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Ethyl 1-benzyl-3-(4-methylphenyl)-1*H*-pyrazole-5-carboxylate

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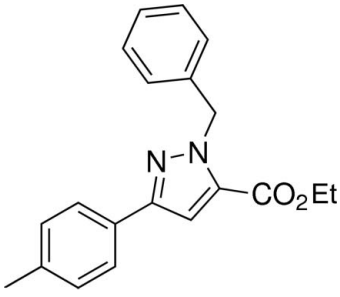
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.066; wR factor = 0.220; data-to-parameter ratio = 14.0.

In the title compound, $C_{20}H_{20}N_2O_2$, the pyrazole ring makes dihedral angles of 15.68 (4) and 83.40 (4)°, respectively, with the tolyl and benzyl rings, respectively.

Related literature

For arelated structure, see: Ge *et al.* (2007). For applications of nitrogen-containing heterocyclic compounds in agrochemicals and pharmaceuticals, see: Ge *et al.* (2009*a*,*b*, 2011).



Experimental

Crystal data

$C_{20}H_{20}N_2O_2$
M_r = 320.38
Triclinic, $P\overline{1}$
a = 7.666 (4) Å
b = 10.160 (6) Å
c = 11.381 (7) Å
α = 83.991 (9)°
β = 87.466 (9)°
γ = 85.47 (1)°
V = 878.2 (9) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 298 K
0.21 × 0.16 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.984, T_{max} = 0.991
4489 measured reflections
3045 independent reflections
2182 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.220
S = 1.06
3045 reflections
217 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2257).

References

Bruker (1997). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Ge, Y.-Q., Dong, W.-L., Xia, Y., Wei, F. & Zhao, B.-X. (2007). *Acta Cryst.* E**63**, o1313–o1314.
Ge, Y. Q., Jia, J., Li, Y., Yin, L. & Wang, J. W. (2009*a*). *Heterocycles*, **42**, 197–206.
Ge, Y. Q., Jia, J., Yang, H., Tao, X. T. & Wang, J. W. (2011). *Dyes Pigm.* **88**, 344–349.
Ge, Y. Q., Jia, J., Yang, H., Zhao, G. L., Zhan, F. X. & Wang, J. W. (2009*b*). *Heterocycles*, **72**, 725–736.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2011). E**67**, o468 [**doi:10.1107/S1600536811002339**]

Ethyl 1-benzyl-3-(4-methylphenyl)-1*H*-pyrazole-5-carboxylate

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Comment

Synthesis of nitrogen-containing heterocyclic compounds has been a subject of great interest due to the wide application in agrochemical and pharmaceutical fields (Ge *et al.*; 2011, 2009*a*, 2009*b*). Some pyrazole derivatives which belong to this category have been of interest for their biological activities. Considerable efforts have been devoted to the development of novel pyrazole compounds. We report here the crystal structure of the title compound, (I) (Fig. 1)

Experimental

A mixture of ethyl 3-*p*-tolyl-1*H*-pyrazole-5-carboxylate (0.02 mol), benzyl chloride (0.0024 mol) and potassium carbonate (0.02 mol) in acetonitrile (100 ml) was heated to reflux for 10 h. The solvent was removed under reduced pressure and an product was isolated by column chromatography on silica gel (yield 82%). Crystals of (I) suitable for X-ray diffraction were obtained by slow cooling of the refluxed solution of the product in ethyl acetate at room temperature for 2 d.

Refinement

All H atoms were placed in geometrically calculated positions and refined using a riding model with C—H = 0.97 Å (for CH_2 groups) and 0.96 Å (for CH_3 groups), their isotropic displacement parameters were set to 1.2 times (1.5 times for CH_3 groups) the equivalent displacement parameter of their parent atoms.

