Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-1-(2-Hydroxy-4,6-dimethoxyphenyl)-3-(4-methoxyphenyl)prop-2-en-1-one from *Kaempferia rotunda* Val.

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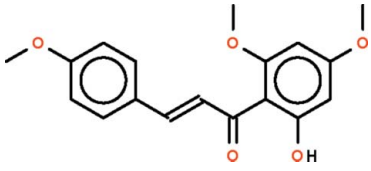
Received 20 October 2010; accepted 20 October 2010

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.101; data-to-parameter ratio = 16.1.

The planar –CH=CHC(=O)– fragment (r.m.s. deviation = 0.074 Å) in the title compound, $C_{18}H_{18}O_5$, connects the planar hydroxydimethoxyphenyl (r.m.s. deviation = 0.039 Å) and methoxylphenyl (r.m.s. deviation = 0.021 Å) parts. The central fragment forms a dihedral angle of 13.7 (1)° with the hydroxydimethoxyphenyl part and 32.0 (1)° with the methoxyphenyl part. The hydroxy group forms an intramolecular hydrogen bond to the carbonyl O atom.

Related literature

For the isolation of the compound from *Kaempferia rotunda*, see: Sirat *et al.* (2001); Stevenson *et al.* (2007).



Experimental

Crystal data

$C_{18}H_{18}O_5$
M_r = 314.32
Monoclinic, $P2_1/n$
a = 12.8502 (8) Å
b = 8.3226 (5) Å
c = 14.1865 (9) Å
β = 97.765 (1)°
V = 1503.29 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100 K
0.45 × 0.40 × 0.05 mm

Data collection

Bruker SMART APEX diffractometer
13992 measured reflections
3464 independent reflections
2989 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.101
S = 1.01
3464 reflections
215 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O4	0.87 (1)	1.65 (1)	2.465 (1)	156 (2)

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); 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, 2010).

We thank the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5385).

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supporting information

Acta Cryst. (2010). E**66**, o2944 [https://doi.org/10.1107/S1600536810042674]

(*E*)-1-(2-Hydroxy-4,6-dimethoxyphenyl)-3-(4-methoxyphenyl)prop-2-en-1-one from *Kaempferia rotunda* Val.

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S1. Comment

Kaempferia rotunda is one of the four Malaysian *Kaempferia* of the Zingiberaceae family; among the constitutents isolated is 1-(2-hydroxy-4,6-dimethoxyphenyl)-3-(4-methoxyphenyl)-2-propen-1-one (Scheme I) (Sirat *et al.*, 2001; Stevenson *et al.*, 2007). The planar –CH═CHC(═O)– fragment (r.m.s. deviation 0.074 Å) connects the planar hydroxyldimethoxyphenyl (r.m.s. deviation 0.039 Å) and methoxylphenyl (r.m.s. deviation 0.021 Å) parts. The fragment is aligned at 13.7 (1) ° with the hydroxydimethoxyphenyl part and at 32.0 (1) ° with the methoxyphenyl part. The hydroxy group forms an intramolecular hydrogen bond to the carbonyl oxygen atom of the fragment (Fig. 1).

S2. Experimental

Kaempferia rotunda rhizomes were purchased from a market in Kempas, Johor. The rhizomes were dried and then grounded. The grounded rhizomes were extracted with *n*-hexane (4.5 *L*), ethyl acetate (4.5 *L*) and methanol (4.5 *L*) in a soxhlet extractor for 16 h. The extracts were concentrated to give a dark brown semi-solid from the *n*-hexane extract (2.32 g), a dark brown oil from the ethyl acetate extract (6.80 g) and a dark brown viscous liquid from the methanol extract (15.27 g). The *n*-hexane extract was purified by column chromatography (93.0 g, column size: 4.5 × 45.0 cm) with *n*-hexane:ether (99:1, 98:2 and 97:3) as eluents to give 20 fractions. Fractions 13 (0.08 g) and 14 (0.02 g) were combined and recrystallized by from an *n*-hexane: ether mixture to afford the title compound (6.9 mg) as yellow crystals.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95–0.98 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2–15*U*(C).

