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1-(5-Methyl-3-phenylisoxazol-4-yl)-ethanone

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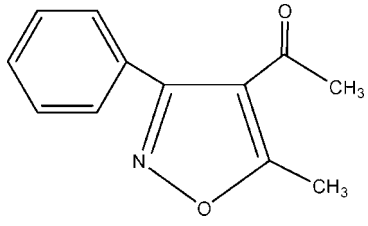
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.061; wR factor = 0.257; data-to-parameter ratio = 17.6.

In the title compound, $C_{12}H_{11}NO_2$, synthesized by 1,3-dipolar cycloaddition reaction of nitrile oxides with sodium pentane-2,4-dionate, all bond lengths and angles are normal. In the molecule, the isoxazole and phenyl rings make a dihedral angle of 84.8 (1)°.

Related literature

The synthesis of 1-(5-methyl-3-phenylisoxazol-4-yl)ethanone was described by Doyle *et al.* (1963); for the crystal structures of related complexes see Higgins *et al.* (1997).

For related literature, see: Hanson & Mohamed (1997); Lin *et al.* (1997); Martins *et al.* (2000).



Experimental

Crystal data

$C_{12}H_{11}NO_2$
M_r = 201.22
Monoclinic, $P2_1/n$
a = 14.090 (3) Å
b = 5.2504 (11) Å
c = 14.280 (3) Å
β = 92.60 (3)°
V = 1055.3 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
0.70 × 0.50 × 0.20 mm

Data collection

Rigaku R-AXIS SPIDER diffractometer
Absorption correction: multi-scan (using intensity measurements) (Higashi, 1995)
T_{min} = 0.942, T_{max} = 0.983
10361 measured reflections
2410 independent reflections
1444 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.257
S = 1.05
2410 reflections
137 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Data collection: *RAPID-AUTO* (Rigaku 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); 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: CV2244).

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supplementary materials

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1-(5-Methyl-3-phenylisoxazol-4-yl)ethanone

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Comment

Isoxazole derivatives exhibit anticonvulsant, antibacterial, antiasthmatic, and other pharmacological activities (Lin *et al.*, 1997). Isoxazoles are typically prepared by the reaction of nitrile oxides with alkynes (Hanson & Mohamed, 1997) or olefine. In addition they are synthesized by cyclization of the adducts of alpha, beta-unsaturated ketones (or aldehydes) and hydroxylamines (Martins *et al.*, 2000). In this article, we report here the crystal structure of 1-(5-methyl-3-phenyl-isoxazol −4-yl)ethanone synthesized by 1,3-Dipolar Cycloaddition reaction of nitrile oxideswith sodioacetylacetone (Doyle *et al.*, 1963).

Experimental

A solution of alpha-chlorobenzaldoxime(0.02 mol) in methanol was added slowly to a stirred solution of sodio-acetylacetone(0.026 mol) at 268–273 K in ice-salt bath. The mixture was stirred for 2 h and allowed to warm to room temperature, then kept on stirring for 2 h. After finished the reaction, the residue was shaken with water(200 ml), filtrated. The solid was crystallized from ethanol and water to give colorless prism crystals (yield 63.8%). *M*.p. 336 k. Analysis, found (calculated for $C_{12}H_{11}NO_2$): C 71.63 (71.61%) H 5.51(5.54%) N 6.96(6.94%). Crystals were grown from a solution of ethanol by slow evaporation.

Refinement

All H atoms were geometrically fixed with C—H = 0.93–0.96 Å, and were treated as riding with U_{iso}(H) = 1.2–1.5Ueq(parent atom).

Figures

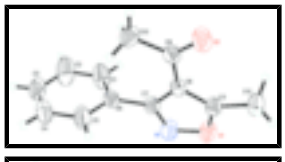

Figure 1
A view of the molecule structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

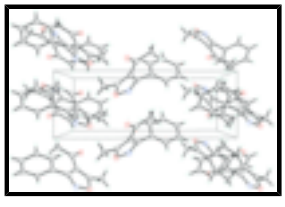

Figure 2 The packing structure of the title compound viewed down the *a* axis.

