Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# (*E*)-2,2-Dimethyl-5-(3-phenylallylidene)-1,3-dioxane-4,6-dione

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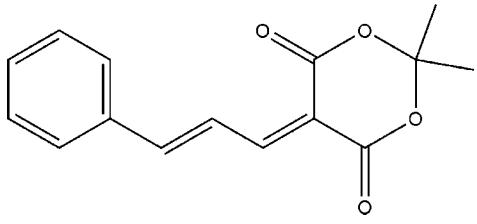
Received 9 October 2010; accepted 20 October 2010

Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.055; $wR$ factor = 0.170; data-to-parameter ratio = 17.5.

The title compound, $C_{15}H_{14}O_4$, was prepared by the reaction of 2,2-dimethyl-1,3-dioxane-4,6-dione and (*Z*)-3-phenylacrylaldehyde in ethanol. The dioxane ring is in a sofa conformation with the C atom bonded to the two methyl groups forming the flap. With the exception of the flap atom and the methyl group C atoms, all other non-H atoms are essentially planar, with an r.m.s. deviation of 0.067 (1) Å. The crystal structure is stabilized by weak intermolecular C—H···O hydrogen bonds.

## Related literature

For background to Meldrum's acid, 2,2-dimethyl-1,3-dioxane-4,6-dione, see: Kuhn *et al.* (2003); Casadesus *et al.* (2006). For a related structure, see: Zeng & Jian (2009).



## Experimental

### Crystal data

$C_{15}H_{14}O_4$
$M_r$ = 258.26
Triclinic, $P\overline{1}$
$a$ = 6.9171 (14) Å
$b$ = 7.0961 (14) Å
$c$ = 13.732 (3) Å
$\alpha$ = 94.79 (3)°
$\beta$ = 90.79 (3)°
$\gamma$ = 98.31 (3)°
$V$ = 664.4 (2) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 293 K
0.20 × 0.15 × 0.10 mm

### Data collection

Bruker SMART CCD area-detector diffractometer
6475 measured reflections
3006 independent reflections
2319 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.045

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.170
$S$ = 1.25
3006 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.33 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.30 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C15—H15$B$···O3$^{i}$ | 0.96 | 2.41 | 3.2991 (19) | 155 |
| C15—H15$C$···O3$^{ii}$ | 0.96 | 2.57 | 3.486 (2) | 159 |

Symmetry codes: (i) $x, y+1, z$; (ii) $-x, -y+1, -z+2$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); 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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5147).

## References

Bruker (1997). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Casadesus, M., Coogan, M. P. & Ooi, L. L. (2006). *Org. Biomol. Chem.*. **58**, 3822–3830.
Kuhn, N., Al-Sheikh, A. & Steimann, M. (2003). *Z. Naturforsch*. **58**, 381–384.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Zeng, W.-L. & Jian, F.-F. (2009). *Acta Cryst.* E**65**, o2587.

# supplementary materials

*Acta Cryst.* (2010). E**66**, o2943 [ **doi:10.1107/S1600536810042534** ]

# (*E*)-2,2-Dimethyl-5-(3-phenylallylidene)-1,3-dioxane-4,6-dione

**W.-L. Zeng**

## Comment

Starting with its discovery and correct structural assignment, Meldrum's acid has become a widely used reagent in organic synthesis (Kuhn *et al.*, 2003; Casadesus *et al.*, 2006) owing to the interesting conformational features of the products. We have recently reported the crystal structure of 5-(2-fluorobenzylidene)-2,2-dimethyl-1,3-dioxane-4,6-dione (Zeng *et al.* 2009). As part of our search for new Meldrum's acid, the title compound (I) has been synthesized and its structure is reported herein. The molecular structure of (I) is shown in Fig. 1. The dioxane ring is in a sofa conformation with the C atom bonded to the two methyl groups forming the flap. With the exception of the flap atom and the methyl group C atoms, all other non-hydrogen atoms are essentially planar with an rms deviation of 0.067 (1)Å. The deviation of atom C13 from the mean-plane formed by O1/O2/C11/C12/C10 is 0.270 (1)Å. The crystal structure is stabilized by weak intermolecular C—H···O hydrogen bonds (Table 1).

