

4-(2,4,6-Trimethylphenyl)butan-2-one

Lang-Jing Zhang, Jun-Feng Kou and Feng-Yi Liu*

College of Chemistry and Chemical Engineering, Yunnan Normal University, Kunming 650500, People's Republic of China. *Correspondence e-mail: lfy20110407@163.com

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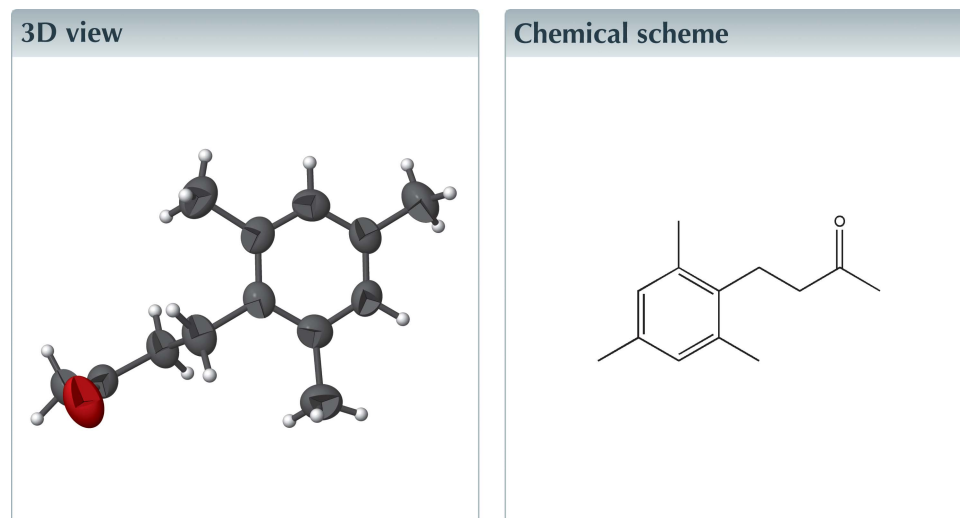
Edited by D.-J. Xu, Zhejiang University (Yuquan Campus), China

Keywords: crystal structure; π - π stacking; hydrolysis of β -diketone.

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Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{13}H_{18}O$, the 3-oxobutyl group is approximately planar [maximum deviation = 0.029 (4) Å] and its mean plane is twisted with respect to the benzene ring at 84.0 (2)°. In the crystal, weak C—H... π interactions link the molecules into supramolecular chains propagating along the *a* axis.



Structure description

The β -diketone motif is a versatile metal coordinating ligand that is readily derivatized and able to form complexes with a wide range of metal ions (Bray *et al.*, 2007; Chen *et al.*, 2010). It has also been shown to be an attractive ligand for the preparation of metallo-supramolecular structures. However, our attempts at forming lanthanide-transition metal complexes using the normal procedure of heating with a lanthanide salt in the presence of base were unsuccessful. Rather than obtaining the desired lanthanide metal complex upon cooling the reaction mixture, we isolated solids that contained no metal ion. An X-ray crystal structure determination confirmed that this product was the ketone derivative of compound (II) (Fig. 2), which could be attributed to the alkaline hydrolysis of the β -diketone groups (Hauser *et al.*, 1948; Pearson & Mayerle, 1951). Herein we report the synthesis and structure of the title compound.

In the molecule (Fig. 1), the 3'-oxo-butyl group is nearly planar, the maximum deviation being 0.029 (4) Å (C1 atom), and its mean plane is oriented at 84.0 (2)° with respect to the benzene ring.

In the crystal, weak C—H... π interactions (Table 1) link the molecules into supramolecular chains propagating along the *a*-axis direction.

