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Ethyl 2-acetylhydrazono-2-phenylacetate

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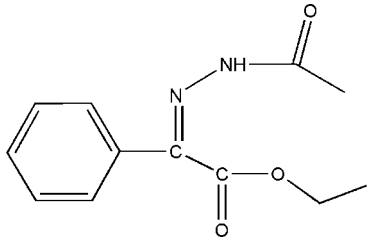
Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–C) = 0.002 Å; R factor = 0.037; wR factor = 0.110; data-to-parameter ratio = 14.1.

The title compound, $C_{12}H_{14}N_2O_3$, was synthesized as an intermediate for the synthesis of metamitron. The benzene ring forms dihedral angles of 86.3 (2) and 10.0 (3)° with the ethyl group and the acetylimino plane, respectively. The crystal structure involves intermolecular C—H···O and N—H···O hydrogen bonds.

Related literature

For related literature, see: Glaser *et al.* (1993); Javier *et al.* (2006); Pan & Gao (2007).



Experimental

Crystal data

$C_{12}H_{14}N_2O_3$
M_r = 234.25
Orthorhombic, *Pbca*
a = 9.3039 (19) Å
b = 15.752 (3) Å
c = 17.129 (3) Å
V = 2510.3 (8) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 153 (2) K
0.32 × 0.22 × 0.10 mm

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi 1995)
T_{min} = 0.972, T_{max} = 0.991
18227 measured reflections
2206 independent reflections
1870 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.110
S = 1.09
2206 reflections
156 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O3^{i}	0.86	2.03	2.8737 (16)	165
C9—H9B···O3^{i}	0.97	2.55	3.2023 (19)	124

Symmetry code: (i) $-x+2, -y, -z+1$.

Data collection: *RAPID-AUTO* (Rigaku 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2352).

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supporting information

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S1. Comment

Metamitron(Trade name: Goltix) is a widely used herbicide for the control of grass and broad-leaved weeds in sugar and red beets, fodder beet, and certain strawberry varieties. The dose rates for metamitron are 0.35–4.2 kg active ingredient/ha for all crops. The currently used weed control strategy in sugarbeet involves a mixture of herbicides(phenmedipham, ethofumesate, metamitron, chloridazon *etc*) to control dicotyledonus weeds. 70% wettable powderand has been used for the control of morel goosefoot chickweed Lamium barbatum *etc*. Metamitron can be used before and after the plantingis. It can be applied to the control of the entire crop growing period with better efficacy when it cooperate with others herbicides and pesticides(Javier *et al.*, 2006). The title compound (I) was synthesized as an intermediate for the synthesis of metamitron. We report here the crystal structure of (I).

In (I) (Fig. 1), all bond lengths and angles are normal and in a good agreement with those reported previously (Glaser *et al.*, 1993). The benzene ring plane forms dihedral angles of 86.3 (2)° and 10.0 (3)° with the ethyl plane (O1/O2/C7/C8/C9) and the acetylimino plane (O3/N1/N2/C4/C5/C6/C7/C11/C12), respectively. The crystal structure is stabilized by intermolecular C–H–O and N–H–O hydrogen bonds.

S2. Experimental

Ethyl benzoylformate 12.1 g (6.8 mmol), was dissolved in 20 ml e thanol in a flask equipped with stirrer and reflux condenser. Acethydrazide 5.1 g(6.8 mmol) was slowly added from a dropping-funnel during 30 minutes while maintaining the temperature at 75–80°C for eight hours. Evaporation of portion of the solvent and cooling down the remaining solution in ice water yielded white crystals out after three hours (11.9 g, yield 78.9%) (Pan *et al.*, 2007). Single crystals suitable for X-ray measurement were obtained by recrystallization from petroleum ether at room temperature.

S3. Refinement

All H atoms were found on difference maps. All H atoms were positioned geometrically [N—H = 0.86 Å(NH). C—H = 0.93Å (CH), C—H = 0.97Å (CH_2). C—H = 0.96Å (CH_3). U_{iso}(H) = 1.5 *x* (Methyl) or U_{iso}(H) = 1.2 *x* (other groups)].

