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## Structure Reports

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# (*E,E*)-2-[3,4-Bis(4-methylbenzylidene)-5-oxotetrahydrofuran-2-ylidene]propane-dinitrile

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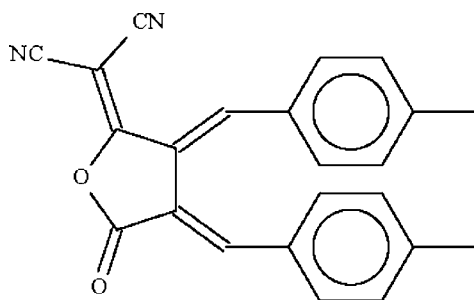
Received 5 March 2009; accepted 9 March 2009

 Key indicators: single-crystal X-ray study;  $T = 123$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.044;  $wR$  factor = 0.119; data-to-parameter ratio = 16.5.

In the title molecule,  $\text{C}_{23}\text{H}_{16}\text{N}_2\text{O}_2$ , the two exocyclic  $\text{C}=\text{C}$  bonds bearing the tolyl groups have an *E* configuration and the benzene rings are oriented at 22.1 (1) and 24.8 (1)° with respect to the mean plane of the atoms of the furan ring.

## Related literature

The compound belongs to a class of photochromic fulgicides; for similar structures, see: Asiri *et al.* (2000); Heller *et al.* (1994); Liang *et al.* (2003).



## Experimental

## Crystal data

$\text{C}_{23}\text{H}_{16}\text{N}_2\text{O}_2$	$V = 1766.00$ (8) Å <sup>3</sup>
$M_r = 352.38$	$Z = 4$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 6.7908$ (2) Å	$\mu = 0.09$ mm <sup>-1</sup>
$b = 22.0814$ (5) Å	$T = 123$ K
$c = 11.8626$ (3) Å	$0.22 \times 0.12 \times 0.08$ mm
$\beta = 96.877$ (2)°	

## Data collection

Bruker SMART APEX diffractometer	4051 independent reflections
Absorption correction: none	2971 reflections with $I > 2\sigma(I)$
16672 measured reflections	$R_{\text{int}} = 0.040$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$	246 parameters
$wR(F^2) = 0.119$	H-atom parameters constrained
$S = 1.02$	$\Delta\rho_{\text{max}} = 0.29$ e Å <sup>-3</sup>
4051 reflections	$\Delta\rho_{\text{min}} = -0.24$ e Å <sup>-3</sup>

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2785).

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**supplementary materials**

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**(*E,E*)-2-[3,4-Bis(4-methylbenzylidene)-5-oxotetrahydrofuran-2-ylidene]propanedinitrile**

**A. M. Asiri and S. W. Ng**

**Comment**

The molecular structure of the title compound is shown in Fig. 1.

**Experimental**

Diethylamine (0.73 g, 10.0 mmol) was added dropwise *E,E*-3,4-bis(4-tolylmethylene) succinic anhydride (1.52 g, 5.0 mmol) and malononitrile (0.33 g, 5.0 mmol) in THF (20 ml) at 273 K. The mixture was kept at this temperature for 6 h. Diethyl ether (15 ml) was added to quench the reaction. The product was dissolved in dichloromethane (20 ml) and cyclized by acetyl chloride (10 ml) at 293 K. The reaction was kept at this temperature for 10 h. The solvent and excess acetyl chloride were removed in vacuum, and the residual was chromatographed on silica gel. Elution by a 3:7 mixture of ethyl acetate and light petroleum give the title compound as an orange compound (1.41 g, 80% yield), m.p. 478–479 K. Crystals were grown with ether/light petroleum ether as solvent.

**Refinement**

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 0.98 Å) and were included in the refinement in the riding model approximation, with  $U(\text{H})$  set to 1.2 to 1.5 $U(\text{C})$ .

**Figures**

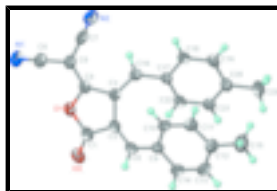


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of  $\text{C}_{23}\text{H}_{16}\text{N}_2\text{O}_2$ ; probability levels are set at 70% and H-atoms are drawn as spheres of arbitrary radius.

