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Methyl 2-hydroxy-3-(2-nitrophenyl)-2-(2-oxocyclopentyl)propanoate

Long He

College of Chemistry and Chemical Engineering, China West Normal University, Nanchong 637002, People's Republic of China
Correspondence e-mail: helongcwnu@yahoo.com.cn

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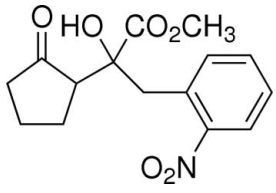
Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.008 Å; R factor = 0.076; wR factor = 0.220; data-to-parameter ratio = 12.9.

In the title compound, $C_{15}H_{17}NO_6$, the nitro group is tilted at a dihedral angle of 44.1 (3)° with respect to the benzene ring. The five-membered cyclopentanone ring adopts an envelope conformation. Intramolecular O—H···O hydrogen bonding is observed.

Related literature

For general background, see: Basavaiah *et al.* (2003); Elier *et al.* (1956). For related structures, see: Langner *et al.* (2005); Desimoni *et al.* (2006).



Experimental

Crystal data

$C_{15}H_{17}NO_6$
M_r = 307.30
Monoclinic, $P2_1/c$
a = 14.556 (4) Å
b = 5.540 (2) Å
c = 18.194 (6) Å
β = 95.12 (3)°
V = 1461.3 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 292 (2) K
0.25 × 0.22 × 0.05 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
2595 measured reflections
2591 independent reflections
949 reflections with $I > 2\sigma(I)$
R_{int} = 0.010
3 standard reflections
every 300 reflections
intensity decay: 4.8%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.076
$wR(F^2)$ = 0.220
S = 0.91
2591 reflections
201 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O5	0.82	2.49	3.283 (7)	162
O2—H2···O6	0.82	2.45	3.018 (5)	127

Data collection: *DIFRAC* (Gabe *et al.*, 1993); cell refinement: *DIFRAC*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia,1997); software used to prepare material for publication: *SHELXL97*.

The diffraction measurements were made at the Centre for Testing and Analysis, Sichuan University, China. We are grateful for financial support from China West Normal University (grant No. 05B022).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2357).

References

Basavaiah, D., Jaganmohan, R. A. & Satyanarayana, T. (2003). *Chem. Rev.* **103**, 811–892.
Desimoni, G., Faita, G., Piccinini, F. & Toscanini, M. (2006). *Eur. J. Org. Chem.* 5228–5230.
Elier, E. L., Fisk, M. T. & Prosser, T. (1956). *Org. Synth.* **36**, 3–6.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.
Gabe, E. J., White, P. S. & Enright, G. D. (1993). *DIFRAC*. Steacie Institute for Molecular Sciences, NRC, Ottawa, Ontario, Canada, and Department of Chemisty, University of North Carolina, Chapel Hill, North Carolina, USA.
Langner, M., Remy, P. & Bolm, C. (2005). *Chem. Eur. J.* **11**, 6254–6255.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

supplementary materials

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Methyl 2-hydroxy-3-(2-nitrophenyl)-2-(2-oxocyclopentyl)propanoate

L. He

Comment

α-Hydroxy ester can be transferred to a large variety of important product. The title compound is a very important intermediate for the construction of α,β-unsaturated carbonyl compound, which has broad utility in organic synthesis (Basavaiah *et al.*, 1996; Elier *et al.*, 1956). Its crystal structure is reported here.

The molecular structure is shown in Fig. 1. Bond lengths and angles are normal. The C15-containing ester shows an extended planar conformation and makes a dihedral angle of 49.8 (2)° with the benzene plane, The nitro group is not coplanar with attached benzene ring and tilted with respect to the benzene ring with a angle of 44.1 (3)°. Intramolecular O—H···O hydrogen bonding is observed in the structure (Table 1).

Experimental

To a mixture of anhydrous cyclopentanone (1 ml) and toluene (3 ml) was added the 3-(2-nitrophenyl)-2-oxopropanoic acid (0.5 mmol) and *L-N*-(pyridin-2-yl)pyrrolidine-2-carboxamide (2 mmol) and the resulting mixture was stirred at 273 K for 12 h. The reaction mixture was treated with CH_2N_2 solution in ether for 20 min. After removal of solvent, the residue was purified through flash column chromatography on a silica gel to give the title compound. Colourless single crystals suitable for X-ray diffraction were obtained by recrystallization from an ethanol solution.