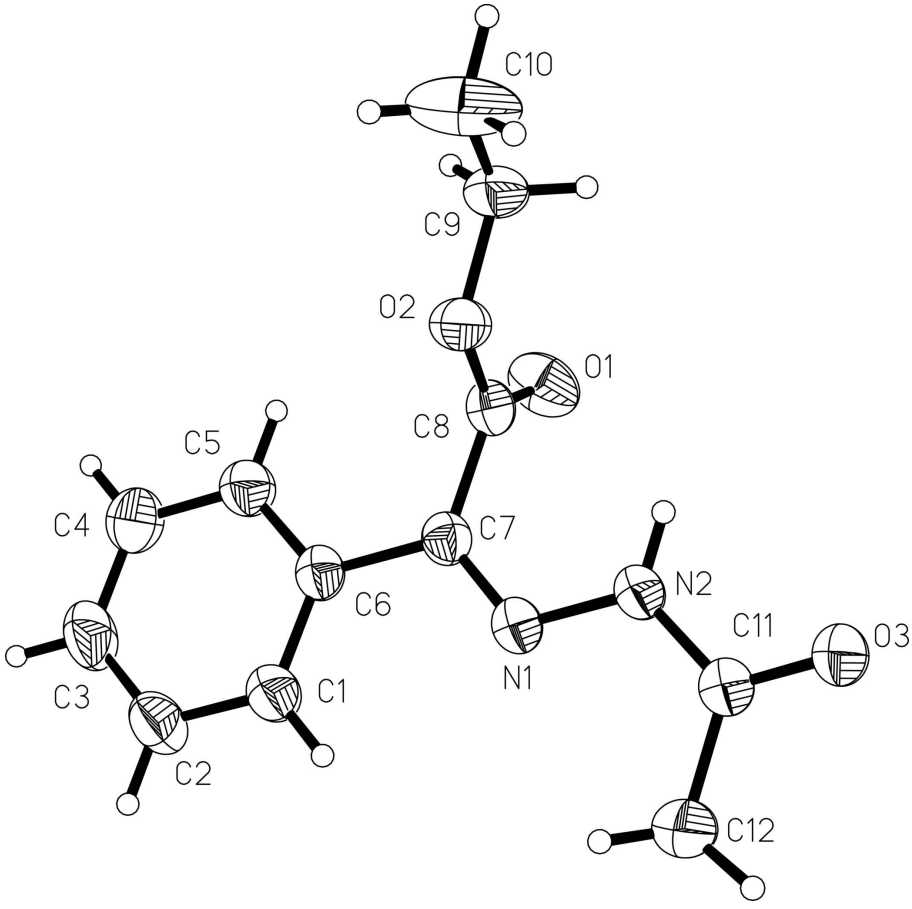

Figure 1

View of the title compound (I), with displacement ellipsoids drawn at the 35% probability level.