**(*E,E*)-2-[3,4-Bis(4-methylbenzylidene)-5-oxotetrahydrofuran-2-ylidene]propanedinitrile**

*Crystal data*

$\text{C}_{23}\text{H}_{16}\text{N}_2\text{O}_2$

$M_r = 352.38$

Monoclinic,  $P2_1/n$

Hall symbol: -P 2yn

$a = 6.7908$  (2) Å

$b = 22.0814$  (5) Å

$F_{000} = 736$

$D_x = 1.325$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 2995 reflections

$\theta = 2.5$ – $25.4^\circ$

$\mu = 0.09$  mm<sup>-1</sup>

# supplementary materials

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$c = 11.8626$  (3) Å  
 $\beta = 96.877$  (2)°  
 $V = 1766.00$  (8) Å<sup>3</sup>  
 $Z = 4$

$T = 123$  K  
Prism, orange  
 $0.22 \times 0.12 \times 0.08$  mm

## Data collection

Bruker SMART APEX diffractometer	2971 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{\text{int}} = 0.040$
Monochromator: graphite	$\theta_{\text{max}} = 27.5^\circ$
$T = 123$ K	$\theta_{\text{min}} = 1.8^\circ$
$\omega$ scans	$h = -8 \rightarrow 8$
Absorption correction: None	$k = -28 \rightarrow 28$
16672 measured reflections	$l = -15 \rightarrow 15$
4051 independent reflections	

## Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.044$	H-atom parameters constrained
$wR(F^2) = 0.119$	$w = 1/[\sigma^2(F_o^2) + (0.0555P)^2 + 0.4567P]$
$S = 1.02$	where $P = (F_o^2 + 2F_c^2)/3$
4051 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
246 parameters	$\Delta\rho_{\text{max}} = 0.29 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.24 \text{ e \AA}^{-3}$
	Extinction correction: none

## Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.81235 (17)	0.53159 (5)	0.60804 (9)	0.0311 (3)
O2	0.8934 (2)	0.56246 (6)	0.78870 (10)	0.0434 (3)
N1	0.7342 (2)	0.40469 (7)	0.44380 (13)	0.0404 (4)
N2	0.5427 (3)	0.55698 (7)	0.22059 (13)	0.0419 (4)
C1	0.8373 (2)	0.57691 (7)	0.69372 (14)	0.0311 (4)
C2	0.7782 (2)	0.63512 (7)	0.64023 (13)	0.0255 (3)
C3	0.7583 (2)	0.62428 (7)	0.51673 (13)	0.0239 (3)
C4	0.7526 (2)	0.55846 (7)	0.50622 (13)	0.0257 (3)
C5	0.6993 (2)	0.52050 (7)	0.41733 (14)	0.0279 (3)
C6	0.7187 (2)	0.45625 (7)	0.43229 (14)	0.0309 (4)
C7	0.6140 (3)	0.54142 (7)	0.30845 (15)	0.0310 (4)
C8	0.7414 (2)	0.68084 (7)	0.71076 (13)	0.0269 (3)
H8	0.7747	0.6724	0.7892	0.032*
C9	0.6596 (2)	0.74052 (7)	0.68603 (12)	0.0242 (3)