Refinement

H atoms were placed in calculated positions with C–H = 0.93–0.98 Å and O—H = 0.82 Å, and refined using a riding model with U_{iso}(H) =1.2U_{eq}(C,*O*).

Figures

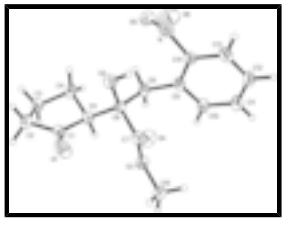

Fig. 1. The molecular structure of (I) with 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

Methyl 2-hydroxy-3-(2-nitrophenyl)-2-(2-oxocyclopentyl)propanoate

Crystal data

$C_{15}H_{17}NO_6$ F_{000} = 648

M_r = 307.30	D_x = 1.397 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 19 reflections
a = 14.556 (4) Å	θ = 5.0–9.3º
b = 5.540 (2) Å	μ = 0.11 mm^{-1}
c = 18.194 (6) Å	T = 292 (2) K
β = 95.12 (3)º	Block, colourless
V = 1461.3 (8) Å^3	0.25 × 0.22 × 0.05 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.010
Radiation source: fine-focus sealed tube	θ_{max} = 25.1º
Monochromator: graphite	θ_{min} = 1.4º
T = 292(2) K	h = −17→17
ω/2θ scan	k = 0→6
Absorption correction: none	l = −4→21
2595 measured reflections	3 standard reflections
2591 independent reflections	every 300 reflections
949 reflections with $I > 2\sigma(I)$	intensity decay: 4.8%

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.076	H-atom parameters constrained
$wR(F^2)$ = 0.220	$w = 1/[\sigma^2(F_o^2) + (0.107P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.91	$(\Delta/\sigma)_{max}$ = 0.001
2591 reflections	$\Delta\rho_{max}$ = 0.34 e Å^{-3}
201 parameters	$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ is used only for calculat-

