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(2*E*,5*E*)-2,5-Difurfurylidenecyclopentanone

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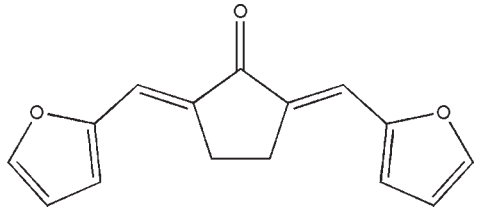
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.050; wR factor = 0.129; data-to-parameter ratio = 12.7.

In the title molecule, $C_{15}H_{12}O_3$, the three five-membered rings are nearly coplanar: the dihedral angles between the cyclopentanone ring and the furan rings are 3.5 (2) and 9.7 (2)°, and the two furan rings form a dihedral angle of 7.2 (2)°. In the crystal structure, weak intermolecular C—H···O hydrogen bonds help to consolidate the crystal packing.

Related literature

For 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: Du *et al.* (2007); Sun & Cui (2007); Wei *et al.* (2008).



Experimental

Crystal data

$C_{15}H_{12}O_3$ M_r = 240.25
Monoclinic, $P2_1/c$
a = 5.9280 (9) Å
b = 8.5031 (13) Å
c = 23.280 (3) Å
β = 92.139 (3)°
V = 1172.6 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 K
0.12 × 0.08 × 0.05 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.989, T_{max} = 0.995
5793 measured reflections
2076 independent reflections
1083 reflections with $I > 2\sigma(I)$
R_{int} = 0.059

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.129
S = 0.99
2076 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C15—H15···O1^{i}	0.93	2.49	3.221 (4)	136
C4—H4A···O1^{ii}	0.97	2.45	3.393 (3)	165

Symmetry codes: (i) $-x+2, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $x-1, y, z$.

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: CV2656).

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supporting information

Acta Cryst. (2009). E**65**, o3084 [doi:10.1107/S1600536809047278]

(2*E*,5*E*)-2,5-Difurfurylidenecyclopentanone

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S1. 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 title molecule adopts an *E*-configuration about the central olefinic bonds (Fig.1). The cyclopentanone ring and the furan rings are alomst coplanar. All bond lengths and angles are normal and correspond to those observed in the related substituted cyclopentanone and cyclohexanone analogues reported by Du *et al.* (2007), Sun & Cui (2007) and Wei *et al.* (2008). The crystal packing exhibits weak intermolecular C—H···O hydrogen bonds (Table 1).

S2. Experimental

Tetrabutylammonium bromide (0.3 mmol) and NaOH (5 mmol) were dissolved in the mixture of water (5 ml) and ethanol (2 ml). The solution was stirred at room temperature for 10 min,followed by added dropwise the mixture of furaldehyde (10 mmol) and cyclopentanone (5 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. Analysis calculated for $C_{15}H_{12}O_3$: C 75.00,H 5.00%; found: C 74.90,H 5.03%. Crystals of (I) suitable for single-crystal X-ray analysis were selected after recrystallization.

S3. 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).

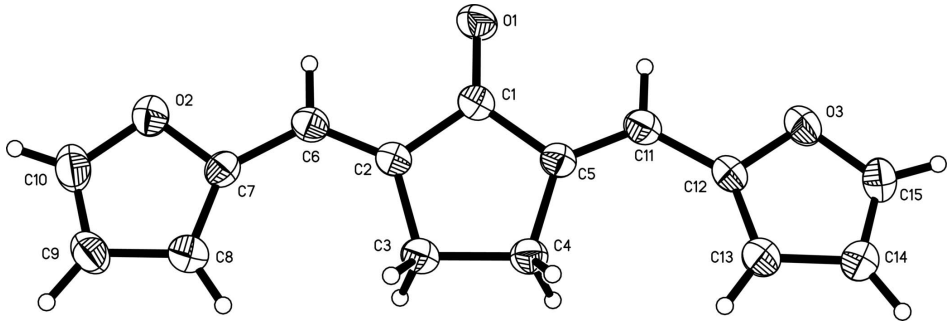

Figure 1

The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level.