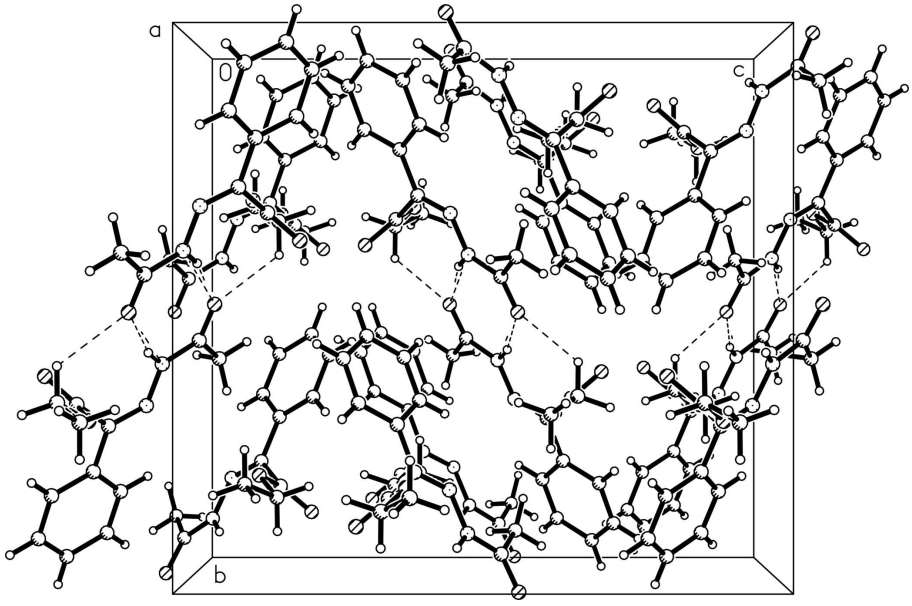

Figure 2

A packing diagram of the molecule of the title compound, viewed down the *a* axis. Hydrogen bonds are shown as dashed lines.

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$C_{12}H_{14}N_2O_3$
$M_r = 234.25$
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Hall symbol: -P 2ac 2ab
$a = 9.3039\ (19)$ Å
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$c = 17.129\ (3)$ Å
$V = 2510.3\ (8)$ Å^3
$Z = 8$
$F(000) = 992$
$D_x = 1.240$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2998 reflections
$\theta = 2.3$–$21.9°$
$\mu = 0.09$ mm^{-1}
$T = 153$ K
Block, colorless
0.32 × 0.22 × 0.10 mm

Data collection

Rigaku R-AXIS Rapid IP area-detector diffractometer
Radiation source: Rotating Anode
Graphite monochromator
ω Oscillation scans
Absorption correction: multi-scan (*ABSCOR*; Higashi 1995)
$T_{min} = 0.972$, $T_{max} = 0.991$
18227 measured reflections
2206 independent reflections
1870 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.2°$
$h = -11 \rightarrow 11$
$k = -18 \rightarrow 18$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.110$
$S = 1.09$
2206 reflections
156 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
$w = 1/[\sigma^2(F_o^2) + (0.0576P)^2 + 0.4358P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.12$ e Å^{-3}
Extinction correction: *SHELXL*, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.030 (2)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.98377 (12)	0.11865 (7)	0.70415 (6)	0.0676 (4)
O2	0.84719 (10)	0.19284 (6)	0.62123 (6)	0.0504 (3)
O3	1.14302 (12)	−0.00717 (6)	0.44126 (6)	0.0628 (3)
N1	1.16667 (12)	0.17432 (7)	0.55542 (6)	0.0456 (3)
N2	1.11668 (13)	0.09905 (7)	0.52543 (6)	0.0485 (3)
H2A	1.0369	0.0782	0.5420	0.058*
C1	1.26329 (16)	0.33271 (9)	0.60630 (8)	0.0514 (4)
H1B	1.3104	0.3071	0.5646	0.062*
C2	1.30914 (19)	0.41054 (10)	0.63304 (9)	0.0616 (4)
H2B	1.3875	0.4369	0.6096	0.074*
C3	1.2401 (2)	0.44963 (10)	0.69408 (10)	0.0682 (5)
H3A	1.2713	0.5024	0.7117	0.082*
C4	1.1246 (2)	0.41045 (10)	0.72922 (10)	0.0675 (5)
H4A	1.0776	0.4368	0.7706	0.081*
C5	1.07824 (17)	0.33195 (9)	0.70302 (8)	0.0538 (4)
H5A	1.0005	0.3057	0.7271	0.065*
C6	1.14696 (14)	0.29215 (8)	0.64113 (7)	0.0416 (3)
C7	1.09697 (14)	0.20901 (8)	0.61165 (7)	0.0405 (3)
C8	0.97033 (15)	0.16787 (8)	0.65146 (7)	0.0422 (3)
C9	0.71683 (16)	0.15866 (11)	0.65747 (9)	0.0574 (4)
H9A	0.7085	0.1788	0.7108	0.069*
H9B	0.7202	0.0971	0.6582	0.069*
C10	0.5936 (2)	0.18798 (17)	0.61093 (13)	0.1044 (9)
H10A	0.5062	0.1667	0.6334	0.157*
H10B	0.6029	0.1675	0.5584	0.157*
H10C	0.5914	0.2489	0.6107	0.157*
C11	1.19254 (16)	0.05777 (9)	0.46999 (8)	0.0494 (4)
C12	1.33423 (19)	0.09243 (12)	0.44570 (12)	0.0758 (5)
H12A	1.3687	0.0617	0.4011	0.114*