C10	0.5317 (2)	0.75352 (7)	0.58741 (12)	0.0250 (3)
H10	0.4849	0.7216	0.5374	0.030*
C11	0.4733 (2)	0.81213 (7)	0.56248 (13)	0.0276 (3)
H11	0.3863	0.8201	0.4953	0.033*
C12	0.5393 (2)	0.86024 (7)	0.63392 (14)	0.0306 (4)
C13	0.6552 (2)	0.84664 (8)	0.73535 (14)	0.0317 (4)
H13	0.6955	0.8783	0.7873	0.038*
C14	0.7129 (2)	0.78790 (7)	0.76190 (13)	0.0283 (4)
H14	0.7897	0.7795	0.8325	0.034*
C15	0.4873 (3)	0.92448 (8)	0.60006 (17)	0.0445 (5)
H15A	0.5459	0.9521	0.6595	0.067*
H15B	0.3428	0.9293	0.5902	0.067*
H15C	0.5394	0.9339	0.5285	0.067*
C16	0.7738 (2)	0.66042 (7)	0.42565 (13)	0.0246 (3)
H16	0.7519	0.6408	0.3540	0.030*
C17	0.8187 (2)	0.72444 (7)	0.42155 (12)	0.0223 (3)
C18	0.7433 (2)	0.75755 (7)	0.32513 (12)	0.0252 (3)
H18	0.6720	0.7372	0.2624	0.030*
C19	0.7713 (2)	0.81920 (7)	0.32030 (13)	0.0274 (3)
H19	0.7157	0.8410	0.2551	0.033*
C20	0.8803 (2)	0.85031 (7)	0.40968 (13)	0.0273 (3)
C21	0.9648 (2)	0.81676 (7)	0.50266 (13)	0.0261 (3)
H21	1.0441	0.8367	0.5629	0.031*
C22	0.9353 (2)	0.75505 (7)	0.50862 (12)	0.0242 (3)
H22	0.9949	0.7331	0.5727	0.029*
C23	0.9098 (3)	0.91778 (8)	0.40462 (16)	0.0382 (4)
H23A	0.9312	0.9342	0.4819	0.057*
H23B	0.7918	0.9365	0.3632	0.057*
H23C	1.0257	0.9266	0.3654	0.057*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0370 (7)	0.0231 (6)	0.0326 (6)	0.0003 (5)	0.0017 (5)	0.0047 (5)
O2	0.0597 (9)	0.0357 (7)	0.0323 (7)	-0.0004 (6)	-0.0051 (6)	0.0116 (5)
N1	0.0444 (9)	0.0266 (8)	0.0501 (10)	0.0001 (6)	0.0057 (8)	-0.0024 (7)
N2	0.0523 (10)	0.0350 (8)	0.0375 (9)	0.0054 (7)	0.0009 (7)	-0.0061 (7)
C1	0.0314 (9)	0.0286 (9)	0.0327 (9)	-0.0034 (7)	0.0014 (7)	0.0057 (7)
C2	0.0258 (8)	0.0245 (8)	0.0256 (8)	-0.0029 (6)	0.0010 (6)	0.0040 (6)
C3	0.0219 (7)	0.0238 (8)	0.0261 (8)	-0.0003 (6)	0.0034 (6)	-0.0005 (6)
C4	0.0229 (8)	0.0246 (8)	0.0300 (8)	0.0009 (6)	0.0051 (6)	0.0029 (6)
C5	0.0262 (8)	0.0228 (8)	0.0355 (9)	0.0012 (6)	0.0068 (7)	-0.0006 (6)
C6	0.0276 (9)	0.0271 (9)	0.0384 (9)	0.0003 (6)	0.0054 (7)	-0.0031 (7)
C7	0.0345 (9)	0.0215 (8)	0.0378 (10)	0.0016 (6)	0.0076 (8)	-0.0067 (7)
C8	0.0284 (8)	0.0313 (9)	0.0208 (7)	-0.0053 (6)	0.0023 (6)	0.0033 (6)
C9	0.0237 (8)	0.0274 (8)	0.0224 (7)	-0.0032 (6)	0.0066 (6)	-0.0014 (6)
C10	0.0230 (8)	0.0295 (8)	0.0233 (7)	-0.0020 (6)	0.0059 (6)	-0.0060 (6)
C11	0.0229 (8)	0.0338 (9)	0.0262 (8)	0.0029 (6)	0.0038 (6)	-0.0012 (6)

