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(1*E*,4*E*)-1,5-Bis(2,4-dimethylphenyl)-penta-1,4-dien-3-one

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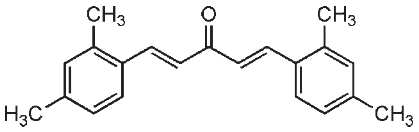
Key indicators: single-crystal X-ray study; *T* = 293 K; mean σ(C–C) = 0.003 Å; *R* factor = 0.065; *wR* factor = 0.173; data-to-parameter ratio = 15.4.

In the title compound, $C_{21}H_{22}O$, a derivative of the biologically active compound curcumin, the dihedral angle between the aromatic ring planes is 20.57 (11)°.

Related literature

For backgound to cucucmin and its biological properties, see: Began *et al.* (1999); Gautam *et al.* (2007); Liang *et al.* (2008); Liang, Shao *et al.* (2009); Liang, Tian *et al.* (2007); Liang, Yang *et al.* (2007); Liang, Zhou *et al.* (2009); Maheshwari *et al.* (2006); Zhao *et al.* (2009).



Experimental

Crystal data

$C_{21}H_{22}O$
$M_r = 290.39$
Monoclinic, $P2_1/c$
$a = 4.9548$ (7) Å
$b = 26.555$ (4) Å
$c = 12.9632$ (19) Å
$\beta = 94.090$ (3)°
$V = 1701.3$ (4) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.07$ mm^{-1}
$T = 293$ K
0.42 × 0.37 × 0.26 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.769$, $T_{max} = 1.000$
8842 measured reflections
3128 independent reflections
2007 reflections with $I > 2\sigma(I)$
$R_{int} = 0.095$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.173$
$S = 0.97$
3128 reflections
203 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); 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: HB5050).

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(1*E*,4*E*)-1,5-Bis(2,4-dimethylphenyl)penta-1,4-dien-3-one

Zhiguo Feng, Junhua Li and Yi Lin

S1. Comment

The title compound, $C_{21}H_{22}O$, (1E,4E)-1,5-bis(2,4-dimethylphenyl)penta-1,4-dien-3-one (I), is a mono-carbonyl analogue of curcumin. Curcumin has been found to possess a variety of pharmaceutical applications, for example, inhibiting the mutations and the formation of tumors, antioxidation, anti-inflammation and anti-virus (Began *et al.*, 1999; Maheshwari *et al.*, 2006; Gautam *et al.*, 2007). According to the structural disadvantages of curcumin which is considered to be responsible for its weak pharmacokinetic profiles, a series of mono-carbonyl analogues have been designed and synthesized, and their biological activity *in vitro* and *in vivo* were evaluated.

Derivatives of dibenzylidene acetone, cyclopentanone and cyclohexanone exhibit potent anti-inflammatory, antibacterial and anti-cancer activity.(Liang *et al.*, 2008; Liang *et al.*, 2008; Liang, Shao *et al.*, 2009; Liang, Zhou *et al.*, 2009). This fact leads to the significance of these synthetic mono-carbonyl analogues of curcumin. Several of these derivatives have been reported their crystal structures (Liang, Tian *et al.*, 2007; Liang, Yang *et al.*, 2007; Zhao *et al.*, 2009). In the present paper, we describe the crystal structure of the title compound $C_{21}H_{22}O$ (I) here. Its geometrical parameters of are normal, the dihedral angle between the six-membered aromatic ring planes is 20.57 (11)°.

