)	0.0584 (4)
H8A	0.3042	0.6875	−0.1755	0.070*
H8B	0.4376	0.8363	−0.1426	0.070*
C2	−0.1606 (2)	1.52867 (18)	0.63955 (15)	0.0585 (4)
H2A	−0.0563	1.6015	0.6649	0.070*
C7	0.1938 (2)	0.86284 (17)	−0.06957 (15)	0.0551 (4)
H7A	0.2471	0.9577	−0.0115	0.066*
H7B	0.1140	0.8803	−0.1390	0.066*
C1	−0.3313 (3)	1.55903 (18)	0.67735 (15)	0.0590 (4)
H1A	−0.3420	1.6523	0.7274	0.071*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O4	0.0515 (5)	0.0329 (5)	0.0487 (6)	0.0045 (4)	0.0164 (4)	0.0102 (4)
O3	0.0393 (5)	0.0384 (5)	0.0451 (6)	0.0004 (4)	0.0101 (4)	0.0006 (4)
C18	0.0396 (6)	0.0387 (7)	0.0341 (7)	0.0034 (5)	0.0056 (5)	0.0072 (6)
O2	0.0517 (6)	0.0479 (6)	0.0653 (8)	−0.0055 (5)	0.0172 (5)	0.0136 (5)
C17	0.0412 (7)	0.0411 (7)	0.0386 (7)	0.0026 (6)	0.0063 (6)	0.0112 (6)
C11	0.0407 (6)	0.0300 (6)	0.0389 (7)	0.0024 (5)	0.0092 (5)	0.0059 (5)
C12	0.0454 (7)	0.0429 (7)	0.0438 (8)	0.0112 (6)	0.0078 (6)	0.0113 (6)
C10	0.0470 (7)	0.0359 (6)	0.0493 (8)	0.0123 (6)	0.0113 (6)	0.0110 (6)
C16	0.0415 (7)	0.0416 (7)	0.0428 (8)	0.0034 (6)	0.0089 (6)	0.0024 (6)
C4	0.0470 (7)	0.0489 (7)	0.0366 (7)	0.0114 (6)	0.0134 (6)	0.0144 (6)
C14	0.0458 (7)	0.0465 (7)	0.0407 (8)	0.0060 (6)	0.0115 (6)	0.0082 (6)
C15	0.0420 (7)	0.0462 (7)	0.0384 (7)	0.0034 (6)	0.0086 (6)	0.0112 (6)
O1	0.0542 (6)	0.0493 (6)	0.0873 (9)	−0.0122 (5)	0.0238 (6)	−0.0238 (6)
C13	0.0448 (7)	0.0481 (7)	0.0426 (8)	0.0063 (6)	0.0109 (6)	0.0118 (6)
C5	0.0522 (8)	0.0555 (8)	0.0493 (9)	0.0097 (7)	0.0199 (7)	0.0151 (7)
C9	0.0492 (8)	0.0500 (8)	0.0612 (10)	0.0158 (7)	0.0235 (7)	0.0145 (7)
C3	0.0492 (8)	0.0571 (9)	0.0440 (9)	0.0071 (7)	0.0147 (6)	0.0104 (7)
C6	0.0644 (10)	0.0632 (9)	0.0533 (10)	0.0229 (8)	0.0298 (8)	0.0165 (8)
C8	0.0696 (10)	0.0579 (9)	0.0577 (10)	0.0154 (8)	0.0298 (8)	0.0216 (8)
C2	0.0685 (10)	0.0551 (9)	0.0474 (9)	0.0000 (8)	0.0143 (8)	0.0086 (8)
C7	0.0685 (10)	0.0530 (8)	0.0541 (10)	0.0214 (7)	0.0191 (8)	0.0253 (8)
C1	0.0802 (11)	0.0530 (9)	0.0458 (9)	0.0170 (8)	0.0236 (8)	0.0099 (7)

Geometric parameters (Å, º)