Synthesis and crystallization

The synthesis procedures for the title compound are shown in Fig. 2. Acetylacetone (9 mmol, 900 mg) was added to 30 ml *tert*-butanol in the presence of *t*-BuOK. After 1 h,

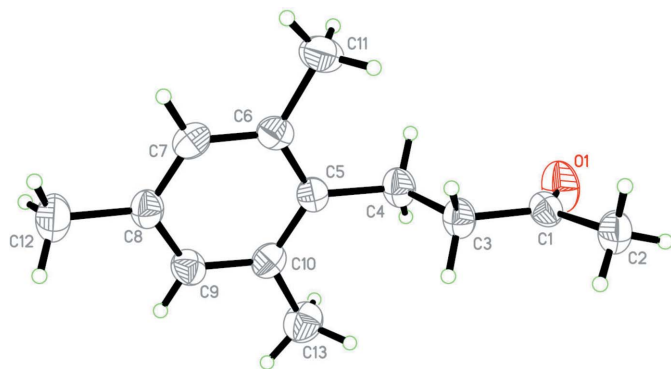


Figure 1
The molecular structure of the title compound, shown with atom labels and 30% probability displacement ellipsoids.

2-(bromomethyl)-1,3,5-trimethylbenzene (9 mmol, 1917 mg) was added to the above mixture, then 0.25 g KI was added. The mixture was then refluxed under argon for 72 h. After cooling to room temperature, the solvent was evaporated. The product was extracted with dichloromethane (50 ml), and the organic phase was washed with water (50 ml). Then the organic phase was dried by magnesium sulfate and filtered. White solids were obtained after the dichloromethane solvent was evaporated [compound (II)]. Yield: 63% (based on acetylacetone). Compound (II) (6 mmol, 1392 mg) was dissolved in 20 ml ethanol, then 6 mmol NaOH (0.1 mol l^{-1}) was added to adjust the pH of the mixture to 7. Then 2 mmol europium(III) chloride, (0.2 mol l^{-1} in ethanol) was added drop by drop. The mixture was refluxed for 2 h. The filtrate was dissolved in dichloromethane/ether. Colorless block-shaped crystals were obtained by slow evaporation after one week.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Funding information

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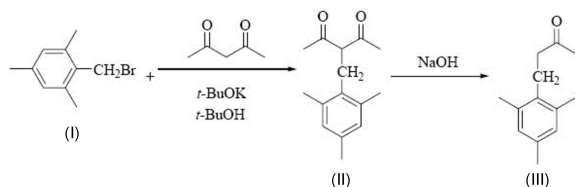


Figure 2
Steps in the synthesis of the title compound.

Table 1
Hydrogen-bond geometry (\AA , $^\circ$).

Cg1 is the centroid of the benzene ring.

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C11-H11B \cdots Cg1^1$	0.96	2.76	3.642 (5)	154

Symmetry code: (i) $x + 1, y, z$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{18}O$
M_r	190.27
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	293
a, b, c (\AA)	4.8462 (10), 7.8042 (16), 30.996 (6)
V (\AA^3)	1172.3 (4)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.07
Crystal size (mm)	$0.28 \times 0.25 \times 0.23$
Data collection	
Diffractometer	Rigaku <i>MM007</i> -HF CCD (Saturn 724+)
Absorption correction	Multi-scan (<i>SADABS</i> ; Sheldrick, 1996)
T_{\min}, T_{\max}	0.982, 0.985
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11150, 2673, 1626
R_{int}	0.078
$(\sin \theta/\lambda)_{\text{max}}$ (\AA^{-1})	0.649
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.081, 0.168, 1.09
No. of reflections	2673
No. of parameters	131
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ ($e \text{ \AA}^{-3}$)	0.12, -0.13
Absolute structure	Flack x determined using 411 quotients $[(I^+) - (I^-)] / [(I^+) + (I^-)]$ (Parsons <i>et al.</i> , 2013)
Absolute structure parameter	0.5 (10)

Computer programs: *CrystalStructure* (Rigaku/MSO, 2006), *SHELXT* (Sheldrick, 2015a), *SHELXL2014* (Sheldrick, 2015b) and *SHELXTL* (Sheldrick, 2008).

