

Experimental

Crystals were grown by diffusion of an acetonitrile solution of the title compound into water.

Crystal data

$C_{31}H_{24}N_4O_8$
 $M_r = 580.54$
 Orthorhombic
 $P2_12_12_1$
 $a = 9.849 (2) \text{ \AA}$
 $b = 10.800 (2) \text{ \AA}$
 $c = 25.682 (3) \text{ \AA}$
 $V = 2731.8 (8) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.412 \text{ Mg m}^{-3}$
 D_m not measured

Cu $K\alpha$ radiation
 $\lambda = 1.54180 \text{ \AA}$
 Cell parameters from 25 reflections
 $\theta = 5\text{--}22^\circ$
 $\mu = 0.868 \text{ mm}^{-1}$
 $T = 293 (2) \text{ K}$
 Plate
 $0.60 \times 0.10 \times 0.05 \text{ mm}$
 Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω - 2θ scans
 Absorption correction: none
 3148 measured reflections
 3026 independent reflections
 2527 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.016$
 $\theta_{\text{max}} = 72.95^\circ$
 $h = 0 \rightarrow 11$
 $k = 0 \rightarrow 13$
 $l = 0 \rightarrow 31$
 3 standard reflections every 100 reflections
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.117$
 $S = 0.691$
 3026 reflections
 413 parameters
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.1326P)^2 + 0.8103P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.023$
 $\Delta\rho_{\text{max}} = 0.164 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.166 \text{ e \AA}^{-3}$
 Extinction correction: *SHELXL97* (Sheldrick, 1997b)
 Extinction coefficient: 0.0032 (4)
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Hydrogen-bonding geometry (\AA , $^\circ$)

D—H...A	D—H	H...A	D...A	D—H...A
N1—H1...N7 ⁱ	0.86	2.15	2.956 (3)	157
C2—H2...O6 ⁱ	0.93	2.56	3.057 (4)	114

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Data reduction: *CAD-4 Software*. Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997b). Molecular graphics: *INSIGHTII* (Biosym Technologies, 1995) and *Xtal.GX* (Hall & du Boulay, 1995). Software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: DE1094). Services for accessing these data are described at the back of the journal.

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1-Acetyl-5-(4-methoxyphenyl)-3-(4-methoxystyryl)-2-pyrazoline and 1-acetyl-5-(2-chlorophenyl)-3-(2-chlorostyryl)-2-pyrazoline

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Abstract

The structural details of two methoxy- and chloro-substituted pyrazoline derivatives ($C_{21}H_{22}N_2O_3$ and $C_{19}H_{16}Cl_2N_2O$, respectively) are presented. The two structures show considerable differences in the orientation of the phenyl ring attached to the heterocyclic ring. While the packing of the molecules in the methoxy-substituted derivative is characterized by a C—H...O hydrogen bond, the packing in the chloro-substituted derivative is characterized by short Cl...Cl contacts and C—H...Cl intramolecular hydrogen bonds.

Comment

Pyrazolines are known to exhibit important biological and industrial properties (Wang *et al.*, 1995; El-

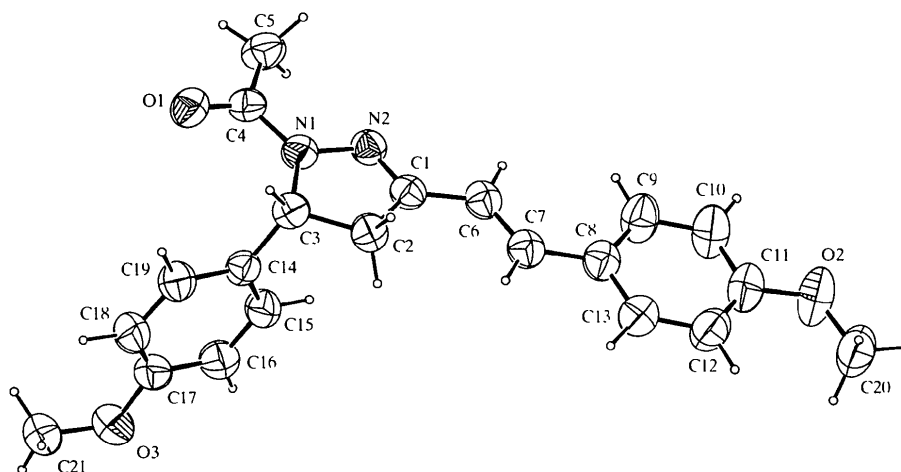


Fig. 1. The molecular structure and atom labelling of (I). Displacement ellipsoids are plotted at the 50% probability level.