H12B	1.4015	0.0867	0.4878	0.114*
H12C	1.3240	0.1513	0.4325	0.114*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0664 (7)	0.0730 (7)	0.0634 (6)	−0.0111 (6)	−0.0089 (5)	0.0261 (6)
O2	0.0413 (6)	0.0526 (6)	0.0575 (6)	0.0010 (4)	0.0043 (4)	0.0092 (4)
O3	0.0619 (7)	0.0551 (6)	0.0715 (7)	−0.0139 (5)	0.0159 (5)	−0.0241 (5)
N1	0.0468 (6)	0.0391 (6)	0.0508 (6)	−0.0053 (5)	0.0006 (5)	−0.0052 (5)
N2	0.0485 (7)	0.0406 (6)	0.0564 (7)	−0.0102 (5)	0.0089 (5)	−0.0100 (5)
C1	0.0531 (8)	0.0463 (8)	0.0548 (8)	−0.0067 (6)	0.0027 (7)	−0.0031 (6)
C2	0.0647 (10)	0.0519 (9)	0.0682 (10)	−0.0166 (8)	0.0005 (8)	−0.0013 (7)
C3	0.0787 (12)	0.0473 (9)	0.0785 (11)	−0.0158 (8)	−0.0044 (9)	−0.0145 (8)
C4	0.0750 (11)	0.0591 (9)	0.0683 (10)	−0.0048 (8)	0.0055 (8)	−0.0229 (8)
C5	0.0539 (9)	0.0521 (8)	0.0553 (8)	−0.0055 (7)	0.0032 (7)	−0.0076 (7)
C6	0.0435 (7)	0.0372 (7)	0.0442 (7)	0.0002 (5)	−0.0062 (5)	−0.0004 (5)
C7	0.0406 (7)	0.0372 (7)	0.0436 (7)	−0.0002 (5)	−0.0036 (5)	0.0007 (5)
C8	0.0473 (8)	0.0376 (7)	0.0416 (7)	−0.0023 (5)	−0.0030 (6)	−0.0020 (6)
C9	0.0470 (9)	0.0641 (9)	0.0610 (9)	−0.0050 (7)	0.0153 (7)	−0.0018 (7)
C10	0.0464 (11)	0.174 (3)	0.0925 (14)	−0.0096 (13)	−0.0001 (10)	0.0327 (15)
C11	0.0495 (8)	0.0437 (7)	0.0551 (8)	−0.0041 (6)	0.0056 (6)	−0.0057 (6)
C12	0.0617 (10)	0.0691 (11)	0.0964 (13)	−0.0160 (8)	0.0270 (9)	−0.0234 (10)

Geometric parameters (Å, º)