S2. Experimental

To a solution of 15 mmol 2,4-dimethylbenzaldehyde in MeOH (10 ml) was added 7.5 mmol acetone. The solution was stirred at room temperature for 20 min, followed by added dropwise 20% (*w*/*v*) NaOH (1.5 ml, 7.5 mmol). The mixture was stirred at RT and monitored with TLC. When the reaction finished, the residue was poured into saturated NH_4Cl solution and filtered. The precipitate was washed and purified by chromatography over silica gel using CH_2Cl_2 / CH_3OH as the eluent to afford the pure product (yield: 38.2%). Colourless blokcs of (I) were grown in a $CH_2Cl_2—CH_3OH$ mixture (6:2 *v*/*v*) by slow evaporation (mp 436–437 K). 1*H*-NMR ($CDCl_3$): 2.34 (s, 6H, $Ar_4—CH_3$), 2.44 (s, 6H, $Ar_2—CH_3$), 6.96 (d, 2H, J=16.0 Hz, =CH—C=O), 7.05 (m, 4H, $Ar—H_{3,5}$), 7.56 (d, 2H, J=8.0 Hz, $Ar—H_6$), 8.0 (d, 2H, J=16.0 Hz, Ar—CH=C). ESI-MS m/*z*: 603.8 (2*M*+Na)+, calcd for $C_{21}H_{22}O$: 290.4.

S3. Refinement

The H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C).

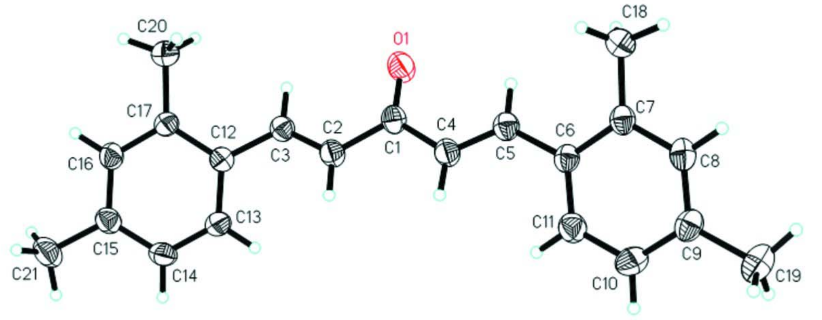

Figure 1

The molecular structure of (I) shoiwng 50% displacement ellipsoids for the non-hydrogen atoms.

(1*E*,4*E*)-1,5-Bis(2,4-dimethylphenyl)penta-1,4-dien-3-one

Crystal data

$C_{21}H_{22}O$
$M_r = 290.39$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 4.9548\ (7)$ Å
$b = 26.555\ (4)$ Å
$c = 12.9632\ (19)$ Å
$\beta = 94.090\ (3)°$
$V = 1701.3\ (4)$ Å^3
$Z = 4$

$F(000) = 624$
$D_x = 1.134$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1845 reflections
$\theta = 4.4–44.1°$
$\mu = 0.07$ mm^{-1}
$T = 293$ K
Prism, green
0.42 × 0.37 × 0.26 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{\min} = 0.769$, $T_{\max} = 1.000$