Figures

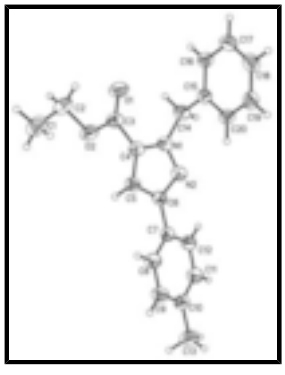


Fig. 1. The molecular structure of (I), showing displacement ellipsoids drawn at the 50% probability level.

Ethyl 1-benzyl-3-(4-methylphenyl)-1*H*-pyrazole-5-carboxylate

Crystal data

$C_{20}H_{20}N_2O_2$	$Z = 2$
$M_r = 320.38$	$F(000) = 340$

Triclinic, $P\overline{1}$	$D_x = 1.212$ Mg m^{-3}
$a = 7.666$ (4) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 10.160$ (6) Å	Cell parameters from 1778 reflections
$c = 11.381$ (7) Å	$\theta = 2.6$–$24.2°$
$\alpha = 83.991$ (9)°	$\mu = 0.08$ mm^{-1}
$\beta = 87.466$ (9)°	$T = 298$ K
$\gamma = 85.47$ (1)°	BLOCK, white
$V = 878.2$ (9) Å^3	0.21 × 0.16 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer	3045 independent reflections
Radiation source: fine-focus sealed tube	2182 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.032$
phi and ω scans	$\theta_{max} = 25.1°$, $\theta_{min} = 1.8°$
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)	$h = -9 \rightarrow 8$
$T_{min} = 0.984$, $T_{max} = 0.991$	$k = -12 \rightarrow 9$
4489 measured reflections	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.066$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.220$	H-atom parameters constrained
$S = 1.06$	$w = 1/[\sigma^2(F_o^2) + (0.1348P)^2 + 0.1525P]$ where $P = (F_o^2 + 2F_c^2)/3$
3045 reflections	$(\Delta/\sigma)_{max} < 0.001$
217 parameters	$\Delta\rho_{max} = 0.34$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.21$ e Å^{-3}