(2*E*,5*E*)-2,5-Difurfurylidenecyclopentanone

Crystal data

$C_{15}H_{12}O_3$
$M_r = 240.25$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 5.9280$ (9) Å
$b = 8.5031$ (13) Å
$c = 23.280$ (3) Å
$\beta = 92.139$ (3)°
$V = 1172.6$ (3) Å^3
$Z = 4$
$F(000) = 504$
$D_x = 1.361$ Mg m^{-3}
Melting point: 405 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 461 reflections
$\theta = 3.0$–$18.7°$
$\mu = 0.10$ mm^{-1}
$T = 298$ K
Block, yellow
0.12 × 0.08 × 0.05 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.989$, $T_{max} = 0.995$
5793 measured reflections
2076 independent reflections
1083 reflections with $I > 2\sigma(I)$
$R_{int} = 0.059$
$\theta_{max} = 25.1°$, $\theta_{min} = 1.8°$
$h = -7 \rightarrow 7$
$k = -10 \rightarrow 9$
$l = -27 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.129$
$S = 0.99$
2076 reflections
164 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
$w = 1/[\sigma^2(F_o^2) + (0.049P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.015 (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_{iso}*/U_{eq}
O1	1.2374 (3)	0.2225 (2)	0.62045 (7)	0.0606 (6)
O2	1.3568 (3)	0.4626 (2)	0.42876 (8)	0.0648 (6)

O3	0.7570 (3)	−0.1375 (2)	0.73472 (8)	0.0640 (6)
C1	1.0690 (5)	0.1894 (3)	0.58984 (11)	0.0443 (7)
C2	1.0293 (4)	0.2376 (3)	0.52998 (10)	0.0416 (7)
C3	0.8083 (4)	0.1745 (3)	0.50780 (10)	0.0496 (7)
H3A	0.8296	0.1048	0.4755	0.059*
H3B	0.7091	0.2597	0.4954	0.059*
C4	0.7065 (4)	0.0834 (3)	0.55845 (10)	0.0487 (7)
H4A	0.5655	0.1311	0.5692	0.058*
H4B	0.6780	−0.0252	0.5478	0.058*
C5	0.8774 (4)	0.0925 (3)	0.60707 (11)	0.0430 (7)
C6	1.1804 (4)	0.3239 (3)	0.50282 (11)	0.0476 (7)
H6	1.3098	0.3508	0.5244	0.057*
C7	1.1698 (5)	0.3802 (3)	0.44512 (12)	0.0459 (7)
C8	1.0169 (5)	0.3795 (3)	0.40086 (12)	0.0569 (8)
H8	0.8755	0.3320	0.4004	0.068*
C9	1.1100 (6)	0.4640 (3)	0.35521 (12)	0.0652 (9)
H9	1.0427	0.4831	0.3192	0.078*
C10	1.3128 (6)	0.5103 (4)	0.37419 (13)	0.0704 (10)
H10	1.4122	0.5684	0.3526	0.084*
C11	0.8754 (4)	0.0258 (3)	0.65893 (11)	0.0499 (7)
H11	1.0011	0.0448	0.6830	0.060*
C12	0.7047 (5)	−0.0711 (3)	0.68205 (11)	0.0465 (7)
C13	0.4940 (5)	−0.1134 (3)	0.66577 (12)	0.0579 (8)
H13	0.4170	−0.0844	0.6319	0.069*
C14	0.4101 (5)	−0.2101 (4)	0.70957 (13)	0.0657 (9)
H14	0.2682	−0.2566	0.7101	0.079*
C15	0.5736 (5)	−0.2212 (4)	0.74968 (13)	0.0642 (9)
H15	0.5640	−0.2786	0.7835	0.077*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0480 (12)	0.0831 (16)	0.0497 (12)	−0.0054 (11)	−0.0089 (9)	0.0018 (10)
O2	0.0632 (13)	0.0725 (15)	0.0587 (14)	−0.0127 (11)	0.0024 (10)	0.0110 (11)
O3	0.0670 (14)	0.0815 (16)	0.0430 (12)	−0.0039 (12)	−0.0042 (10)	0.0100 (10)
C1	0.0404 (16)	0.0489 (18)	0.0437 (17)	0.0062 (14)	0.0016 (13)	−0.0033 (13)
C2	0.0419 (16)	0.0414 (17)	0.0413 (16)	0.0049 (13)	0.0006 (12)	−0.0041 (12)
C3	0.0478 (17)	0.0550 (19)	0.0456 (17)	0.0013 (14)	−0.0021 (13)	−0.0022 (13)
C4	0.0449 (17)	0.0526 (18)	0.0484 (17)	−0.0014 (14)	−0.0006 (13)	−0.0019 (13)
C5	0.0422 (16)	0.0451 (17)	0.0417 (17)	0.0039 (13)	0.0022 (13)	−0.0042 (13)
C6	0.0472 (17)	0.0489 (19)	0.0466 (17)	0.0040 (14)	−0.0018 (13)	−0.0021 (13)
C7	0.0457 (17)	0.0408 (17)	0.0515 (18)	−0.0017 (13)	0.0048 (14)	−0.0021 (13)
C8	0.059 (2)	0.059 (2)	0.0522 (19)	−0.0027 (15)	−0.0028 (16)	−0.0025 (15)
C9	0.090 (3)	0.062 (2)	0.0430 (19)	0.0013 (18)	−0.0044 (17)	0.0024 (16)
C10	0.094 (3)	0.064 (2)	0.054 (2)	−0.0035 (19)	0.0106 (19)	0.0113 (17)
C11	0.0482 (17)	0.058 (2)	0.0435 (17)	0.0025 (14)	−0.0019 (13)	−0.0027 (14)
C12	0.0539 (19)	0.0477 (18)	0.0377 (16)	0.0054 (15)	−0.0009 (13)	0.0013 (13)
C13	0.058 (2)	0.066 (2)	0.0493 (18)	−0.0032 (16)	−0.0052 (15)	0.0082 (15)