8842 measured reflections
3128 independent reflections
2007 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.095$
$\theta_{\max} = 25.5°$, $\theta_{\min} = 1.5°$
$h = -5 \rightarrow 6$
$k = -31 \rightarrow 32$
$l = -7 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.173$
$S = 0.97$
3128 reflections
203 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
$w = 1/[\sigma^2(F_o^2) + (0.0858P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.057$
$\Delta\rho_{\max} = 0.21$ 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*	$U_{\rm iso}$*/$U_{\rm eq}$
O1	1.4353 (4)	0.35616 (7)	0.08357 (12)	0.0700 (5)
C1	1.2047 (5)	0.36963 (8)	0.09662 (16)	0.0510 (6)
C2	1.0758 (5)	0.35782 (8)	0.19171 (16)	0.0528 (6)
H2	0.9031	0.3703	0.1994	0.063*
C3	1.1920 (4)	0.33037 (8)	0.26714 (16)	0.0479 (6)
H3	1.3621	0.3173	0.2566	0.057*
C4	1.0435 (5)	0.39838 (8)	0.01739 (16)	0.0545 (6)
H4	0.8698	0.4083	0.0313	0.065*
C5	1.1329 (5)	0.41070 (8)	−0.07216 (16)	0.0516 (6)
H5	1.3019	0.3982	−0.0860	0.062*
C6	0.9942 (4)	0.44212 (7)	−0.15263 (16)	0.0473 (6)
C7	1.0565 (4)	0.43873 (7)	−0.25633 (16)	0.0493 (6)
C8	0.9237 (5)	0.47084 (8)	−0.32771 (17)	0.0557 (6)
H8	0.9633	0.4684	−0.3966	0.067*
C9	0.7367 (5)	0.50614 (8)	−0.30192 (18)	0.0570 (6)
C10	0.6759 (5)	0.50852 (9)	−0.19985 (19)	0.0645 (7)
H10	0.5482	0.5316	−0.1801	0.077*
C11	0.8023 (5)	0.47709 (9)	−0.12683 (18)	0.0603 (6)
H11	0.7577	0.4794	−0.0585	0.072*
C12	1.0812 (4)	0.31833 (7)	0.36552 (15)	0.0455 (5)
C13	0.8956 (5)	0.34978 (8)	0.40688 (17)	0.0564 (6)
H13	0.8429	0.3789	0.3710	0.068*
C14	0.7855 (5)	0.33986 (9)	0.49876 (18)	0.0614 (7)
H14	0.6607	0.3620	0.5239	0.074*
C15	0.8597 (5)	0.29692 (9)	0.55428 (16)	0.0544 (6)
C16	1.0474 (4)	0.26532 (9)	0.51378 (16)	0.0535 (6)
H16	1.0998	0.2365	0.5506	0.064*
C17	1.1613 (4)	0.27453 (8)	0.42090 (15)	0.0466 (5)
C18	1.2581 (5)	0.40116 (8)	−0.29188 (17)	0.0626 (7)
H18A	1.2595	0.4025	−0.3658	0.094*
H18B	1.2081	0.3679	−0.2712	0.094*
H18C	1.4350	0.4091	−0.2613	0.094*
C19	0.5986 (6)	0.54084 (9)	−0.3819 (2)	0.0795 (8)
H19A	0.6863	0.5383	−0.4453	0.119*
H19B	0.6098	0.5749	−0.3572	0.119*

H19C	0.4120	0.5313	−0.3938	0.119*
C20	1.3587 (5)	0.23772 (9)	0.38199 (18)	0.0640 (7)
H20A	1.3890	0.2110	0.4313	0.096*
H20B	1.5267	0.2545	0.3725	0.096*
H20C	1.2871	0.2240	0.3172	0.096*
C21	0.7377 (5)	0.28438 (12)	0.65406 (18)	0.0784 (8)
H21A	0.6361	0.3127	0.6761	0.118*
H21B	0.8792	0.2765	0.7060	0.118*
H21C	0.6198	0.2559	0.6438	0.118*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0565 (11)	0.0924 (13)	0.0619 (11)	0.0062 (9)	0.0100 (9)	0.0172 (9)
C1	0.0505 (14)	0.0541 (13)	0.0482 (13)	−0.0077 (11)	0.0023 (11)	0.0032 (10)
C2	0.0491 (13)	0.0618 (14)	0.0476 (13)	−0.0010 (11)	0.0044 (11)	0.0058 (11)
C3	0.0476 (13)	0.0479 (12)	0.0482 (13)	−0.0034 (10)	0.0043 (10)	−0.0009 (10)
C4	0.0522 (14)	0.0625 (14)	0.0492 (13)	0.0007 (11)	0.0061 (11)	0.0072 (11)
C5	0.0558 (14)	0.0491 (13)	0.0498 (13)	−0.0050 (10)	0.0036 (11)	−0.0002 (10)
C6	0.0546 (14)	0.0411 (11)	0.0459 (13)	−0.0053 (10)	0.0016 (10)	−0.0001 (9)
C7	0.0548 (13)	0.0456 (12)	0.0474 (13)	−0.0087 (10)	0.0037 (11)	0.0022 (10)
C8	0.0668 (16)	0.0528 (13)	0.0474 (13)	−0.0088 (12)	0.0034 (11)	0.0045 (11)
C9	0.0682 (16)	0.0427 (12)	0.0588 (15)	−0.0048 (12)	−0.0049 (12)	0.0009 (11)
C10	0.0678 (17)	0.0532 (14)	0.0716 (17)	0.0092 (12)	−0.0008 (13)	−0.0069 (13)
C11	0.0715 (17)	0.0579 (14)	0.0520 (14)	−0.0001 (12)	0.0084 (12)	−0.0018 (11)
C12	0.0447 (12)	0.0481 (12)	0.0435 (12)	−0.0012 (10)	0.0020 (10)	−0.0009 (10)
C13	0.0607 (15)	0.0549 (13)	0.0539 (14)	0.0141 (11)	0.0074 (12)	0.0058 (11)
C14	0.0598 (15)	0.0696 (16)	0.0564 (15)	0.0172 (13)	0.0156 (12)	0.0002 (12)
C15	0.0517 (14)	0.0657 (15)	0.0461 (13)	0.0025 (12)	0.0056 (11)	0.0027 (11)
C16	0.0531 (14)	0.0586 (14)	0.0487 (13)	0.0035 (11)	0.0031 (11)	0.0115 (11)
C17	0.0455 (12)	0.0462 (12)	0.0479 (13)	0.0023 (10)	0.0015 (10)	0.0012 (10)
C18	0.0700 (16)	0.0639 (15)	0.0549 (14)	0.0062 (13)	0.0110 (12)	−0.0017 (12)
C19	0.095 (2)	0.0616 (16)	0.0795 (19)	0.0047 (14)	−0.0140 (16)	0.0103 (14)
C20	0.0720 (17)	0.0580 (14)	0.0632 (15)	0.0108 (13)	0.0140 (13)	0.0003 (12)
C21	0.0716 (18)	0.107 (2)	0.0586 (16)	0.0085 (16)	0.0199 (13)	0.0149 (15)