References

- Bray, D. J., Jolliffe, K. A., Lindoy, L. F. & McMurtrie, J. C. (2007). *Tetrahedron*, **63**, 1953–1958.
- Chen, X.-Y., Yang, X.-P. & Holliday, B. J. (2010). *Inorg. Chem.* **49**, 2583–2585.
- Hauser, C. R., Swamer, F. W. & Ringler, B. I. (1948). *J. Am. Chem. Soc.* **70**, 4023–4026.
- Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* **B69**, 249–259.
- Pearson, R. G. & Mayerle, E. A. (1951). *J. Am. Chem. Soc.* **73**, 926–930.
- Rigaku/MSO (2006). *CrystalStructure*. Rigaku Americas, The Woodlands.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.
- Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.

full crystallographic data

IUCrData (2017). 2, x170675 [https://doi.org/10.1107/S2414314617006757]

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Crystal data

$C_{13}H_{18}O$

$M_r = 190.27$

Orthorhombic, $P2_12_12_1$

$a = 4.8462$ (10) Å

$b = 7.8042$ (16) Å

$c = 30.996$ (6) Å

$V = 1172.3$ (4) Å³

$Z = 4$

$F(000) = 416$

$D_x = 1.078$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 11221 reflections

$\theta = 3.3$ – 27.5°

$\mu = 0.07$ mm⁻¹

$T = 293$ K

Block, colorless

$0.28 \times 0.25 \times 0.23$ mm

Data collection

Rigaku MM007-HF CCD (Saturn 724+) diffractometer

Radiation source: rotating anode

ω scans at fixed $\chi = 45^\circ$

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{\min} = 0.982$, $T_{\max} = 0.985$

11150 measured reflections

2673 independent reflections

1626 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.078$

$\theta_{\max} = 27.5^\circ$, $\theta_{\min} = 3.3^\circ$

$h = -6 \rightarrow 5$

$k = -10 \rightarrow 10$

$l = -40 \rightarrow 39$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.081$

$wR(F^2) = 0.168$

$S = 1.09$

2673 reflections

131 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

$w = 1/[\sigma^2(F_o^2) + (0.0612P)^2 + 0.1143P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.12$ e Å⁻³

$\Delta\rho_{\min} = -0.13$ e Å⁻³

Absolute structure: Flack x determined using

411 quotients $[(F^-)-(F^+)]/[(F^-)+(F^+)]$ (Parsons *et al.*, 2013)