ing *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	0.0421 (3)	0.2211 (8)	0.2122 (3)	0.0735 (15)
O2	0.1788 (3)	0.1796 (7)	0.0950 (2)	0.0445 (10)
H2	0.2285	0.2059	0.0785	0.053*
O3	0.2303 (2)	−0.0964 (6)	0.27028 (19)	0.0421 (10)
O4	0.2620 (3)	0.2696 (7)	0.2269 (2)	0.0583 (12)
O5	0.3485 (4)	0.3703 (9)	0.0026 (3)	0.0834 (16)
O6	0.2893 (3)	0.0276 (9)	−0.0299 (2)	0.0691 (14)
N1	0.3379 (4)	0.1526 (11)	0.0124 (3)	0.0549 (14)
C1	0.0238 (4)	0.0522 (11)	0.1718 (3)	0.0456 (15)
C2	−0.0695 (4)	−0.0067 (12)	0.1355 (3)	0.0541 (17)
H2A	−0.0909	0.1195	0.1012	0.065*
H2B	−0.1137	−0.0252	0.1720	0.065*
C3	−0.0574 (4)	−0.2430 (12)	0.0950 (3)	0.0548 (17)
H3A	−0.0713	−0.3797	0.1254	0.066*
H3B	−0.0973	−0.2487	0.0494	0.066*
C4	0.0428 (4)	−0.2444 (11)	0.0796 (3)	0.0471 (15)
H4A	0.0523	−0.1489	0.0363	0.056*
H4B	0.0643	−0.4077	0.0723	0.056*
C5	0.0921 (3)	−0.1321 (10)	0.1492 (3)	0.0392 (14)
H5	0.0981	−0.2572	0.1874	0.047*
C6	0.1875 (4)	−0.0227 (9)	0.1435 (3)	0.0365 (13)
C7	0.2303 (4)	0.0695 (10)	0.2176 (3)	0.0379 (13)
C8	0.2536 (4)	−0.2062 (10)	0.1124 (3)	0.0400 (14)
H8A	0.2485	−0.3581	0.1382	0.048*
H8B	0.2334	−0.2342	0.0608	0.048*
C9	0.3530 (4)	−0.1341 (10)	0.1178 (3)	0.0362 (13)
C10	0.4123 (4)	−0.2346 (10)	0.1729 (3)	0.0412 (14)
H10	0.3888	−0.3479	0.2039	0.049*
C11	0.5037 (4)	−0.1768 (12)	0.1844 (3)	0.0558 (17)
H11	0.5413	−0.2555	0.2208	0.067*
C12	0.5397 (4)	−0.0019 (12)	0.1417 (3)	0.0517 (17)
H12	0.6016	0.0408	0.1497	0.062*
C13	0.4844 (4)	0.1078 (11)	0.0880 (3)	0.0509 (17)
H13	0.5083	0.2277	0.0595	0.061*
C14	0.3933 (4)	0.0434 (10)	0.0753 (3)	0.0414 (14)
C15	0.2697 (4)	−0.0246 (11)	0.3430 (3)	0.0530 (17)
H15A	0.3355	−0.0110	0.3429	0.064*
H15B	0.2553	−0.1437	0.3785	0.064*
H15C	0.2445	0.1284	0.3557	0.064*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.078 (3)	0.058 (3)	0.087 (4)	−0.003 (3)	0.021 (3)	−0.036 (3)
O2	0.052 (2)	0.035 (2)	0.048 (2)	0.002 (2)	0.0130 (19)	0.0112 (19)
O3	0.061 (3)	0.026 (2)	0.039 (2)	−0.0021 (19)	−0.0014 (17)	0.0054 (18)
O4	0.081 (3)	0.035 (3)	0.056 (3)	−0.010 (2)	−0.008 (2)	−0.003 (2)
O5	0.132 (5)	0.052 (3)	0.066 (3)	−0.001 (3)	0.008 (3)	0.020 (3)
O6	0.075 (3)	0.087 (4)	0.044 (3)	−0.005 (3)	−0.003 (2)	−0.004 (3)
N1	0.073 (4)	0.051 (4)	0.043 (3)	0.009 (3)	0.017 (3)	0.007 (3)
C1	0.051 (4)	0.038 (4)	0.049 (4)	0.005 (3)	0.011 (3)	−0.001 (3)
C2	0.047 (4)	0.064 (5)	0.050 (4)	0.011 (3)	0.004 (3)	0.002 (3)
C3	0.041 (4)	0.066 (4)	0.056 (4)	−0.004 (3)	−0.003 (3)	−0.001 (3)
C4	0.050 (4)	0.041 (4)	0.049 (3)	−0.003 (3)	−0.001 (3)	−0.005 (3)
C5	0.037 (3)	0.047 (4)	0.033 (3)	−0.003 (3)	0.000 (2)	0.004 (3)
C6	0.049 (3)	0.031 (3)	0.030 (3)	0.007 (3)	0.006 (2)	0.005 (3)
C7	0.039 (3)	0.023 (3)	0.052 (4)	−0.001 (3)	0.007 (3)	−0.005 (3)
C8	0.053 (4)	0.028 (3)	0.040 (3)	0.003 (3)	0.008 (3)	−0.004 (3)
C9	0.040 (3)	0.033 (3)	0.036 (3)	0.003 (3)	0.004 (2)	−0.007 (3)
C10	0.041 (3)	0.032 (3)	0.050 (3)	0.004 (3)	0.001 (3)	0.001 (3)
C11	0.058 (4)	0.052 (4)	0.056 (4)	0.004 (4)	−0.001 (3)	0.003 (3)
C12	0.038 (3)	0.062 (4)	0.053 (4)	−0.005 (3)	−0.003 (3)	−0.017 (4)
C13	0.058 (4)	0.049 (4)	0.048 (4)	−0.020 (3)	0.019 (3)	−0.012 (3)
C14	0.051 (4)	0.039 (4)	0.035 (3)	0.007 (3)	0.010 (3)	−0.008 (3)
C15	0.057 (4)	0.063 (4)	0.037 (3)	−0.001 (3)	−0.002 (3)	0.009 (3)

Geometric parameters (Å, °)