Geometric parameters (Å, º)

O1—C1	1.221 (3)	C12—C17	1.409 (3)
C1—C2	1.462 (3)	C13—C14	1.370 (3)
C1—C4	1.469 (3)	C13—H13	0.9300
C2—C3	1.319 (3)	C14—C15	1.384 (3)
C2—H2	0.9300	C14—H14	0.9300
C3—C12	1.459 (3)	C15—C16	1.383 (3)
C3—H3	0.9300	C15—C21	1.504 (3)
C4—C5	1.313 (3)	C16—C17	1.388 (3)
C4—H4	0.9300	C16—H16	0.9300
C5—C6	1.468 (3)	C17—C20	1.496 (3)

C5—H5	0.9300	C18—H18A	0.9600
C6—C11	1.387 (3)	C18—H18B	0.9600
C6—C7	1.403 (3)	C18—H18C	0.9600
C7—C8	1.389 (3)	C19—H19A	0.9600
C7—C18	1.507 (3)	C19—H19B	0.9600
C8—C9	1.376 (3)	C19—H19C	0.9600
C8—H8	0.9300	C20—H20A	0.9600
C9—C10	1.379 (3)	C20—H20B	0.9600
C9—C19	1.513 (3)	C20—H20C	0.9600
C10—C11	1.379 (3)	C21—H21A	0.9601
C10—H10	0.9300	C21—H21B	0.9601
C11—H11	0.9300	C21—H21C	0.9601
C12—C13	1.379 (3)		

