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2-[Hydroxy(4-nitrophenyl)methyl]-4-methylcyclohexanone

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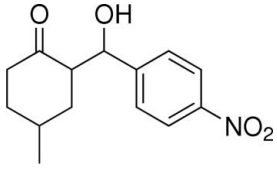
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.009 Å; R factor = 0.069; wR factor = 0.205; data-to-parameter ratio = 7.6.

In the title compound, $C_{14}H_{17}NO_4$, the cyclohexane ring displays a chair conformation. The crystal packing is stabilized by O—H···O hydrogen bonding.

Related literature

For general background, see: Basavaiah *et al.* (1996).



Experimental

Crystal data

$C_{14}H_{17}NO_4$
M_r = 263.29
Monoclinic, $P2_1$
a = 9.917 (3) Å
b = 6.927 (3) Å
c = 10.518 (3) Å
β = 109.35 (3)°
V = 681.7 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
0.25 × 0.20 × 0.18 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
2439 measured reflections
1321 independent reflections
773 reflections with $I > 2\sigma(I)$
R_{int} = 0.124
3 standard reflections
every 300 reflections
intensity decay: 0.6%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.205
S = 0.97
1321 reflections
174 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O4^{i}	0.82	2.31	2.947 (7)	135
O3—H3···O2^{ii}	0.82	2.60	3.206 (8)	132

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

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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2309).

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supplementary materials

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2-[Hydroxy(4-nitrophenyl)methyl]-4-methylcyclohexanone

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Comment

Beta-hydroxy ketone can be transferred to a large variety of important product. The title compound, (I), is an very important intermediate for the construction of α,β-unsaturated carbonyl compound, which are important synthetic building blocks for the construction of many natural products (Basavaiah *et al.*, 1996). We report here the crystal structure of the title compound, (I).

The molecular structure of (I) is shown in Fig. 1. Bond lengths and angles in (I) are normal. The benzene ring and nitro group is slightly non-planar, with a dihedral angle of 4.14 (8)°. The dihedral angle between the benzene and O4/C8/C9/C10 planes is 11.1 (3)°. The crystal packing is stabilized by O—H···O hydrogen bonding (Table 1).

Experimental

To a solution of 4-nitrobenzaldehyde (0.5 mmol) and 4-methylcyclohexanone (5 mmol) in anhydrous dicloromethane (2 ml) was added *L*—*N*-phenylpyrrolidine-2-carboxamide (19 mg, 0.1 mmol). The resulting mixture was stirred at 273 K for 24 h. The reaction mixture was treated with saturated ammonium chloride solution and the aqueous layer was extracted with ethyl acetate, dried over anhydrous $MgSO_4$. After filtration and removal of solvent under reduced pressure, the residue was purified through column chromatography on silica gel to give the title compound. Colourless single crystals of the title compound were obtained by recrystallization from an ethanol solution.

Refinement

Hydroxy and methyl H atoms were placed in calculated positions with O—H = 0.82 and C—H = 0.96 Å, and torsion angles were refined, U_{iso}(H) = 1.5U_{eq}(O,*C*). Other H atoms were placed in calculated positions with C—H = 0.93 to 0.98 Å and refined using a riding model with U_{iso}(H) =1.2U_{eq}(C). In the absence of significant anomalous scattering effects, Friedel pairs were averaged.

Figures

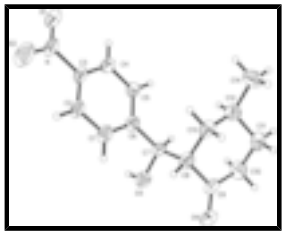

Fig. 1. The molecular structure of (I) with 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

2-[Hydroxy(4-nitrophenyl)methyl]-4-methylcyclohexanone

Crystal data

$C_{14}H_{17}NO_4$	$F_{000} = 280$
$M_r = 263.29$	$D_x = 1.283$ Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2yb	Cell parameters from 28 reflections
$a = 9.917$ (3) Å	$\theta = 4.9$–9.2^o
$b = 6.927$ (3) Å	$\mu = 0.09$ mm^{-1}
$c = 10.518$ (3) Å	$T = 293$ (2) K
$\beta = 109.35$ (3)^o	Block, colourless
$V = 681.7$ (4) Å^3	0.25 × 0.20 × 0.18 mm
$Z = 2$	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.124$
Radiation source: fine-focus sealed tube	$\theta_{max} = 25.2^o$
Monochromator: graphite	$\theta_{min} = 2.0^o$
$T = 293$(2) K	$h = -11 \rightarrow 0$
$\omega/2\theta$ scans	$k = -8 \rightarrow 8$
Absorption correction: none	$l = -11 \rightarrow 12$
2439 measured reflections	3 standard reflections
1321 independent reflections	every 300 reflections
773 reflections with $I > 2\sigma(I)$	intensity decay: 0.6%

