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Racemic 5-(2,4-dichlorophenyl)-3-(2-naphthyl)-1-phenyl-2-pyrazoline dichloromethane 0.25-solvate

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.006 Å
Disorder in solvent or counterion
R factor = 0.052
wR factor = 0.142
Data-to-parameter ratio = 13.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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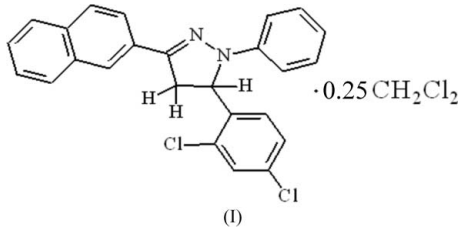
The racemic title compound, $C_{25}H_{18}Cl_2N_2\cdot0.25CH_2Cl_2$, contains two pyrazoline molecules in the asymmetric unit, with similar conformations. A disordered dichloromethane solvent molecule completes the structure.

Comment

Pyrazoline derivatives have applications as chiral antimicrobial and immunosuppressive agents (Brown *et al.*, 1972; Lombardino *et al.*, 1981; Ramalingham *et al.*, 1977). The title compound, (I), was prepared and structurally characterized as part of our studies of these compounds.



(I)

As shown in Fig. 1, there are two pyrazoline molecules in the asymmetric unit, accompanied by a half-occupancy dichloromethane solvent molecule. The pyrazoline molecule is chiral; in the arbitrarily chosen asymmetric unit, C7 in the first molecule has the *R* configuration and C32 in the second molecule the S configuration, but crystal symmetry generates racemates for both molecules.

In the first molecule, the mean planes of aromatic rings C1–C6 (*A*) and C20–C25 (*B*) and the naphthalene ring system C10–C19 make dihedral angles of 78.9 (3), 8.0 (3) and 6.7 (3)°, respectively, with the mean plane of the N1/N2/C7–C9 pyrazoline ring. Rings *A* and *B* are inclined to each other at an angle of 81.2 (3)° and the dihedral angle between the naphthalene ring system and ring *A* is 79.4 (3)°.

For the second molecule, aromatic rings C26–C31 (*C*) and C45–C50 (*D*) and the naphthalene ring system C35–C44 make dihedral angles of 73.8 (3), 5.2 (3) and 10.3 (3)°, respectively, with the N3/N4/C32–C34 pyrazoline ring. Rings *C* and *D*, and ring *C* and the naphthalene ring system are inclined to each other with angles of 74.8 (3) and 80.2 (3)°, respectively. Overall, the two molecules have very similar conformations.

A dichloromethane solvent molecule, positionally disordered about an inversion centre, completes the structure.

Experimental

A mixture of 3-(2,4-dichlorophenyl)-1-(2-naphthyl)prop-2-en-1-one (5.0 mmol), phenylhydrazine (25.0 mmol) and acetic acid (30 ml) was

heated at reflux for 5 h, then poured on to crushed ice. The precipitate was separated by filtration, washed with water, and crystallized from dichloromethane–methanol (1:1 *v*/*v*) to obtain the product (m.p. 382–383 K). The product (40 mg) was dissolved in a mixture of dichloromethane (10 ml) and methanol (10 ml) and the solution was kept at room temperature for 15 d. Natural evaporation of the solution gave colourless crystals of (I) suitable for X-ray analysis.

Crystal data

$C_{25}H_{18}Cl_2N_2\cdot0.25(CH_2Cl_2)$	V = 2170.7 (11) Å^3
M_r = 438.55	Z = 4
Triclinic, $P\overline{1}$	D_x = 1.342 Mg m^{-3}
a = 11.171 (3) Å	Mo $K\alpha$ radiation
b = 13.464 (4) Å	μ = 0.38 mm^{-1}
c = 15.414 (4) Å	T = 294 (2) K
α = 69.656 (5)°	Prism, colourless
β = 86.978 (5)°	0.26 × 0.22 × 0.18 mm
γ = 88.595 (6)°	

Data collection

Bruker SMART1000 CCD diffractometer	11102 measured reflections
	7622 independent reflections
ω scans	3525 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)	R_{int} = 0.034
	θ_{max} = 25.0°
T_{min} = 0.898, T_{max} = 0.936	

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.053	$w = 1/[\sigma^2(F_o^2) + (0.0554P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.142	$(\Delta/\sigma)_{max}$ = 0.001
S = 0.98	$\Delta\rho_{max}$ = 0.30 e Å^{-3}
7622 reflections	$\Delta\rho_{min}$ = −0.32 e Å^{-3}
550 parameters	

All H atoms were placed in idealized positions (C—H = 0.93–0.98 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C). The CH_2Cl_2 molecule lies close to an inversion centre and was refined with 0.5 site occupancy for all atoms.

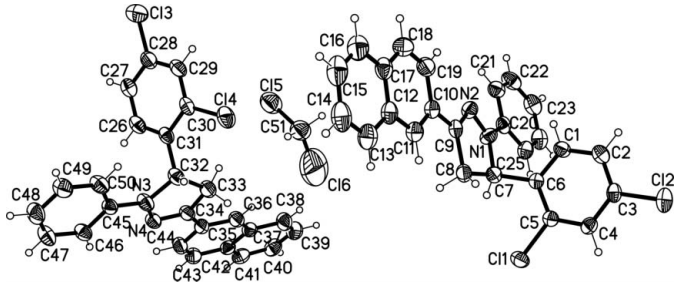


Figure 1
The asymmetric unit of (I), shown with 30% probability displacement ellipsoids (arbitrary spheres for H atoms). Only one orientation of the disordered dichloromethane solvent molecule is shown.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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