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(2*E*,6*E*)-2,6-Difurfurylidenecyclohexanone

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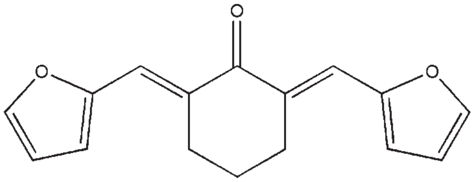
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.047; wR factor = 0.122; data-to-parameter ratio = 12.6.

The complete molecule of the title compound, $C_{16}H_{14}O_3$, is generated by crystallographic mirror symmetry, with two C atoms and one O atom lying on the mirror plane. The molecule adopts an *E* configuration about the C=C bond and the dihedral angle between the furan rings is 16.1 (2)°.

Related literature

For general background to the use of bis(arylmethylidene)cycloalkanones as building blocks for the synthesis of biologically active heterocycles, see: Guilford *et al.* (1999). For related structures, see: Liu & Chen (2009); Liu (2009); Shi *et al.* (2008).



Experimental

Crystal data

$C_{16}H_{14}O_3$	V = 1251.5 (3) $Å^3$
M_r = 254.27	Z = 4
Orthorhombic, *Pnma*	Mo $K\alpha$ radiation
a = 7.7313 (11) Å	μ = 0.09 mm^{-1}
b = 15.658 (2) Å	T = 295 K
c = 10.3388 (14) Å	0.15 × 0.10 × 0.06 mm

Data collection

Siemens SMART CCD diffractometer	6025 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	1158 independent reflections
T_{min} = 0.986, T_{max} = 0.995	731 reflections with $I > 2\sigma(I)$
	R_{int} = 0.072

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047	92 parameters
$wR(F^2)$ = 0.122	H-atom parameters constrained
S = 1.03	$\Delta\rho_{max}$ = 0.17 e $Å^{-3}$
1158 reflections	$\Delta\rho_{min}$ = −0.16 e $Å^{-3}$

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5212).

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supplementary materials

Acta Cryst. (2009). E**65**, o3085 [**doi:10.1107/S160053680904728X**]

(2*E*,6*E*)-2,6-Difurfurylidenecyclohexanone

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Comment

Bis(arylmethylidene)cycloalkanones are widely used as building blocks for the synthesis of biologically active heterocycles (Guilford *et al.*, 1999). In the present paper, we describe the crystal stucture of the title compound. The molecule posseses normal geometric parameters and adopts an *E*-configuration about the central olefinic bonds (Fig. 1). The cyclohexanone ring and the furan rings are alomst coplanar which allows conjugation. Similar structures have been observed in the related substituted cyclopentanone and cyclohexanone analogues reported by Liu & Chen (2009); Liu (2009); Shi *et al.* (2008).

Experimental

Tetrabutylammonium bromide (0.5 mmol) and NaOH (10 mmol) were dissolved in the mixture of water (10 ml) and ethanol (4 ml). The solution was stirred at room temperature for 10 min, followed by dropwise addition of a mixture of furaldehyde (20 mmol) and cyclohexanone (10 mmol).The mixture was stirred at the temperature of 303 K for 2 h. When the reaction was complete, the residue was filtered. The precipitate was washed by water and recrystallized from ethyl acetate to yield yellow blocks of (I). Analysis calculated for $C_{16}H_{14}O_3$: C 75.59, H 5.51%; found: C 75.65, H 5.46%.

Refinement

All H-atoms were initially located in a difference Fourier map and were placed in geometrically idealized positions, with C—H = 0.93 - 0.97 Å and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

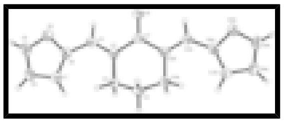

Fig. 1. The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level. Atoms with the suffix A are generated by (x, 3/2–y, z).

(2*E*,6*E*)-2,6-Difurfurylidenecyclohexanone

Crystal data

$C_{16}H_{14}O_3$	$D_x = 1.349$ Mg m^{-3}
$M_r = 254.27$	Melting point: 417 K
Orthorhombic, *Pnma*	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: -P 2ac 2n	Cell parameters from 472 reflections
a = 7.7313 (11) Å	θ = 2.6–19.2°
b = 15.658 (2) Å	μ = 0.09 mm^{-1}
c = 10.3388 (14) Å	*T* = 295 K

$V = 1251.5$ (3) Å^3
$Z = 4$
$F_{000} = 536$

Block, yellow
$0.15 \times 0.10 \times 0.06$ mm

Data collection

Siemens SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Monochromator: graphite
$T = 295$ K
ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
$T_{\rm min} = 0.986$, $T_{\rm max} = 0.995$
6025 measured reflections

1158 independent reflections
731 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.072$
$\theta_{\rm max} = 25.1°$
$\theta_{\rm min} = 2.4°$
$h = -9\rightarrow9$
$k = -15\rightarrow18$
$l = -12\rightarrow9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.122$
$S = 1.03$
1158 reflections
92 parameters
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
$w = 1/[\sigma^2(F_o^2) + (0.0454P)^2 + 0.2592P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.17$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.16$ e Å^{-3}
Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.017 (2)