The hydroxy H-atom was located in a difference Fourier map, and was refined isotropically with the O–H distance restrained to 0.84±0.01 Å.

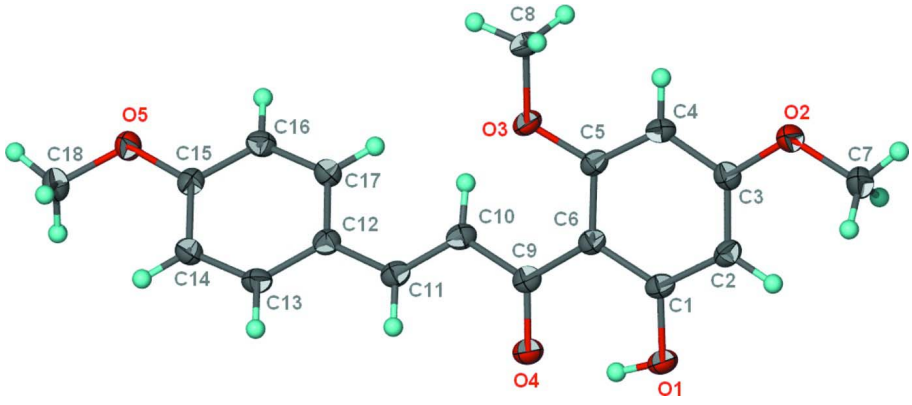

Figure 1

Anisotropic displacement ellipsoid plot (Barbour, 2001) of $C_{18}H_{18}O_5$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