## Experimental

The mixture of malonic acid (6.24 g, 0.06 mol) and acetic anhydride(9 ml) in strong sulfuric acid (0.25 ml) was stirred with water at 303K, After dissolving, propan-2-one (3.48 g, 0.06 mol) was added dropwise into solution for 1 h. The reaction was allowed to proceed for 2 h. The mixture was cooled and filtered, and then an ethanol solution of (*Z*)-3-phenylacrylaldehyde (7.92g,0.06 mol) was added. The solution was then filtered and concentrated. Single crystals were obtained by evaporation of an petroleum ether-ethylacetate (4:1 *v*/*v*) solution of (I) at room temperature over a period of several days.

## Refinement

The H atoms were placed in calculated positions (C—H = 0.93 and 0.96 Å), and refined as riding with $U_{iso}$(H) = 1.2$U_{eq}$(C) or 1.5$U_{eq}$(methyl C).

## Figures

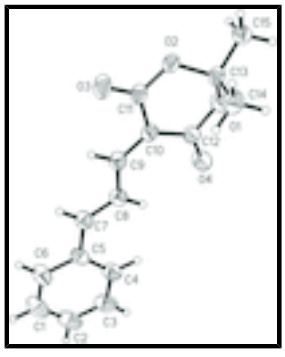


Fig. 1. The molecular structure of (I), drawn with 30% probability ellipsoids and spheres of arbitrary size for the H atoms.