O1—C1—C2	121.6 (2)	C12—C13—H13	118.7
O1—C1—C4	121.5 (2)	C13—C14—C15	120.2 (2)
C2—C1—C4	116.9 (2)	C13—C14—H14	119.9
C3—C2—C1	123.4 (2)	C15—C14—H14	119.9
C3—C2—H2	118.3	C14—C15—C16	117.6 (2)
C1—C2—H2	118.3	C14—C15—C21	121.4 (2)
C2—C3—C12	126.7 (2)	C16—C15—C21	121.0 (2)
C2—C3—H3	116.6	C15—C16—C17	123.3 (2)
C12—C3—H3	116.7	C15—C16—H16	118.4
C5—C4—C1	123.1 (2)	C17—C16—H16	118.4
C5—C4—H4	118.4	C16—C17—C12	118.1 (2)
C1—C4—H4	118.4	C16—C17—C20	119.59 (19)
C4—C5—C6	126.8 (2)	C12—C17—C20	122.3 (2)
C4—C5—H5	116.6	C7—C18—H18A	109.5
C6—C5—H5	116.6	C7—C18—H18B	109.5
C11—C6—C7	118.4 (2)	H18A—C18—H18B	109.5
C11—C6—C5	120.2 (2)	C7—C18—H18C	109.5
C7—C6—C5	121.4 (2)	H18A—C18—H18C	109.5
C8—C7—C6	118.2 (2)	H18B—C18—H18C	109.5
C8—C7—C18	119.7 (2)	C9—C19—H19A	109.5
C6—C7—C18	122.09 (19)	C9—C19—H19B	109.5
C9—C8—C7	123.5 (2)	H19A—C19—H19B	109.5
C9—C8—H8	118.3	C9—C19—H19C	109.5
C7—C8—H8	118.3	H19A—C19—H19C	109.5
C8—C9—C10	117.5 (2)	H19B—C19—H19C	109.5
C8—C9—C19	121.9 (2)	C17—C20—H20A	109.5
C10—C9—C19	120.7 (2)	C17—C20—H20B	109.5
C11—C10—C9	120.8 (2)	H20A—C20—H20B	109.5
C11—C10—H10	119.6	C17—C20—H20C	109.5
C9—C10—H10	119.6	H20A—C20—H20C	109.5
C10—C11—C6	121.7 (2)	H20B—C20—H20C	109.5
C10—C11—H11	119.2	C15—C21—H21A	109.5
C6—C11—H11	119.1	C15—C21—H21B	109.5
C13—C12—C17	118.2 (2)	H21A—C21—H21B	109.5

C13—C12—C3	120.67 (19)	C15—C21—H21C	109.5
C17—C12—C3	121.08 (19)	H21A—C21—H21C	109.5
C14—C13—C12	122.6 (2)	H21B—C21—H21C	109.5
C14—C13—H13	118.7		

O1—C1—C2—C3	−3.1 (3)	C9—C10—C11—C6	0.1 (4)
C4—C1—C2—C3	176.8 (2)	C7—C6—C11—C10	−0.8 (3)
C1—C2—C3—C12	177.8 (2)	C5—C6—C11—C10	177.5 (2)
O1—C1—C4—C5	1.1 (4)	C2—C3—C12—C13	−26.1 (3)
C2—C1—C4—C5	−178.8 (2)	C2—C3—C12—C17	154.2 (2)
C1—C4—C5—C6	−175.49 (19)	C17—C12—C13—C14	−0.5 (3)
C4—C5—C6—C11	24.6 (3)	C3—C12—C13—C14	179.7 (2)
C4—C5—C6—C7	−157.2 (2)	C12—C13—C14—C15	0.1 (4)
C11—C6—C7—C8	0.4 (3)	C13—C14—C15—C16	0.4 (4)
C5—C6—C7—C8	−177.83 (18)	C13—C14—C15—C21	−178.5 (2)
C11—C6—C7—C18	−178.7 (2)	C14—C15—C16—C17	−0.4 (3)
C5—C6—C7—C18	3.0 (3)	C21—C15—C16—C17	178.4 (2)
C6—C7—C8—C9	0.7 (3)	C15—C16—C17—C12	0.1 (3)
C18—C7—C8—C9	179.9 (2)	C15—C16—C17—C20	−178.8 (2)
C7—C8—C9—C10	−1.4 (3)	C13—C12—C17—C16	0.4 (3)
C7—C8—C9—C19	179.3 (2)	C3—C12—C17—C16	−179.83 (19)
C8—C9—C10—C11	1.0 (4)	C13—C12—C17—C20	179.2 (2)
C19—C9—C10—C11	−179.7 (2)	C3—C12—C17—C20	−1.0 (3)