

# Ethyl (4-oxo-1-phenyl-1,4-dihydro-5H-pyrazolo[3,4-d]pyrimidin-5-yl)acetate

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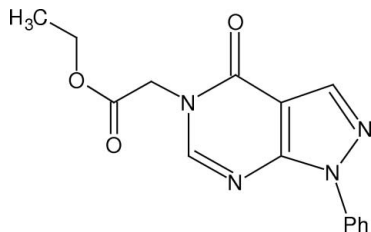
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Key indicators: single-crystal X-ray study;  $T = 173$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.033;  $wR$  factor = 0.089; data-to-parameter ratio = 13.2.

Geometric parameters of the title compound,  $\text{C}_{14}\text{H}_{12}\text{N}_4\text{O}_3$ , a pyrazolopyrimidine derivative, are in the usual ranges. The dihedral angle between the pyrazolopyrimidine system and the phenyl ring is  $4.64$  ( $5^\circ$ ). The non-H atoms of the ester side chain lie in a common plane (r.m.s. deviation =  $0.028$  Å) and this plane is almost perpendicular [ $77.69$  ( $4^\circ$ )] to the central ring system.

## Related literature

For related structures, see: Wen *et al.* (2004); Oliveira-Campos *et al.* (2006), Portilla *et al.* (2005), Yathirajan *et al.*, (2007). For related literature, see: Garg *et al.* (1990); El-Feky & Abd El-Samii (1996); Ismail, *et al.* (2003); Devesa, *et al.* (2004) and Russo *et al.* (1993).



## Experimental

### Crystal data

$\text{C}_{15}\text{H}_{14}\text{N}_4\text{O}_3$   
 $M_r = 298.30$   
Monoclinic,  $P2_1/c$   
 $a = 7.5794$  (8) Å  
 $b = 11.1125$  (9) Å  
 $c = 16.9767$  (14) Å  
 $\beta = 99.654$  ( $8^\circ$ )  
 $V = 1409.6$  (2) Å<sup>3</sup>  
 $Z = 4$   
Mo  $K\alpha$  radiation  
 $\mu = 0.10$  mm<sup>-1</sup>  
 $T = 173$  (2) K  
 $0.42 \times 0.42 \times 0.39$  mm

### Data collection

Stoe IPDSII two-circle diffractometer  
Absorption correction: none  
13151 measured reflections  
2639 independent reflections  
2246 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.037$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$   
 $wR(F^2) = 0.089$   
 $S = 1.02$   
2639 reflections  
200 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.22$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.18$  e Å<sup>-3</sup>

Data collection: *X-Area* (Stoe & Cie, 2001); cell refinement: *X-Area*; data reduction: *X-Area*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2372).

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**supplementary materials**

*Acta Cryst.* (2007). E63, o2718 [ doi:10.1107/S1600536807020284 ]

## Ethyl (4-oxo-1-phenyl-1,4-dihydro-5H-pyrazolo[3,4-d]pyrimidin-5-yl)acetate

H. S. Yathirajan, S. Bindya, B. K. Sarojini, B. Narayana and M. Bolte

### Comment

Pyrazolo[3,4-d]pyrimidines and their derivatives are of interest as potential bioactive molecules. Various pyrazolopyrimidine derivatives are reported to have antileishmanial, antihypertensive, antibacterial and antifungal, antiangiogenic, antiinflammatory and analgesic activities. A new pyrazolopyrimidine derivative, (I),  $C_{15}H_{14}N_4O_3$ , has been synthesized and the crystal structure is reported. The dihedral angle between the pyrazolopyrimidine moiety and the phenyl ring is  $4.64(5)^\circ$ . The non-H atoms of the ester side chain lie in a common plane (r.m.s. deviation  $0.028 \text{ \AA}$ ) and this plane is almost perpendicular [ $77.69(4)^\circ$ ] to the central ring system.