O1—C1	1.205 (7)	C5—C6	1.527 (7)
O2—C6	1.426 (6)	C5—H5	0.9800
O2—H2	0.8200	C6—C7	1.521 (7)
O3—C7	1.329 (6)	C6—C8	1.542 (7)
O3—C15	1.449 (6)	C8—C9	1.496 (7)
O4—C7	1.206 (6)	C8—H8A	0.9700
O5—N1	1.230 (6)	C8—H8B	0.9700
O6—N1	1.214 (6)	C9—C10	1.380 (7)
N1—C14	1.470 (7)	C9—C14	1.410 (7)
C1—C2	1.492 (8)	C10—C11	1.365 (8)
C1—C5	1.508 (8)	C10—H10	0.9300
C2—C3	1.520 (8)	C11—C12	1.374 (8)
C2—H2A	0.9700	C11—H11	0.9300
C2—H2B	0.9700	C12—C13	1.353 (8)
C3—C4	1.509 (7)	C12—H12	0.9300
C3—H3A	0.9700	C13—C14	1.373 (7)
C3—H3B	0.9700	C13—H13	0.9300
C4—C5	1.530 (7)	C15—H15A	0.9600
C4—H4A	0.9700	C15—H15B	0.9600
C4—H4B	0.9700	C15—H15C	0.9600

C6—O2—H2	109.5	C7—C6—C8	109.1 (4)
C7—O3—C15	116.3 (4)	C5—C6—C8	111.3 (4)
O6—N1—O5	122.7 (6)	O4—C7—O3	124.0 (5)
O6—N1—C14	120.4 (6)	O4—C7—C6	123.7 (5)
O5—N1—C14	116.7 (6)	O3—C7—C6	112.4 (4)
O1—C1—C2	125.7 (5)	C9—C8—C6	115.5 (4)
O1—C1—C5	125.2 (5)	C9—C8—H8A	108.4
C2—C1—C5	109.1 (5)	C6—C8—H8A	108.4
C1—C2—C3	105.2 (5)	C9—C8—H8B	108.4
C1—C2—H2A	110.7	C6—C8—H8B	108.4
C3—C2—H2A	110.7	H8A—C8—H8B	107.5
C1—C2—H2B	110.7	C10—C9—C14	114.6 (5)
C3—C2—H2B	110.7	C10—C9—C8	118.6 (5)
H2A—C2—H2B	108.8	C14—C9—C8	126.6 (5)
C4—C3—C2	104.5 (5)	C11—C10—C9	123.6 (6)
C4—C3—H3A	110.9	C11—C10—H10	118.2
C2—C3—H3A	110.9	C9—C10—H10	118.2
C4—C3—H3B	110.9	C10—C11—C12	119.6 (6)
C2—C3—H3B	110.9	C10—C11—H11	120.2
H3A—C3—H3B	108.9	C12—C11—H11	120.2
C3—C4—C5	103.6 (4)	C13—C12—C11	119.5 (5)
C3—C4—H4A	111.0	C13—C12—H12	120.3
C5—C4—H4A	111.0	C11—C12—H12	120.3
C3—C4—H4B	111.0	C12—C13—C14	120.6 (6)
C5—C4—H4B	111.0	C12—C13—H13	119.7
H4A—C4—H4B	109.0	C14—C13—H13	119.7
C1—C5—C6	112.1 (5)	C13—C14—C9	122.1 (5)
C1—C5—C4	103.2 (4)	C13—C14—N1	118.6 (5)
C6—C5—C4	117.9 (4)	C9—C14—N1	119.2 (5)
C1—C5—H5	107.7	O3—C15—H15A	109.5
C6—C5—H5	107.7	O3—C15—H15B	109.5
C4—C5—H5	107.7	H15A—C15—H15B	109.5
O2—C6—C7	107.0 (4)	O3—C15—H15C	109.5
O2—C6—C5	108.8 (4)	H15A—C15—H15C	109.5
C7—C6—C5	112.1 (4)	H15B—C15—H15C	109.5
O2—C6—C8	108.3 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O5	0.82	2.49	3.283 (7)	162
O2—H2···O6	0.82	2.45	3.018 (5)	127

Fig. 1