Hashash *et al.*, 1995). X-ray structure analysis of the title compounds, (I) 1-acetyl-5-(4-methoxyphenyl)-3-(4-methoxystyryl)-2-pyrazoline, and (II) 1-acetyl-5-(2-chlorophenyl)-3-(2-chlorostyryl)-2-pyrazoline, were undertaken in order to study the influence of the substituent groups on the conformation of the pyrazolines. The present study is the second in a series of investigations being carried out on pyrazolines and their derivatives in our laboratory (Krishnakumar *et al.*, 1998).

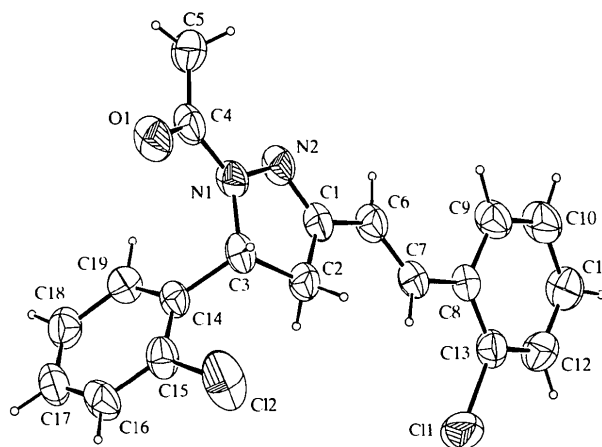
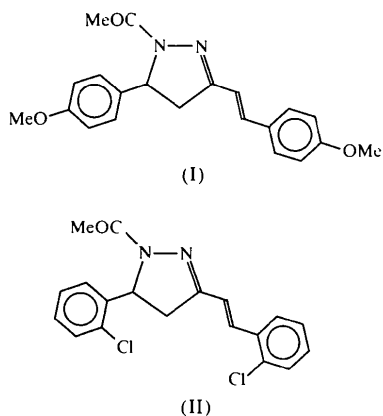


Fig. 2. The molecular structure and atom labelling of (II). Displacement ellipsoids are plotted at the 50% probability level.

The torsion angles about the dienes (N2=C1—C6=C7) in compounds (I) and (II) are found to be -173.6 (1) and -176.9 (4) $^\circ$, respectively, indicating that they exist in an *s-trans* configuration. Though planarity of the rings is maintained in both compounds, there is a significant difference in the orientation of the six-membered phenyl ring attached to the pyrazoline. Selected torsion angles are given in Tables 1 and 3.

Figs. 1 and 2 show the molecular structure and atom labelling of (I) and (II), respectively. The molecular

packing (Fig. 3) within the unit cell of (I) is in a zigzag head-to-tail fashion, characterized by a C—H \cdots O hydrogen bond. The molecular packing in (II) (Fig. 4) is stabilized by Cl \cdots Cl interactions. The two short Cl \cdots Cl contacts observed [Cl1 \cdots Cl2($-x, -y, -z$) 3.665 (2) and Cl2 \cdots Cl2($-x + 1, -y, -z$) 3.796 (3) Å] agree well with the values expected for planar chloroaromatic compounds (Sarma & Desiraju, 1986). In addition, two intramolecular C—H \cdots Cl hydrogen bonds are present. Details of hydrogen-bond parameters are given in Tables 2 and 4.

In compound (I), the five-membered dinitrogen heterocyclic ring, the ethenyl C6 and C7 atoms and the aryl ring of the styryl group all lie in the same plane, with the 4-methoxyphenyl ring attached perpendicular to the heterocyclic ring. This has possibly aided efficient intermolecular stacking of the molecules parallel to the longer *c* axis. The conformation of (II) differs

significantly from that of (I), and this may be attributed to the fact that each of the two 2-substituted Cl atoms, Cl1 and Cl2, prefer sterically favourable intramolecular C—H...Cl interactions. Consequently, the twist of the aryl ring of the styryl group by about 23° in (II) may be responsible for the difference in the packing modes of the molecules in the two structures. This twist of the 2-chlorophenyl ring in (II) has been postulated to explain the ¹⁵N chemical shift of the imine-N atom, which is of a higher value than that observed for (I) (Vijayabaskar *et al.*, 1999).