### Experimental

4-Hydroxy-1-phenyl pyrazolo[3,4-d]pyrimidine (21.2 g, 0.1 mol) in 180 ml acetone was stirred with (16.5 g, 0.12 mol) of anhydrous potassium carbonate at room temperature and ethyl chloroacetate (10.8 g, 0.1 mol) was added to it in drops. The reaction mixture was then refluxed for 8 h. Progress of the reaction was monitored by TLC. The acetone was distilled out and the residue was diluted with 250 ml water. The solid obtained was filtered, washed with water and then recrystallized from methanol to obtain the compound as white needles (Yield : 95%; m.p.: 401-404 K). Crystals suitable for X-ray diffraction were obtained from acetone by slow evaporation. Analysis for  $C_{15}H_{14}N_4O_3$ : Found( Calculated): C: 60.28 (60.40); H: 4.61 (4.73); N: 18.63% (18.78%).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  1.28 (t, 3H,  $\text{CH}_3$ ),  $\delta$  4.24 (q, 2H,  $\text{CH}_2$ ),  $\delta$  4.72 (s, 2H,  $-\text{CH}_2-$ ), 7.34 (t, 1H, ArH), 7.49 (t, 2H, ArH), 7.97 (s, 1H, ArH), 8.01 (d ( $J=8.4$ ), 1H, ArH), 8.24 (s, 1H, ArH).

### Refinement

H atoms were found in a difference map, but they were refined using a riding model with  $C_{\text{aromatic}}-\text{H} = 0.95 \text{ \AA}$ ,  $C_{\text{methyl}}-\text{H} = 0.98 \text{ \AA}$  or  $C_{\text{methylene}}-\text{H} = 0.99 \text{ \AA}$  and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  or  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$ .

### Figures

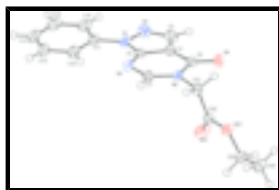


Fig. 1. The molecular structure of (I) with the atom numbering; displacement ellipsoids are at the 50% probability level.

## Ethyl (4-oxo-1-phenyl-1,4-dihydro-5H-pyrazolo[3,4-d]pyrimidin-5-yl)acetate

### Crystal data

$C_{15}H_{14}N_4O_3$	$F_{000} = 624$
$M_r = 298.30$	$D_x = 1.406 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
Hall symbol: -P 2ybc	$\lambda = 0.71073 \text{ \AA}$
$a = 7.5794 (8) \text{ \AA}$	Cell parameters from 13263 reflections
$b = 11.1125 (9) \text{ \AA}$	$\theta = 3.7\text{--}25.7^\circ$
$c = 16.9767 (14) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$\beta = 99.654 (8)^\circ$	$T = 173 (2) \text{ K}$
$V = 1409.6 (2) \text{ \AA}^3$	Block, colourless
$Z = 4$	$0.42 \times 0.42 \times 0.39 \text{ mm}$

### Data collection

Stoe IPDSII two-circle diffractometer	2246 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{\text{int}} = 0.037$
Monochromator: graphite	$\theta_{\text{max}} = 25.6^\circ$
$T = 173(2) \text{ K}$	$\theta_{\text{min}} = 3.7^\circ$
$\omega$ scans	$h = -8 \rightarrow 9$
Absorption correction: none	$k = -13 \rightarrow 12$
13151 measured reflections	$l = -20 \rightarrow 20$
2639 independent reflections	

### Refinement

Refinement on $F^2$	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0484P)^2 + 0.3352P]$
$R[F^2 > 2\sigma(F^2)] = 0.033$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.089$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.02$	$\Delta\rho_{\text{max}} = 0.22 \text{ e \AA}^{-3}$
2639 reflections	$\Delta\rho_{\text{min}} = -0.18 \text{ e \AA}^{-3}$
200 parameters	Extinction correction: SHELXL97,
Primary atom site location: structure-invariant direct methods	$F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.020 (2)
Hydrogen site location: inferred from neighbouring sites	

# Special details

## Experimental. ;

**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.** 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 ( $\text{\AA}^2$ )