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.069$	$w = 1/[\sigma^2(F_o^2) + (0.1226P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.205$	$(\Delta/\sigma)_{max} < 0.001$
$S = 0.97$	$\Delta\rho_{max} = 0.28$ e Å^{-3}
1321 reflections	$\Delta\rho_{min} = -0.22$ e Å^{-3}
174 parameters	Extinction correction: none
1 restraint	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

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 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}
O1	0.3277 (6)	−0.0005 (10)	0.9818 (5)	0.0900 (17)
O2	0.2949 (8)	0.2523 (11)	1.0846 (6)	0.112 (2)
O3	0.4801 (5)	0.7878 (7)	0.6164 (5)	0.0666 (12)
H3	0.5562	0.7380	0.6605	0.100*
O4	0.2824 (6)	1.0356 (8)	0.3885 (5)	0.0797 (15)
N	0.3154 (6)	0.1751 (11)	0.9901 (5)	0.0728 (19)
C1	0.3416 (7)	0.6009 (11)	0.7890 (6)	0.0640 (18)
H1	0.3413	0.7348	0.7957	0.077*
C2	0.3250 (7)	0.4906 (10)	0.8923 (6)	0.0621 (17)
H2	0.3114	0.5478	0.9671	0.075*
C3	0.3294 (6)	0.2947 (11)	0.8807 (6)	0.0546 (15)
C4	0.3480 (7)	0.2079 (9)	0.7711 (6)	0.0615 (18)
H4	0.3524	0.0741	0.7664	0.074*
C5	0.3602 (7)	0.3203 (10)	0.6679 (6)	0.0581 (16)
H5	0.3696	0.2616	0.5917	0.070*
C6	0.3587 (6)	0.5168 (9)	0.6757 (5)	0.0493 (14)
C7	0.3760 (6)	0.6408 (9)	0.5629 (6)	0.0491 (14)
H7	0.4063	0.5594	0.5011	0.059*
C8	0.2362 (6)	0.7441 (9)	0.4842 (6)	0.0562 (16)
H8	0.2112	0.8309	0.5465	0.067*
C9	0.2614 (6)	0.8666 (10)	0.3738 (6)	0.0573 (16)
C10	0.2588 (9)	0.7609 (13)	0.2488 (7)	0.078 (2)
H10A	0.3451	0.6842	0.2680	0.094*
H10B	0.2581	0.8539	0.1797	0.094*
C11	0.1309 (8)	0.6310 (13)	0.1962 (6)	0.0726 (19)
H11A	0.1376	0.5592	0.1194	0.087*
H11B	0.0449	0.7090	0.1660	0.087*
C12	0.1203 (6)	0.4908 (11)	0.3032 (6)	0.0654 (17)
H12	0.2088	0.4152	0.3332	0.078*
C13	0.1114 (6)	0.6068 (11)	0.4240 (6)	0.0636 (17)
H13A	0.0235	0.6810	0.3962	0.076*
H13B	0.1067	0.5175	0.4935	0.076*
C14	−0.0036 (9)	0.3504 (14)	0.2514 (9)	0.093 (3)

H14A	0.0054	0.2806	0.1757	0.140*
H14B	−0.0921	0.4206	0.2240	0.140*
H14C	−0.0025	0.2613	0.3216	0.140*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.120 (4)	0.083 (4)	0.070 (3)	−0.017 (3)	0.035 (3)	0.020 (3)
O2	0.166 (6)	0.118 (5)	0.080 (4)	0.025 (5)	0.078 (4)	0.025 (4)
O3	0.065 (2)	0.069 (3)	0.067 (3)	−0.009 (2)	0.025 (2)	0.009 (2)
O4	0.104 (4)	0.053 (3)	0.078 (3)	−0.001 (3)	0.024 (3)	0.013 (3)
N	0.086 (4)	0.089 (6)	0.050 (3)	0.005 (3)	0.031 (3)	0.022 (3)
C1	0.080 (4)	0.063 (4)	0.055 (4)	0.017 (3)	0.031 (3)	0.007 (3)
C2	0.084 (4)	0.066 (5)	0.045 (3)	0.008 (4)	0.032 (3)	0.008 (3)
C3	0.053 (3)	0.068 (5)	0.044 (3)	−0.004 (3)	0.018 (3)	0.012 (3)
C4	0.076 (4)	0.051 (4)	0.057 (4)	−0.005 (3)	0.022 (3)	0.008 (3)
C5	0.080 (4)	0.054 (4)	0.047 (3)	−0.007 (3)	0.031 (3)	−0.002 (3)
C6	0.054 (3)	0.051 (4)	0.048 (3)	0.002 (3)	0.023 (3)	0.004 (3)
C7	0.059 (3)	0.045 (3)	0.049 (3)	0.004 (3)	0.026 (3)	0.005 (3)
C8	0.073 (4)	0.053 (4)	0.051 (3)	0.009 (3)	0.033 (3)	0.005 (3)
C9	0.066 (4)	0.059 (5)	0.050 (3)	0.009 (3)	0.023 (3)	0.015 (3)
C10	0.106 (5)	0.090 (6)	0.054 (4)	−0.017 (5)	0.047 (4)	0.006 (4)
C11	0.081 (4)	0.085 (5)	0.052 (4)	0.006 (4)	0.022 (3)	0.005 (4)
C12	0.062 (3)	0.070 (5)	0.062 (4)	−0.006 (4)	0.020 (3)	0.003 (3)
C13	0.063 (4)	0.073 (5)	0.059 (4)	0.006 (3)	0.027 (3)	0.017 (3)
C14	0.083 (5)	0.085 (6)	0.105 (6)	−0.022 (4)	0.022 (4)	−0.009 (5)