## supplementary materials

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C12	0.0280 (8)	0.0277 (9)	0.0384 (9)	0.0007 (6)	0.0132 (7)	-0.0027 (7)
C13	0.0314 (9)	0.0323 (9)	0.0327 (9)	-0.0066 (7)	0.0094 (7)	-0.0113 (7)
C14	0.0269 (8)	0.0356 (9)	0.0227 (8)	-0.0043 (7)	0.0047 (6)	-0.0046 (6)
C15	0.0500 (12)	0.0306 (10)	0.0552 (12)	0.0064 (8)	0.0152 (10)	-0.0012 (8)
C16	0.0229 (8)	0.0260 (8)	0.0252 (7)	0.0011 (6)	0.0044 (6)	-0.0022 (6)
C17	0.0215 (7)	0.0237 (8)	0.0226 (7)	0.0008 (6)	0.0067 (6)	0.0011 (6)
C18	0.0251 (8)	0.0298 (8)	0.0212 (7)	-0.0005 (6)	0.0046 (6)	0.0005 (6)
C19	0.0287 (8)	0.0284 (8)	0.0255 (8)	0.0030 (6)	0.0047 (6)	0.0071 (6)
C20	0.0254 (8)	0.0256 (8)	0.0323 (8)	-0.0014 (6)	0.0091 (7)	0.0035 (6)
C21	0.0229 (8)	0.0295 (8)	0.0264 (8)	-0.0040 (6)	0.0046 (6)	-0.0012 (6)
C22	0.0225 (8)	0.0269 (8)	0.0233 (7)	0.0000 (6)	0.0033 (6)	0.0035 (6)
C23	0.0431 (10)	0.0278 (9)	0.0436 (10)	-0.0045 (7)	0.0055 (8)	0.0057 (8)

### *Geometric parameters (Å, °)*

O1—C4	1.3631 (19)	C13—C14	1.381 (2)
O1—C1	1.422 (2)	C13—H13	0.9500
O2—C1	1.1890 (19)	C14—H14	0.9500
N1—C6	1.150 (2)	C15—H15A	0.9800
N2—C7	1.148 (2)	C15—H15B	0.9800
C1—C2	1.468 (2)	C15—H15C	0.9800
C2—C8	1.353 (2)	C16—C17	1.448 (2)
C2—C3	1.475 (2)	C16—H16	0.9500
C3—C16	1.357 (2)	C17—C22	1.398 (2)
C3—C4	1.459 (2)	C17—C18	1.402 (2)
C4—C5	1.361 (2)	C18—C19	1.377 (2)
C5—C7	1.428 (2)	C18—H18	0.9500
C5—C6	1.434 (2)	C19—C20	1.399 (2)
C8—C9	1.446 (2)	C19—H19	0.9500
C8—H8	0.9500	C20—C21	1.394 (2)
C9—C14	1.399 (2)	C20—C23	1.505 (2)
C9—C10	1.401 (2)	C21—C22	1.380 (2)
C10—C11	1.375 (2)	C21—H21	0.9500
C10—H10	0.9500	C22—H22	0.9500
C11—C12	1.399 (2)	C23—H23A	0.9800
C11—H11	0.9500	C23—H23B	0.9800
C12—C13	1.390 (2)	C23—H23C	0.9800
C12—C15	1.505 (2)		
C4—O1—C1	108.93 (12)	C13—C14—C9	120.75 (15)
O2—C1—O1	119.06 (15)	C13—C14—H14	119.6
O2—C1—C2	133.21 (16)	C9—C14—H14	119.6
O1—C1—C2	107.71 (13)	C12—C15—H15A	109.5
C8—C2—C1	116.55 (14)	C12—C15—H15B	109.5
C8—C2—C3	137.18 (14)	H15A—C15—H15B	109.5
C1—C2—C3	105.97 (13)	C12—C15—H15C	109.5
C16—C3—C2	121.42 (14)	H15A—C15—H15C	109.5
C16—C3—C2	133.53 (14)	H15B—C15—H15C	109.5
C4—C3—C2	104.18 (12)	C3—C16—C17	129.59 (14)
C5—C4—O1	116.21 (14)	C3—C16—H16	115.2

