Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

H. S. Yathirajan,[a] S. Bindya,[a] B. K. Sarojini,[b] B. Narayana[c] and Michael Bolte[d]*

[a]Department of Studies in Chemistry, University of Mysore, Manasagangotri, Mysore 570 006, India, [b]Department of Chemistry, P. A. College of Engineering, Nadupadavu, Mangalore 574 153, India, [c]Department of Studies in Chemistry, Mangalore University, Mangalagangotri 574 199, India, and [d]Institut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Max-von-Laue-Strasse 7, 60438 Frankfurt/Main, Germany
Correspondence e-mail: bolte@chemie.uni-frankfurt.de

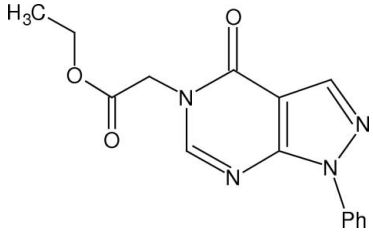
Received 20 April 2007; accepted 23 April 2007

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.033; wR factor = 0.089; data-to-parameter ratio = 13.2.

Geometric parameters of the title compound, $C_{14}H_{12}N_4O_3$, a pyrazolopyrimidine derivative, are in the usual ranges. The dihedral angle between the pyrazolopyrimidine system and the phenyl ring is 4.64 (5)°. 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)°] 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

$C_{15}H_{14}N_4O_3$
M_r = 298.30
Monoclinic, $P2_1/c$
a = 7.5794 (8) Å
b = 11.1125 (9) Å
c = 16.9767 (14) Å
β = 99.654 (8)°
V = 1409.6 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 173 (2) K
0.42 × 0.42 × 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_{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
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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*.

SB thanks the University of Mysore for research facilities.

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

References

Devesa, I., Alcaraz, M. J., Riguera, R. & Ferrandiz, M. L. (2004). *Eur. J. Pharmacol.* **488**, 225–230..
El-Feky, S. A. & Abd El-Samii, Z. K. (1996). *Pharmazie*, **51**, 540–543.
Garg, N., Avasthi, K., Pratap, R., Bhakuni, D. S. & Guru, P. Y. (1990). *Ind. J. Chem. Sect. B*, **29**, 859–864.
Ismail, Z. H., Abdel-Gawad, S. M., Abdel-Aziem, A. & Ghorab, M. M. (2003). *Phosphorus Sulfur Silicon*, **178**, 1795–1805.
Oliveira-Campos, A. M. F., Rodrigues, L. M., Kaja, M., Guilardi, S., Franca, E. de F. & Ellena, J. (2006). *Acta Cryst.* E**62**, o5246–o5248.
Portilla, J., Quiroga, J., Cobo, J., Low, J. N. & Glidewell, C. (2005). *Acta Cryst.* C**61**, o452–o456.
Russo, F., Guccione, S., Romeo, G., Baretta, G. U., Pucci, S., Caruso, A., Roxas, M. A. & Cutuli, V. (1993). *Eur. J. Med. Chem.* **28**, 363–376.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.
Wen, L.-R., Wang, S.-W., Xu, H.-Z., Zhang, X.-L., Li, M. & Liu, J.-H. (2004). *Acta Cryst.* E**60**, o1294–o1295.
Yathirajan, H. S., Bindya, S., Sarojini, B. K., Narayana, B. & Bolte, M. (2007). *Acta Cryst.* E**63**, o2566.

  doi:10.1107/S1600536807020284

supplementary materials

Acta Cryst. (2007). E**63**, o2718 [**doi:10.1107/S1600536807020284**]

Ethyl (4-oxo-1-phenyl-1,4-dihydro-5*H*-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 ist 4.64 (5)°. 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)°] 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(Calaculated): C: 60.28 (60.40); H: 4.61 (4.73); N: 18.63% (18.78%). ^{1}H-NMR (400 MHz, $CDCl_3$): δ 1.28 (t, 3H, CH_3), δ 4.24 (q, 2H, CH_2), δ 4.72 (s, 2H, -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 $C_{aromatic}$—H = 0.95 Å, C_{methyl}—H = 0.98Å or $C_{methylene}$—H = 0.99Å and U_{iso}(H) = 1.2U_{eq}(C) or U_{iso}(H) = 1.5U_{eq}(C_{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-5*H*-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 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 13263 reflections
a = 7.5794 (8) Å	θ = 3.7–25.7º
b = 11.1125 (9) Å	μ = 0.10 mm^{-1}
c = 16.9767 (14) Å	T = 173 (2) K
β = 99.654 (8)º	Block, colourless
V = 1409.6 (2) Å^3	0.42 × 0.42 × 0.39 mm
Z = 4	

Data collection

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

Refinement

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

	x	*y*	*z*	U_{iso}*/U_{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*

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 (Å^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)

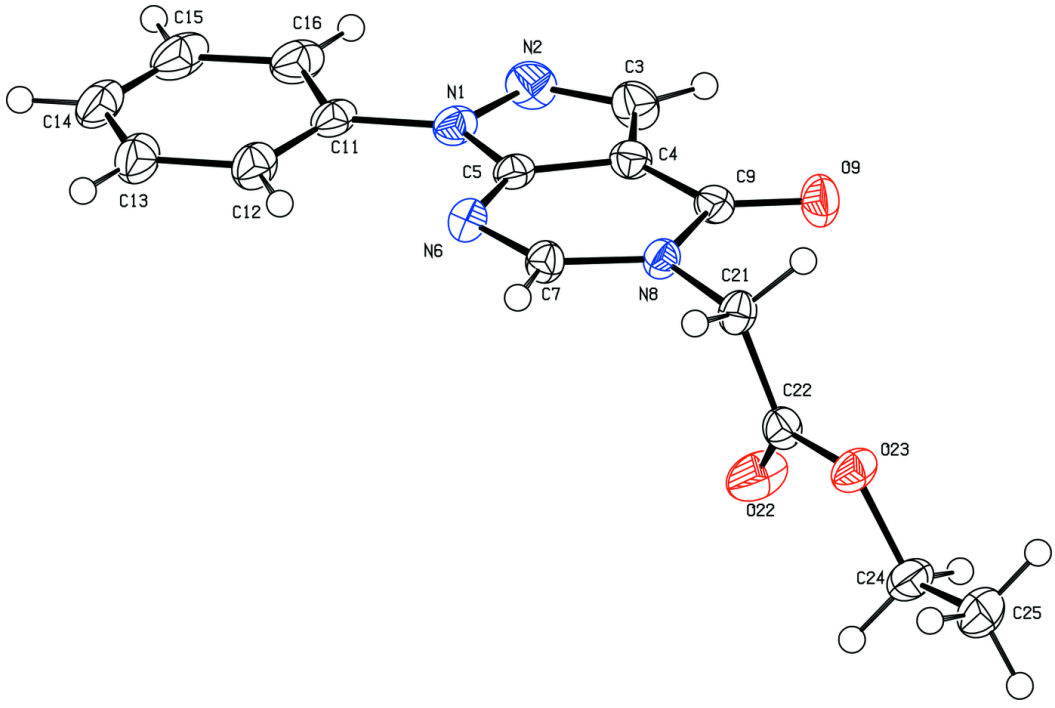
Geometric parameters (Å, °)

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)

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


C15
C16
N2
C3
N1
C14
C11
C4
C5
C9
O9
C13
C12
N6
C7
N8
C21
C22
O23
O22
C24
C25