Geometric parameters (Å, °)

O1—N	1.229 (8)	C8—C13	1.523 (9)
O2—N	1.204 (8)	C8—C9	1.524 (8)
O3—C7	1.426 (8)	C8—H8	0.9800
O3—H3	0.8200	C9—C10	1.498 (9)
O4—C9	1.190 (9)	C10—C11	1.503 (11)
N—C3	1.461 (8)	C10—H10A	0.9700
C1—C2	1.382 (9)	C10—H10B	0.9700
C1—C6	1.387 (8)	C11—C12	1.516 (9)
C1—H1	0.9300	C11—H11A	0.9700
C2—C3	1.364 (10)	C11—H11B	0.9700
C2—H2	0.9300	C12—C14	1.519 (10)
C3—C4	1.366 (9)	C12—C13	1.531 (9)
C4—C5	1.373 (9)	C12—H12	0.9800
C4—H4	0.9300	C13—H13A	0.9700
C5—C6	1.365 (9)	C13—H13B	0.9700
C5—H5	0.9300	C14—H14A	0.9600
C6—C7	1.520 (7)	C14—H14B	0.9600
C7—C8	1.535 (8)	C14—H14C	0.9600
C7—H7	0.9800		

C7—O3—H3	109.5	O4—C9—C10	123.1 (6)
O2—N—O1	123.1 (6)	O4—C9—C8	120.9 (6)
O2—N—C3	119.0 (7)	C10—C9—C8	116.0 (6)
O1—N—C3	117.9 (6)	C9—C10—C11	112.5 (5)
C2—C1—C6	121.6 (7)	C9—C10—H10A	109.1
C2—C1—H1	119.2	C11—C10—H10A	109.1
C6—C1—H1	119.2	C9—C10—H10B	109.1
C3—C2—C1	117.7 (6)	C11—C10—H10B	109.1
C3—C2—H2	121.2	H10A—C10—H10B	107.8
C1—C2—H2	121.2	C10—C11—C12	111.6 (5)
C2—C3—C4	122.0 (6)	C10—C11—H11A	109.3
C2—C3—N	118.6 (6)	C12—C11—H11A	109.3
C4—C3—N	119.4 (6)	C10—C11—H11B	109.3
C3—C4—C5	119.4 (6)	C12—C11—H11B	109.3
C3—C4—H4	120.3	H11A—C11—H11B	108.0
C5—C4—H4	120.3	C11—C12—C14	113.0 (6)
C6—C5—C4	120.8 (6)	C11—C12—C13	108.5 (6)
C6—C5—H5	119.6	C14—C12—C13	111.9 (5)
C4—C5—H5	119.6	C11—C12—H12	107.8
C5—C6—C1	118.6 (6)	C14—C12—H12	107.8
C5—C6—C7	120.7 (5)	C13—C12—H12	107.8
C1—C6—C7	120.8 (6)	C8—C13—C12	114.3 (4)
O3—C7—C6	110.5 (5)	C8—C13—H13A	108.7
O3—C7—C8	106.6 (5)	C12—C13—H13A	108.7
C6—C7—C8	111.6 (4)	C8—C13—H13B	108.7
O3—C7—H7	109.4	C12—C13—H13B	108.7
C6—C7—H7	109.4	H13A—C13—H13B	107.6
C8—C7—H7	109.4	C12—C14—H14A	109.5
C13—C8—C9	110.3 (5)	C12—C14—H14B	109.5
C13—C8—C7	113.4 (5)	H14A—C14—H14B	109.5
C9—C8—C7	108.9 (4)	C12—C14—H14C	109.5
C13—C8—H8	108.0	H14A—C14—H14C	109.5
C9—C8—H8	108.0	H14B—C14—H14C	109.5
C7—C8—H8	108.0		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O4^{i}	0.82	2.31	2.947 (7)	135
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Fig. 1