Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

Four substituted pyrazolines

Deepak Chopra,[a]* T. P. Mohan,[b] B. Vishalakshi[b] and T. N. Guru Row[a]

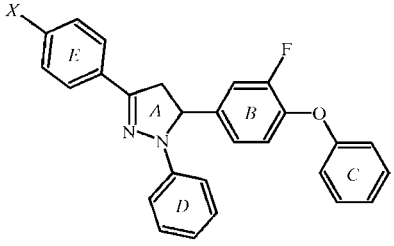
[a]Solid State and Structural Chemistry Unit, Indian Institute of Science, Bangalore 560 012, Karnataka, India, and [b]Department of Chemistry, Mangalore University, Bangalore 574 199, Karnataka, India
Correspondence e-mail: deepak@sscu.iisc.ernet.in

Received 26 September 2007
Accepted 24 October 2007
Online 24 November 2007

In the title compounds 5-(3-fluoro-4-phenoxyphenyl)-1,3-biphenyl-4,5-dihydro-1*H*-pyrazole, $C_{27}H_{21}FN_2O$, (I), 3-(4-chlorophenyl)-5-(3-fluoro-4-phenoxyphenyl)-1-phenyl-4,5-dihydro-1*H*-pyrazole, $C_{27}H_{20}ClFN_2O$, (II), 5-(3-fluoro-4-phenoxyphenyl)-3-(4-methylphenyl)-1-phenyl-4,5-dihydro-1*H*-pyrazole, $C_{28}H_{23}FN_2O$, (III), and 5-(3-fluoro-4-phenoxyphenyl)-3-(4-methoxyphenyl)-1-phenyl-4,5-dihydro-1*H*-pyrazole, $C_{28}H_{23}FN_2O_2$, (IV), the five-membered pyrazole ring exists in an envelope conformation. The crystal structure of (I) has three independent C—H$\cdots\pi$ intermolecular interactions. In (II), an intermolecular C—Cl$\cdots\pi$ contact is present, forming molecular chains. Replacement of this chloro group in (II) by a methyl group yields an isomorphic crystal structure, (III).

Comment

Pyrazolines are an important class of compounds found to possess extensive physiological properties, including antibacterial (Nauduri & Reddy, 1998; Azarifar & Shaabanzadeh, 2002), anti-inflammatory (Kuroda *et al.*, 1992; Udupi, Kushnoor & Bhat, 1998; Udupi, Rao & Bhat, 1998), antifungal



(I) X=H
(II) X=Cl
(III) X=methyl
(IV) X=methoxy

(Korgaokar *et al.*, 1996), analgesic (Udupi, Kushnoor & Bhat, 1998; Udupi, Rao & Bhat, 1998), antitumour (Chen *et al.*, 1997), antidepressant (Bilgin *et al.*, 1993) and antiprotozoal (Cetin *et al.*, 2003). They also possess fungicidal or herbicidal properties (Takao *et al.*, 1994; Ohvchi & Okada, 1998; Malhotra *et al.*, 1997), and are used as scintillation solutes and antioxidants in lubrication oils (Beher *et al.*, 1967). Previous studies have demonstrated the monoamine oxidase (MAO) inhibitory activities of 1,3,5-triphenyl-2-pyrazolines (Soni *et al.*, 1987; Chimenti *et al.*, 2004). Several recent structural determinations of this class of compounds have also been carried out (Köysal *et al.*, 2005; Langer *et al.*, 2007).

In view of the diverse applications of this class of compounds, we report here the crystal structures of four substituted pyrazoles, namely 5-(3-fluoro-4-phenoxyphenyl)-1,3-biphenyl-4,5-dihydro-1*H*-pyrazole, (I), 3-(4-chlorophenyl)-5-(3-fluoro-4-phenoxyphenyl)-1-phenyl-4,5-dihydro-1*H*-pyrazole, (II), 5-(3-fluoro-4-phenoxyphenyl)-3-(4-methylphenyl)-1-phenyl-4,5-dihydro-1*H*-pyrazole, (III), and 5-(3-fluoro-4-phenoxyphenyl)-3-(4-methoxyphenyl)-1-phenyl-4,5-dihydro-1*H*-pyrazole, (IV). The effect of varying molecular substituents on the molecular geometry, the conformational changes and the associated packing features have also been analysed. All four structures have pyrazoline (ring *A*), 3-fluoro-4-