### (*E*)-2,2-Dimethyl-5-(3-phenylallylidene)-1,3-dioxane-4,6-dione

*Crystal data*

| | |
|---|---|
| $C_{15}H_{14}O_4$ | $Z = 2$ |
| $M_r = 258.26$ | $F(000) = 272$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.291$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 6.9171$ (14) Å | Cell parameters from 2319 reflections |
| $b = 7.0961$ (14) Å | $\theta = 3.2$–$27.5°$ |
| $c = 13.732$ (3) Å | $\mu = 0.09$ mm$^{-1}$ |
| $\alpha = 94.79$ (3)° | $T = 293$ K |
| $\beta = 90.79$ (3)° | Block, yellow |
| $\gamma = 98.31$ (3)° | 0.20 × 0.15 × 0.10 mm |
| $V = 664.4$ (2) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD area-detector diffractometer | 2319 reflections with $I > 2\sigma(I)$ |
| Radiation source: fine-focus sealed tube | $R_{int} = 0.045$ |
| graphite | $\theta_{max} = 27.5°$, $\theta_{min} = 3.2°$ |
| phi and ω scans | $h = -8 \rightarrow 8$ |
| 6475 measured reflections | $k = -9 \rightarrow 9$ |
| 3006 independent reflections | $l = -17 \rightarrow 17$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.055$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.170$ | H-atom parameters constrained |
| $S = 1.25$ | $w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 3006 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 172 parameters | $\Delta\rho_{max} = 0.33$ e Å$^{-3}$ |
| 0 restraints | $\Delta\rho_{min} = -0.30$ 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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O2 | 0.11039 (12) | 0.49034 (12) | 0.88464 (7) | 0.0556 (3) |
| O1 | 0.26632 (13) | 0.73340 (13) | 0.79292 (7) | 0.0576 (3) |
| C10 | 0.40643 (17) | 0.44465 (17) | 0.80181 (9) | 0.0489 (3) |
| C9 | 0.53406 (18) | 0.32290 (19) | 0.77683 (9) | 0.0533 (3) |
| H9A | 0.5087 | 0.2047 | 0.8024 | 0.064* |
| O4 | 0.52765 (15) | 0.70212 (16) | 0.70801 (9) | 0.0790 (4) |
| O3 | 0.21672 (17) | 0.21587 (14) | 0.89068 (9) | 0.0782 (4) |
| C11 | 0.24288 (19) | 0.37290 (18) | 0.86240 (9) | 0.0534 (3) |
| C8 | 0.70169 (18) | 0.3474 (2) | 0.71739 (10) | 0.0546 (3) |
| H8A | 0.7361 | 0.4617 | 0.6891 | 0.066* |
| C12 | 0.41250 (18) | 0.63399 (19) | 0.76415 (10) | 0.0537 (3) |
| C15 | −0.0208 (2) | 0.7761 (2) | 0.87670 (11) | 0.0621 (4) |
| H15A | −0.0951 | 0.7213 | 0.8191 | 0.093* |
| H15B | 0.0082 | 0.9121 | 0.8745 | 0.093* |
| H15C | −0.0953 | 0.7481 | 0.9335 | 0.093* |
| C7 | 0.80960 (19) | 0.2062 (2) | 0.70229 (9) | 0.0569 (3) |
| H7A | 0.7673 | 0.0944 | 0.7315 | 0.068* |
| C5 | 0.98573 (19) | 0.2077 (2) | 0.64538 (9) | 0.0553 (3) |
| C13 | 0.16573 (17) | 0.69346 (17) | 0.88135 (9) | 0.0503 (3) |
| C4 | 1.06834 (19) | 0.3651 (2) | 0.59900 (11) | 0.0648 (4) |
| H4A | 1.0124 | 0.4769 | 0.6049 | 0.078* |
| C6 | 1.0760 (2) | 0.0447 (2) | 0.63695 (11) | 0.0726 (4) |
| H6A | 1.0236 | −0.0620 | 0.6680 | 0.087* |
| C14 | 0.2935 (2) | 0.7718 (2) | 0.96872 (12) | 0.0717 (4) |
| H14A | 0.4106 | 0.7137 | 0.9673 | 0.108* |
| H14B | 0.2248 | 0.7443 | 1.0273 | 0.108* |
| H14C | 0.3271 | 0.9077 | 0.9675 | 0.108* |
| C3 | 1.2336 (2) | 0.3567 (3) | 0.54389 (13) | 0.0784 (5) |
| H3A | 1.2869 | 0.4624 | 0.5123 | 0.094* |
| C2 | 1.3187 (2) | 0.1942 (3) | 0.53569 (14) | 0.0887 (6) |
| H2A | 1.4286 | 0.1886 | 0.4979 | 0.106* |
| C1 | 1.2416 (3) | 0.0393 (3) | 0.58337 (14) | 0.0903 (6) |
| H1A | 1.3018 | −0.0700 | 0.5794 | 0.108* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O2 | 0.0584 (5) | 0.0430 (5) | 0.0644 (6) | 0.0006 (4) | 0.0176 (4) | 0.0074 (4) |
| O1 | 0.0619 (5) | 0.0547 (5) | 0.0613 (6) | 0.0155 (4) | 0.0176 (4) | 0.0212 (4) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C10 | 0.0530 (6) | 0.0473 (7) | 0.0473 (6) | 0.0056 (5) | 0.0066 (5) | 0.0115 (5) |
| C9 | 0.0593 (7) | 0.0520 (7) | 0.0500 (7) | 0.0098 (5) | 0.0023 (5) | 0.0097 (5) |
| O4 | 0.0782 (7) | 0.0716 (7) | 0.0972 (8) | 0.0208 (5) | 0.0424 (6) | 0.0429 (6) |
| O3 | 0.1022 (8) | 0.0478 (6) | 0.0891 (8) | 0.0118 (5) | 0.0430 (6) | 0.0245 (5) |
| C11 | 0.0652 (7) | 0.0439 (7) | 0.0509 (7) | 0.0035 (5) | 0.0112 (6) | 0.0082 (5) |
| C8 | 0.0546 (7) | 0.0582 (7) | 0.0525 (7) | 0.0106 (5) | 0.0027 (5) | 0.0086 (5) |
| C12 | 0.0531 (6) | 0.0531 (7) | 0.0577 (7) | 0.0087 (5) | 0.0127 (5) | 0.0180 (6) |
| C15 | 0.0607 (7) | 0.0590 (8) | 0.0679 (9) | 0.0116 (6) | 0.0127 (6) | 0.0063 (6) |
| C7 | 0.0603 (7) | 0.0609 (8) | 0.0519 (7) | 0.0148 (6) | 0.0021 (6) | 0.0098 (6) |
| C5 | 0.0556 (7) | 0.0672 (8) | 0.0463 (7) | 0.0197 (6) | −0.0027 (5) | 0.0050 (6) |
| C13 | 0.0567 (6) | 0.0423 (6) | 0.0510 (7) | 0.0014 (5) | 0.0104 (5) | 0.0081 (5) |
| C4 | 0.0582 (7) | 0.0763 (10) | 0.0642 (8) | 0.0191 (7) | 0.0015 (6) | 0.0144 (7) |
| C6 | 0.0820 (9) | 0.0768 (10) | 0.0665 (9) | 0.0339 (8) | 0.0090 (8) | 0.0096 (8) |
| C14 | 0.0797 (9) | 0.0637 (9) | 0.0678 (9) | 0.0026 (7) | −0.0088 (7) | −0.0016 (7) |
| C3 | 0.0592 (8) | 0.1082 (14) | 0.0715 (10) | 0.0144 (8) | 0.0096 (7) | 0.0236 (9) |
| C2 | 0.0677 (9) | 0.1327 (18) | 0.0731 (10) | 0.0386 (10) | 0.0170 (8) | 0.0082 (11) |
| C1 | 0.0914 (12) | 0.1067 (14) | 0.0844 (12) | 0.0537 (11) | 0.0177 (10) | 0.0057 (10) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O2—C11 | 1.3468 (15) | C7—C5 | 1.456 (2) |
| O2—C13 | 1.4415 (14) | C7—H7A | 0.9300 |
| O1—C12 | 1.3590 (15) | C5—C4 | 1.387 (2) |
| O1—C13 | 1.4362 (15) | C5—C6 | 1.389 (2) |
| C10—C9 | 1.3501 (17) | C13—C14 | 1.4992 (19) |
| C10—C11 | 1.4702 (18) | C4—C3 | 1.385 (2) |
| C10—C12 | 1.4746 (17) | C4—H4A | 0.9300 |
| C9—C8 | 1.4256 (19) | C6—C1 | 1.374 (2) |
| C9—H9A | 0.9300 | C6—H6A | 0.9300 |
| O4—C12 | 1.1959 (16) | C14—H14A | 0.9600 |
| O3—C11 | 1.2004 (15) | C14—H14B | 0.9600 |
| C8—C7 | 1.3375 (18) | C14—H14C | 0.9600 |
| C8—H8A | 0.9300 | C3—C2 | 1.366 (2) |
| C15—C13 | 1.4955 (17) | C3—H3A | 0.9300 |
| C15—H15A | 0.9600 | C2—C1 | 1.372 (3) |
| C15—H15B | 0.9600 | C2—H2A | 0.9300 |
| C15—H15C | 0.9600 | C1—H1A | 0.9300 |
| C11—O2—C13 | 119.15 (10) | C6—C5—C7 | 118.85 (14) |
| C12—O1—C13 | 119.85 (9) | O1—C13—O2 | 110.44 (10) |
| C9—C10—C11 | 116.66 (11) | O1—C13—C15 | 106.78 (10) |
| C9—C10—C12 | 123.71 (12) | O2—C13—C15 | 106.17 (10) |
| C11—C10—C12 | 119.36 (11) | O1—C13—C14 | 110.25 (11) |
| C10—C9—C8 | 130.02 (12) | O2—C13—C14 | 109.54 (10) |
| C10—C9—H9A | 115.0 | C15—C13—C14 | 113.55 (13) |
| C8—C9—H9A | 115.0 | C3—C4—C5 | 120.42 (15) |
| O3—C11—O2 | 118.16 (12) | C3—C4—H4A | 119.8 |
| O3—C11—C10 | 124.58 (13) | C5—C4—H4A | 119.8 |
| O2—C11—C10 | 117.22 (10) | C1—C6—C5 | 120.81 (17) |
| C7—C8—C9 | 120.10 (13) | C1—C6—H6A | 119.6 |