1-(5-methyl-3-phenylisoxazol-4-yl)ethanone

Crystal data

$C_{12}H_{11}NO_2$	F_{000} = 424
M_r = 201.22	D_x = 1.266 Mg m^{-3}
Monoclinic, $P2_1/n$	Melting point: 336 K
Hall symbol: -P 2yn	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 14.090 (3) Å	Cell parameters from 5329 reflections
b = 5.2504 (11) Å	θ = 3.3–27.5º
c = 14.280 (3) Å	μ = 0.09 mm^{-1}
β = 92.60 (3)º	T = 293 (2) K
V = 1055.3 (4) Å^3	Prism, colourless
Z = 4	0.70 × 0.50 × 0.20 mm

Data collection

Rigaku R-AXIS SPIDER diffractometer	2410 independent reflections
Radiation source: Rotating Anode	1444 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.025
T = 293(2) K	θ_{max} = 27.5º
ω oscillation scans	θ_{min} = 4.0º
Absorption correction: multi-scan Higashi (1995)	h = −18→18
T_{min} = 0.942, T_{max} = 0.983	k = −6→6
10361 measured reflections	l = −18→18

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.15P)^2 + 0.2275P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.061	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.257	$\Delta\rho_{max}$ = 0.24 e Å^{-3}
S = 1.05	$\Delta\rho_{min}$ = −0.22 e Å^{-3}
2410 reflections	Extinction correction: SHELXL
137 parameters	Extinction coefficient: 0.063 (14)

Special details

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	0.25512 (14)	0.1587 (4)	0.82721 (12)	0.0768 (6)
O2	0.46364 (16)	0.6933 (5)	0.89829 (16)	0.0964 (8)
N1	0.19230 (16)	0.2155 (5)	0.89941 (16)	0.0764 (7)
C1	0.1178 (3)	0.6683 (8)	1.0260 (2)	0.1056 (12)
H1	0.1011	0.7383	0.9678	0.127*
C2	0.0734 (3)	0.7530 (11)	1.1046 (3)	0.1268 (16)
H2	0.0286	0.8828	1.0989	0.152*
C3	0.0946 (3)	0.6488 (8)	1.1894 (2)	0.0954 (11)
H3	0.0626	0.7004	1.2417	0.114*
C4	0.1624 (4)	0.4698 (8)	1.1971 (2)	0.1256 (16)
H4	0.1793	0.4015	1.2556	0.151*
C5	0.2073 (4)	0.3863 (7)	1.1188 (2)	0.1178 (15)
H5	0.2535	0.2602	1.1250	0.141*
C6	0.18481 (17)	0.4861 (5)	1.03262 (16)	0.0601 (6)
C7	0.23385 (16)	0.3934 (5)	0.94913 (15)	0.0572 (6)
C8	0.32306 (15)	0.4644 (4)	0.91255 (14)	0.0536 (6)
C9	0.39362 (17)	0.6557 (5)	0.94341 (16)	0.0607 (6)
C10	0.3788 (2)	0.8049 (6)	1.0298 (2)	0.0759 (8)
H10C	0.4307	0.9216	1.0404	0.114*
H10B	0.3759	0.6913	1.0823	0.114*
H10A	0.3204	0.8984	1.0226	0.114*
C11	0.33123 (17)	0.3083 (5)	0.83683 (15)	0.0596 (6)
C12	0.4041 (2)	0.2697 (6)	0.76707 (18)	0.0747 (8)
H12C	0.3831	0.1407	0.7232	0.112*
H12B	0.4626	0.2171	0.7984	0.112*
H12A	0.4141	0.4264	0.7342	0.112*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0791 (12)	0.0874 (13)	0.0643 (11)	−0.0131 (10)	0.0068 (9)	−0.0224 (9)
O2	0.0895 (15)	0.1154 (17)	0.0872 (14)	−0.0362 (12)	0.0347 (12)	−0.0201 (12)
N1	0.0676 (13)	0.0920 (16)	0.0700 (14)	−0.0163 (12)	0.0102 (11)	−0.0163 (12)
C1	0.097 (2)	0.149 (3)	0.0723 (18)	0.047 (2)	0.0205 (16)	0.013 (2)
C2	0.100 (3)	0.185 (4)	0.098 (3)	0.049 (3)	0.031 (2)	−0.010 (3)
C3	0.093 (2)	0.114 (3)	0.083 (2)	−0.023 (2)	0.0432 (17)	−0.0238 (18)
C4	0.200 (5)	0.117 (3)	0.0626 (18)	0.023 (3)	0.041 (2)	0.0083 (19)
C5	0.189 (4)	0.109 (3)	0.0579 (16)	0.054 (3)	0.031 (2)	0.0109 (17)
C6	0.0593 (13)	0.0657 (13)	0.0562 (12)	−0.0088 (11)	0.0110 (9)	−0.0029 (10)
C7	0.0600 (13)	0.0618 (13)	0.0495 (11)	−0.0031 (10)	0.0014 (9)	−0.0015 (10)
C8	0.0594 (12)	0.0574 (12)	0.0443 (10)	−0.0003 (9)	0.0039 (8)	0.0016 (9)
C9	0.0608 (13)	0.0638 (13)	0.0580 (13)	−0.0030 (10)	0.0079 (10)	0.0016 (10)