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.3059 (3)	0.5585 (2)	0.70282 (17)	0.0826 (7)
O2	0.2483 (3)	0.6693 (2)	0.86211 (17)	0.0703 (6)
N1	0.3539 (3)	0.3185 (2)	0.86746 (18)	0.0548 (6)
N2	0.3537 (3)	0.2290 (2)	0.96361 (19)	0.0569 (6)
C1	0.1960 (6)	0.9004 (4)	0.8654 (3)	0.0955 (11)
H1A	0.1004	0.8824	0.9206	0.143*
H1B	0.1698	0.9836	0.8191	0.143*
H1C	0.3006	0.9049	0.9077	0.143*
C2	0.2227 (5)	0.7932 (3)	0.7865 (3)	0.0764 (9)
H2A	0.1213	0.7919	0.7387	0.092*
H2B	0.3246	0.8065	0.7342	0.092*
C3	0.2870 (3)	0.5601 (3)	0.8081 (2)	0.0597 (7)
C4	0.3021 (3)	0.4439 (3)	0.8954 (2)	0.0528 (6)
C5	0.2658 (3)	0.4323 (3)	1.0152 (2)	0.0551 (7)
H5	0.2268	0.5002	1.0609	0.066*
C6	0.2990 (3)	0.2986 (3)	1.0543 (2)	0.0517 (6)
C7	0.2828 (3)	0.2321 (3)	1.1751 (2)	0.0523 (6)
C8	0.2692 (4)	0.3059 (3)	1.2712 (2)	0.0625 (7)
H8	0.2707	0.3978	1.2586	0.075*
C9	0.2535 (4)	0.2459 (3)	1.3851 (2)	0.0669 (8)
H9	0.2442	0.2982	1.4478	0.080*
C10	0.2512 (4)	0.1096 (3)	1.4083 (2)	0.0616 (7)
C11	0.2643 (4)	0.0367 (3)	1.3121 (3)	0.0717 (8)
H11	0.2620	−0.0551	1.3247	0.086*
C12	0.2806 (4)	0.0958 (3)	1.1980 (2)	0.0648 (8)
H12	0.2903	0.0433	1.1355	0.078*
C13	0.2361 (5)	0.0423 (4)	1.5327 (3)	0.0880 (10)
H13A	0.2140	−0.0490	1.5301	0.132*
H13B	0.1414	0.0861	1.5752	0.132*
H13C	0.3434	0.0471	1.5719	0.132*
C14	0.4189 (3)	0.2742 (3)	0.7545 (2)	0.0608 (7)
H14A	0.3443	0.3163	0.6925	0.073*
H14B	0.4110	0.1791	0.7574	0.073*
C15	0.6048 (3)	0.3050 (2)	0.7234 (2)	0.0485 (6)
C16	0.6535 (4)	0.3469 (3)	0.6073 (2)	0.0596 (7)
H16	0.5692	0.3606	0.5501	0.071*
C17	0.8246 (4)	0.3682 (3)	0.5762 (3)	0.0730 (9)
H17	0.8563	0.3945	0.4979	0.088*
C18	0.9483 (4)	0.3509 (4)	0.6601 (3)	0.0766 (9)
H18	1.0637	0.3675	0.6390	0.092*
C19	0.9039 (4)	0.3090 (3)	0.7758 (3)	0.0692 (8)
H19	0.9892	0.2966	0.8324	0.083*
C20	0.7326 (3)	0.2855 (3)	0.8074 (2)	0.0555 (7)
H20	0.7025	0.2566	0.8853	0.067*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.1089 (17)	0.0879 (17)	0.0500 (12)	−0.0083 (13)	−0.0052 (10)	−0.0005 (11)
O2	0.0972 (15)	0.0575 (12)	0.0542 (11)	−0.0067 (10)	−0.0058 (9)	0.0058 (9)
N1	0.0577 (12)	0.0589 (14)	0.0494 (12)	−0.0112 (10)	0.0018 (9)	−0.0093 (10)
N2	0.0608 (13)	0.0548 (13)	0.0561 (13)	−0.0100 (10)	0.0022 (10)	−0.0083 (10)
C1	0.131 (3)	0.061 (2)	0.091 (2)	−0.0049 (19)	−0.015 (2)	0.0112 (18)
C2	0.091 (2)	0.065 (2)	0.0695 (19)	−0.0083 (16)	−0.0136 (16)	0.0169 (16)
C3	0.0604 (16)	0.0674 (18)	0.0520 (16)	−0.0112 (13)	−0.0059 (11)	−0.0031 (13)
C4	0.0542 (14)	0.0551 (16)	0.0501 (14)	−0.0110 (11)	−0.0027 (11)	−0.0037 (12)
C5	0.0643 (16)	0.0517 (15)	0.0500 (14)	−0.0065 (12)	0.0008 (11)	−0.0082 (11)
C6	0.0545 (14)	0.0512 (15)	0.0509 (14)	−0.0089 (11)	0.0025 (11)	−0.0098 (11)
C7	0.0545 (14)	0.0491 (15)	0.0531 (14)	−0.0051 (11)	0.0026 (11)	−0.0056 (11)
C8	0.0851 (19)	0.0470 (15)	0.0561 (16)	−0.0106 (13)	0.0011 (13)	−0.0061 (12)
C9	0.089 (2)	0.0595 (18)	0.0530 (16)	−0.0082 (15)	0.0023 (14)	−0.0104 (13)
C10	0.0716 (17)	0.0582 (17)	0.0534 (15)	−0.0028 (13)	0.0007 (12)	−0.0007 (12)
C11	0.103 (2)	0.0443 (16)	0.0660 (18)	−0.0029 (14)	0.0045 (16)	−0.0023 (13)
C12	0.091 (2)	0.0462 (15)	0.0567 (16)	−0.0007 (13)	0.0029 (14)	−0.0081 (12)
C13	0.123 (3)	0.079 (2)	0.0594 (19)	−0.008 (2)	−0.0044 (18)	0.0073 (16)
C14	0.0632 (16)	0.0726 (19)	0.0512 (14)	−0.0156 (13)	−0.0016 (12)	−0.0198 (13)
C15	0.0543 (14)	0.0459 (14)	0.0472 (13)	−0.0054 (10)	−0.0033 (10)	−0.0120 (10)
C16	0.0669 (17)	0.0637 (17)	0.0481 (14)	−0.0030 (13)	−0.0076 (12)	−0.0048 (12)
C17	0.0712 (19)	0.087 (2)	0.0594 (17)	−0.0095 (16)	0.0107 (14)	−0.0026 (15)
C18	0.0563 (17)	0.092 (2)	0.081 (2)	−0.0046 (15)	0.0081 (15)	−0.0122 (18)
C19	0.0549 (16)	0.084 (2)	0.0699 (18)	0.0027 (14)	−0.0142 (13)	−0.0154 (16)
C20	0.0669 (16)	0.0551 (15)	0.0456 (13)	−0.0038 (12)	−0.0063 (11)	−0.0096 (11)