| | | | |
|---|---|---|---|
| C7—C8—H8A | 119.9 | C5—C6—H6A | 119.6 |
| C9—C8—H8A | 119.9 | C13—C14—H14A | 109.5 |
| O4—C12—O1 | 118.08 (11) | C13—C14—H14B | 109.5 |
| O4—C12—C10 | 126.04 (12) | H14A—C14—H14B | 109.5 |
| O1—C12—C10 | 115.79 (11) | C13—C14—H14C | 109.5 |
| C13—C15—H15A | 109.5 | H14A—C14—H14C | 109.5 |
| C13—C15—H15B | 109.5 | H14B—C14—H14C | 109.5 |
| H15A—C15—H15B | 109.5 | C2—C3—C4 | 120.37 (17) |
| C13—C15—H15C | 109.5 | C2—C3—H3A | 119.8 |
| H15A—C15—H15C | 109.5 | C4—C3—H3A | 119.8 |
| H15B—C15—H15C | 109.5 | C3—C2—C1 | 119.80 (16) |
| C8—C7—C5 | 127.42 (13) | C3—C2—H2A | 120.1 |
| C8—C7—H7A | 116.3 | C1—C2—H2A | 120.1 |
| C5—C7—H7A | 116.3 | C2—C1—C6 | 120.36 (16) |
| C4—C5—C6 | 118.21 (13) | C2—C1—H1A | 119.8 |
| C4—C5—C7 | 122.93 (13) | C6—C1—H1A | 119.8 |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C15—H15B···O3$^{i}$ | 0.96 | 2.41 | 3.2991 (19) | 155 |
| C15—H15C···O3$^{ii}$ | 0.96 | 2.57 | 3.486 (2) | 159 |

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

**Fig. 1**