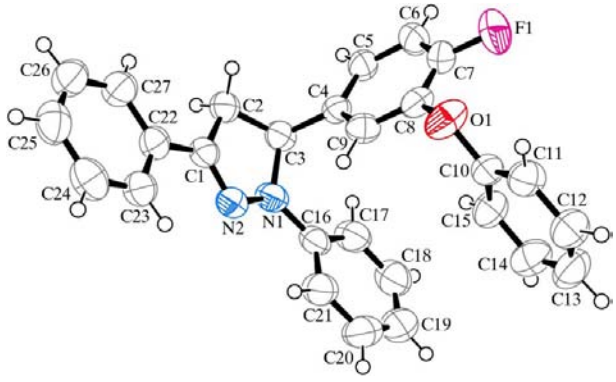


Figure 1
A view of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

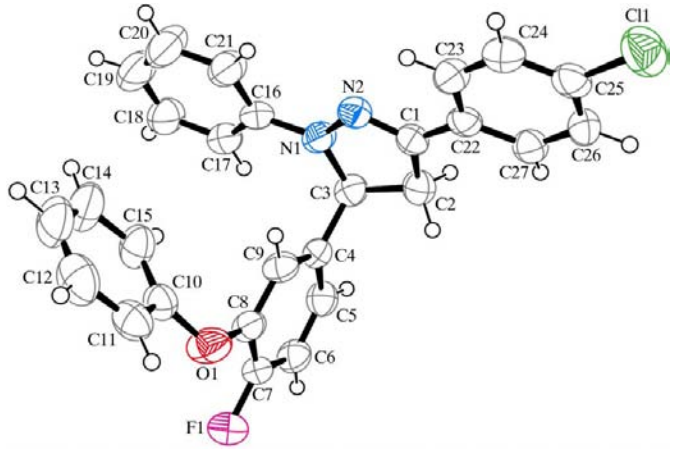


Figure 2
A view of (II), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

  DOI: 10.1107/S0108270107052705

phenoxyphenyl (attached to C3, rings *B* and *C*), phenyl (attached to N1, ring *D*) and substituted phenyl (attached to C1, ring *E*) groups in the molecule. The different groups present on ring *E* are the chloro, methyl and methoxy substituents in compounds (II)–(IV), respectively. All four compounds crystallize with one molecule in the asymmetric unit. In contrast with the unsubstituted pyrazoline, (I), which crystallizes in a noncentrosymmetric space group, the other three compounds crystallize in centrosymmetric space groups. The absolute configuration in (I) was not defined and so is shown to match the configurations shown for the other three structures.

Figs. 1–4 depict the molecular structures of the four pyrazolines with their different substitutions and show the corresponding atom-numbering schemes. In all four compounds, the central ring *A* exists in an envelope conformation with the chiral atom C3 (crystallographic numbering) forming the flap; deviations from the least-squares planes for (I)–(IV) are given in Table 8. The remaining atoms of ring *A* (N1, N2, C1 and C2) are coplanar, with the out-of-plane distances being −0.0004 (2) and −0.003 (3) Å for atom N2 in (I) and (IV), respectively. The corresponding distances are 0.002 (2) and 0.005 (2) Å for atom C2 in (III) and (IV), respectively. The bond lengths and torsion angles of the five-membered ring (Tables 1, 3, 4 and 5) in all four compounds are in agreement with the expected values reported in the literature (Allen *et al.*, 1987; Allen, 2002). The N—N bond lengths of the pyrazoline ring in (I) [1.383 (2) Å] and (IV) [1.390 (3) Å] are similar. In the case of (II) [1.363 (3) Å] and (III) [1.365 (2) Å], these are shorter, indicating greater double-bond character due to resonance delocalization between the lone pair of electrons on atom N1 and the C=N double bond of ring *A* and extending to ring *E*.