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.24290 (13)	0.61443 (10)	0.49115 (6)	0.0247 (2)
N2	0.22207 (16)	0.73702 (10)	0.47946 (7)	0.0325 (3)
C3	0.26957 (19)	0.76037 (12)	0.40955 (8)	0.0314 (3)
H3	0.2687	0.8383	0.3865	0.038*
C4	0.32179 (16)	0.65424 (11)	0.37357 (7)	0.0238 (3)
C5	0.30268 (15)	0.56255 (11)	0.42729 (7)	0.0216 (3)
N6	0.33727 (14)	0.44297 (9)	0.41782 (6)	0.0238 (2)
C7	0.38414 (16)	0.41908 (11)	0.34901 (7)	0.0235 (3)
H7	0.4084	0.3373	0.3386	0.028*
N8	0.40173 (14)	0.50088 (9)	0.29023 (6)	0.0216 (2)
C9	0.37669 (16)	0.62594 (11)	0.29877 (7)	0.0228 (3)
O9	0.40183 (13)	0.69497 (8)	0.24495 (5)	0.0322 (2)
C11	0.19631 (16)	0.56162 (13)	0.56226 (7)	0.0265 (3)
C12	0.20112 (18)	0.43756 (13)	0.57351 (8)	0.0310 (3)
H12	0.2372	0.3861	0.5344	0.037*
C13	0.15252 (18)	0.38940 (15)	0.64270 (8)	0.0354 (3)
H13	0.1561	0.3048	0.6507	0.043*
C14	0.09882 (18)	0.46431 (15)	0.70013 (8)	0.0380 (4)
H14	0.0646	0.4312	0.7469	0.046*
C15	0.09596 (19)	0.58721 (16)	0.68826 (8)	0.0400 (4)
H15	0.0597	0.6385	0.7274	0.048*
C16	0.14538 (18)	0.63769 (14)	0.61984 (8)	0.0347 (3)
H16	0.1443	0.7225	0.6126	0.042*
C21	0.45008 (17)	0.45945 (11)	0.21501 (7)	0.0233 (3)
H21A	0.5033	0.3781	0.2227	0.028*
H21B	0.5416	0.5141	0.1995	0.028*
C22	0.28981 (17)	0.45528 (11)	0.14821 (7)	0.0236 (3)
O22	0.13704 (13)	0.47698 (11)	0.15424 (6)	0.0420 (3)
O23	0.34468 (12)	0.42116 (8)	0.08081 (5)	0.0279 (2)
C24	0.20836 (18)	0.41720 (13)	0.00853 (7)	0.0313 (3)
H24A	0.1594	0.4987	−0.0047	0.038*