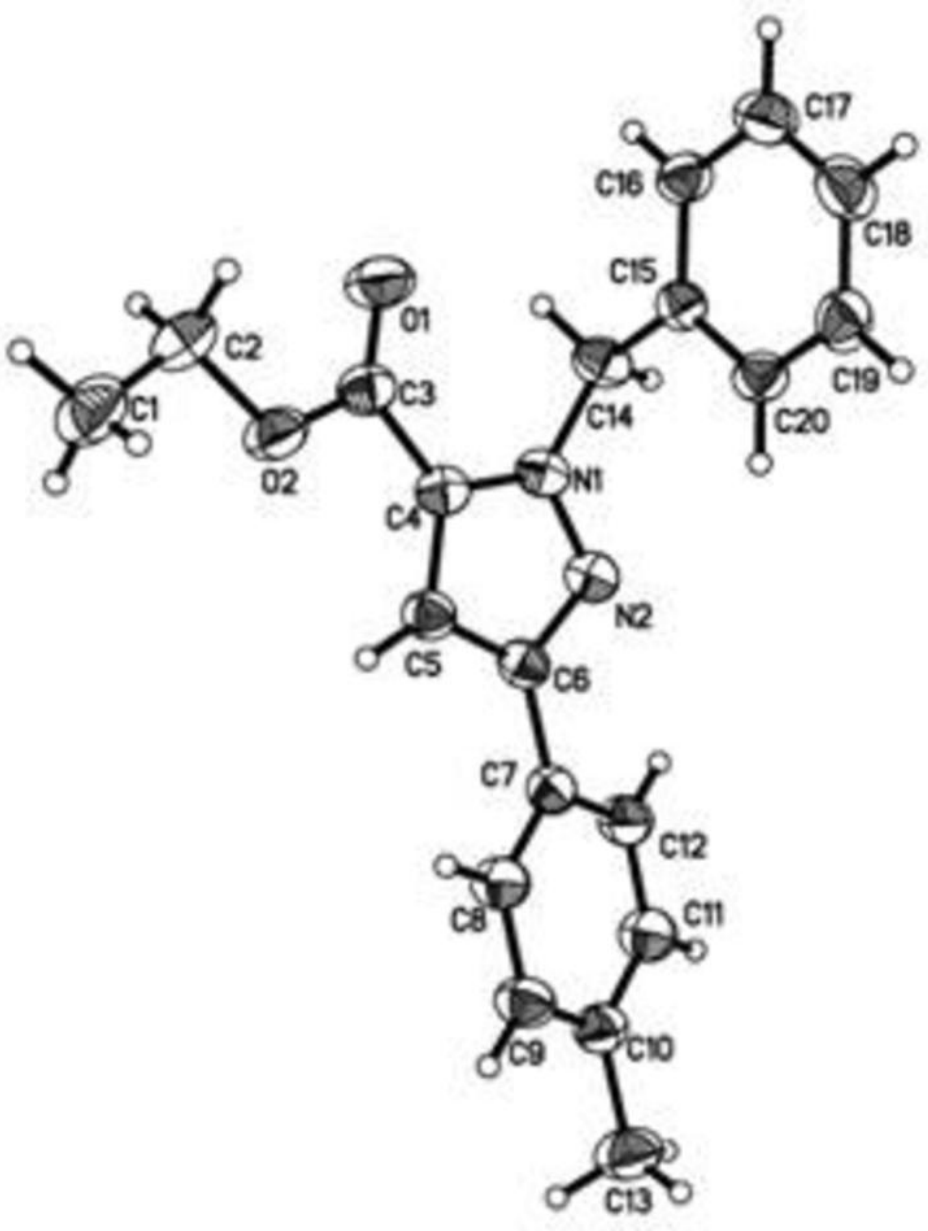
Geometric parameters (Å, °)