(*E*)-1-(2-Hydroxy-4,6-dimethoxyphenyl)-3-(4-methoxyphenyl)prop-2-en-1-one

Crystal data

$C_{18}H_{18}O_5$
$M_r = 314.32$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 12.8502$ (8) Å
$b = 8.3226$ (5) Å
$c = 14.1865$ (9) Å
$\beta = 97.765$ (1)°
$V = 1503.29$ (16) Å^3
$Z = 4$
$F(000) = 664$
$D_x = 1.389$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5893 reflections
$\theta = 2.3$–$28.2°$
$\mu = 0.10$ mm^{-1}
$T = 100$ K
Irregular block, yellow
0.45 × 0.40 × 0.05 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
13992 measured reflections
3464 independent reflections
2989 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.0°$
$h = -16 \rightarrow 15$
$k = -10 \rightarrow 10$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.101$
$S = 1.01$
3464 reflections
215 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0545P)^2 + 0.5468P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.71889 (6)	0.58291 (10)	0.67517 (6)	0.02051 (19)
H1	0.7343 (14)	0.536 (2)	0.6242 (9)	0.049 (5)*
O2	0.44911 (7)	0.93407 (10)	0.74558 (6)	0.0224 (2)
O3	0.45229 (6)	0.75473 (10)	0.42926 (5)	0.01955 (19)
O4	0.72544 (6)	0.50207 (10)	0.50890 (6)	0.02089 (19)
O5	0.61863 (7)	0.54719 (10)	−0.08390 (5)	0.01985 (19)
C1	0.63266 (9)	0.66965 (13)	0.64416 (8)	0.0166 (2)
C2	0.58866 (9)	0.75693 (13)	0.71251 (8)	0.0181 (2)
H2	0.6199	0.7560	0.7771	0.022*
C3	0.49855 (9)	0.84502 (13)	0.68446 (8)	0.0176 (2)
C4	0.45027 (9)	0.84618 (14)	0.58972 (8)	0.0177 (2)
H4	0.3869	0.9041	0.5724	0.021*
C5	0.49567 (9)	0.76227 (13)	0.52174 (8)	0.0163 (2)
C6	0.59130 (8)	0.67424 (13)	0.54579 (7)	0.0160 (2)
C7	0.49545 (11)	0.93606 (16)	0.84345 (8)	0.0257 (3)
H7A	0.4487	0.9924	0.8815	0.039*
H7B	0.5632	0.9917	0.8491	0.039*
H7C	0.5061	0.8255	0.8665	0.039*
C8	0.35464 (9)	0.83628 (15)	0.40224 (8)	0.0210 (2)
H8A	0.3332	0.8236	0.3337	0.032*
H8B	0.3632	0.9507	0.4176	0.032*
H8C	0.3008	0.7903	0.4370	0.032*
C9	0.64968 (9)	0.59271 (13)	0.47819 (8)	0.0170 (2)
C10	0.62915 (9)	0.61886 (14)	0.37466 (8)	0.0180 (2)
H10	0.5914	0.7110	0.3497	0.022*
C11	0.66420 (9)	0.51130 (14)	0.31598 (8)	0.0179 (2)
H11	0.6976	0.4182	0.3446	0.022*
C12	0.65630 (8)	0.52262 (14)	0.21240 (8)	0.0170 (2)
C13	0.67036 (9)	0.38511 (14)	0.15961 (8)	0.0194 (2)
H13	0.6880	0.2870	0.1922	0.023*
C14	0.65939 (9)	0.38694 (14)	0.06058 (8)	0.0191 (2)
H14	0.6686	0.2912	0.0262	0.023*
C15	0.63480 (8)	0.53098 (14)	0.01267 (8)	0.0166 (2)
C16	0.62576 (9)	0.67210 (14)	0.06464 (8)	0.0192 (2)
H16	0.6127	0.7714	0.0322	0.023*
C17	0.63562 (9)	0.66808 (14)	0.16271 (8)	0.0183 (2)
H17	0.6284	0.7645	0.1971	0.022*
C18	0.63148 (10)	0.40641 (15)	−0.13935 (8)	0.0219 (2)
H18A	0.6141	0.4319	−0.2071	0.033*
H18B	0.5847	0.3215	−0.1222	0.033*
H18C	0.7045	0.3696	−0.1267	0.033*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0192 (4)	0.0242 (4)	0.0175 (4)	0.0058 (3)	0.0000 (3)	0.0029 (3)
O2	0.0276 (5)	0.0221 (4)	0.0174 (4)	0.0066 (3)	0.0027 (3)	−0.0003 (3)
O3	0.0168 (4)	0.0253 (4)	0.0156 (4)	0.0041 (3)	−0.0011 (3)	0.0016 (3)
O4	0.0204 (4)	0.0229 (4)	0.0191 (4)	0.0052 (3)	0.0017 (3)	0.0024 (3)
O5	0.0246 (4)	0.0197 (4)	0.0156 (4)	0.0016 (3)	0.0042 (3)	0.0000 (3)
C1	0.0150 (5)	0.0154 (5)	0.0188 (5)	−0.0015 (4)	0.0007 (4)	0.0041 (4)
C2	0.0200 (6)	0.0183 (5)	0.0152 (5)	−0.0012 (4)	−0.0001 (4)	0.0025 (4)
C3	0.0200 (5)	0.0147 (5)	0.0185 (5)	−0.0016 (4)	0.0044 (4)	0.0006 (4)
C4	0.0166 (5)	0.0172 (5)	0.0191 (5)	0.0017 (4)	0.0018 (4)	0.0037 (4)
C5	0.0168 (5)	0.0159 (5)	0.0156 (5)	−0.0025 (4)	0.0002 (4)	0.0032 (4)
C6	0.0160 (5)	0.0157 (5)	0.0160 (5)	−0.0013 (4)	0.0016 (4)	0.0023 (4)
C7	0.0348 (7)	0.0229 (6)	0.0188 (6)	0.0044 (5)	0.0013 (5)	−0.0027 (5)
C8	0.0170 (5)	0.0242 (6)	0.0207 (5)	0.0033 (4)	−0.0019 (4)	0.0034 (4)
C9	0.0161 (5)	0.0156 (5)	0.0189 (5)	−0.0026 (4)	0.0013 (4)	0.0021 (4)
C10	0.0162 (5)	0.0194 (5)	0.0184 (5)	0.0002 (4)	0.0023 (4)	0.0038 (4)
C11	0.0173 (5)	0.0180 (5)	0.0181 (5)	−0.0008 (4)	0.0011 (4)	0.0031 (4)
C12	0.0142 (5)	0.0198 (6)	0.0171 (5)	0.0005 (4)	0.0022 (4)	0.0012 (4)
C13	0.0199 (6)	0.0167 (5)	0.0211 (6)	0.0025 (4)	0.0011 (4)	0.0030 (4)
C14	0.0187 (5)	0.0179 (6)	0.0206 (5)	0.0021 (4)	0.0024 (4)	−0.0014 (4)
C15	0.0131 (5)	0.0210 (6)	0.0160 (5)	−0.0005 (4)	0.0030 (4)	0.0003 (4)
C16	0.0207 (6)	0.0167 (5)	0.0205 (5)	0.0021 (4)	0.0039 (4)	0.0027 (4)
C17	0.0191 (5)	0.0169 (6)	0.0190 (5)	0.0008 (4)	0.0035 (4)	−0.0005 (4)
C18	0.0253 (6)	0.0220 (6)	0.0193 (5)	−0.0011 (5)	0.0060 (4)	−0.0033 (4)