In all four compounds, the 3-fluoro-4-phenoxyphenyl group occupies a pseudo-axial position and hence is approximately perpendicular to the mean plane of the pyrazoline ring, the dihedral angle being 80.25 (6)° in (I) (Table 7). This value is comparable with that observed in 3-(4-bromophenyl)-5-[4-(dimethylamino)phenyl]-1-phenyl-2-pyrazoline (Langer *et al.*, 2007), in which the 4-(dimethylamino)phenyl group makes an angle of 74.3 (1)° with the central pyrazoline ring. Similar geometric characteristics are observed in *N*-substituted 3,5-diphenyl-2-pyrazoline-1-thiocarboxamides (Köysal *et al.*, 2005), where the methoxyphenyl and methylphenyl groups make dihedral angles of 89.29 (8) and 80.39 (10)°, respectively. A similar orientation at the C3 atom is also observed in spiro compounds (Bruno *et al.*, 2004), where the isoxazoline ring is connected to the substituted pyrazoline ring with the dihedral

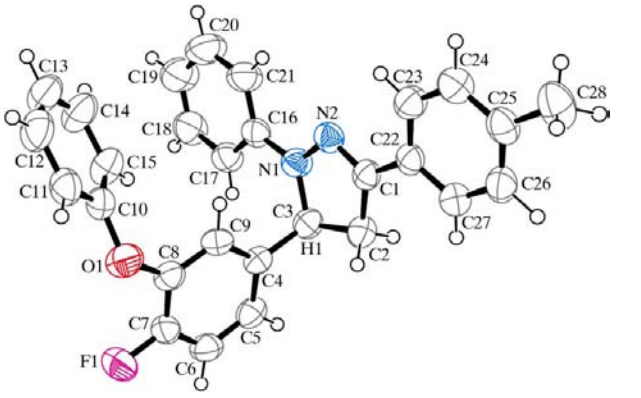


Figure 3
A view of (III), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

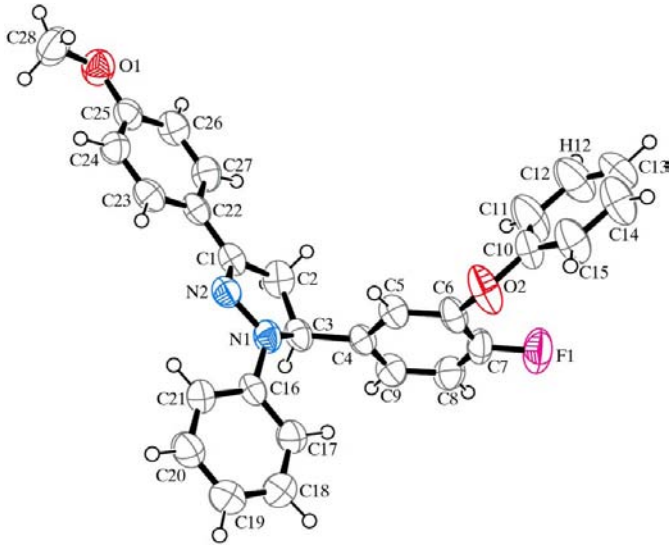


Figure 4
A view of (IV), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

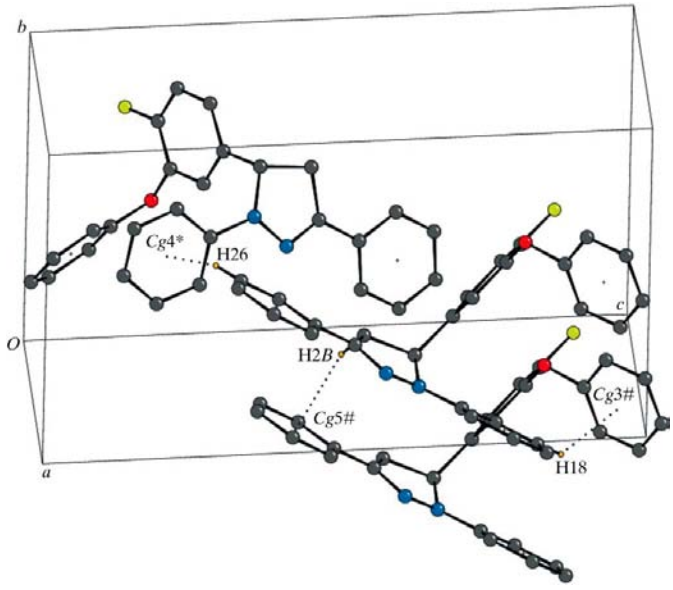


Figure 5
A partial packing diagram for (I), depicting the formation of C—H···π interactions (dotted lines) with respect to the reference molecule at (x, y, z). *Cg*3, *Cg*4 and *Cg*5 are the centroids of rings *C*, *D* and *E*, respectively. Molecules labelled with hash (#) or asterisk (*) suffixes are at the symmetry positions ($x+1$, y, z) and ($-x+1$, $y+\frac{1}{2}$, $-z+1$), respectively.