O1—C3	1.202 (3)	C9—H9	0.9300
O2—C3	1.332 (3)	C10—C11	1.381 (4)
O2—C2	1.453 (3)	C10—C13	1.510 (4)
N1—N2	1.348 (3)	C11—C12	1.376 (4)
N1—C4	1.369 (3)	C11—H11	0.9300
N1—C14	1.461 (3)	C12—H12	0.9300
N2—C6	1.345 (3)	C13—H13A	0.9600
C1—C2	1.480 (5)	C13—H13B	0.9600
C1—H1A	0.9600	C13—H13C	0.9600
C1—H1B	0.9600	C14—C15	1.502 (3)
C1—H1C	0.9600	C14—H14A	0.9700
C2—H2A	0.9700	C14—H14B	0.9700
C2—H2B	0.9700	C15—C20	1.388 (4)
C3—C4	1.463 (4)	C15—C16	1.389 (4)
C4—C5	1.374 (3)	C16—C17	1.372 (4)
C5—C6	1.392 (4)	C16—H16	0.9300
C5—H5	0.9300	C17—C18	1.364 (4)
C6—C7	1.471 (4)	C17—H17	0.9300

C7—C12	1.384 (4)	C18—C19	1.377 (4)
C7—C8	1.386 (4)	C18—H18	0.9300
C8—C9	1.378 (4)	C19—C20	1.379 (4)
C8—H8	0.9300	C19—H19	0.9300
C9—C10	1.384 (4)	C20—H20	0.9300
C3—O2—C2	116.6 (2)	C11—C10—C13	121.0 (3)
N2—N1—C4	111.8 (2)	C9—C10—C13	122.1 (3)
N2—N1—C14	118.5 (2)	C12—C11—C10	122.0 (3)
C4—N1—C14	129.5 (2)	C12—C11—H11	119.0
C6—N2—N1	105.3 (2)	C10—C11—H11	119.0
C2—C1—H1A	109.5	C11—C12—C7	121.0 (3)
C2—C1—H1B	109.5	C11—C12—H12	119.5
H1A—C1—H1B	109.5	C7—C12—H12	119.5
C2—C1—H1C	109.5	C10—C13—H13A	109.5
H1A—C1—H1C	109.5	C10—C13—H13B	109.5
H1B—C1—H1C	109.5	H13A—C13—H13B	109.5
O2—C2—C1	106.8 (3)	C10—C13—H13C	109.5
O2—C2—H2A	110.4	H13A—C13—H13C	109.5
C1—C2—H2A	110.4	H13B—C13—H13C	109.5
O2—C2—H2B	110.4	N1—C14—C15	113.41 (19)
C1—C2—H2B	110.4	N1—C14—H14A	108.9
H2A—C2—H2B	108.6	C15—C14—H14A	108.9
O1—C3—O2	124.5 (3)	N1—C14—H14B	108.9
O1—C3—C4	125.5 (3)	C15—C14—H14B	108.9
O2—C3—C4	110.0 (2)	H14A—C14—H14B	107.7
N1—C4—C5	106.1 (2)	C20—C15—C16	118.7 (2)
N1—C4—C3	123.6 (2)	C20—C15—C14	121.2 (2)
C5—C4—C3	130.3 (3)	C16—C15—C14	119.9 (2)
C4—C5—C6	106.2 (2)	C17—C16—C15	120.7 (2)
C4—C5—H5	126.9	C17—C16—H16	119.7
C6—C5—H5	126.9	C15—C16—H16	119.7
N2—C6—C5	110.6 (2)	C18—C17—C16	119.9 (3)
N2—C6—C7	120.6 (2)	C18—C17—H17	120.0
C5—C6—C7	128.8 (2)	C16—C17—H17	120.0
C12—C7—C8	117.3 (2)	C17—C18—C19	120.6 (3)
C12—C7—C6	122.4 (2)	C17—C18—H18	119.7
C8—C7—C6	120.2 (2)	C19—C18—H18	119.7
C9—C8—C7	121.3 (3)	C18—C19—C20	119.7 (3)
C9—C8—H8	119.4	C18—C19—H19	120.1
C7—C8—H8	119.4	C20—C19—H19	120.1
C8—C9—C10	121.5 (3)	C19—C20—C15	120.3 (2)
C8—C9—H9	119.3	C19—C20—H20	119.9
C10—C9—H9	119.3	C15—C20—H20	119.9
C11—C10—C9	116.9 (3)		
C4—N1—N2—C6	0.6 (3)	C12—C7—C8—C9	−0.2 (4)
C14—N1—N2—C6	176.1 (2)	C6—C7—C8—C9	179.8 (2)
C3—O2—C2—C1	−175.4 (3)	C7—C8—C9—C10	0.2 (5)
C2—O2—C3—O1	1.8 (4)	C8—C9—C10—C11	−0.4 (5)