C14	0.061 (2)	0.078 (2)	0.058 (2)	−0.0164 (17)	0.0007 (17)	0.0083 (17)
C15	0.072 (2)	0.071 (2)	0.050 (2)	−0.0090 (19)	0.0120 (17)	0.0091 (16)

Geometric parameters (Å, º)

O1—C1	1.237 (3)	C6—C7	1.425 (3)
O2—C10	1.350 (3)	C6—H6	0.9300
O2—C7	1.377 (3)	C7—C8	1.347 (3)
O3—C15	1.356 (3)	C8—C9	1.412 (4)
O3—C12	1.375 (3)	C8—H8	0.9300
C1—C2	1.463 (3)	C9—C10	1.326 (4)
C1—C5	1.471 (3)	C9—H9	0.9300
C2—C6	1.335 (3)	C10—H10	0.9300
C2—C3	1.490 (3)	C11—C12	1.426 (4)
C3—C4	1.552 (3)	C11—H11	0.9300
C3—H3A	0.9700	C12—C13	1.341 (3)
C3—H3B	0.9700	C13—C14	1.415 (4)
C4—C5	1.493 (3)	C13—H13	0.9300
C4—H4A	0.9700	C14—C15	1.324 (3)
C4—H4B	0.9700	C14—H14	0.9300
C5—C11	1.335 (3)	C15—H15	0.9300
C10—O2—C7	106.5 (2)	C8—C7—C6	136.4 (3)
C15—O3—C12	106.8 (2)	O2—C7—C6	115.1 (2)
O1—C1—C2	125.6 (3)	C7—C8—C9	107.6 (3)
O1—C1—C5	125.8 (2)	C7—C8—H8	126.2
C2—C1—C5	108.6 (2)	C9—C8—H8	126.2
C6—C2—C1	121.2 (2)	C10—C9—C8	106.0 (3)
C6—C2—C3	129.1 (2)	C10—C9—H9	127.0
C1—C2—C3	109.7 (2)	C8—C9—H9	127.0
C2—C3—C4	106.2 (2)	C9—C10—O2	111.4 (3)
C2—C3—H3A	110.5	C9—C10—H10	124.3
C4—C3—H3A	110.5	O2—C10—H10	124.3
C2—C3—H3B	110.5	C5—C11—C12	128.1 (2)
C4—C3—H3B	110.5	C5—C11—H11	115.9
H3A—C3—H3B	108.7	C12—C11—H11	115.9
C5—C4—C3	106.2 (2)	C13—C12—O3	108.6 (2)
C5—C4—H4A	110.5	C13—C12—C11	135.6 (3)
C3—C4—H4A	110.5	O3—C12—C11	115.7 (2)
C5—C4—H4B	110.5	C12—C13—C14	107.5 (3)
C3—C4—H4B	110.5	C12—C13—H13	126.2
H4A—C4—H4B	108.7	C14—C13—H13	126.2
C11—C5—C1	121.2 (2)	C15—C14—C13	106.4 (3)
C11—C5—C4	129.4 (2)	C15—C14—H14	126.8
C1—C5—C4	109.3 (2)	C13—C14—H14	126.8
C2—C6—C7	128.6 (2)	C14—C15—O3	110.8 (3)
C2—C6—H6	115.7	C14—C15—H15	124.6
C7—C6—H6	115.7	O3—C15—H15	124.6

C8—C7—O2	108.4 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C15—H15···O1^{i}	0.93	2.49	3.221 (4)	136
C4—H4*A*···O1^{ii}	0.97	2.45	3.393 (3)	165

Symmetry codes: (i) −*x*+2, *y*−1/2, −*z*+3/2; (ii) *x*−1, *y*, *z*.