angle lying in the range 87.7 (1)–89.0 (1)°. In the isomorphic compounds (II) and (III), the dihedral angles between ring *A* and rings *D* and *E* are similar to those observed in 3-(4-bromophenyl)-5-[4-(dimethylamino)phenyl]-1-phenyl-2-pyrazoline (Langer *et al.*, 2007), where rings *D* and *E* are taken as the phenyl and 4-bromo groups, respectively (Table 7). A ring-puckering analysis (Cremer & Pople, 1975) of the five-membered pyrazoline ring gives the parameters listed in Table 8.

In the absence of strong hydrogen-bond donors in (I), the crystal packing is controlled by the involvement of weak C—H$\cdots\pi$ intermolecular interactions (Nishio *et al.*, 1995; Takahashi *et al.*, 2000; Umezawa *et al.*, 1999). The molecules in (I) are held together by three different weak yet directional C—H$\cdots\pi$ interactions, involving atoms H18 and H2*B* with rings *C* and *E* and forming chains along the crystallographic *a* axis, coupled with the formation of chains along *b* axis involving atom H26 and ring *D* (see Fig. 5 and Table 2).

In the crystal structure of (II), the packing is controlled by a noncovalent C—Cl$\cdots\pi$ interaction involving the lone pair of electrons on the Cl atom with the antibonding orbitals of ring *B* [C25—Cl1$\cdots$*Cg*2 = 137.2 (1)°; *Cg*2 is the centroid of ring *B*] (Fig. 6 and Table 3). These interactions are well documented in the literature and have been extensively studied in small molecules and in proteins (Prasanna & Guru Row, 2000; Saraogi *et al.*, 2003) using the Cambridge Structural Database (Allen, 2002). In (II), these interactions form molecular chains along the crystallographic screw *b* axis.

In compound (IV), the presence of a methoxy group results in a change in the molecular conformation around the C16—N1 bond, with the torsion angle increasing from −8.1 (3)° in the case of (III) to 35.7 (4)° in (IV). There are no unusually short intermolecular contacts in (IV). The H atoms of methyl atom C28 are not well resolved by the calculated position for H28*A* is 2.77 Å from the centroid of the pyrazoline ring at $(2-x, -\frac{1}{2}+y, \frac{1}{2}-z)$.

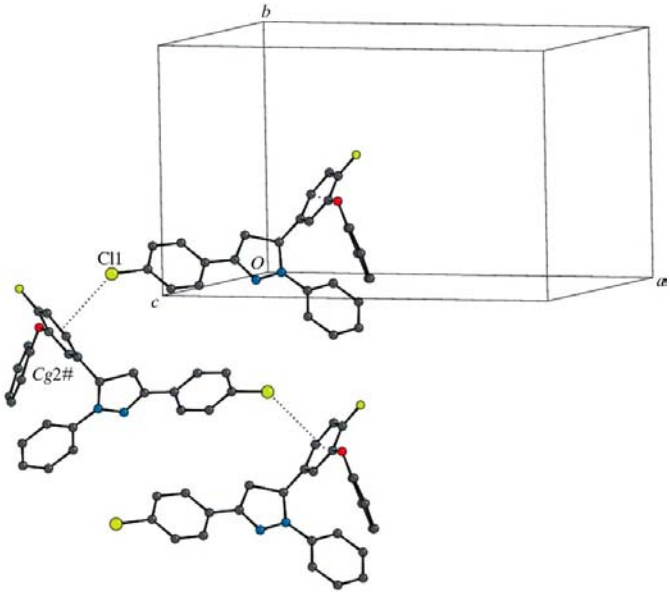


Figure 6
A partial packing diagram for (II), depicting C—Cl$\cdots\pi$ intermolecular contacts (dotted lines) along the *b* axis. *Cg*2 is the centroid of ring *B*. The hash (#) suffix indicates the symmetry-related molecule at $(-x, y-\frac{1}{2}, -z+\frac{3}{2})$.