C2—O2—C3—C4	−177.9 (2)	C8—C9—C10—C13	179.3 (3)
N2—N1—C4—C5	−0.6 (3)	C9—C10—C11—C12	0.6 (5)
C14—N1—C4—C5	−175.4 (2)	C13—C10—C11—C12	−179.1 (3)
N2—N1—C4—C3	−178.8 (2)	C10—C11—C12—C7	−0.7 (5)
C14—N1—C4—C3	6.4 (4)	C8—C7—C12—C11	0.4 (4)
O1—C3—C4—N1	5.4 (4)	C6—C7—C12—C11	−179.6 (3)
O2—C3—C4—N1	−174.9 (2)	N2—N1—C14—C15	−99.0 (3)
O1—C3—C4—C5	−172.3 (3)	C4—N1—C14—C15	75.5 (3)
O2—C3—C4—C5	7.4 (4)	N1—C14—C15—C20	42.9 (4)
N1—C4—C5—C6	0.4 (3)	N1—C14—C15—C16	−140.7 (3)
C3—C4—C5—C6	178.4 (2)	C20—C15—C16—C17	0.2 (4)
N1—N2—C6—C5	−0.4 (3)	C14—C15—C16—C17	−176.3 (3)
N1—N2—C6—C7	−179.9 (2)	C15—C16—C17—C18	−1.3 (5)
C4—C5—C6—N2	0.0 (3)	C16—C17—C18—C19	1.5 (5)
C4—C5—C6—C7	179.5 (2)	C17—C18—C19—C20	−0.6 (5)
N2—C6—C7—C12	−16.0 (4)	C18—C19—C20—C15	−0.5 (4)
C5—C6—C7—C12	164.6 (3)	C16—C15—C20—C19	0.7 (4)
N2—C6—C7—C8	164.0 (2)	C14—C15—C20—C19	177.2 (2)
C5—C6—C7—C8	−15.4 (4)		

Fig. 1


C17
C16
C18
C15
O1
C2
C19
C1
C3
C14
C20
O2
N1
C4
N2
C5
C6
C7
C12
C8
C11
C9
C10
C13