Absolute structure parameter: 0.5 (10)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. H atoms were geometrically fixed and refined in riding mode with C—H = 0.93–0.97 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms and $1.2U_{\text{eq}}(\text{C})$ for the others.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.2549 (8)	0.2228 (5)	0.24165 (11)	0.0573 (9)
C2	0.1217 (9)	0.2873 (6)	0.28200 (12)	0.0719 (12)
H2A	0.1694	0.2136	0.3057	0.108*
H2B	−0.0750	0.2883	0.2784	0.108*
H2C	0.1852	0.4014	0.2879	0.108*
C3	0.1343 (8)	0.2873 (5)	0.20018 (11)	0.0595 (10)
H3A	0.1267	0.4114	0.2013	0.071*
H3B	−0.0537	0.2456	0.1978	0.071*
C4	0.2930 (8)	0.2343 (5)	0.15972 (11)	0.0644 (11)
H4A	0.4828	0.2724	0.1623	0.077*
H4B	0.2940	0.1103	0.1576	0.077*
C5	0.1684 (7)	0.3087 (5)	0.11887 (11)	0.0503 (9)
C6	0.2388 (7)	0.4738 (5)	0.10572 (11)	0.0556 (10)
C7	0.1185 (8)	0.5412 (5)	0.06896 (11)	0.0594 (10)
H7	0.1681	0.6510	0.0603	0.071*
C8	−0.0712 (8)	0.4524 (5)	0.04478 (11)	0.0591 (10)
C9	−0.1382 (8)	0.2888 (5)	0.05782 (12)	0.0604 (10)
H9	−0.2638	0.2257	0.0416	0.073*
C10	−0.0231 (8)	0.2157 (5)	0.09455 (11)	0.0565 (10)
C11	0.4462 (9)	0.5809 (6)	0.13046 (14)	0.0804 (13)
H11A	0.4481	0.6953	0.1191	0.121*
H11B	0.6264	0.5311	0.1276	0.121*
H11C	0.3954	0.5841	0.1604	0.121*
C12	−0.2043 (10)	0.5318 (6)	0.00512 (13)	0.0871 (15)
H12A	−0.1128	0.4908	−0.0203	0.131*
H12B	−0.1884	0.6542	0.0066	0.131*
H12C	−0.3957	0.5003	0.0041	0.131*
C13	−0.1044 (11)	0.0326 (6)	0.10584 (15)	0.0824 (14)
H13A	0.0522	−0.0414	0.1023	0.124*
H13B	−0.2506	−0.0043	0.0871	0.124*
H13C	−0.1662	0.0281	0.1353	0.124*
O1	0.4465 (7)	0.1257 (4)	0.24275 (10)	0.0920 (11)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.058 (2)	0.057 (2)	0.057 (2)	0.000 (2)	−0.010 (2)	0.0008 (19)
C2	0.081 (3)	0.081 (3)	0.053 (2)	0.002 (3)	−0.005 (2)	0.007 (2)
C3	0.056 (2)	0.070 (2)	0.052 (2)	0.018 (2)	−0.0019 (18)	0.0011 (19)
C4	0.059 (2)	0.078 (3)	0.056 (2)	0.017 (2)	−0.002 (2)	0.000 (2)
C5	0.0426 (18)	0.062 (2)	0.0466 (18)	0.0125 (18)	0.0052 (17)	−0.0037 (17)
C6	0.0446 (19)	0.068 (3)	0.054 (2)	0.0000 (19)	0.0102 (17)	−0.0121 (19)

C7	0.062 (2)	0.055 (2)	0.062 (2)	0.001 (2)	0.013 (2)	0.0027 (19)
C8	0.062 (2)	0.067 (3)	0.0481 (19)	0.007 (2)	0.0015 (18)	0.0006 (19)
C9	0.066 (2)	0.062 (2)	0.053 (2)	-0.003 (2)	-0.0032 (19)	-0.0114 (19)
C10	0.062 (2)	0.054 (2)	0.054 (2)	0.005 (2)	0.012 (2)	-0.0034 (18)
C11	0.063 (2)	0.095 (3)	0.082 (3)	-0.017 (3)	0.001 (2)	-0.014 (2)
C12	0.099 (3)	0.097 (3)	0.066 (3)	0.007 (3)	-0.009 (3)	0.018 (3)
C13	0.103 (3)	0.061 (3)	0.083 (3)	-0.004 (3)	0.003 (3)	0.002 (2)
O1	0.089 (2)	0.105 (2)	0.081 (2)	0.048 (2)	-0.0164 (18)	0.0056 (17)

Geometric parameters (Å, °)