O4—C17	1.3536 (17)	C14—C15	1.428 (2)
O4—C11	1.4344 (16)	C14—H14A	0.9300
O3—C16	1.3515 (17)	C15—H15A	0.9300
O3—C11	1.4437 (16)	C13—H13A	0.9300
C18—C15	1.3575 (19)	C5—C6	1.381 (2)
C18—C16	1.4665 (19)	C5—H5A	0.9300
C18—C17	1.4765 (19)	C9—C8	1.522 (2)
O2—C17	1.2062 (17)	C9—H9A	0.9700
C11—C10	1.5080 (18)	C9—H9B	0.9700

C11—C12	1.5174 (18)	C3—C2	1.378 (2)
C12—C7	1.522 (2)	C3—H3A	0.9300
C12—H12A	0.9700	C6—C1	1.369 (2)
C12—H12B	0.9700	C6—H6A	0.9300
C10—C9	1.524 (2)	C8—C7	1.526 (2)
C10—H10A	0.9700	C8—H8A	0.9700
C10—H10B	0.9700	C8—H8B	0.9700
C16—O1	1.2045 (17)	C2—C1	1.384 (2)
C4—C3	1.394 (2)	C2—H2A	0.9300
C4—C5	1.395 (2)	C7—H7A	0.9700
C4—C13	1.457 (2)	C7—H7B	0.9700
C14—C13	1.344 (2)	C1—H1A	0.9300

C17—O4—C11	118.14 (10)	C14—C15—H15A	115.5
C16—O3—C11	119.54 (10)	C14—C13—C4	127.39 (14)
C15—C18—C16	123.28 (12)	C14—C13—H13A	116.3
C15—C18—C17	117.99 (12)	C4—C13—H13A	116.3
C16—C18—C17	118.63 (12)	C6—C5—C4	121.11 (15)
O2—C17—O4	118.86 (12)	C6—C5—H5A	119.4
O2—C17—C18	124.36 (14)	C4—C5—H5A	119.4
O4—C17—C18	116.68 (12)	C8—C9—C10	111.66 (12)
O4—C11—O3	110.01 (11)	C8—C9—H9A	109.3
O4—C11—C10	107.54 (10)	C10—C9—H9A	109.3
O3—C11—C10	106.20 (10)	C8—C9—H9B	109.3
O4—C11—C12	109.82 (10)	C10—C9—H9B	109.3
O3—C11—C12	110.77 (10)	H9A—C9—H9B	107.9
C10—C11—C12	112.40 (12)	C2—C3—C4	120.55 (15)
C11—C12—C7	111.29 (12)	C2—C3—H3A	119.7
C11—C12—H12A	109.4	C4—C3—H3A	119.7
C7—C12—H12A	109.4	C1—C6—C5	119.95 (15)
C11—C12—H12B	109.4	C1—C6—H6A	120.0
C7—C12—H12B	109.4	C5—C6—H6A	120.0
H12A—C12—H12B	108.0	C9—C8—C7	110.64 (13)
C11—C10—C9	111.33 (10)	C9—C8—H8A	109.5
C11—C10—H10A	109.4	C7—C8—H8A	109.5
C9—C10—H10A	109.4	C9—C8—H8B	109.5
C11—C10—H10B	109.4	C7—C8—H8B	109.5
C9—C10—H10B	109.4	H8A—C8—H8B	108.1
H10A—C10—H10B	108.0	C3—C2—C1	120.34 (16)
O1—C16—O3	117.67 (13)	C3—C2—H2A	119.8
O1—C16—C18	125.75 (14)	C1—C2—H2A	119.8
O3—C16—C18	116.55 (12)	C12—C7—C8	111.50 (12)
C3—C4—C5	118.04 (14)	C12—C7—H7A	109.3
C3—C4—C13	122.64 (13)	C8—C7—H7A	109.3
C5—C4—C13	119.32 (14)	C12—C7—H7B	109.3
C13—C14—C15	121.18 (14)	C8—C7—H7B	109.3
C13—C14—H14A	119.4	H7A—C7—H7B	108.0
C15—C14—H14A	119.4	C6—C1—C2	120.00 (16)

C18—C15—C14	128.93 (13)	C6—C1—H1A	120.0
C18—C15—H15A	115.5	C2—C1—H1A	120.0

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10*A*···O2^{i}	0.97	2.52	3.440 (2)	158

Symmetry code: (i) −*x*, −*y*+1, −*z*.