Geometric parameters (Å, º)

O1—C1	1.3457 (13)	C8—H8A	0.9800
O1—H1	0.867 (9)	C8—H8B	0.9800
O2—C3	1.3615 (14)	C8—H8C	0.9800
O2—C7	1.4349 (14)	C9—C10	1.4728 (15)
O3—C5	1.3558 (13)	C10—C11	1.3411 (16)
O3—C8	1.4326 (13)	C10—H10	0.9500
O4—C9	1.2618 (14)	C11—C12	1.4623 (15)
O5—C15	1.3644 (13)	C11—H11	0.9500
O5—C18	1.4331 (14)	C12—C13	1.3927 (16)
C1—C2	1.3913 (16)	C12—C17	1.4079 (16)
C1—C6	1.4250 (15)	C13—C14	1.3929 (16)
C2—C3	1.3828 (16)	C13—H13	0.9500
C2—H2	0.9500	C14—C15	1.3932 (16)
C3—C4	1.4023 (15)	C14—H14	0.9500
C4—C5	1.3820 (16)	C15—C16	1.3999 (16)
C4—H4	0.9500	C16—C17	1.3804 (16)
C5—C6	1.4318 (15)	C16—H16	0.9500
C6—C9	1.4619 (15)	C17—H17	0.9500
C7—H7A	0.9800	C18—H18A	0.9800