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 (Å^2)

	x	y	z	$U_{\rm iso}$*/$U_{\rm eq}$

O1	0.0245 (3)	0.7500	0.0550 (2)	0.0606 (7)
O2	0.1187 (2)	0.44953 (11)	0.12636 (15)	0.0689 (6)
C1	0.0568 (4)	0.7500	0.1720 (3)	0.0461 (8)
C2	0.0806 (3)	0.66875 (14)	0.24340 (19)	0.0434 (6)
C3	0.0950 (3)	0.67029 (14)	0.3886 (2)	0.0509 (7)
H3A	0.2160	0.6668	0.4129	0.061*
H3B	0.0367	0.6206	0.4239	0.061*
C4	0.0172 (4)	0.7500	0.4467 (3)	0.0527 (9)
H4A	0.0366	0.7500	0.5394	0.063*
H4B	−0.1067	0.7500	0.4319	0.063*
C5	0.0954 (3)	0.59655 (14)	0.1739 (2)	0.0494 (6)
H5	0.0794	0.6022	0.0852	0.059*
C6	0.1324 (3)	0.51243 (15)	0.2189 (2)	0.0497 (7)
C7	0.1845 (4)	0.47563 (15)	0.3310 (2)	0.0602 (7)
H7	0.2048	0.5036	0.4089	0.072*
C8	0.2024 (4)	0.38729 (16)	0.3081 (2)	0.0646 (8)
H8	0.2366	0.3460	0.3674	0.078*
C9	0.1606 (4)	0.37468 (17)	0.1841 (3)	0.0729 (9)
H9	0.1603	0.3218	0.1432	0.087*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0879 (19)	0.0532 (15)	0.0406 (13)	0.000	−0.0145 (12)	0.000
O2	0.1102 (16)	0.0485 (11)	0.0481 (10)	0.0097 (10)	0.0021 (10)	−0.0052 (8)
C1	0.051 (2)	0.051 (2)	0.0365 (17)	0.000	−0.0044 (15)	0.000
C2	0.0445 (14)	0.0456 (14)	0.0402 (12)	−0.0018 (11)	−0.0005 (10)	−0.0007 (11)
C3	0.0656 (17)	0.0479 (14)	0.0391 (12)	−0.0026 (13)	−0.0014 (11)	0.0010 (11)
C4	0.061 (2)	0.059 (2)	0.0387 (17)	0.000	0.0036 (16)	0.000
C5	0.0538 (15)	0.0524 (16)	0.0421 (13)	−0.0002 (12)	−0.0027 (11)	0.0021 (11)
C6	0.0596 (17)	0.0451 (14)	0.0445 (13)	−0.0001 (12)	0.0003 (11)	−0.0024 (11)
C7	0.0767 (19)	0.0525 (16)	0.0513 (14)	−0.0008 (14)	−0.0073 (13)	−0.0004 (12)
C8	0.081 (2)	0.0479 (16)	0.0650 (17)	0.0033 (14)	0.0018 (15)	0.0110 (13)
C9	0.109 (2)	0.0432 (16)	0.0669 (18)	0.0100 (16)	0.0138 (17)	0.0018 (14)

Geometric parameters (Å, °)

O1—C1	1.235 (3)	C4—H4A	0.9700
O2—C9	1.355 (3)	C4—H4B	0.9700
O2—C6	1.377 (2)	C5—C6	1.426 (3)
C1—C2^{i}	1.482 (3)	C5—H5	0.9300
C1—C2	1.482 (3)	C6—C7	1.355 (3)
C2—C5	1.344 (3)	C7—C8	1.410 (3)
C2—C3	1.506 (3)	C7—H7	0.9300
C3—C4	1.510 (3)	C8—C9	1.336 (3)
C3—H3A	0.9700	C8—H8	0.9300
C3—H3B	0.9700	C9—H9	0.9300
C4—C3^{i}	1.510 (3)		

C9—O2—C6	107.08 (19)	C3^{i}—C4—H4B	109.3
O1—C1—C2^{i}	120.85 (13)	H4A—C4—H4B	108.0
O1—C1—C2	120.85 (13)	C2—C5—C6	128.3 (2)
C2^{i}—C1—C2	118.2 (3)	C2—C5—H5	115.9
C5—C2—C1	117.8 (2)	C6—C5—H5	115.9
C5—C2—C3	122.7 (2)	C7—C6—O2	108.2 (2)
C1—C2—C3	119.5 (2)	C7—C6—C5	137.0 (2)
C2—C3—C4	112.3 (2)	O2—C6—C5	114.73 (19)
C2—C3—H3A	109.1	C6—C7—C8	107.6 (2)
C4—C3—H3A	109.1	C6—C7—H7	126.2
C2—C3—H3B	109.1	C8—C7—H7	126.2
C4—C3—H3B	109.1	C9—C8—C7	106.4 (2)
H3A—C3—H3B	107.9	C9—C8—H8	126.8
C3—C4—C3^{i}	111.5 (3)	C7—C8—H8	126.8
C3—C4—H4A	109.3	C8—C9—O2	110.7 (2)
C3^{i}—C4—H4A	109.3	C8—C9—H9	124.7
C3—C4—H4B	109.3	O2—C9—H9	124.7
O1—C1—C2—C5	11.1 (4)	C9—O2—C6—C7	0.9 (3)
C2^{i}—C1—C2—C5	−166.16 (18)	C9—O2—C6—C5	179.0 (2)
O1—C1—C2—C3	−171.5 (3)	C2—C5—C6—C7	−10.1 (5)
C2^{i}—C1—C2—C3	11.2 (4)	C2—C5—C6—O2	172.5 (2)
C5—C2—C3—C4	−161.0 (2)	O2—C6—C7—C8	−0.5 (3)
C1—C2—C3—C4	21.8 (3)	C5—C6—C7—C8	−178.0 (3)
C2—C3—C4—C3^{i}	−55.1 (3)	C6—C7—C8—C9	0.0 (3)
C1—C2—C5—C6	174.6 (2)	C7—C8—C9—O2	0.6 (3)
C3—C2—C5—C6	−2.7 (4)	C6—O2—C9—C8	−0.9 (3)

Symmetry codes: (i) x, $-y$+3/2, z.

Fig. 1