## supplementary materials

H24B	0.1089	0.3634	0.0167	0.038*
C25	0.2988 (2)	0.37021 (14)	−0.05772 (8)	0.0376 (4)
H25A	0.2118	0.3661	−0.1074	0.056*
H25B	0.3467	0.2896	−0.0438	0.056*
H25C	0.3968	0.4243	−0.0651	0.056*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N1	0.0240 (5)	0.0289 (6)	0.0211 (5)	−0.0003 (4)	0.0039 (4)	−0.0042 (4)
N2	0.0374 (6)	0.0280 (6)	0.0335 (6)	−0.0008 (5)	0.0106 (5)	−0.0084 (5)
C3	0.0388 (8)	0.0233 (7)	0.0339 (7)	−0.0021 (6)	0.0110 (6)	−0.0046 (5)
C4	0.0242 (6)	0.0231 (6)	0.0240 (6)	−0.0024 (5)	0.0035 (5)	−0.0023 (5)
C5	0.0178 (6)	0.0269 (6)	0.0194 (6)	−0.0010 (5)	0.0010 (4)	−0.0022 (5)
N6	0.0260 (5)	0.0252 (6)	0.0205 (5)	0.0025 (4)	0.0046 (4)	0.0017 (4)
C7	0.0263 (6)	0.0216 (6)	0.0226 (6)	0.0012 (5)	0.0039 (5)	0.0020 (5)
N8	0.0263 (5)	0.0204 (5)	0.0184 (5)	0.0008 (4)	0.0041 (4)	0.0000 (4)
C9	0.0244 (6)	0.0203 (6)	0.0231 (6)	−0.0016 (5)	0.0022 (5)	−0.0005 (5)
O9	0.0470 (6)	0.0232 (5)	0.0277 (5)	−0.0013 (4)	0.0104 (4)	0.0044 (4)
C11	0.0172 (6)	0.0423 (8)	0.0196 (6)	−0.0021 (5)	0.0019 (5)	−0.0033 (5)
C12	0.0276 (7)	0.0421 (8)	0.0244 (6)	0.0002 (6)	0.0075 (5)	−0.0007 (6)
C13	0.0284 (7)	0.0506 (9)	0.0276 (7)	−0.0014 (6)	0.0057 (6)	0.0059 (6)
C14	0.0252 (7)	0.0679 (11)	0.0209 (6)	−0.0039 (7)	0.0040 (5)	0.0024 (6)
C15	0.0299 (7)	0.0674 (11)	0.0234 (7)	−0.0003 (7)	0.0065 (6)	−0.0119 (7)
C16	0.0291 (7)	0.0485 (9)	0.0268 (7)	−0.0025 (6)	0.0053 (6)	−0.0095 (6)
C21	0.0266 (6)	0.0245 (6)	0.0200 (6)	0.0025 (5)	0.0073 (5)	−0.0001 (5)
C22	0.0276 (7)	0.0221 (6)	0.0226 (6)	−0.0014 (5)	0.0084 (5)	−0.0018 (5)
O22	0.0274 (5)	0.0700 (8)	0.0298 (5)	0.0030 (5)	0.0080 (4)	−0.0127 (5)
O23	0.0274 (5)	0.0380 (5)	0.0190 (4)	0.0019 (4)	0.0060 (4)	−0.0049 (4)
C24	0.0307 (7)	0.0412 (8)	0.0211 (6)	−0.0028 (6)	0.0013 (5)	−0.0027 (5)
C25	0.0491 (9)	0.0415 (8)	0.0230 (7)	−0.0044 (7)	0.0087 (6)	−0.0049 (6)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

N1—C5	1.3703 (15)	C13—C14	1.394 (2)
N1—N2	1.3818 (16)	C13—H13	0.9500
N1—C11	1.4386 (16)	C14—C15	1.380 (2)
N2—C3	1.3224 (17)	C14—H14	0.9500
C3—C4	1.4148 (17)	C15—C16	1.396 (2)
C3—H3	0.9500	C15—H15	0.9500
C4—C5	1.3911 (17)	C16—H16	0.9500
C4—C9	1.4359 (16)	C21—C22	1.5172 (17)
C5—N6	1.3692 (16)	C21—H21A	0.9900
N6—C7	1.3039 (15)	C21—H21B	0.9900
C7—N8	1.3728 (15)	C22—O22	1.2038 (16)
C7—H7	0.9500	C22—O23	1.3362 (14)
N8—C9	1.4134 (16)	O23—C24	1.4663 (15)
N8—C21	1.4608 (14)	C24—C25	1.5055 (18)
C9—O9	1.2321 (15)	C24—H24A	0.9900