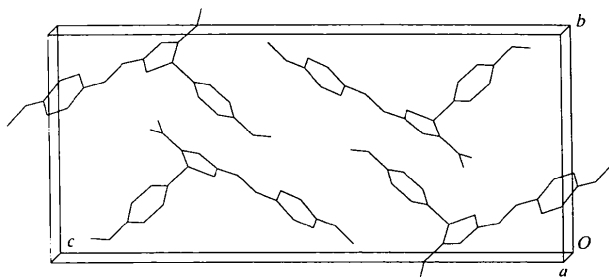


Fig. 3. The packing of the molecules within the unit cell of (I).

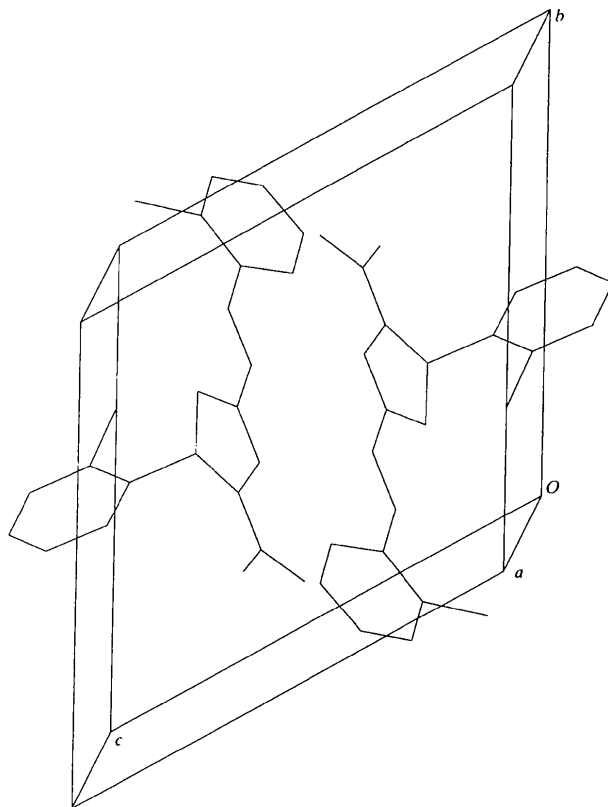


Fig. 4. The packing of the molecules within the unit cell of (II).

Experimental

For (I), analysis found: C 71.90, H 6.27, N 7.91%; M^+ 350; calculated for $C_{21}H_{22}N_2O_3$: C 71.9, H 6.33, N 7.99%; M^+ 350; m.p. 434 K. For (II), analysis found: C 63.44, H 4.53, N 7.72%; M^+ 359; calculated for $C_{19}H_{16}Cl_2N_2O$: C 63.52, H 4.49, N 7.80%; M^+ 359; m.p. 422 K.

Compound (I)

Crystal data

$C_{21}H_{22}N_2O_3$
 $M_r = 350.41$
 Monoclinic
 $P2_1/n$
 $a = 5.720 (1) \text{ \AA}$
 $b = 12.085 (4) \text{ \AA}$
 $c = 26.654 (4) \text{ \AA}$
 $\beta = 91.78 (2)^\circ$
 $V = 1841.6 (7) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.264 \text{ Mg m}^{-3}$
 D_m not measured

Cu $K\alpha$ radiation
 $\lambda = 1.54180 \text{ \AA}$
 Cell parameters from 25 reflections
 $\theta = 10\text{--}19^\circ$
 $\mu = 0.686 \text{ mm}^{-1}$
 $T = 293 (2) \text{ K}$
 Plate
 $0.50 \times 0.25 \times 0.25 \text{ mm}$
 Pale yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: none
 3846 measured reflections
 3478 independent reflections
 3012 reflections with $I > 2\sigma(I)$

$R_{int} = 0.015$
 $\theta_{max} = 70^\circ$
 $h = 0 \rightarrow 6$
 $k = 0 \rightarrow 14$
 $l = -32 \rightarrow 32$
 2 standard reflections
 every 200 reflections
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.127$
 $S = 1.138$
 3478 reflections
 324 parameters
 All H atoms refined
 $w = 1/[\sigma^2(F_o^2) + (0.0568P)^2 + 0.3905P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.166 \text{ e \AA}^{-3}$
 $\Delta\rho_{min} = -0.155 \text{ e \AA}^{-3}$
 Extinction correction: *SHELXL97* (Sheldrick, 1997a)
 Extinction coefficient: 0.0073 (5)
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Selected torsion angles ($^\circ$)

N1—C3—C14—C15	−113.5 (2)	N1—C3—C14—C19	64.8 (2)
C2—C3—C14—C15	133.7 (2)	C2—C3—C14—C19	−48.0 (2)