O1—C8	1.1964 (16)	C4—H4A	0.9300
O2—C8	1.3173 (16)	C5—C6	1.3878 (19)
O2—C9	1.4650 (17)	C5—H5A	0.9300
O3—C11	1.2251 (16)	C6—C7	1.4787 (18)
N1—C7	1.2832 (17)	C7—C8	1.5078 (19)
N1—N2	1.3733 (15)	C9—C10	1.471 (2)
N2—C11	1.3501 (18)	C9—H9A	0.9700
N2—H2A	0.8600	C9—H9B	0.9700
C1—C2	1.377 (2)	C10—H10A	0.9600
C1—C6	1.391 (2)	C10—H10B	0.9600
C1—H1B	0.9300	C10—H10C	0.9600
C2—C3	1.373 (2)	C11—C12	1.486 (2)
C2—H2B	0.9300	C12—H12A	0.9600
C3—C4	1.378 (2)	C12—H12B	0.9600
C3—H3A	0.9300	C12—H12C	0.9600
C4—C5	1.384 (2)		
C8—O2—C9	116.34 (11)	C6—C7—C8	118.16 (11)
C7—N1—N2	118.50 (11)	O1—C8—O2	125.51 (13)
C11—N2—N1	120.12 (11)	O1—C8—C7	122.55 (12)
C11—N2—H2A	119.9	O2—C8—C7	111.94 (11)
N1—N2—H2A	119.9	O2—C9—C10	107.46 (13)

C2—C1—C6	120.50 (14)	O2—C9—H9A	110.2
C2—C1—H1B	119.8	C10—C9—H9A	110.2
C6—C1—H1B	119.8	O2—C9—H9B	110.2
C3—C2—C1	120.52 (15)	C10—C9—H9B	110.2
C3—C2—H2B	119.7	H9A—C9—H9B	108.5
C1—C2—H2B	119.7	C9—C10—H10A	109.5
C2—C3—C4	119.78 (15)	C9—C10—H10B	109.5
C2—C3—H3A	120.1	H10A—C10—H10B	109.5
C4—C3—H3A	120.1	C9—C10—H10C	109.5
C3—C4—C5	120.10 (15)	H10A—C10—H10C	109.5
C3—C4—H4A	120.0	H10B—C10—H10C	109.5
C5—C4—H4A	120.0	O3—C11—N2	119.22 (13)
C4—C5—C6	120.51 (15)	O3—C11—C12	121.86 (13)
C4—C5—H5A	119.7	N2—C11—C12	118.92 (13)
C6—C5—H5A	119.7	C11—C12—H12A	109.5
C5—C6—C1	118.59 (12)	C11—C12—H12B	109.5
C5—C6—C7	121.07 (12)	H12A—C12—H12B	109.5
C1—C6—C7	120.33 (12)	C11—C12—H12C	109.5
N1—C7—C6	118.33 (12)	H12A—C12—H12C	109.5
N1—C7—C8	123.46 (11)	H12B—C12—H12C	109.5

C7—N1—N2—C11	−175.43 (12)	C1—C6—C7—N1	2.49 (19)
C6—C1—C2—C3	−0.5 (2)	C5—C6—C7—C8	−0.87 (19)
C1—C2—C3—C4	0.4 (3)	C1—C6—C7—C8	−179.93 (12)
C2—C3—C4—C5	0.0 (3)	C9—O2—C8—O1	1.9 (2)
C3—C4—C5—C6	−0.3 (3)	C9—O2—C8—C7	−178.08 (11)
C4—C5—C6—C1	0.2 (2)	N1—C7—C8—O1	85.17 (18)
C4—C5—C6—C7	−178.90 (14)	C6—C7—C8—O1	−92.28 (16)
C2—C1—C6—C5	0.2 (2)	N1—C7—C8—O2	−94.86 (15)
C2—C1—C6—C7	179.29 (13)	C6—C7—C8—O2	87.69 (14)
N2—N1—C7—C6	−177.00 (11)	C8—O2—C9—C10	−174.94 (15)
N2—N1—C7—C8	5.56 (19)	N1—N2—C11—O3	−176.56 (13)
C5—C6—C7—N1	−178.46 (12)	N1—N2—C11—C12	3.6 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O3^{i}	0.86	2.03	2.8737 (16)	165
C9—H9*B*···O3^{i}	0.97	2.55	3.2023 (19)	124

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