C11—C12	1.391 (2)	C24—H24B	0.9900
C11—C16	1.3948 (18)	C25—H25A	0.9800
C12—C13	1.3954 (18)	C25—H25B	0.9800
C12—H12	0.9500	C25—H25C	0.9800
C5—N1—N2	110.25 (10)	C15—C14—C13	119.22 (13)
C5—N1—C11	130.73 (11)	C15—C14—H14	120.4
N2—N1—C11	118.98 (10)	C13—C14—H14	120.4
C3—N2—N1	106.29 (10)	C14—C15—C16	121.27 (13)
N2—C3—C4	111.22 (12)	C14—C15—H15	119.4
N2—C3—H3	124.4	C16—C15—H15	119.4
C4—C3—H3	124.4	C11—C16—C15	118.94 (14)
C5—C4—C3	105.08 (11)	C11—C16—H16	120.5
C5—C4—C9	119.79 (11)	C15—C16—H16	120.5
C3—C4—C9	135.07 (12)	N8—C21—C22	112.10 (10)
N6—C5—N1	126.62 (11)	N8—C21—H21A	109.2
N6—C5—C4	126.22 (11)	C22—C21—H21A	109.2
N1—C5—C4	107.16 (11)	N8—C21—H21B	109.2
C7—N6—C5	112.90 (10)	C22—C21—H21B	109.2
N6—C7—N8	126.21 (11)	H21A—C21—H21B	107.9
N6—C7—H7	116.9	O22—C22—O23	124.86 (12)
N8—C7—H7	116.9	O22—C22—C21	126.25 (11)
C7—N8—C9	123.05 (10)	O23—C22—C21	108.88 (10)
C7—N8—C21	119.74 (10)	C22—O23—C24	116.63 (10)
C9—N8—C21	117.21 (10)	O23—C24—C25	106.88 (11)
O9—C9—N8	119.73 (11)	O23—C24—H24A	110.3
O9—C9—C4	128.55 (11)	C25—C24—H24A	110.3
N8—C9—C4	111.71 (10)	O23—C24—H24B	110.3
C12—C11—C16	120.54 (12)	C25—C24—H24B	110.3
C12—C11—N1	120.99 (11)	H24A—C24—H24B	108.6
C16—C11—N1	118.46 (13)	C24—C25—H25A	109.5
C11—C12—C13	119.41 (13)	C24—C25—H25B	109.5
C11—C12—H12	120.3	H25A—C25—H25B	109.5
C13—C12—H12	120.3	C24—C25—H25C	109.5
C14—C13—C12	120.60 (15)	H25A—C25—H25C	109.5
C14—C13—H13	119.7	H25B—C25—H25C	109.5
C12—C13—H13	119.7		
C5—N1—N2—C3	−0.40 (14)	C3—C4—C9—O9	4.3 (2)
C11—N1—N2—C3	−178.39 (11)	C5—C4—C9—N8	0.99 (16)
N1—N2—C3—C4	0.25 (15)	C3—C4—C9—N8	−175.48 (14)
N2—C3—C4—C5	−0.01 (15)	C5—N1—C11—C12	−2.80 (19)
N2—C3—C4—C9	176.81 (13)	N2—N1—C11—C12	174.71 (11)
N2—N1—C5—N6	−179.59 (11)	C5—N1—C11—C16	177.43 (12)
C11—N1—C5—N6	−1.9 (2)	N2—N1—C11—C16	−5.06 (16)
N2—N1—C5—C4	0.39 (13)	C16—C11—C12—C13	0.71 (19)
C11—N1—C5—C4	178.07 (11)	N1—C11—C12—C13	−179.06 (11)
C3—C4—C5—N6	179.75 (12)	C11—C12—C13—C14	0.3 (2)
C9—C4—C5—N6	2.33 (19)	C12—C13—C14—C15	−0.7 (2)
C3—C4—C5—N1	−0.23 (13)	C13—C14—C15—C16	0.1 (2)

## supplementary materials

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C9—C4—C5—N1	−177.65 (10)	C12—C11—C16—C15	−1.22 (19)
N1—C5—N6—C7	176.75 (11)	N1—C11—C16—C15	178.56 (11)
C4—C5—N6—C7	−3.22 (18)	C14—C15—C16—C11	0.8 (2)
C5—N6—C7—N8	0.80 (18)	C7—N8—C21—C22	102.44 (13)
N6—C7—N8—C9	2.53 (19)	C9—N8—C21—C22	−78.11 (13)
N6—C7—N8—C21	−178.06 (11)	N8—C21—C22—O22	−3.42 (19)
C7—N8—C9—O9	176.98 (11)	N8—C21—C22—O23	177.69 (10)
C21—N8—C9—O9	−2.45 (17)	O22—C22—O23—C24	3.73 (19)
C7—N8—C9—C4	−3.23 (16)	C21—C22—O23—C24	−177.36 (10)
C21—N8—C9—C4	177.34 (10)	C22—O23—C24—C25	−176.90 (11)
C5—C4—C9—O9	−179.25 (12)		



Fig. 1