C10	0.0687 (16)	0.0788 (17)	0.0808 (17)	−0.0105 (13)	0.0092 (13)	−0.0204 (14)
C11	0.0646 (14)	0.0658 (13)	0.0481 (11)	0.0042 (11)	0.0014 (10)	−0.0008 (10)
C12	0.0772 (17)	0.0907 (19)	0.0568 (13)	0.0156 (14)	0.0102 (12)	−0.0067 (13)

Geometric parameters (Å, °)

O1—C11	1.331 (3)	C5—H5	0.9300
O1—N1	1.421 (3)	C6—C7	1.487 (3)
O2—C9	1.219 (3)	C7—C8	1.432 (3)
N1—C7	1.297 (3)	C8—C11	1.366 (3)
C1—C6	1.344 (4)	C8—C9	1.467 (3)
C1—C2	1.383 (5)	C9—C10	1.484 (4)
C1—H1	0.9300	C10—H10C	0.9600
C2—C3	1.350 (6)	C10—H10B	0.9600
C2—H2	0.9300	C10—H10A	0.9600
C3—C4	1.341 (5)	C11—C12	1.477 (3)
C3—H3	0.9300	C12—H12C	0.9600
C4—C5	1.381 (5)	C12—H12B	0.9600
C4—H4	0.9300	C12—H12A	0.9600
C5—C6	1.362 (4)		
C11—O1—N1	109.08 (17)	C8—C7—C6	130.9 (2)
C7—N1—O1	105.53 (19)	C11—C8—C7	103.9 (2)
C6—C1—C2	121.0 (3)	C11—C8—C9	124.6 (2)
C6—C1—H1	119.5	C7—C8—C9	131.5 (2)
C2—C1—H1	119.5	O2—C9—C8	120.4 (2)
C3—C2—C1	120.5 (4)	O2—C9—C10	120.1 (2)
C3—C2—H2	119.8	C8—C9—C10	119.5 (2)
C1—C2—H2	119.8	C9—C10—H10C	109.5
C4—C3—C2	119.1 (3)	C9—C10—H10B	109.5
C4—C3—H3	120.5	H10C—C10—H10B	109.5
C2—C3—H3	120.5	C9—C10—H10A	109.5
C3—C4—C5	120.4 (4)	H10C—C10—H10A	109.5
C3—C4—H4	119.8	H10B—C10—H10A	109.5
C5—C4—H4	119.8	O1—C11—C8	109.8 (2)
C6—C5—C4	120.9 (4)	O1—C11—C12	115.5 (2)
C6—C5—H5	119.5	C8—C11—C12	134.6 (2)
C4—C5—H5	119.5	C11—C12—H12C	109.5
C1—C6—C5	118.1 (3)	C11—C12—H12B	109.5
C1—C6—C7	121.8 (2)	H12C—C12—H12B	109.5
C5—C6—C7	120.1 (3)	C11—C12—H12A	109.5
N1—C7—C8	111.7 (2)	H12C—C12—H12A	109.5
N1—C7—C6	117.4 (2)	H12B—C12—H12A	109.5
C11—O1—N1—C7	−0.6 (3)	N1—C7—C8—C11	−0.9 (3)
C6—C1—C2—C3	−1.9 (7)	C6—C7—C8—C11	178.0 (2)
C1—C2—C3—C4	2.9 (7)	N1—C7—C8—C9	179.0 (2)
C2—C3—C4—C5	−2.5 (7)	C6—C7—C8—C9	−2.1 (4)
C3—C4—C5—C6	1.0 (8)	C11—C8—C9—O2	3.5 (4)
C2—C1—C6—C5	0.4 (6)	C7—C8—C9—O2	−176.3 (3)
C2—C1—C6—C7	−179.6 (4)	C11—C8—C9—C10	−176.9 (2)