C7—H7B	0.9800	C18—H18B	0.9800
C7—H7C	0.9800	C18—H18C	0.9800
C1—O1—H1	103.7 (12)	O4—C9—C6	119.43 (10)
C3—O2—C7	117.01 (9)	O4—C9—C10	117.29 (10)
C5—O3—C8	117.88 (9)	C6—C9—C10	123.16 (10)
C15—O5—C18	117.20 (9)	C11—C10—C9	119.37 (10)
O1—C1—C2	116.51 (10)	C11—C10—H10	120.3
O1—C1—C6	121.08 (10)	C9—C10—H10	120.3
C2—C1—C6	122.39 (10)	C10—C11—C12	126.67 (10)
C3—C2—C1	118.66 (10)	C10—C11—H11	116.7
C3—C2—H2	120.7	C12—C11—H11	116.7
C1—C2—H2	120.7	C13—C12—C17	117.82 (10)
O2—C3—C2	123.37 (10)	C13—C12—C11	119.40 (10)
O2—C3—C4	115.00 (10)	C17—C12—C11	122.77 (10)
C2—C3—C4	121.63 (10)	C12—C13—C14	122.04 (10)
C5—C4—C3	119.44 (10)	C12—C13—H13	119.0
C5—C4—H4	120.3	C14—C13—H13	119.0
C3—C4—H4	120.3	C15—C14—C13	119.13 (10)
O3—C5—C4	122.78 (10)	C15—C14—H14	120.4
O3—C5—C6	115.74 (10)	C13—C14—H14	120.4
C4—C5—C6	121.46 (10)	O5—C15—C14	124.66 (10)
C1—C6—C5	116.20 (10)	O5—C15—C16	115.70 (10)
C1—C6—C9	118.26 (10)	C14—C15—C16	119.64 (10)
C5—C6—C9	125.53 (10)	C17—C16—C15	120.50 (10)
O2—C7—H7A	109.5	C17—C16—H16	119.7
O2—C7—H7B	109.5	C15—C16—H16	119.7
H7A—C7—H7B	109.5	C16—C17—C12	120.72 (10)
O2—C7—H7C	109.5	C16—C17—H17	119.6
H7A—C7—H7C	109.5	C12—C17—H17	119.6
H7B—C7—H7C	109.5	O5—C18—H18A	109.5
O3—C8—H8A	109.5	O5—C18—H18B	109.5
O3—C8—H8B	109.5	H18A—C18—H18B	109.5
H8A—C8—H8B	109.5	O5—C18—H18C	109.5
O3—C8—H8C	109.5	H18A—C18—H18C	109.5
H8A—C8—H8C	109.5	H18B—C18—H18C	109.5
H8B—C8—H8C	109.5		
O1—C1—C2—C3	−178.53 (10)	C5—C6—C9—O4	−170.97 (10)
C6—C1—C2—C3	3.23 (17)	C1—C6—C9—C10	−165.63 (10)
C7—O2—C3—C2	0.85 (16)	C5—C6—C9—C10	13.18 (17)
C7—O2—C3—C4	179.78 (10)	O4—C9—C10—C11	22.06 (16)
C1—C2—C3—O2	179.81 (10)	C6—C9—C10—C11	−162.02 (11)
C1—C2—C3—C4	0.96 (17)	C9—C10—C11—C12	−176.23 (10)
O2—C3—C4—C5	178.59 (10)	C10—C11—C12—C13	−162.90 (11)
C2—C3—C4—C5	−2.46 (17)	C10—C11—C12—C17	17.58 (18)
C8—O3—C5—C4	−0.74 (16)	C17—C12—C13—C14	−3.23 (17)
C8—O3—C5—C6	177.90 (10)	C11—C12—C13—C14	177.23 (10)

C3—C4—C5—O3	178.40 (10)	C12—C13—C14—C15	0.76 (18)
C3—C4—C5—C6	−0.17 (17)	C18—O5—C15—C14	−2.19 (16)
O1—C1—C6—C5	176.25 (10)	C18—O5—C15—C16	177.52 (10)
C2—C1—C6—C5	−5.59 (16)	C13—C14—C15—O5	−177.73 (10)
O1—C1—C6—C9	−4.83 (16)	C13—C14—C15—C16	2.57 (17)
C2—C1—C6—C9	173.33 (10)	O5—C15—C16—C17	176.90 (10)
O3—C5—C6—C1	−174.67 (9)	C14—C15—C16—C17	−3.38 (17)
C4—C5—C6—C1	4.00 (16)	C15—C16—C17—C12	0.84 (17)
O3—C5—C6—C9	6.50 (16)	C13—C12—C17—C16	2.41 (17)
C4—C5—C6—C9	−174.84 (11)	C11—C12—C17—C16	−178.06 (11)
C1—C6—C9—O4	10.22 (16)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O4	0.87 (1)	1.65 (1)	2.465 (1)	156 (2)