Experimental

A mixture of chalcone (with different substitutions; 0.01 mol), acetic acid (20 ml) and phenylhydrazine (0.01 mol) was refluxed for 12 h. The resulting mixture was cooled to 283 K and filtered. The progress of the reaction and the purity of the products were monitored by thin-layer chromatography. The crude products thus obtained were recrystallized from propan-2-ol (Mogilaiah & Sudhakar, 2003; Dandia *et al.*, 1993; Bhatt *et al.*, 2001; Mohan, 2006). All four compounds were characterized using mass, NMR and IR spectra, melting-point measurements and elemental analyses. A small quantity of each sample was dissolved in a suitable solvent or solvent combination, such as dichloromethane, dichloromethane–hexane (1:1), acetonitrile, ethyl acetate or ethyl acetate–hexane (1:1). The crystals used for data collection were obtained from ethyl acetate–hexane (1:1).

Compound (I)

Crystal data

$C_{27}H_{21}FN_2O$
M_r = 408.46
Monoclinic, $P2_1$
a = 5.9774 (14) Å
b = 10.484 (3) Å
c = 16.896 (4) Å
β = 90.762 (4)°
V = 1058.7 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 290 (2) K
0.34 × 0.23 × 0.09 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.941, T_{max} = 0.992
8402 measured reflections
2272 independent reflections
1984 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.087
S = 1.26
2272 reflections
364 parameters
1 restraint
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

Table 1
Selected geometric parameters (Å, °) for (I).

N1—N2	1.383 (2)	N1—C3	1.472 (3)
N1—C16	1.403 (2)	N2—C1	1.284 (2)
C27—C22—C1—C2	5.3 (3)	C2—C3—C4—C5	−93.1 (2)
C3—N1—C16—C17	−33.3 (3)	N2—C1—C2—C3	−13.3 (2)

Table 2
Short intermolecular contact geometry (Å, °) for (I).

*Cg*3, *Cg*4 and *Cg*5 are the centroids of rings *C*, *D* and *E*, respectively.

D—H$\cdots$*A*	*D*—H	H$\cdots$*A*	*D*$\cdots$*A*	*D*—H$\cdots$*A*
C18—H18$\cdots$*Cg*3^{i}	0.95 (3)	2.84 (2)	3.566 (3)	134 (2)
C26—H26$\cdots$*Cg*4^{ii}	0.96 (3)	2.93 (3)	3.755 (3)	145 (2)
C2—H2*B*$\cdots$*Cg*5^{i}	1.00 (2)	2.80 (2)	3.764 (3)	162 (2)

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+1, y+\frac{1}{2}, -z+1$.

Compound (II)

Crystal data

$C_{27}H_{20}ClFN_2O$
M_r = 442.90
Monoclinic, $P2_1/c$
a = 17.728 (3) Å
b = 11.0132 (17) Å
c = 11.2709 (17) Å
β = 95.729 (3)°
V = 2189.6 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 290 (2) K
0.29 × 0.17 × 0.07 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.937, T_{max} = 0.986
17206 measured reflections
4469 independent reflections
2393 reflections with $I > 2\sigma(I)$
R_{int} = 0.067

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.071
$wR(F^2)$ = 0.135
S = 1.06
4469 reflections
369 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Table 3
Selected geometric parameters (Å, °) for (II).

*Cg*2 is the centroid of ring *B*.

Cl1—C25	1.742 (3)	N1—C3	1.467 (3)
N1—N2	1.363 (3)	N2—C1	1.291 (3)
N1—C16	1.394 (3)		
Cl1···*Cg*2^{i}	3.560 (2)		
C3—N1—C16—C17	−9.4 (4)	C27—C22—C1—C2	5.2 (4)
C5—C4—C3—C2	−92.1 (3)	N2—C1—C2—C3	−7.4 (4)

Symmetry code: (i) $-x, y-\frac{1}{2}, -z+\frac{3}{2}$.