C4—C5—C6—C1	0.1 (7)	C7—C8—C9—C10	3.3 (4)
C4—C5—C6—C7	−179.9 (4)	N1—O1—C11—C8	0.1 (3)
O1—N1—C7—C8	0.9 (3)	N1—O1—C11—C12	179.3 (2)
O1—N1—C7—C6	−178.2 (2)	C7—C8—C11—O1	0.4 (3)
C1—C6—C7—N1	−85.0 (4)	C9—C8—C11—O1	−179.4 (2)
C5—C6—C7—N1	95.1 (4)	C7—C8—C11—C12	−178.6 (3)
C1—C6—C7—C8	96.2 (4)	C9—C8—C11—C12	1.5 (4)
C5—C6—C7—C8	−83.8 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
?—?···?	?	?	?	?

Fig. 1

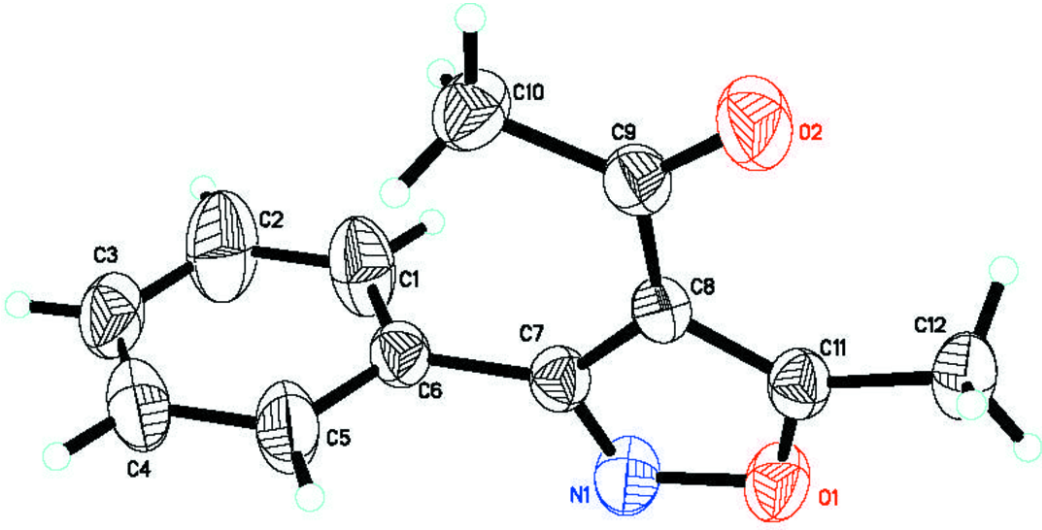

Fig. 2