Table 2. Hydrogen-bonding geometry ($\text{\AA}, ^\circ$) for (I)

D—H...A	D—H	H...A	D...A	D—H...A
C2—H22...O1'	0.97 (2)	2.58 (2)	3.545 (2)	174 (1)

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

Compound (II)

Crystal data

$C_{19}H_{16}Cl_2N_2O$
 $M_r = 359.24$

Mo $K\alpha$ radiation
 $\lambda = 0.71073 \text{ \AA}$

Triclinic

$P\bar{1}$
 $a = 9.296(2) \text{ \AA}$
 $b = 10.524(3) \text{ \AA}$
 $c = 11.044(3) \text{ \AA}$
 $\alpha = 113.89(2)^\circ$
 $\beta = 100.15(2)^\circ$
 $\gamma = 109.75(2)^\circ$
 $V = 866.8(4) \text{ \AA}^3$
 $Z = 2$
 $D_x = 1.376 \text{ Mg m}^{-3}$
 D_m not measured

Data collection

Enraf–Nonius CAD-4
 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: none
 3245 measured reflections
 3039 independent reflections
 2060 reflections with
 $I > 2\sigma(I)$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.067$
 $wR(F^2) = 0.150$
 $S = 1.232$
 3039 reflections
 281 parameters
 All H atoms refined
 $w = 1/[\sigma^2(F_o^2) + (0.0359P)^2 + 0.9770P]$
 where $P = (F_o^2 + 2F_c^2)/3$

Cell parameters from 25
 reflections
 $\theta = 5\text{--}12^\circ$
 $\mu = 0.382 \text{ mm}^{-1}$
 $T = 293(2) \text{ K}$
 Plate
 $0.6 \times 0.3 \times 0.1 \text{ mm}$
 Pale yellow

$R_{\text{int}} = 0.023$
 $\theta_{\text{max}} = 24.98^\circ$
 $h = 0 \rightarrow 11$
 $k = -12 \rightarrow 11$
 $l = -13 \rightarrow 12$
 2 standard reflections
 every 200 reflections
 intensity decay: none

$(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.239 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.298 \text{ e \AA}^{-3}$
 Extinction correction: none
 Scattering factors from
*International Tables for
 Crystallography* (Vol. C)

Table 3. Selected torsion angles ($^\circ$)

N1—C3—C14—C15	-158.3(4)	N1—C3—C14—C19	21.9(5)
C2—C3—C14—C15	87.0(5)	C2—C3—C14—C19	-92.8(5)

Table 4. Hydrogen-bonding geometry ($\text{\AA}, ^\circ$) for (II)

D—H...A	D—H	H...A	D...A	D—H...A
C3—H3...C11	0.95(5)	2.68(3)	3.032(4)	102.9(3)
C7—H7...C12	0.93(4)	2.62(5)	3.046(5)	109.0(3)

Two standard reflections ($12\bar{4}$ and $21\bar{2}$) were used to monitor crystal decay in (II). All H atoms were unambiguously located in difference Fourier calculations and they were included in the refinement with isotropic displacement parameters.

For both compounds, data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *CAD-4 Software*; program(s) used to solve structures: *SHELXS97* (Sheldrick, 1997b); program(s) used to refine structures: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *PLUTO* (Motherwell & Clegg, 1978); software used to prepare material for publication: *SHELXL97*.

RVK thanks the CSIR, India, for financial assistance in the form of an SRF. SP thanks CSIR, India for a major research project. The authors thank the Regional Sophisticated Instrumentation Centre, Indian Institute of Technology, Chennai, for X-ray data collection using *CAD-4* equipment.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: DE1102). Services for accessing these data are described at the back of the journal.

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Ethyl 1-(2-cyanoethyl)-3,5-dimethyl-1H-indole-2-carboxylate

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Abstract

The title compound, C₁₆H₁₈N₂O₂, is an important precursor in the synthesis of 1,2,3,4-tetrahydropyrazinoindoles, which show excellent antihistamine, antihypertensive and central nervous system depressant properties. The carbethoxy group attached to C2 and the planar cyanoethyl group attached to N1 make dihedral angles of 11.0(4) and 75.0(3)°, respectively, with the mean plane of the indole ring. The C—C=N chain is linear with a bond angle of 179.3(4)°.

Comment

The title compound belongs to the family of indole derivatives. Interest in these compounds is increasing with the realisation of their role in obtaining phar-