Compound (III)

Crystal data

$C_{28}H_{23}FN_2O$
M_r = 422.48
Monoclinic, $P2_1/c$
a = 17.742 (6) Å
b = 11.099 (4) Å
c = 11.187 (4) Å
β = 95.293 (6)°
V = 2193.5 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 290 (2) K
0.40 × 0.40 × 0.12 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.921, T_{max} = 0.990
16874 measured reflections
4427 independent reflections
2845 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.116
S = 1.06
4427 reflections
370 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Table 4
Selected geometric parameters (Å, °) for (III).

N2—C1	1.289 (2)	C16—N1	1.388 (2)
N2—N1	1.365 (2)	N1—C3	1.468 (2)
C17—C16—N1—C3	−8.1 (3)	C5—C4—C3—C2	−90.6 (2)
C2—C1—C22—C27	2.9 (3)	N2—C1—C2—C3	−7.3 (2)

Compound (IV)

Crystal data

$C_{28}H_{23}FN_2O_2$
M_r = 438.48
Monoclinic, $P2_1/c$
a = 20.274 (4) Å
b = 5.7588 (12) Å
c = 21.241 (4) Å
β = 116.438 (4)°
V = 2220.6 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 290 (2) K
0.40 × 0.35 × 0.09 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.943, T_{max} = 0.992
16344 measured reflections
4445 independent reflections
2297 reflections with $I > 2\sigma(I)$
R_{int} = 0.084

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.076
$wR(F^2)$ = 0.135
S = 1.18
4445 reflections
379 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Table 5
Selected geometric parameters (Å, °) for (IV).

N1—N2	1.390 (3)	N1—C3	1.476 (4)
N1—C16	1.405 (4)	N2—C1	1.286 (4)
C27—C22—C1—C2	−4.0 (5)	C5—C4—C3—C2	−101.5 (4)
C3—N1—C16—C17	35.7 (4)	N2—C1—C2—C3	5.2 (4)

Table 6
Displacement D (Å) of atom C3 from the least-squares plane formed by atoms C1/C2/N1/N2 for compounds (I)–(IV).

Compound	*D*
(I)	−0.342 (2)
(II)	−0.182 (3)
(III)	0.169 (2)
(IV)	−0.146 (4)

Table 7
Dihedral angles (°) between the least-squares planes for compounds (I)–(IV).

1 is the least-squares plane through atoms C1–C3/N1/N2, 2 is the least-squares plane through atoms C4–C9, 3 is the least-squares plane through atoms C16–C21 and 4 is the least-squares plane through atoms C22–C27.

Compound	1/2	1/3	1/4	2/3	2/4	3/4
(I)	80.3 (1)	17.3 (1)	10.5 (1)	77.8 (2)	77.8 (2)	7.4 (4)
(II)	75.6 (1)	4.3 (1)	7.5 (1)	78.4 (2)	71.8 (1)	7.4 (3)
(III)	74.2 (1)	4.6 (1)	5.0 (1)	77.6 (2)	71.9 (2)	5.9 (3)
(IV)	77.0 (1)	24.3 (1)	7.3 (1)	82.9 (3)	82.0 (2)	17.2 (4)
(V)[a]	74.28 (12)	8.79 (10)	5.75 (12)	82.7 (9)	68.9 (9)	13.8 (9)

Note: (*a*) Langer *et al.* (2007).

Friedel-equivalent relections were averaged before refinement in (I). The H atoms of the methyl groups in (III) and (IV) were fixed in geometrical positions and allowed to ride on their parent C atoms, with C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C). The remaining H atoms in all four structures were located in a difference Fourier map and

Table 8
Cremer & Pople (1975) puckering parameters (Å, °) for the five-membered pyrazole ring of compounds (I)–(IV).

Compound	Q_2	φ
(I)	0.211 (2)	143.2 (5)
(II)	0.111 (2)	321.7 (3)
(III)	0.105 (2)	138.8 (9)
(IV)	0.088 (4)	326 (3)

refined isotropically. The C—H bond lengths are in the range 0.85 (4)–1.03 (3) Å.

For all compounds, data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *PLATON* (Spek, 2003).

The authors thank the Department of Science and Technology, India, for data collection on the CCD Facility set up at the IISc, Bangalore, under the IRHPA–DST programme. DC acknowledges the CSIR, India, for a Junior Research Fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GA3076). Services for accessing these data are described at the back of the journal.

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