C1—O1	1.199 (4)	C7—C8	1.374 (5)
C1—C2	1.495 (5)	C7—H7	0.9300
C1—C3	1.499 (5)	C8—C9	1.378 (5)
C2—H2A	0.9600	C8—C12	1.520 (5)
C2—H2B	0.9600	C9—C10	1.390 (5)
C2—H2C	0.9600	C9—H9	0.9300
C3—C4	1.528 (5)	C10—C13	1.523 (6)
C3—H3A	0.9700	C11—H11A	0.9600
C3—H3B	0.9700	C11—H11B	0.9600
C4—C5	1.518 (5)	C11—H11C	0.9600
C4—H4A	0.9700	C12—H12A	0.9600
C4—H4B	0.9700	C12—H12B	0.9600
C5—C6	1.394 (5)	C12—H12C	0.9600
C5—C10	1.399 (5)	C13—H13A	0.9600
C6—C7	1.384 (5)	C13—H13B	0.9600
C6—C11	1.515 (5)	C13—H13C	0.9600
O1—C1—C2	121.6 (3)	C6—C7—H7	118.7
O1—C1—C3	122.6 (3)	C7—C8—C9	117.7 (4)
C2—C1—C3	115.9 (3)	C7—C8—C12	121.3 (4)
C1—C2—H2A	109.5	C9—C8—C12	121.0 (4)
C1—C2—H2B	109.5	C8—C9—C10	121.7 (4)
H2A—C2—H2B	109.5	C8—C9—H9	119.1
C1—C2—H2C	109.5	C10—C9—H9	119.1
H2A—C2—H2C	109.5	C9—C10—C5	119.6 (3)
H2B—C2—H2C	109.5	C9—C10—C13	118.0 (4)
C1—C3—C4	114.6 (3)	C5—C10—C13	122.3 (4)
C1—C3—H3A	108.6	C6—C11—H11A	109.5
C4—C3—H3A	108.6	C6—C11—H11B	109.5
C1—C3—H3B	108.6	H11A—C11—H11B	109.5
C4—C3—H3B	108.6	C6—C11—H11C	109.5
H3A—C3—H3B	107.6	H11A—C11—H11C	109.5
C5—C4—C3	112.4 (3)	H11B—C11—H11C	109.5
C5—C4—H4A	109.1	C8—C12—H12A	109.5
C3—C4—H4A	109.1	C8—C12—H12B	109.5
C5—C4—H4B	109.1	H12A—C12—H12B	109.5
C3—C4—H4B	109.1	C8—C12—H12C	109.5

H4A—C4—H4B	107.9	H12A—C12—H12C	109.5
C6—C5—C10	119.0 (3)	H12B—C12—H12C	109.5
C6—C5—C4	120.0 (3)	C10—C13—H13A	109.5
C10—C5—C4	121.0 (3)	C10—C13—H13B	109.5
C7—C6—C5	119.3 (4)	H13A—C13—H13B	109.5
C7—C6—C11	119.1 (4)	C10—C13—H13C	109.5
C5—C6—C11	121.6 (4)	H13A—C13—H13C	109.5
C8—C7—C6	122.7 (4)	H13B—C13—H13C	109.5
C8—C7—H7	118.7		
O1—C1—C3—C4	-6.0 (6)	C6—C7—C8—C9	-1.1 (5)
C2—C1—C3—C4	173.9 (4)	C6—C7—C8—C12	178.6 (4)
C1—C3—C4—C5	-177.8 (3)	C7—C8—C9—C10	1.1 (5)
C3—C4—C5—C6	84.4 (4)	C12—C8—C9—C10	-178.5 (4)
C3—C4—C5—C10	-93.8 (4)	C8—C9—C10—C5	-0.8 (5)
C10—C5—C6—C7	-0.2 (5)	C8—C9—C10—C13	-178.6 (4)
C4—C5—C6—C7	-178.5 (3)	C6—C5—C10—C9	0.3 (5)
C10—C5—C6—C11	-179.6 (3)	C4—C5—C10—C9	178.6 (3)
C4—C5—C6—C11	2.1 (5)	C6—C5—C10—C13	178.0 (3)
C5—C6—C7—C8	0.6 (5)	C4—C5—C10—C13	-3.7 (5)
C11—C6—C7—C8	-180.0 (3)		

Hydrogen-bond geometry (\AA , $^\circ$)

Cg1 is the centroid of the benzene ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C11—H11B \cdots Cg1 ⁱ	0.96	2.76	3.642 (5)	154

Symmetry code: (i) $x+1, y, z$.