C5—C4—C3	132.95 (15)	C17—C16—H16	115.2
O1—C4—C3	110.84 (13)	C22—C17—C18	118.03 (14)
C4—C5—C7	122.89 (15)	C22—C17—C16	123.37 (13)
C4—C5—C6	120.09 (15)	C18—C17—C16	118.59 (13)
C7—C5—C6	116.96 (14)	C19—C18—C17	120.72 (14)
N1—C6—C5	179.72 (18)	C19—C18—H18	119.6
N2—C7—C5	178.35 (18)	C17—C18—H18	119.6
C2—C8—C9	130.47 (14)	C18—C19—C20	121.15 (14)
C2—C8—H8	114.8	C18—C19—H19	119.4
C9—C8—H8	114.8	C20—C19—H19	119.4
C14—C9—C10	118.09 (14)	C21—C20—C19	117.99 (14)
C14—C9—C8	119.16 (14)	C21—C20—C23	120.89 (15)
C10—C9—C8	122.74 (14)	C19—C20—C23	121.11 (14)
C11—C10—C9	120.42 (14)	C22—C21—C20	121.10 (14)
C11—C10—H10	119.8	C22—C21—H21	119.5
C9—C10—H10	119.8	C20—C21—H21	119.5
C10—C11—C12	121.42 (15)	C21—C22—C17	120.82 (14)
C10—C11—H11	119.3	C21—C22—H22	119.6
C12—C11—H11	119.3	C17—C22—H22	119.6
C13—C12—C11	117.88 (15)	C20—C23—H23A	109.5
C13—C12—C15	121.69 (15)	C20—C23—H23B	109.5
C11—C12—C15	120.43 (16)	H23A—C23—H23B	109.5
C14—C13—C12	121.09 (15)	C20—C23—H23C	109.5
C14—C13—H13	119.5	H23A—C23—H23C	109.5
C12—C13—H13	119.5	H23B—C23—H23C	109.5
C4—O1—C1—O2	-178.40 (16)	C2—C8—C9—C14	-151.98 (17)
C4—O1—C1—C2	3.07 (17)	C2—C8—C9—C10	26.5 (3)
O2—C1—C2—C8	-15.0 (3)	C14—C9—C10—C11	4.9 (2)
O1—C1—C2—C8	163.23 (14)	C8—C9—C10—C11	-173.64 (14)
O2—C1—C2—C3	170.13 (19)	C9—C10—C11—C12	0.1 (2)
O1—C1—C2—C3	-11.63 (17)	C10—C11—C12—C13	-4.4 (2)
C8—C2—C3—C16	32.8 (3)	C10—C11—C12—C15	174.86 (15)
C1—C2—C3—C16	-153.94 (17)	C11—C12—C13—C14	3.6 (2)
C8—C2—C3—C4	-158.09 (19)	C15—C12—C13—C14	-175.61 (16)
C1—C2—C3—C4	15.13 (16)	C12—C13—C14—C9	1.4 (2)
C1—O1—C4—C5	-172.45 (14)	C10—C9—C14—C13	-5.6 (2)
C1—O1—C4—C3	7.04 (16)	C8—C9—C14—C13	172.93 (14)
C16—C3—C4—C5	-23.9 (3)	C4—C3—C16—C17	-167.02 (14)
C2—C3—C4—C5	165.36 (17)	C2—C3—C16—C17	0.5 (3)
C16—C3—C4—O1	156.70 (14)	C3—C16—C17—C22	28.9 (2)
C2—C3—C4—O1	-14.02 (16)	C3—C16—C17—C18	-151.66 (16)
O1—C4—C5—C7	173.23 (14)	C22—C17—C18—C19	-4.7 (2)
C3—C4—C5—C7	-6.1 (3)	C16—C17—C18—C19	175.85 (14)
O1—C4—C5—C6	-3.6 (2)	C17—C18—C19—C20	1.8 (2)
C3—C4—C5—C6	177.04 (15)	C18—C19—C20—C21	1.8 (2)
C4—C5—C6—N1	-164 (100)	C18—C19—C20—C23	-179.62 (15)
C7—C5—C6—N1	19 (40)	C19—C20—C21—C22	-2.6 (2)
C4—C5—C7—N2	-147 (6)	C23—C20—C21—C22	178.83 (15)
C6—C5—C7—N2	30 (7)	C20—C21—C22—C17	-0.3 (2)

# supplementary materials

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C1—C2—C8—C9	-171.89 (15)	C18—C17—C22—C21	3.9 (2)
C3—C2—C8—C9	0.8 (3)	C16—C17—C22—C21	-176.66 (14)

Fig. 1

